# Experimental Setup Used for Fabrication and Characterisation of Thin Films

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**Abstract:** ***This study presents the experimental setup and characterization methods used for the fabrication of thin films intended for semiconductor and infrared detector applications. Thin films were deposited using flash evaporation under controlled vacuum conditions to achieve compositional stability and film uniformity. Chemical composition and homogeneity were evaluated using energy dispersive X-ray analysis, confirming near-stoichiometric deposition with minimal elemental loss. Surface characteristics and film thickness were determined using ellipsometry, while optical properties, including reflectance, transmittance, and absorbance, were measured using UV-VIS-IR spectrophotometry. The combined methodology provides a reliable framework for assessing structural, compositional, and optical properties of thin films and supports their application in advanced optoelectronic materials. (1- 5)***

**Keywords:** Thin films; Flash evaporation; EDAX; Ellipsometry; UV-VIS-IR spectroscopy; Semiconductor materials, Infrared detectors

## 1. Experimental Coating Unit

A simple and convenient technique which can be used to deposit thin films of binary alloys is the flash evaporation method. In this technique, proper selection and control of evaporation boat and substrate temperature provides adequate control of stoichiometry of the films. Here, a small quantity of the multicomponent material, dropped onto the pre-heated boat, cracks instantaneously into its components which then travel towards the substrate. There is practically no time for the more volatile components to be lost preferentially and give rise to the loss of stoichiometry. It is thus obvious that mixtures or alloys prepared over complete range of a particular system could be flash evaporated without loss of stoichiometry. Thin films of were deposited using flash evaporation technique. This technique provides a convenient method for the preparation of stoichiometric films of materials whose constituent elements have widely different vapour pressures. In such a technique, the boat temperature is maintained high enough to ensure complete instantaneous evaporation of small grains of the material fed to the pre-heated boat. The evaporated material condenses on substrates to provide the desired films. Even in cases where the material grains decompose into their constituents, a proper choice of substrate temperature can lead to stoichiometric thin film deposition on the substrate. Experimental set up used by us for the flash evaporation deposition of the films is shown in fig.1 The system was a Hindhivac vacuum coating unit Model 12A4-D.

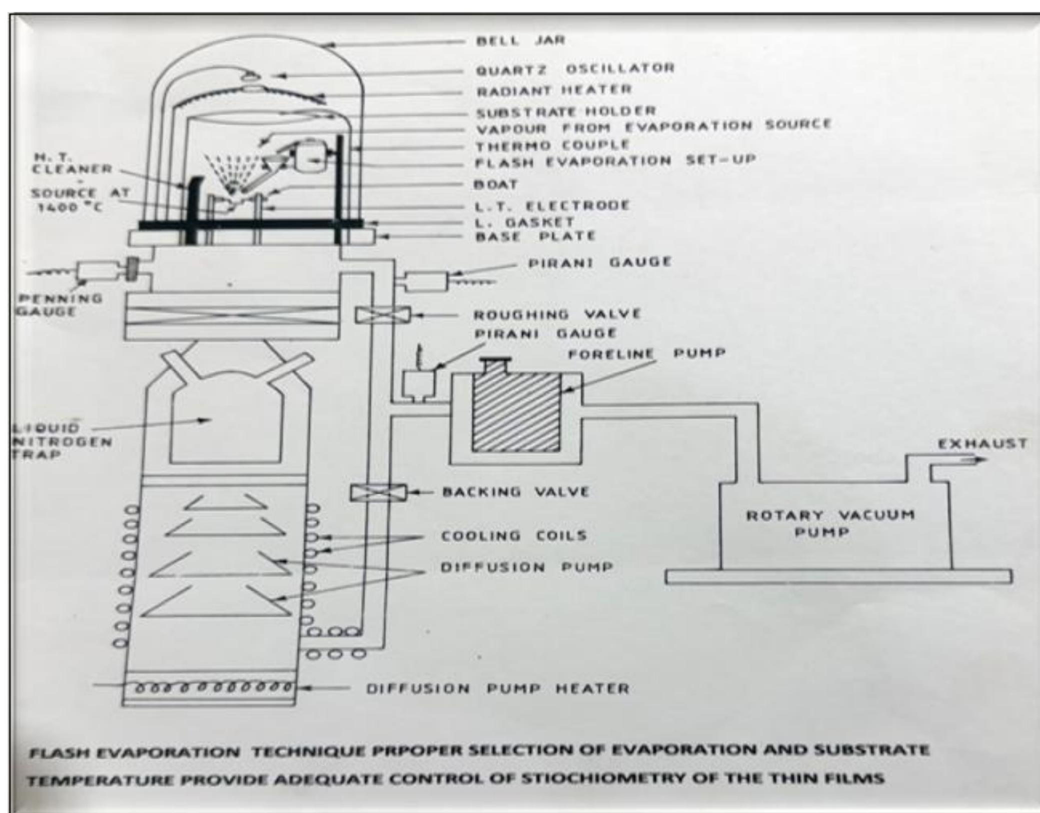


**Figure 1:** Schematic Diagram of Vacuum Coating Unit

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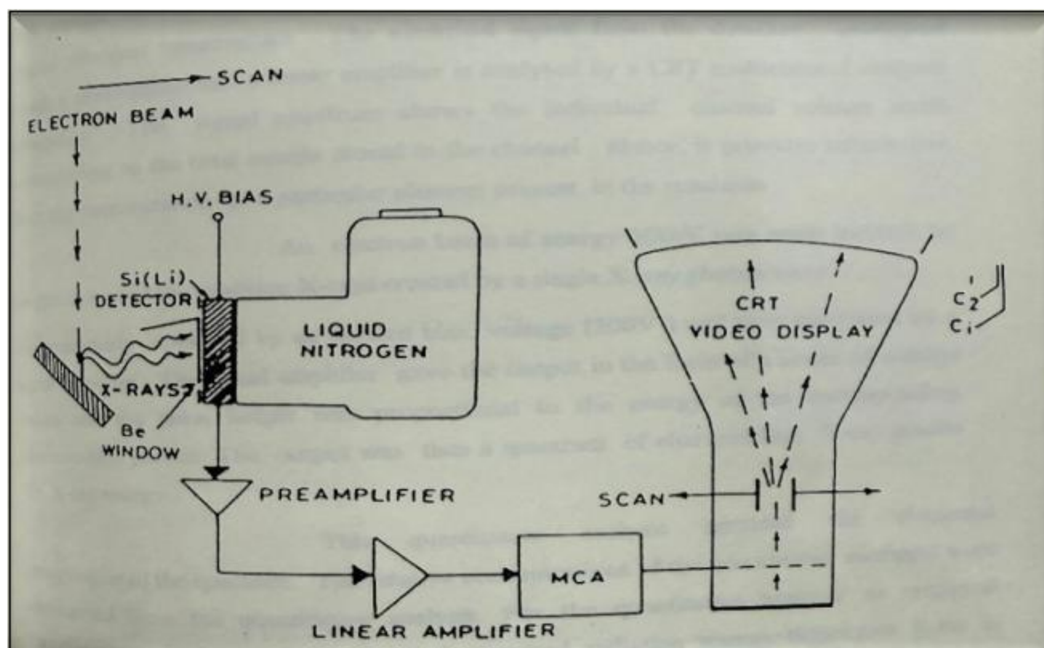


**Figure 2:** Schematic Diagram of Solid State Energy Dispersive X-Ray System

**Energy Dispersive X-Ray Analysis (EDAX):**
Chemical composition/concentration and homogeneity of the films were confirmed by means of EDAX. The compositional analysis was carried out using a PV 990 EDAX attachment on a Philips SEM 515 Model. The EDAX system has a lateral resolution of 0.1%. Measurements were carried out under a vacuum of $10^{-5}$ Torr. A schematic diagram of the instrument is given in fig 2. EDAX is based on the principle that X-ray quanta emitted from a specimen during electron beam excitation have wavelengths/energies characteristic of the constituent elements of the specimen and hence reveal the elemental composition by means of an SEM equipped with a spectrometer capable of analysing energy spectrum of the emitted characteristic X-ray. A beam of 30KeV electrons made incident on selected areas of specimen results in the emission of X-rays which have energy/wavelength spectrum characteristic of the elements constituting the specimen under investigation. While the wavelength/energies are characteristic of the elements, the number of the X-ray photons of particular element provides information about the concentration of the element in the specimen. The X-rays emitted from the specimen are made incident on a biased Si(Li) detector cooled to liquid nitrogen temperature. The electrical signal from the detector, processed through a preamplifier and a linear amplifier is analysed by a CRT multichannel analyser combination. The signal spectrum shows the individual channel voltage levels corresponding to the total counts stored in the channel. Hence, it provides information about the concentration of a particular element present in the specimen. An electron beam of energy 30keV was made incident on the specimen, and the resulting X-rays created by a single X-ray photon were instantaneously collected by an applied bias voltage (300V) and then integrated by a signal amplifier. The signal amplifier gave the output in the form of a series of voltage pulses and the pulse height was proportional to the energy of the corresponding characteristic photon. The output was thus a spectrum of characteristics X-ray counts Vs. X-ray energy. This quantitative analysis revealed the elemental composition of the specimen. The relative concentrations of the constituent elements were determined from the quantitative analysis. For the quantitative analysis an empirical approach was used. The intensity of the emitted radiation energy dispersive X-ray is proportional to the concentration of that element. Certified standards, available with very accurate compositional data were used. Quantitative analysis of the composition of the specimen using X-ray spectrum from the specimen was made by using software provided for the purpose. Certified standards available with very accurate compositional data, were used to calibrate the quantitative program which was then used to relate the observed intensity of the peaks to the concentrations. Due to statistical nature of the X-ray production, the intensity of the emitted radiation is not linearly related to concentration and hence X-ray data was quantified for a given sample and compared to the sample until closest fit was achieved. To negate any effects due to the conducting nature of the sample, an electrode was used to connect the specimen to the base of the specimen holder stub. Since the X-rays originate from a significant depth within the specimen, some X-rays may be absorbed by the other atoms in the specimen. This self- absorption gives rise to secondary emission or X-ray fluorescence (defined as the excitation of lower energy shells by higher energy X-rays). This leads to artificial enhancement of low energy peaks at the cost of higher energy peaks. Errors incorporated due to the 'Absorption-Fluorescence' were corrected by using the quantization program incorporating the ZAF-correction, (Z = atomic number, A= Absorption and F= Fluorescence). Using the EDAX system the author confirmed the chemical composition, concentration and homogeneity of the film samples as well as its source (powdered) material. Towards this end, the EDAX procedure described above was used on various areas of the thin film under analyses and the samples were checked for stoichiometry and homogeneity. The source material for the film sample was then mounted onto the specimen holder and similarly analysed for symmetry and homogeneity. The results from the two were then compared to detect loss of any element during the evaporation process. The results showed that within an error limit of 2-3% all the samples were perfectly homogenous and stoichiometric and

that the deposited films had the same composition as the starting material.

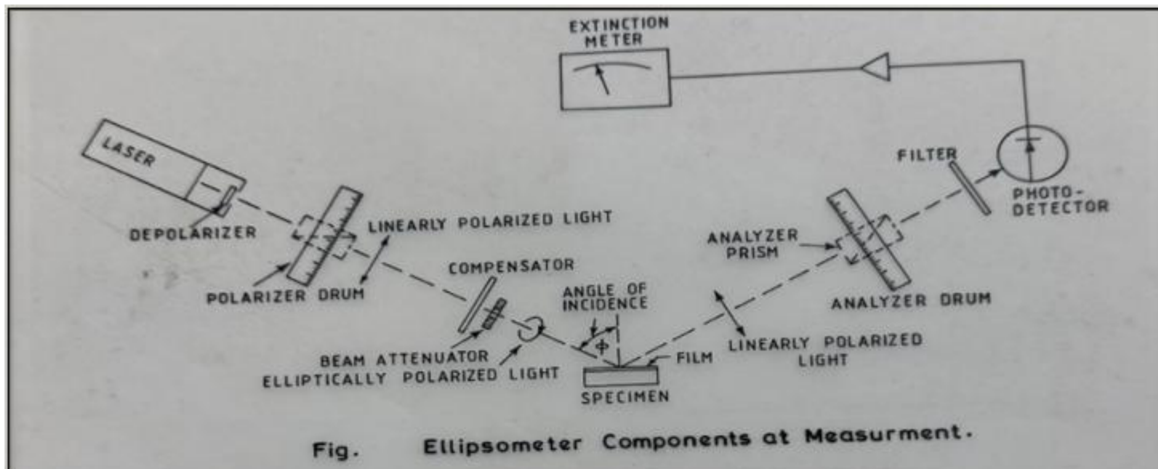


**Figure 3:** Schematic Diagram of Ellipsometer

**Ellipsometry**

Surface properties of MCT crystals after polishing and the thickness of the deposited films were determined by means of ellipsometry. The ellipsometry was done using a Gaertner Ellipsometer. It works on the principle of evaluating the change in the state of polarisation of light reflected from a substrate. The state of polarisation is determined by the relative amplitude of the parallel ($P_p$) and perpendicular ($P_s$) components of radiation and the phase difference between the two components $_p$- $\Delta p$- $\Delta_s$ On reflection from a surface or film, the ratio of the two amplitudes $P_p$ / $P_s$ and the phase difference between the two components, - As, undergo changes which are dependent upon the optical constants of the substrate $n_3$ (refractive index), $k_3$ (extinction coefficient), the angle of incidence $\theta_1$, the optical constants of the film $n_2$(refractive index), $k_2$ (extinction coefficient ), and the film thickness d. Thus, in principle with complete knowledge of the state of polarisation of the incident and reflected light, the refractive index $n_2$ and thickness d can be determined. The main components of the ellipsometer are shown in fig. 3 Light from the helium-neon laser (6328Å) is first linearly passed through the polariser after which it is elliptically polarised by passing through the compensator. When light reflects from the specimen under measurements, the polarisation of the light changes in accordance with the specimen film thickness and optical characteristics of the film and substrate. The light then passes through the analyser and is sensed by the photodetector. A filter mounted in front of the photodetector eliminates unwanted background light so that measurements can be done in normal conditions. The amount of taser light reaching the photodetector is indicated by the extinction meter. There are certain settings of the polariser that cause the larger light reflecting from the surface of the specimen to become completely linearly polarised. When the polariser is at one of the settings the analyser can be rotated to a position where almost no light reaches the photodetector. The extinction meter then moves to its lowest readings. This is the condition for measurement and the analyser and polariser drum setting is recorded. This contributes the first set of readings recorded. The second polariser setting and corresponding analyser positions give the second set of values. The four drum readings are $A_1$, $A_2$ for the analyser drum and $P_1$, $P_2$ for the polariser. The values of Δ (delta) and Ψ(psi) are calculated by using the following relations

$\Psi = 180- (A_1-A_2)/2$
$\Delta = 360 - (P_1+P_2)$

The film thickness and other optical parameters are determined by using graphs and tables provided by Gaertner Scientific Corporation.

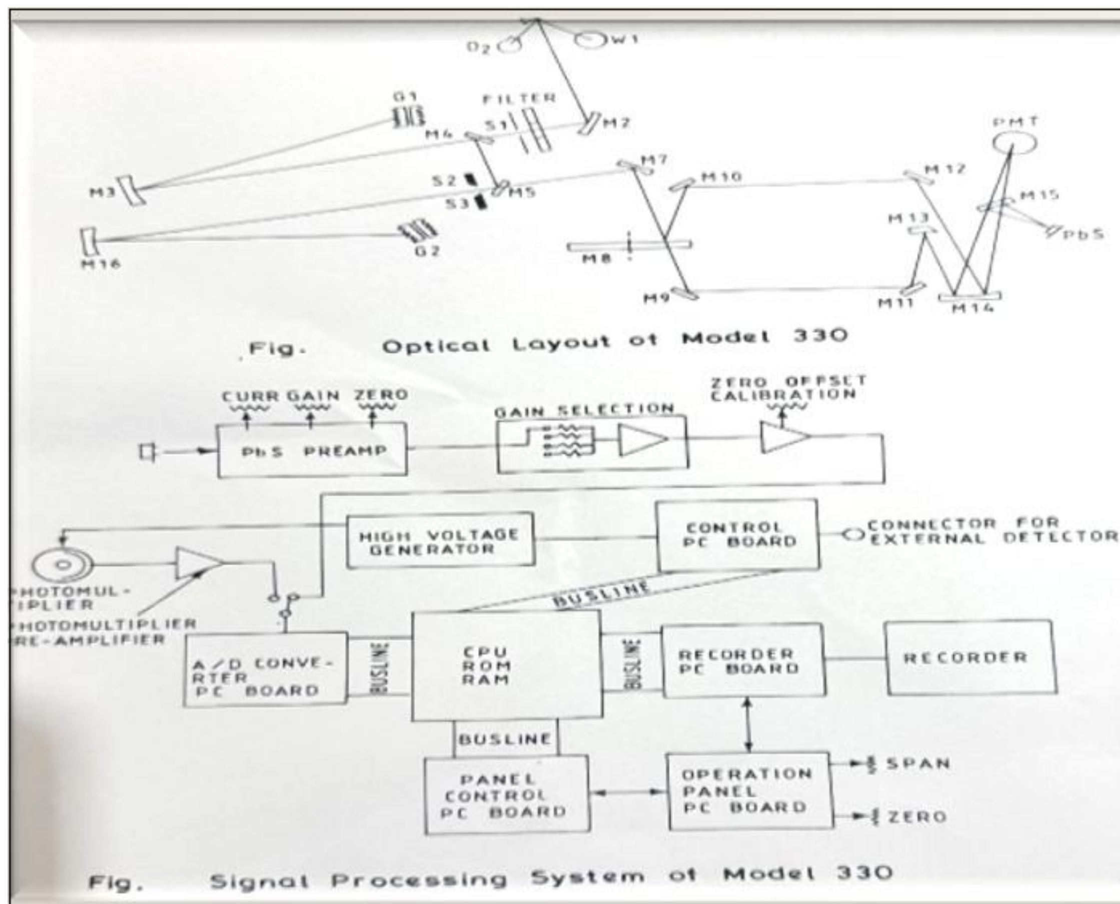


**Figure 4:** Schematic Diagram of UV-VIS-NIR Spectrophotometer

**Spectrophotometer**

The reflectance, transmittance and absorbance spectra of the thin films were recorded by a UV- VIS -IR spectrophotometer of Hitachi model 330. The optical layout and signal processing system of this model are shown in figs. 4, the spectrophotometer operates on the principle that light emitted from the light source passes through two grating monochromators for preparing a monochromatic beam, and then is split into two beams. After passing through the sample compartment, the monochromatic beams are converged and then converted into an electric signal by a detector, photomultiplier, or PbS cell. The electric signal provided from the detector is amplified by a preamplifier and immediately converted into a digital variable by an A/D converter. At subsequent stages, the signal is processed by a CPU and the computational result is displayed directly on the output recorder. Using the Hitachi 330 spectrophotometer, we can measure spectra in the range 187~ 2500 nm having a wavelength accuracy of ~ + 0.2 nm for UV-VIS region. In our case the spectra were analysed in the 500-2000nm range.

## 2. Conclusions

This study demonstrates that flash evaporation is an effective method for preparing homogeneous and near-stoichiometric thin films of multicomponent materials. EDAX confirmed compositional integrity, ellipsometry enabled thickness and surface characterization, and UV-VIS-IR spectroscopy provided optical property assessment. The integrated experimental approach offers a reliable framework for evaluating thin film materials relevant to semiconductor and infrared detector applications.

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