CHAPTER 2 : EXPERIMENTAL TECHNIQUES

The experimental techniques used in the present work are introduced in this chapter. The basic principles, the means and the merits of the techniques are emphasised.

2.1 Sample preparation

Microscope clear glass slides of thickness 1-1.2 mm, were used as substrates for sample preparation. Prior to sample deposition, the substrates were treated with acetone and distilled water and then they were agitated ultrasonically to remove contaminants (such as grease or oil) from the surface of substrates.

The deposition of CdTe was carried out using Edwards AUTO 306 via electron-beam evaporation technique. Masks with dimensions 1 cm x 1 cm were prepared to deposit thin films of the desired dimension onto the substrates.

Edwards AUTO 306, an e-beam apparatus is made up of three major parts. They are vacuum pumping system, vacuum chamber and electron-beam supply. A schematic arrangement of the system is shown in figure 2.1.

Vacuum pumping of the system is achieved by means of diffusion using Edwards ED4/160K diffusion pump. A microprocessor based control system was administered to achieve an automatic switching sequence between roughing and backing processes.

A plan view of the component parts of the six-position electron-beam source is shown in figure 2.2. CdTe granules of 99.99% purity of diameter ~ 3 mm were placed onto the turrets of the e-beam source. The pressure of the chamber was maintained at 1

1.5×10^{-5} torr during degassing and deposition processes to obtain less contaminated samples.

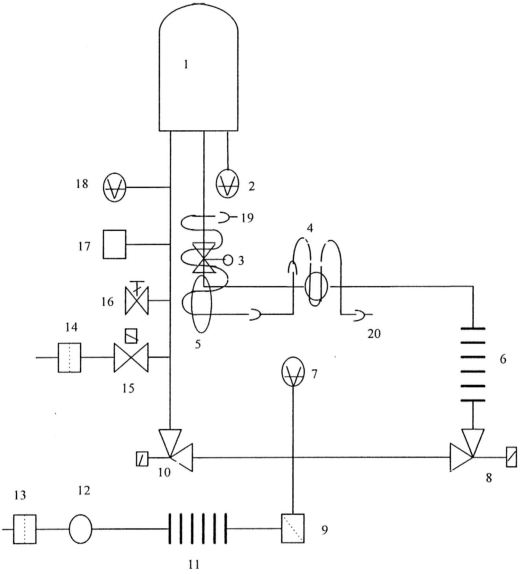
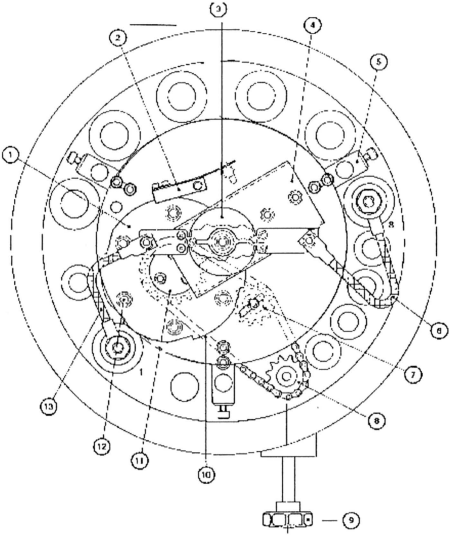


Figure 2.1 : Schematic system of Edwards-Auto 306 Evaporator.

1. Vacuum Chamber
2. Penning Gauge (CP25K)
3. High Vacuum Valve
4. Diffusion Pump
5. Liquid Nitrogen Trap
6. Flexible coupling
7. Pirani Gauge -PRM10K
8. Backing Valve
9. Foreline Trap
10. Roughing Valve
11. Flexible Coupling
12. Rotary Pump
13. Oil Mist Filter
14. Air Admit Filter
15. Air Admit Valve
16. Gas Admit Valve
17. Vacuum Interlock Switch
18. Pirani Gauge PRL10K
17. Water Line (In)
20. Water Line (Out)



1. Turret
2. Index Spring
3. Electron Beam Gun
4. Carrier
5. Tripod Mounting Lug
6. Live Braid
7. Tension Sprocket
8. Drive Sprocket
9. Drive Knob
10. Drive Chain
11. Turret Sprocket
12. Turret Source Holder
13. Live Braid

Figure 2.2: Component Parts of the Six Position Electron Beam Source (Plan View) [55].

2.2 XRD

The structural characteristics of the e-beam evaporated CdTe films were studied using a Philips PW 1840 compact powder X-ray Diffractometer with a CuK_α radiation ($\lambda_{\alpha 1} = 1.54060$ Å and $\lambda_{\alpha 2} = 1.54438$ Å).

XRD analysis enables the differentiation and determination of phases (amorphous and crystalline), unit cell dimension, space group, crystal systems, atomic co-ordinate and crystallite sizes.

The principle of an X-ray Diffractometer is illustrated in figure 2.3 below. X-rays travels from the target to the sample chamber and from the sample to the detector. The rotation angle, θ, is related to the interplanar spacing, d, by the well known equation of Bragg, $n\lambda = 2d \sin\theta$.

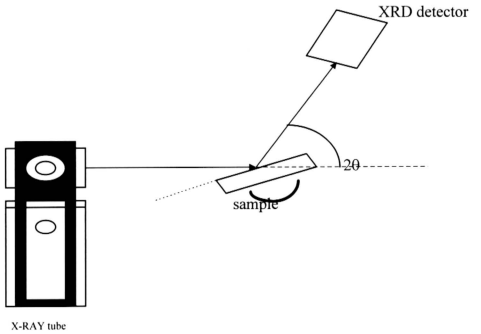


Figure 2.3 : The principle of an X-ray Diffractometer

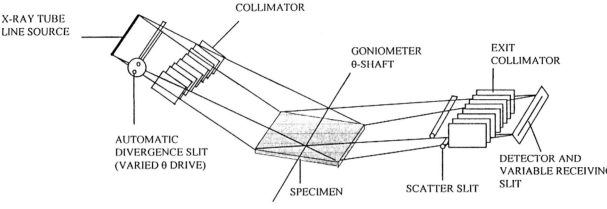


Figure 2.4: Goniometer X-Ray path [56].

A typical X-ray arrangement is shown in figure 2.4 [56]. The primary radiation passes through the entrance collimator and irradiates the sample. The sample scatters the beam. The reflected beam/radiation is measured by a solid state Si-array detector, after passing through the solar slits (secondary beam tunnel).

The diffractogram for thin film CdTe samples were taken in the range $10^{o} \leq 2\theta \leq 100^{o}$ for a trial scan. Subsequent scans were done in the range $20^{o} \leq 2\theta \leq 30^{o}$ as the preferred orientation ([111]) for the crystalline CdTe occurs at $2\theta = 23.8$ [44].

2.3 SEM / EDX / TEM

Scanning electron microscope (Philips Model 515) and an energy dispersive X-ray analyser , EDX, (Philips Model PV 3800) were used to obtain micrographs of sample morphology and to obtain elemental composition of Cadmium and Tellurium in the prepared sample, respectively.

Scanning electron microscope consists of 4 main parts, they are the electron source/gun, a deflection system, a detection system and a vacuum system as shown in figure 2.5. Electron gun produces a source of electrons and accelerates these electrons to an energy in the range 1- 40 keV. Electron lenses are used to reduce the diameter of this source of electrons (spot size, less than 10 nm) and to place a small, focused electron beam on the specimen.

A deflection system consists of two pairs of electromagnetic deflection coils (scan coils) are used to control the raster of beam in order to produce contrast in an image. The interaction of the electron beam with the specimen causes the generation of many signals and the two most often used to produce images are secondary electrons (SE) and backscattered electrons (BSE). These electrons are collected by detectors and converted into an electrical signal. The electron beam traverses the sample surface point-by-point along a line and the back-scattered electron signal is collected and generated into an image.

X-rays emitted from a specimen bombarded with the finely focused electron - beam of the SEM can be used to identify which elements are present in the sample. This technique is known as energy dispersive X-ray analysis. It allows quantitative analysis of chemical composition with an accuracy and precision approaching 1%. Elemental constituents can be determined to concentrations ranging as low as 0.01% (100 ppm) which corresponds to limits of detection in terms of mass 10^{-16} to 10^{-15} g.

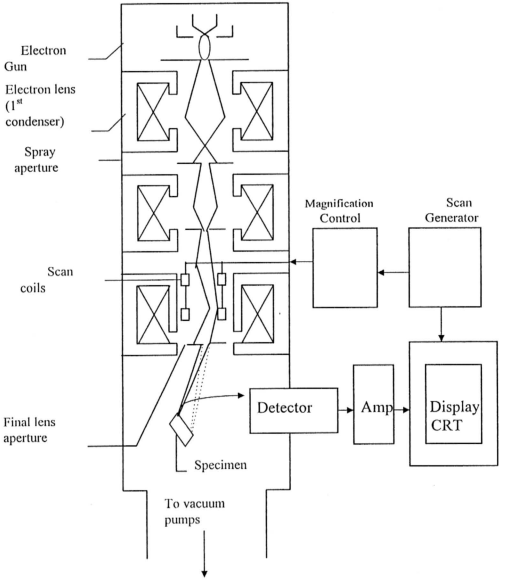


Figure 2.5 : Schematic drawing showing the electron column, the deflection system and the electron detectors of a SEM.

Its limitation are that it cannot distinguish between ionic, non-ionic and isotopic species and EDX in particular cannot detect the low atomic number elements. EDX fitted with beryllium window detectors can detect elements with $Z < 11$ while EDX with windowless detectors can detect elements with $Z > 5$. EDX is a surface analytical technique and because of the vacuum requirements of the SEM, it is not suitable for hydrated samples.

The technique for quantifying results is ZAF. ZAF is an acronym from three separate effects; atomic number (Z), absorption (A) and the fluorescence (F) which the method compensate for. The compositional results were reported as atomic percentage (AT %) and weight percentage (WT %).

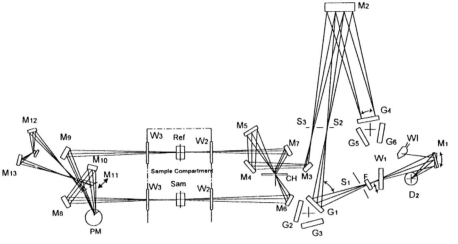
Since CdTe is a non-conductive material, the samples which were mounted on an Aluminium platforms with a conductive carbon cement agar (Agar Aids), were coated with carbon using Bio Rad (mode E5100 series 11) evaporator for the SEM/EDX analysis.

As for the TEM analysis, Philips CM 12 Transmission Electron Microscope was used. Samples were prepared by directly coating the thin films onto copper grids. This analysis allows in depth analysis of thin films, to see the internal structure of thin films and to obtain physical crystallite sizes of CdTe.

2.4 Transmission spectroscopy

Optical properties of thin films were determined from the transmission spectra obtained using a Shimadzu UV 3101-PC UV-VIS-NIR double beam scanning

amplifier to be separated into sample signal, reference and dark level signals before it is detected by the detectors.



D2: Deuterium lamp
W1 Halogen lamp
F: Filter
G1~ G3: Gratings of first monochromator
G4~ G6: Gratings of second monochromator
S1: Entrance slit
S2: Intermediate slit
S3: Exit slit

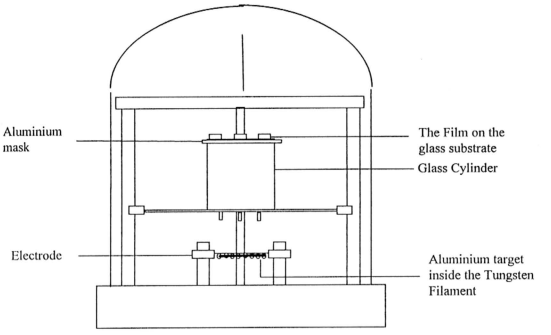
W1~ W3: Window plates (W1, W2, 30 mm dia.;W3, 40mm dia.)
CH: Chopper mirror
M1~ M13: Mirrors (M11 is detector switching mirror)
Ref: Reference cell
Sam: Sample cell
PM: Photomultiplier
PbS: PbS cell

Figure 2.6: Optical Schematics of UV-3101PC [57].

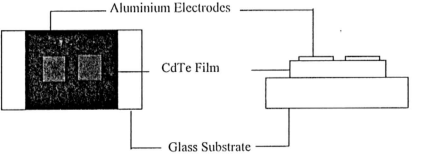
2.5. I-V Measurement

For the purpose of electrical studies aluminium electrodes were deposited onto CdTe film via a thermal evaporation system. The pumping system is basically similar to the e-beam system with a diffusion pump backed by a rotary pump. The arrangement of the chamber is shown in the figure 2.7 (a)

Aluminium was used as contact metal. The source (Al) was placed on a tungsten filament with both ends connected to the anode and cathode. The current



(a)



(b)

Figure 2.7: (a) A cross sectional view of the chamber used in the deposition of the electrodes
(b) A plan and a cross views of the deposited electrodes on the film.

across the two electrodes is controlled by a variac. The CdTe films were placed on an aluminium mask which rests on top of a cylindrically shaped glass. The dimensions of the electrodes are (5 x 4) mm^2 with a 0.2 mm width between the electrodes as shown in the figure 2.7 (b). Silver paste was used to provide a contact between the aluminium electrodes and copper wires which were connected to Keithley 237 d.c. power source.

I-V measurements were carried out at room temperature using a Keithley 237 high voltage power source. The schematic diagram of the circuit is shown in the figure 2.8. To reduce the electromagnetic interference (EMI) the samples were placed in a an aluminium shield and the connection were made. A d.c. voltage was swept across the sample in direct and reversed bias mode and the results were recorded.

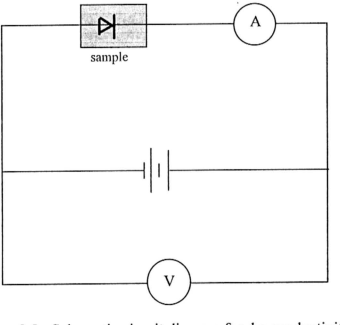


Figure 2.8 : Schematic circuit diagram for d.c.conductivity measurement.