CHAPTER 3
EXPERIMENTAL METHODS

3.1 Introduction

Amorphous silicon film cannot be prepared directly from the melt and solid, but are usually fabricated in the form of thin film by several deposition techniques that have been described in the chapter 2. Direct-current (d.c) plasma glow discharge is one of the well-known techniques that have been used in this work to prepare hydrogenated amorphous silicon (a-Si:H). The dc plasma glow discharge system was preferred to the radio frequency (rf) glow discharge system as it is more economical to build [1].

Good quality a-Si:H can be produced by dc glow discharge decomposition of silane. A specific character of a-Si:H film can be prepared by controlling numerous variables during experiment such as power level, flow-rate of gases, deposition pressure, gas dilution and etc. However, there are still many mysterious characteristics of a-Si:H which can be studied. *D.E. Carlson* [2] was the first to make electronic devices from a-Si:H produced by dc glow discharge in silane. He later made several types of a-Si:H solar cells with excellent photovoltaic performance using these films. Since then, application of dc glow discharge a-Si:H have increased mainly in the fabrication of semiconductor devices such as deposition of passivating films, thin film transistors (TFT), photosensor [3] and etc.

The d.c glow discharge system built for this project for preparing a-Si:H thin film will be briefly presented in this chapter. The experimental details employed in the preparation of a-Si:H thin film begins from substrate cleaning to the deposition of a-Si:H by dc plasma glow discharge technique. This is followed by the optical characterization of film by UV-VIS-NIR transmission spectroscopy while the surface morphology and structural properties are investigated using the Atomic Force Microscopy (AFM).

3.2 Substrates Cleaning Procedure.

The substrates used in this work to deposit the a-Si:H film are glass slides and crystal silicon wafer. The purpose of using these two types of substrates is to study the influence of the substrates on the morphology of a-Si:H film. For the optical transmission measurement, a-Si:H film deposited on glass substrates are used. The size of the substrates prepared in this work is about (2.0 x 1.5) cm^2.

The effective cleaning of the substrates has a strong effect on the adhesion properties of deposited films. Dust, inorganic and organic on the substrates should be cleaned off using a standard cleaning procedure. The glass substrates are ultrasonically cleaned in a beaker of soap solution to remove gross dirt and oil for 15 minutes. The substrates are then rinsed in distilled water. To remove remain oil deposits, the substrate are further rinsed in acetone and finally in ethanol. The cleaned substrates are put in the oven which the heating temperature is set at 60°C for storage. Prior to this, the substrates are dried using a hot-air blower to prevent water spots formation.

The cleaning process of the silicon substrates begins with rinsing the substrates in deionized water, the substrate are then immersed it in a boiling solution of $H_2O:HCl:H_2O_2 = (86:11:3)$ for 10 minutes. After boiling, substrate is rinsed again with deionized water. The rest step is to immerse the substrate in the solution of $H_2O:H_2O_2:NH_4OH = (7:3:3)$ followed by rinsing in deionized water. Finally, the substrates are immersed in the solution of $H_2O:HF = (10:1)$ and then followed a final rinse in deionized water. This step is used to etch away surface silicon oxide (SiO_2) on the substrates. The cleaned silicon substrate is dried using a hot-air blower and placed on the substrate holder in the deposition chamber which is than pumped down. The vacuum will ensure minimum oxide formation on the c-Si substrates.

3.3 Instrumentation of DC Plasma Glow Discharge

The system used in this work is a home built horizontal dc configuration plasma glow discharge system (PGD2). The schematic diagram of the PPG2 system is shown in figure 3.1. This system consists of five major components, which are the reaction chamber, the pumping system, the gas distribution system, the electrical system and the detoxification system. The original configuration of this horizontal dc plasma glow discharge system and reaction chamber is designed by *K.S. Wong* [4].

3.3.1 Reaction Chamber

The cross sectional view of the reaction chamber is shown in figure 3.2. The chamber is made of stainless steel because it is resistant to the corrosive of gases and does not react with the reacting gases. This chamber consists of a stainless steel cylinder tube with front and back plates attached to it on Viton O-rings are used at the seams to prevent any leakage. The dimension of the chamber is 300mm length, 150mm in diameter and the wall are 6mm thick.

The front and back plates carry a stainless steel rotary shaft. The former ones supports the electrode and electrical leadthroughs for the chromel-alumel thermocouple, heater and electrode. The latter supports the electrode, two electrical leadthroughs and a gauge (PRE 10K).

A quartz tube is put inside the reaction chamber where it is supported by three rough Teflon. The dimension of quartz tube is 200mm in length, 96mm in diameter and

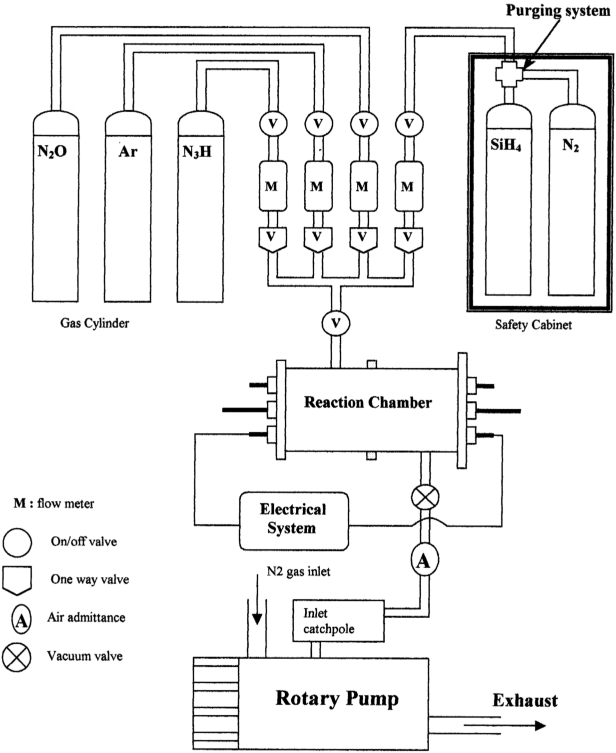
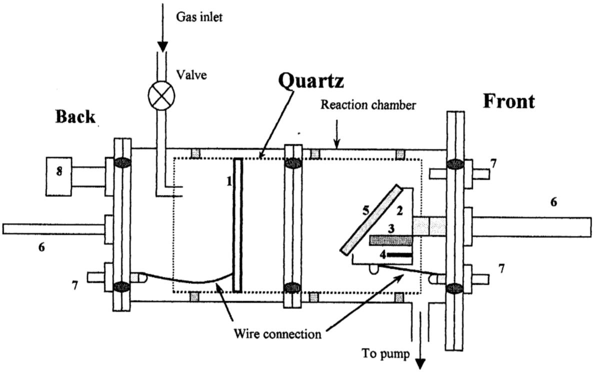


Figure 3.1 : The schematic diagram of horizontal dc plasma glow discharge deposition system.



1. Anode (s.s mesh wires)
2.Cathode (Aluminum block)
3. Catridge heater
4. Chromel-alumel thermocouple
5. Sample holder with substrates
6. Stainless steel rotary shafts
7. Electrical leadthrough
8. Gauge head
▨ Teflon
● Viton O-rings

Figure 3.2 : Schematic diagram of the reaction chamber.

5mm thick. The electrodes are positioned horizontally inside the quartz tube to avoid discharge between electrodes and the stainless steel chamber. The anode is stainless steel mesh wire and the cathode is an aluminum block respectively. The cathode is placed at the front end of the chamber and slanted at 45° as it also acts as a sample holder where the substrate is placed. These electrodes must be insulated from the plates by insulators and a considerable distance should be kept between anode and cathode to avoid short-circuits since the power supply is above hundred volts.

Since the deposition process in this work was done at room temperature, the cartridge heater and Sigma chromel-alumel thermocouple which suppose should be embedded in the cathode block, are not used in this experiment. The gas inlet inside the reaction chamber is used to lead the gas flow to cathode and it is made of Teflon to avoid any discharge from plasma at the inlet during deposition.

3.3.2 The Pumping System

In the horizontal dc glow discharge system, a rotary pump (Edward EM 28) is used to maintaining the required pressure in the reaction chamber during deposition. This rotary pump is able to pump the system down to about 10^{-3}mbar. The pumping unit is attached directly to the reaction chamber via an inlet catchpot and the detoxification system is attached to the exhaust of the pump. Nitrogen gases, which is required for detoxification is directed into the rotary pump through the oil chamber. The pressure in the reaction chamber during deposition is about 10^{-2}mbar, which can be monitored by a special corrosion resistant gauge heads (PRE 10K) and control units (Pirani 501). A vacuum valve is used to control the required pressure in reaction chamber.

3.3.3 The Electrical System

The electrical system comprises of the power supply that triggers the discharge, the electrical control panel and an Iwatsu Oscilloscope that monitors voltage across the electrodes. This electrical system is shown in figure 3.3. The d.c power supplied to the glow discharge system is obtained using a full wave diode rectifier bridge. A setup transformer with input of 240 V and out put of 3 kV is used to set up the 240 V main power supply and then rectified. The transformer is immersed in a container full of transformer oil to keep it cool. A series of resistor is used to control the current during the glow discharge and for protecting the transformer from any current overload. The dc voltage across the electrodes is measured using an Iwatsu Oscilloscope (model SS-5710). A Iwatsu high voltage probe (Modal HV-P30) with reduction ratio at 1000:1, is used to changed the high dc voltage to a suitable level that required by the Oscilloscope.

The power supply system is controlled at the control panel. A voltmeter across the ac power supply is used to monitor the ac voltage that goes into the full wave Rectifier Bridge. A miliammeter is employed for monitoring the discharge current and a switch is used to switch on or off the power supply.

3.3.4 Gas Distribution System

The gas distribution system is shown in figure 3.2, where the source of gas is from the gas cylinders and ends at the gas inlet that leads the mixed gases into the reaction chamber. The gases that can be used in this system are silane (SiH_4), Argon (Ar), nitrous oxide (N_2O), and Ammonia (NH_3). However, only silane and Argon are used in this work. The gases are delivered through gas lines made of ¼ inch stainless steel tubing. Blazer valves and connectors are used for connection. The silane cylinder is placed inside a special silane cabinet to ensure safety. A gas regulator with special purging system is required for the silane gas cylinder. This purging system is connected to an oxygen free nitrogen gas cylinder. This system is to purge excess silane from the gas lines.

The gases from the cylinders are separately delivered through the stainless steel tubing to a gas distribution panel, which then distributes the gases to the other system using valves. Before reaching the reaction chamber, the gases go through 4 separated metering valves and mass flow controllers. On exiting the mass flow controller, the gases pass through one way valves to prevent back flow of the gasses. The gases are mixed only prior to entering the chamber.

3.3.5 Detoxification System

The detoxification system is a hard polymer tank containing a solution of $KMnO_4$ chemical. The residue silane in the chamber will be pumped out by the rotary pump to the detoxification system. Nitrogen gas is fed into the oil chamber of the rotary pump to dilute the excess SiH_4. The outlet of the pump exhaust is connected to the detoxification system and end deep below the surface level of the solution for complete chemical reaction. Figure 3.4 shows the schematic of the detoxification system. The chemical reaction for neutralizing the residue SiH_4 gas is as given below.

$$2KMnO_4 + SiH_4 \longrightarrow 2MnO_2 + K_2SiO_4 + 2H_2\uparrow \qquad (3.1)$$

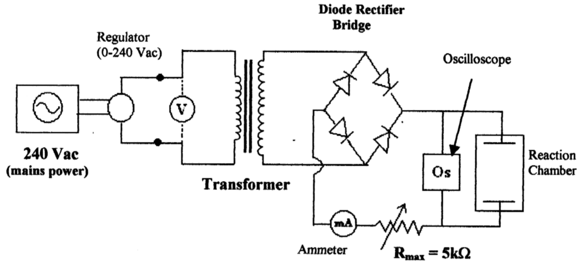


Figure 3.3 : Power supply for d.c Plasma Glow Discharge System.

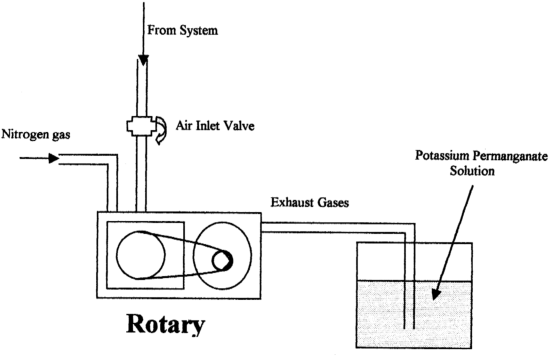


Figure 3.4 : The detoxification system

3.4 Preparation Technique

In this work, a total of 8 sets of a-Si:H thin fim samples were prepared. Two sets were prepared by d.c plasma glow discharge of pure silane at the silane flow-rate of 5sccm and 20sccm without Argon dilution. The remaining six sets of a-Si:H thin film samples were prepared from the discharge of pure silane at flow-rates of 5sccm and 20sccm diluted in argon at flow-rates of 5sccm, 10sccm and 15sccm. For each set of sample, the a-Si:H films were deposited on both crystal silicon and glass substrates. The flow-rate of silane and argon used in preparing the different sets of samples studied in this work is shown in table 3.1.

Samples	A1	A2	A3	A4	A5	A6	A7	A8
Gas SiH_4 (sccm)	20	20	20	20	5	5	5	5
Gas Argon (sccm)	0	5	10	15	0	5	10	15

Table 3.1: Flow-rate of silane and argon used for the samples studied.

The deposition of a-Si:H thin films were done by using the PGD2 home-built system, which is a horizontal dc plasma discharge system. The deposition process occurs within the reaction chamber and is activated when the d.c power supply applied across the electrodes. Basically, the formation of film on the substrate surface can be categorized into two processes: the plasma decomposition of silane and the heterogeneous reactions of the molecules on the substrate surface. The deposition process begin with the pyrolysis of gas silane where electron-impact dissociation of silane into various product. This technique is also referred to as plasma enhanced chemical vapour deposition where the silicon produce from pyrolysis is deposited as amorphous thin film on substrate. This film is referred to as hydrogenated amorphous silicon (a-Si:H) because it contains hydrogen that is incorporated inside the amorphous silicon structure. The most likely reaction [5] involve are:

$$\mathrm{SiH_4} + e^- \longrightarrow \mathrm{SiH_3} + e- + \mathrm{H} \qquad (>3.5\mathrm{eV})$$

$$\mathrm{SiH_4} + e^- \longrightarrow \mathrm{SiH_2} + e- + 2\mathrm{H} \qquad (>6.9\mathrm{eV})$$

$$\mathrm{SiH_4} + e^- \longrightarrow \mathrm{SiH} + e- + 3\mathrm{H} \qquad (>10.4\mathrm{eV})$$

$$\mathrm{SiH_4} + e^- \longrightarrow \mathrm{Si} + e- + 4\mathrm{H} \qquad (>14.0\mathrm{eV})$$

This deposition of amorphous silicon by plasma glow discharge has been pioneered by *Chittick* [6] in 1970.

3.4.1 Pre-deposition Procedure

Deposition of a-Si:H involves a highly toxic gas silane. So, the gas distribution system must be checked prior to deposition for any leakage to ensure a safe operation during deposition. Normal leakage testing procedures such as, introducing nitrogen into the system to a considerably high pressure and then putting soap solution at the join are used to detect big leakage. The formations of bubbles indicate the presence of leak at that particular location. Small leakage can be detected by pumping the system down to the lowest pressure that can be achieved and acetone is used to detect leakage at a particular position. A sudden increase in pressure indicates leakage in this case.

Another important safety step is purging the silane gas line with nitrogen gas to remove air or any excess silane from the line. Purging the system can prevent vapour condensation, decomposition and explosion of hydrocarbon pump oil.

In many thin film devices applications, contamination of the substrate surface with even submicron ($<10^{-4}$ cm^2) particle can affect the performance. Therefore, the atmosphere inside the reaction chamber have to be pumped until a considerable pressure

(~0.04mbar) prior to deposition. To achieve a good pre-deposition pressure, the reaction chamber must be cleaned before every deposition process. Vigorous use of sandpaper on all the metal surface removing a-Si:H is found to be effective in deposits from previous deposition process. The deposition on the surface of the quartz tube is removed by scrubbing it with liquid detergent and wet sandpaper. The chamber and quartz tube are then rinsed thoroughly and dried before substrates are placed into the reaction chamber.

3.4.2 a-Si:H Deposition with Pure Silane Discharge.

Deposition of a-Si:H can proceed once the system is free from leakage and safe to operate. The substrates are placed on the cathode via a sample holder and put inside the quartz tube. The reaction chamber is pumped down using rotary pump to a base pressure of about 0.04mbar. Silane gas is then introduced into the chamber slowly and the required flow-rate is achieved by mass flow controller. Once the pressure in the deposition chamber is stable, the pressure is noted using a Pirani II gangue. Deposition process proceeds when the d.c power supply is applied across the electrodes. The ionization current is fixed at 12mA and the d.c voltage is maintained at around 600V.The deposition time is noted as the period when the power supply is switched on until the times it switched off. During deposition, the parameters such as pressure, flow-rate, ionization current and voltage across the electrodes should be carefully monitored and maintained until the end of the deposition process. When the deposition is completed, the supply of silane gas is turned off.

3.4.3 a-Si:H Deposition with Discharge of Silane Diluted in Argon.

The process of a-Si:H deposition with Argon dilution of silane is similar with deposition described in the previous section. However, argon gas is let into the reaction chamber once the pressure in the chamber stabilized after the introduction of the silane gas. The flow-rate of Ar gas is controlled using the mass flow controller for argon. The mixture of silane and argon gases are then allowed leaving to stabilized. The stabilized pressure is then noted. The power supply is switched on to trigger the discharge of these gases and initiate the deposition process. As mention in the previous section, the deposition parameters are maintained and monitored throughout the deposition on process. Argon is an inert gas and is known to play the role of steric hindrance against not only in H_2 effusion but also thermal re-arrangement of H local environment in a-Si:H thin film [7]. This role of argon in the deposited film will be studied from the effects on the film properties.

3.4.4 Post-deposition Procedure

This operation is important to ensure that the system is free from excess silane gas. The silane lines are purged with nitrogen gas to drive out silane that remains in the lines. The gas lines is then pumped by rotary pump until the flow meter indicates zero flow and the pressure in the chamber goes down to 0.008 mbar. Just before turning off the rotary pump, the air admittance valve is slowly opened to ensure that the rotary pump oil is not sucked up into the lines when the pump is not operating.

3.4.5 Deposition Parameters

The d.c potential necessary to maintain discharge between the anode and the cathode, depends on the pressure and anode-to-cathode spacing as well as on the kinds of gasses and electrode material used. In this system, the spacing between stainless steel anode and cathode are fixed at 9.1 cm. This is the smallest distance between the anode and cathode of this system where the discharge produced is stable. *Uchida* [8] showed that the cathode-to-anode spacing does not change the deposition rate in the dc glow discharge, however, the smallest spacing was chosen to enhance the electric field. In order to determine the effect of the preparation conditions on the characteristics of the a-Si:H, all the parameters should be constant except the gas flow-rate. The parameters maintained during preparation are shown as in table 3.2. The argon and silane flow-rates, partial pressure of argon and silane and the deposition times are presented in table 3.3.

d.c voltage	(600 ± 10) V
Current	(12.0 ± 0.5)mA
Base pressure	(0.04 ± 0.01)mbar
Temperature	27°C (room temperature)

Table 3.2 : The parameters maintained during preparation.

Samples	Flow-rates (sccm)		Gas Pressure (± 0.01mbar)		Deposition Time (± 1min)
	Argon	SiH_4	Argon	SiH_4	
A1	0	20	0.00	0.46	60
A2	5	20	0.18	0.32	60
A3	10	20	0.26	0.24	120
A4	15	20	0.37	0.23	120
A5	0	5	0.00	0.10	60
A6	5	5	0.19	0.07	60
A7	10	5	0.30	0.07	60
A8	15	5	0.40	0.06	60

Table 3.3 : The preparation parameters of flow-rates for argon and silane gases, argon gas pressure, silane gas pressure and deposition time for each sample.

3.5 Optical Characterization

The optical transmission spectrum is indeed a very useful technique in thin film characterization for determining various parameters of an a-Si:H thin film, such as film thickness and the refractive index. In this work, the optical energy gap and Urbach Tail bandwidth are also determined from this spectrum.

3.5.1 Optical Transmission

In this project, the Jasco V-570 UV-VIS-NIR spectrophotometer [9] is used to measure the transmission of amorphous silicon thin film in the range of 250-2500nm wavelength. A schematic diagram of experimental setup is shown in figure 3.5. The details optical system of the Jasco V-570 UV-VIS-NIR spectrophotometer is shown in figure 3.6.

The light source is automatically changed depending on the scanning range needed. Deuterium lamp, D2 and tungsten halogen lamp, W1 and a double grating monochromator are used to produce monochromatic light in the spectral range of 200 - 2500nm. A deuterium source is used for wavelength below 360nm and tungsten halogen source is used for wavelength above 360nm. The detectors are a photomultiplier tube (in the visible and ultraviolet region) and a lead sulphide cell (in the near infrared region) which are switched at the wavelength of 830nm.

The light which is chopped at frequency of 50/60 Hz is split into two beams passing separately into the sample and reference cell before reaching the detector. Output from the detector is passed through a pre-amplifier into an amplifier. The analogue reference and the dark current are digitized before entering computer.

Since the hydrogenated amorphous silicon (a-Si:H) is deposited on glass substrates, therefore two reference glass slides which are similar to the substrate are positioned in both the reference cell and the sample holder during baseline correction. The

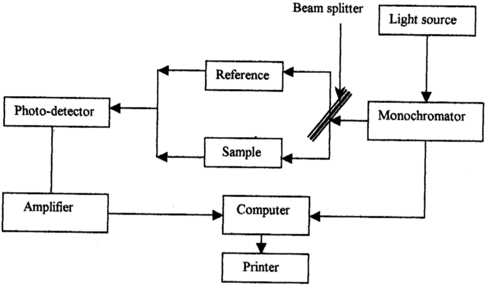
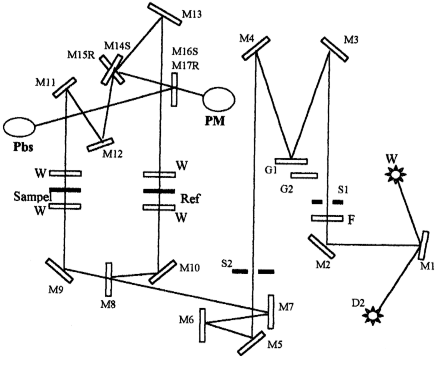


Figure 3.5 : Block diagram of the optical transmission Set-up.



W, D2 : Light source
PM : UV-VIS detector
PBS : NIR detector
Sam : Sample beam
Ref : Reference beam
S : Splitter
G : Jerjak
M : Mirror
F : Filter

Figure 3.6 : Schematic diagram of optical system in Jasco V-570 UV-VIS-NIR spectrophotometer.

baseline is obtained by scanning both glass slides in the range of 200nm to 2500nm. Then, the glass slides in the samples holder is replaced with the sample. Using the same range, the transmission spectrum for the sample is then obtained. If the thickness of the film is from the same order as the wavelength, λ, interference will occur and produces a transmission spectrum of a-Si: H as shown in figure 3.7. A combination of methods proposed by *J.C. Manifacier et. al* [10]and *E.A. Davis et. al* [11] as describe in chapter 4 is used to determine the thickness and refractive index of the film. Next, the various parameters can be calculated from the transmission data. However, for the films have one or not interference fringes at all, an iteration technique propose by *K.M.M. Abo Hassan* in his Phd Thesis [12] uses the transmission, T and reflectance, R relation at normal incidence is utilized to simulate and determine the thickness and refractive index as describe in section 4.3.2.

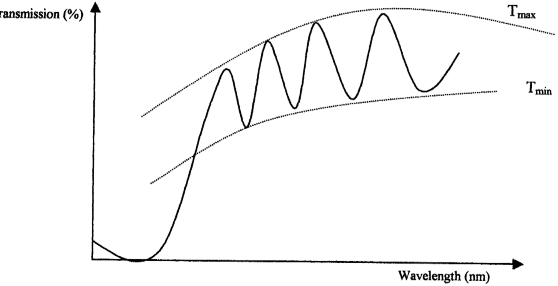


Figure 3.7 : The transmission spectrum which shows the envelope method.

3.6 Atomic Force Microscopy (AFM)

The atomic force microscope (AFM) was developed from the Scanning Tunneling Microscope (STM) in 1986 by *Binning, Quate and Gerber* [13]. Like all the other Scanning Probe Microscopes, the AFM utilizes a sharp probe moving over the surface of a sample in a raster scan. The probe is a tip on the end of a cantilever, which bends in response to the force between the tip and the sample. This AFM is being used to solve processing and materials problems in a wide range of technologies affecting the electronics, telecommunications, biomedical, chemical, automotive, aerospace, and energy industries. The materials under investigation include thin thick film coating, ceramics, composites, glass, synthetic and biological membranes, metals, polymers, and semiconductors.

The first AFM used a scanning tunneling microscope at the end of the cantilever to detect the bending of the lever, but now most AFM employ an optical lever technique. Figure 3.8 is the diagram that illustrates how it works; as the cantilever flexes, the light from the laser is reflected on to the split photo-diode, by measuring the difference signal (A-B), changes in the bending of the cantilever can be measured.

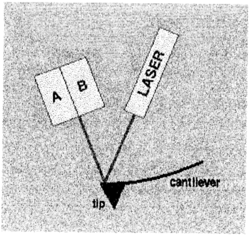


Figure 3.8 : The diagram of optic system in AFM.

Since the Cantilever obeys Hooke's Law for small displacements, the interaction force between the tip and the sample can be found. The movement of the tip or sample is performed by an extremely precise positioning device made from piezo-dielectric ceramics, most often in the form of a tube scanner. The scanner is capable of sub-angstrom resolution in x-, y-, z-directions. The z-axis is conventionally perpendicular to the sample.

3.6.1 Feedback System

Figure 3.9 shows the feedback loop system. The function of this feedback electronic circuit is to control the vertical position of the tip, by controlling the motion of the z-piezoelectric ceramic. It also includes means of sensing the vertical position of the tip. It is also combined with the piezoscanner and the photodiode.

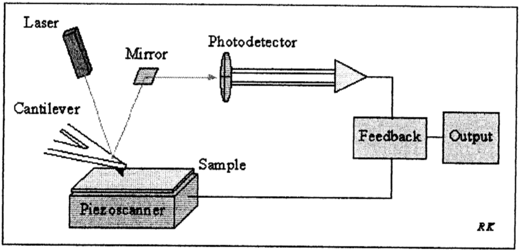


Figure 3.9 : The diagram shows the feedback loop. The sensor output is used to adjust the z-piezoelectric ceramic and generate z-data.

(1). When the feedback system is on, the piezoscanner responds to any changes in force which are detected, and after the tip sample separation to restore the force to pre-determined value. There are two basic AFM feedback methods, those in which the probe is not oscillating, and those that use phase shift or amplitude change in oscillating cantilever systems.

(2). When feedback is off, the microscope operates at constant height or deflection mode.

3.6.2 Tip-sample Interaction

There are three main modes of operation or a tip-sample interaction, as it is also known. The mode of operation is simply the method in the image contrast is obtained. The modes of operation are contact mode, non-contact mode and tapping mode. These three methods can be seen on typical force curve. As discussed in the principle of AFM, while the tip is scanned over the sample a detector measures the cantilever deflection. Several force, most commonly the ***Van der Waals*** force, contribute to the deflection of the cantilever.

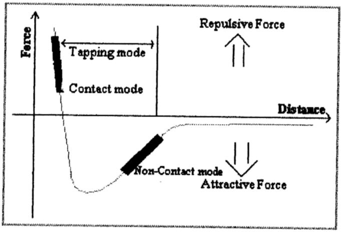


Figure 3.10 : Graph of force versus distance in AFM

Figure3.10 shows the dependence of the Van der Waals force upon the distance between the tip and the sample. The graph shows the force on the cantilever tip as a function of the distance between the tip and the sample. The area below the x-axis is the **Attractive region**. The Attraction is the result of long range Van der Waals interactions means the inter-atomic force between the cantilever and the sample is attractive. This is the non-contact mode.

The area above the x-axis is the **Repulsive region**. The cantilever and the sample surface repel each other, as there is only a few Angstrom between them. This is the contact mode. The tapping region is in between these regions.

3.6.3 Atomic Force Microscope Principles.

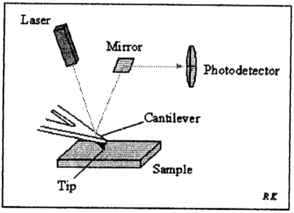


Figure 3.11: Schematic of AFM operation system.

In this section we will be considering the principle of the operation of AFM. Figure3.11 shows the principles of AFM operation system. The sharp tip, mounted on the end of the cantilever spring is scanned over a surface with feedback mechanisms that enable the *piezo-electric scanner* to maintain the tip at a constant force (to obtain height information) or height (to obtain force information) above the sample's surface. The cantilever is a microscopic force sensor which is usually formed by one or more beams of silicon nitride that is 100~500 micrometers long and about 0.5~5 micrometers thick. Mounted on the end of the cantilever is the sharp tip that is used to sense a force between the sample and the tip.

A force between the tip and the sample surface causes the cantilever to bend, or deflect. A detector measures the cantilever deflection as the tip is scanned over the sample, or the sample is scanned under the tip.

The measured cantilever deflection allows a computer system that drives the scanner, to measure the data and convert it into an image (a map of surface topography).

In this work, AFM from Digital Instrument, " Nanoscope III " is using to study the surface structure of the sample. The ***Tapping mode*** is used to scan the samples. The tip used is made of silicon nitride. Image of the sample surface is viewed in 3 dimension with the parameter of 2μm x 2μm area, 0 degree and 200nm height [13].

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