CHAPTER III

EXPERIMENTAL TECHNIQUES AND MEASUREMENT PROCEDURES

3.1 Introduction

In this Chapter, we describe the experimental techniques used for evaporation and the procedures adopted in the measurement of various properties.

3.2 Film Preparation

Amorphous semiconducting films were prepared by evaporating 99.999% pure Silicon, from a pyrolytic graphite boat coated with Silicon carbide, on to corning 7059 or quartz glass substrates. Substrates were cleaned as per the standard procedures\(^1\), consisting of cleaning first in a detergent solution, followed by boiling in deionised water and finally vapour degreasing in methanol and trichloroethylene. All the evaporations were made in a conventional oil diffusion vacuum system, with a liquid nitrogen trap, typical base pressure of which was $10^{-6}$ Torr.

Silicon films were prepared in vacuum and in partial pressure of hydrogen or oxygen. Hydrogenated samples were prepared by bleeding in 6 N pure hydrogen gas through a needle valve, at pressures varying between $1 \times 10^{-5}$ Torr to $5 \times 10^{-4}$ Torr. For oxygenated samples, oxygen gas passed through a molecular sieve column and then through a double walled glass-trap dipped in liquid nitrogen, was used. Typical pressures were $5 \times 10^{-6}$ Torr to $1 \times 10^{-5}$ Torr. Samples were also
prepared in partial pressure of atomic hydrogen, which is obtained, when hydrogen gas is passed over a tungsten filament heated to $2000^\circ\text{C}$. Evaporations were done with small pieces of Silicon, roughly of same mass. The charge was kept on a hopper inside the chamber. Silicon pieces were dropped on to a hot graphite boat by activating the hopper from outside by means of an electromagnet. The temperature of the boat was maintained at $1450^\circ\text{K}$ for all the pieces of Silicon to ensure uniform deposition rates. For conductance measurements pre-deposited contacts of nickel and gold or molybdenum were used. Nickel and gold were evaporated from a resistively heated tungsten filament on to the substrates held at $200^\circ\text{C}$, at a pressure of about $10^{-5}$ Torr. Gold was evaporated subsequently on to a nickel layer to ensure good adhesion to the substrate. Molybdenum deposition was normally done by r.f. sputtering. Contact patterns were made by photolithography and by evaporating through a metal mask. Since the conductance of the sample is very low, an integrated grid pattern, as shown in Fig. 4, consisting of hundred grids connected in parallel, was used to increase the sample conductance. The electrode width and spacing between the electrodes was typically hundred microns. Soldered contacts were used for electrical conductance measurement.

3.3 D.C. Conductivity Measurements

High temperature conductivity measurements were done from room temperature to $600^\circ\text{K}$, in vacuum at $10^{-5}$ Torr pressure. The conductance of the sample was measured at different temperatures after stabilizing the temperature. Electrical leads were soldered to
Fig. 4  The electrode pattern used in D.C. conductance measurement of planar structures. Each electrode has a width of 100 μm. The spacing between two electrodes is also 100 μm.
the sample and conductance was measured by measuring a voltage drop across a standard resistor, by using a Phillips D.C. microvoltmeter model No. PP 9004.

For the low temperature measurements the sample was transferred to a cryostat. The cryostat is shown in Fig.5. It was first evacuated to $10^{-2}$ Torr and then back filled with helium gas to a pressure of one torr. Helium was used as an exchange gas to minimise the temperature gradient across the sample. The sample conductance was measured at various stable temperatures between room temperature and $110^0k$. Temperature was recorded using a calibrated copper-constantan thermocouple fixed on to a glass plate and mounted near the sample. The thickness of the glass was chosen to be the same as that of the substrate used for deposition. The current was measured by using an ECIL Electrometer model No. EA 812.

3.4 Optical Absorption Measurements

Optical absorption was measured using a Cary 170 double beam spectrometer. Films deposited on glass substrates were used for these measurements. In the region of small absorption, the refractive index of the sample was determined by identifying the order of interference peaks obtained from transmission maximum and transmission minimum. An average value of ($n$) refractive index was calculated and used for further calculations.

Following Brodsky et al. for a film of thickness $t$, transmission $T$ is given by
Fig. 5  A schematic illustration of the cryostat and sample holder.
1. ELECTRODES
2,2. THERMO COUPLE FEED THROUGHS
3,3. HEATER ELECTRODES
4. PERSPEX INSULATOR
5. SHIELD
6. SAMPLE
7. THERMO COUPLE
8. GLASS CRYOSTAT
9. DEWAR
\[ T = \frac{(1-R_1)(1-R_2)(1-R_3)}{(1-R_2R_3)} \left\{ 1 - \left[ R_1R_2 + R_1R_3(1-R_1)^2 \right] e^{-\kappa d} \right\} \quad .... \quad 1 \]

Where \( R_1 \) = Film to air reflection coefficient = \( \frac{n-1}{n+n_g} \).

\[ R_2 = \text{Film to substrate reflection coefficient} = \frac{n-n_g}{n+n_g} \]

\[ R_3 = \text{Air to substrate reflection coefficient} = \frac{1-n_g}{1+n_g} \]

\( \kappa \) = Absorption coefficient

\( n \) = Refractive index of glass, and

\( n_g \) = Refractive index of the film.

It is found that \( R_3 \ll R_2 \ll R_1 \), so that the above formula can be approximated to the form

\[ T = (1-R_1)(1-R_2)(1-R_3) e^{-\kappa d} \quad .... \quad 2 \]

From the value of \( T \) measured experimentally, \( \kappa \) can be calculated by using equation 2.

In the region, where interference is important the average of \( T_{\text{max}} \) and \( T_{\text{min}} \) gives the value of \( T \) the transmission. \( \kappa \) can be calculated by using the same equation 2.

3.5 Photoconductivity Measurements

Photoconductivity was measured in vacuum at a pressure of the
order of $10^{-1}$ Torr, by shining white light on the sample. The number of photons incident on the sample is estimated using an RCA photomultiplier tube No. RCA 7102. The tube was placed at the same distance from the source as the sample and a slit of area equal to the sample area was defined in front of the photomultiplier tube. Typical photon flux used for photoconductivity measurements is of the order of $4 \times 10^{18}$ photons/cm$^2$. sec.

3.6 Electron Irradiation

Most of the irradiations were performed with the sample at room temperature, using a pulsed linear accelerator. The electron beam of energy 3.5 MeV, was pulsed at 20 cps, the width of each pulse being 2 μsec. Each pulse corresponds to a flux of $4 \times 10^{11}$ electrons/cm$^2$. The irradiation time for each sample was typically 30 minutes. This corresponds to a flux of $1.4 \times 10^{16}$ electrons/cm$^2$.sec. All the irradiations were performed in vacuum at a pressure of the order of $10^{-5}$ torr, and the conductivity measurements were done in-situ. To minimize any heating effects during irradiation, the samples were mounted on a water cooled substrate holder. The bulk temperature rise for a 30 min. irradiation time was measured to be less than 0.5°C, the temperature being monitored by using a bare thermistor bead, mounted on a glass plate. The suitability of various glass substrates like pyrex, quartz, 7059 corning, was investigated. After irradiation, the resistance of 7059 corning glass substrate was found to be higher than that of the amorphous Silicon film deposited on to it. Hence, corning 7059 glass substrates were suitable for irradiation experiments. In an attempt to freeze the number of
defects produced during irradiation a few experiments were carried out with the sample at $125^\circ$K, by means of a cold finger.

3.7 I.R. Measurements

The amorphous Silicon films were deposited on chemically polished Silicon substrates of resistivity 100 ohm cm. Film thickness was typically 1/\mu m. Another identical substrate was used as reference and the infra-red transmission spectra were recorded using a Perkin-Elmer 457 double beam infra-red spectrophotometer at room temperature.
REFERENCES

1. Hand Book of Thin Film Technology, ed. by L.I. Maissel and

2. "Scientific Foundations of Vacuum-technique", by Dushman, Saul,

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