CHAPTER - I

INTRODUCTION

1.1 General

Several interesting applications in solid state devices of elemental semiconductors Si and Ge as well asthe compound semiconductors such as GaAs, InP and GaP, belonging to III-V group, CdTe, CdS and ZnSe, belonging to II-VI group are already well known. The wealth of information obtained from the exhaustive studies on the opto-electronic properties of these materials forms generally the guideline for the basic understanding of the other compound semiconductors, being developed and studied in recent years. The materials of the present investigation namely, Zn_3P_2 and Cd_3P_2 , and their ternaries $(Zn_xCd_{1-x})_3P_2$ with different compositions x which belong to the II-V group are being actively studied for possible applications in Photovoltaic devices (mainly for solar energy conversion), IR sensors and lasers (7,12 & 13). Zn_3P_2 and Cd_3P_2which possess tetragonal crystal structure with eight molecules in a unit cell, are direct band gap materials (E_g $\simeq$ 1.50 and 0.50 eV for Zn_3P_2 and Cd_3P_2respectively). Homojunction (p-n) devices could not be developed using these compounds because Zn_3P_2 is always intrinsically p-type and Cd_3P_2 is n-type. While large amount of experimental data on the structural, electrical and optical properties of bulk materials of Zn_3P_2 - Cd_3P_2 systems have been reported in the literature similar systematic

studies have not been so extensively carried out on the thin films of these systems. Studies on the ternary films of these systems are particularly very meagre.

Thin films can be grown on glass as well as crystalline substrates using various deposition techniques such as vacuum thermal evaporation, hot wall epitaxy, electron beam evaporation, chemical vapour deposition, RF sputtering etc. Majority of the studies on Zn_3P_2 have been made with the thin films prepared using either vacuum thermal evaporation or hot wall epitaxy technique. Practically no work has been done on the e-beam deposited thin films of Zn_3P_2- Cd_3P_2 systems.

In the present study polycrystalline thin films of Zn_3P_2, Cd_3P_2 and ternaries $(Zn_xCd_{1-x})_3P_2$ with different compositions x have been deposited on glass substrate under different conditions by e-beam evaporation technique. The bulk materials used in the deposition of thin films, have also been prepared in the laboratory using the specpure constituent elements. The structural and chemical characterization of these films have been carried out with the help of XRD, EDAX and XPS.

Electrical conductivity, Hall mobility, optical absorption and photoluminescence have also been studied and these results are reported in Chapter IV. Optical constants (n, the refractive index and k, the extinction coefficient),

optical band gap (E_g) and the band splitting parameters have been calculated and these data is first of its kind on these systems.

The salient features observed in this investigation have been explained in Chapter V considering the role of the native defects of these systems. The background informations regarding the deposition techniques and other different aspects of this investigation are briefly discussed in the following section.

1.2 Thin Film Deposition Techniques

Deposition techniques commonly used for obtaining semiconductor films can be grouped conveniently into three catagories namely, physical vapour deposition (PVD), chemical vapour deposition (CVD) and electro-chemical deposition techniques. The main steps involved in the formation of a deposit are (i) generation of the depositing species in forms of vapour streams or jets, (ii) transport of these species from source to substrate, and (iii) film growth on the substrate by condensation and nucleation process.

The essential requirements in the most PVD techniques is initial high vacuum ($< 10^{-6}$ Torr) while chemical reactions of the species has to be affected under equilibrium thermodynamic conditions in the case of CVD techniques. The

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latter technique is commonly used for preparing epitaxial films of compound semiconductors. A suitable electrolyte (acqueous) has to be selected to initiate the deposition process under optimal conditions in the case of electro deposition technique. In all these processes the quality (homogeneous, well-adherent and chemically stable) of the films depends upon the nature of the substrate surface (smooth and well-cleaned), temperature of the substrate and deposition rate. The different techniques in each of these above categories are briefly discussed in the following sub sections.

1.2.1 Vacuum Thermal Evaporation

Thermal evaporation is a widely used method in the laboratory for depositing metals, alloys and other low melting compounds. In this method, the material is created in a vapour form by means of resistive or RF heating. The resistive heating of materials is done with the help of filament, or boat generally made of refractory metals such as W, Mo, Ta and Nb. The choice of the support material is primarily determind by the evaporation temperature and resistance to alloying and chemical reaction with the evaporant[1 & 5]. On heating a material in vacuum, it evaporates at a rate (m_e) given by the well-known Langmuir-Dushman equation,

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$m_e = 5.83 \times 10^{-2} p_e (M/T)^{1/2}$ g/cm^2-sec where p_e (<10^{-2} Torr)

is the equilibrium vapour pressure of the evaporants at a temperature T, and M is the molecular weight. The vapours thus created are, transported through vacuum to get deposited on the substrates. Only at pressure < 10^{-5} Torr, due to the large mean free path at the low pressures, vapours arrive at the substrate unscattered and get deposited. The deposition rate depends on the source geometry, its position relative to the substrate, and the condensation coefficient. Further, rotation of the substrate during evaporation yields uniform thickness of the films.

1.2.2 Electron Beam Evaporation

. The simple resistive heating process described in section 1.2.1 suffers from the disadvantages of possible contamination from the support materials and the limitations of the input power, which make it difficult to evaporate high-melting point materials. In electron beam evaporation, temperature as high as 3000°C could be obtained due to the striking of high energy (4 to 10 KV) electrons at the focussed spot on the materials. Rest of the material remains cool and the interaction between the material and the support is eliminated, thereby reducing the contamination effects [1,5 & 15], the problems mentioned above in the case of thermal evaporation technique are mostly overcome in this

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case. Further, since the input power is very high (several kW), extremely high rates of evaporation can be achieved, even for high melting materials.

The electron gun used for evaporation consists of a heated W filament for electron emission. The filament is normally shielded to prevent any sputtering by the vapour species and gaseous ions. Depending on how these emitted electrons are accelerated, the guns are called work-accelerated or self-accelerated. In work-accelerated guns, which can be operated only at voltages < 10 kV, the electric field is applied between the cathode (i.e. the filament) and the evaporant. If a separate anode is used, having an aperture to allow the passage of electron beam, the gun is called self-accelerated. These guns can be used at higher voltages as compared to the work-accelerated guns. The electron-source filament is generally heated by a low-tension (6 to 12 V, 100 mA) transformer, while the high tension (0 to 20KV with current upto 500 mA) transformer supplies the power necessary to accelerate the electrons. The same geometric considerations mentioned for thermal evaporation, have to be taken care in this case also in order to obtain good deposition of the film on the substrate. The other experimental details of the vacuum coating unit and EBS are given in Chapter - III.

1.2.3 Laser Evaporation

In this method a high power (∼MW) laser beam strikes a small portion of the material for a very short duration of time (μs to ns) and affects the evaporation process at a very fast rate. The laser source is generally kept outside the vacuum system and the beam is focussed onto the surface of the material to be evaporated. The temperature of the vapour source can be raised by the laser beam upto 10^4 K in such a short time. Laser processing is similar in many respects to e-beam evaporation and thin films of particularly high melting materials can be grown by both these techniques. In recent years laser processing or laser annealing technology is being widely used to improve the crystalline properties of ion implanted amorphous or polycrystalline layers on a (single crystal) semiconductor substrate. This method has distinct advantages because of very short annealing time, local area annealing and providing adequate heat treatment to the films without impairing the characteristics.

1.2.4 Hot Wall Deposition Technique

Hot wall deposition (HWD), also known as hot wall epitaxy (HWE) when the emphasis is on epitaxial growth, is also a frequently used method by which the growth of thin semiconductor films occurs under conditions that are very close to thermodynamic equilibrium. This techniques uses a

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heated liner (hot wall) which serves to enclose and direct the vapour from the source to the substrate and thus avoids the material loss, maintains the high vapour pressure of the compound or its components. Since the temperature difference between the source and the substrate is reduced to minimum, it is essentially a high temperature growth process. It has been widely used for the growth of epitaxial films and multilayer structures of IV-VI & II - VI compounds. Since wall condensation is prevented and the vapour pressure of the volatile constituents builds up, steady state rates of deposition of films with nearly stoichiometic composition become possible by this method.

1.2.5 Molecular Beam Epitaxy (MBE)

MBE is one of the most versatile techniques for growing epitaxial layers under UHV ($< 10^{-9}$ Torr). In this method molecular and /or atomic beams of constituents from Knudsen (effusion) sources impinge upon the substrate and get condensed. In MBE, the growth temperature is low (600°C) and the growth rate is small (1 to 10 Å/sec) . Thus, it is possible to deposit epitaxial films of compound semiconductors with precisely controlled properties and thickness. Normally, the system is equipped with a mass analyser to monitor the evaporated species. To control the deposition conditions at the substrate, in-situ characterisation techniques such as electron diffraction, SEM

AES, XPS etc. are also included in MBE systems built-in-accessories. MBE technique has been very profitably and widely used to deposit multilayer structures of compound semiconductors to fabricate quantum well super structures, heterojunctions and graded composition structures.

1.2.6 Sputtering Technique

Sputtering is the process in which the atoms of the target (cathode) materials, removed by the bombardment with highly energetic ions (usually Ar^{+}), get deposited on the substrate under suitable conditions. The procurement of a target of highest purity with proper dimensions becomes, therefore a prerequisite in order to make use of profitably the sputtering technique for the deposition of thin films of compound semiconductors. This technique has also been widely used for cleaning the surface of the semiconductors in the characterization technique. Some of the commonly used sputtering techniques are briefly discussed in the following.In DC Sputtering , the cathode each covered with target material to be deposited and the substrate is placed on the anode. High voltage (kV) applied between the two electrodes produces high energitic Ar^{+} ions in the glow discharge (10^{-1} Torr) which sputter the target resulting the deposition of thin film on the substrate. Films of pure metals and alloys can be deposited by this technique. But in the case of RF Sputtering , which is, in general, used for

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deposition of thin films of ceramic materials and semiconductors, ac field (13.56 M Hz) is generated in the system. In the rf glow discharge system the operating pressure ($\sim 10^{-3}$ Torr) is lower compared to dc diode sputtering.

In both these methods the secondary electrons generated during the bombardment near the target surface produce radiation damage and also increase the substrate temperature. In Magnetron Sputtering where magnetic field is applied transverse to the electric field, these effects are minimized. In this process electrons acquire cycloidal motions increasing the collision probability with Ar molecules. This enables to lower the gas pressure as low as 10^{-5} Torr compared to the processes (10^{-1} and 10^{-3} Torr) in the DC and RF sputtering respectively. This technique thus allows the use of substrates that are temperature sensitive (e.g. plastic materials) and surface sensitive (e.g. MOS device) with minimal adverse effects. In addition, Magnetron sputtering source produce higher deposition rates than other conventional source and lends itself to economic, large area industrial applications [15].

1.2.7 Chemical Vapour Deposition (CVD) Technique

CVD is one of the most important and versatile methods for the deposition of a very large number of compound

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semiconductors at relatively low temperatures. Thin films with high degree of perfection and purity can be grown with required stoichiometry by this method [5 & 15]. In this meterial synthesis process, constituent vapours react chemically near or on a substrate surface to form a solid deposit. The vapours are transported to the reactor either by a pressure differential or with the help of a carrier gas to the substrate. The chemical reaction can be accelerated with the help of external agencies such as, light or X-rays (Photo-Enhanced CVD), laser beam (Laser Induced CVD), an electric arc or glow discharge (Plasma Assisted CVD), etc. Apart from films of compound semiconductors, oxides of a large number of elements, silicate glasses, carbides, nitrides and oxycarbides have also been deposited by CVD technique.

Another method for growing device quality III-V compound semiconductor films is metal organic chemical vapour deposition (MOCVD) technique. As for example, trimethylgallium (TMG) is reacted with arsine (AsH_3) at 700°C to form GaAs.

1.2.8 <u>Electro Deposition</u>

Electro deposition is a process of depositing a substance upon an electrode by electrolysis. Thin films of CdSe or CdTe have been deposited by co-deposition of Cd and

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Se/Te from their aqueous solutions. Anodization is also an electrolytic process in which the metal oxides are formed using the metal as the anode in a suitable electrolyte.

1.3 X-Ray Diffraction (XRD)

The XRD gives a wide range of information about the crystal structure, nature of the film (crystalline or non-crystalline), orientation and size of the crystallites etc. The conventional X-ray diffractometer is used to obtain these information in the case of films of thickness of the order of microns. If the film thickenss is < 0.1 μm, the X-ray diffracted intensity becomes very low. In such cases, in order to get a sufficient signal to noise ratio, a grazing angle diffraction method is used for increasing the sampling volume. The interplanar distance (d_{hkl}), the lattice constants and particle size can be estimated from the XRD data and these details are discussed in Chapter - III.

1.4 Electron Probe Microanalysis (EPM)

EPM is an accessary to a conventional SEM. The electron beam which impinges on the specimen, generates characteristic X-rays from the constituents of the material. These X-ray photons can be detected and analysed in two ways namely, wavelength dispersive (WDAX) and energy dispersive (EDAX)

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modes. In the former case, a crystal diffracts the incoming X-rays at different angles corresponding to the different wavelengths of the characteristic X-rays, which are detected by PM tube and recorded. Thus, it allows the detection of one element at a time with good wavelength resolution. The energy dispersive mode on the other hand, employs a solid state detector (Li drifted Si) connected to a multichannel analyser in this mode all elements are detected simultaneously and conveniently displayed as peaks in the multichannel analyser. Quantitive analysis of the composition of the material can be obtained from the measured intensity using the standards for the observed elements in the material. The limitations of the EPM technique include difficulty in detecting elements with atomic number less than 8 and matrix effects in multicomponent films.

1.5 X-Ray Photoelectron Spectroscopy (XPS)

In XPS (or ESCA), X-rays of appropriate energy $h\nu$ (usually MgK_{α} or AlK_{α}) strike the surface of a solid sample kept in UHV (10^{-9} to 10^{-11} Torr), and electrons are ejected with different kinetic energies in this process. The kinetic energy E_K of these photoelectrons is given by

$$E_K = h\nu - E_B$$

where E_B is the binding energy of the photoelectrons. The

XPS spectrum consists of a plot of N (E) versus E_B where N(E) is the number of electron counts per second. From these data the elements ($Z \geq 3$) present in the materials are identified. The chemical states of the atoms can then be determined by calculating the chemical shifts. This information is very valuable when dealing with multicomponent materials as often encountered in thin film technology. In conjunction with a sputter etching gun, XPS gives chemical state information and its variation as a function of depth. XPS has been widely used for absorption-desorption studies, catalysis, corrosion etc [11 & 15]. The details of these calculations are given in Chapter - IV.

1.6 Electrical Conductivity and Hall Effect of Semiconductor Thin Films

In an intrinsic semiconductor, in equilibrium, all free charge carriers are due to thermal generation of electron hole pairs. In such a case, the electrical conductivity (σ) is given by

$$\sigma_i (T) = n_i (T) |q| (\mu_e + \mu_h)$$

where $$n_i(T) = 2 \left(\frac{2 \pi K T}{h^2}\right)^{3/2} (m_e m_h)^{3/4} \exp(-E_g/2KT)$$

and, μ_e and μ_h are mobility of electron and holes respectively.

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Introducing impurities, produces significant changes in the carrier concentration of the intrinsic semiconductors which may exhibit either n or p type conductivity, depending upon the nature of the dopants (donor or acceptor like). The mobility is also significantly affected by various scattering machanisms, each of which is operative in a particular temperature range. The scattering can be by the impurity atoms or ions, phonons, point/line defects, grain boundaries, crystal surfaces etc.

In reality all practical semiconducting materials (elemental or compound) used in the fabrication of devices, do contain either intentionally doped or background impurities and defects. Therefore, the nature of the temperature variation of the electrical conductivity of these practical semiconductors can be understood by considering broadly two temperature regions namely, the intrinsic (contributes at high T) and extrinsic (at lower T) regions. Thus, the electrical conductivity at high temperatures (intrinsic region) will be dominated by the exponential term $\exp(-E_g/2KT)$ where E_g is the band gap of the materials. At low temperature (extrinsic region) σ varies as $\exp(-\varepsilon/2KT)$ where ε is the thermal activation energy of the involved impurity/defect states.

In order to estimate the carrier concentration and to identify their nature the Hall coeffcient (R_H) can be

determined which is given by

$$R_H = r\left(-\frac{1}{q\,n}\right) \text{ for } n >> p \quad \text{for electrons}$$

$$R_H = r\left(+\frac{1}{q\,p}\right) \text{ for } p >> n \quad \text{for holes}$$

where $r = \frac{\langle \tau^2 \rangle}{\langle \tau \rangle^2}$, τ is the mean free time. When both types (n and p) of carriers are present R_H can be written as

$$R_H = r\,\frac{1}{q}\,\frac{p - nb^2}{(p + nb)^2}\,, \quad b = \frac{\mu_e}{\mu_h}$$

Then the Hall mobility (μ_H) is given by

$$\mu_H = |R_H|\,\sigma$$

In the case of thin films because of the large value of the surface to volume ratio, surface scattering effects play a very dominant role in the control of the electrical properties. The grains and the grain boundaries in polycrystalline thin films and the disorder produced in amorphous films, further moidfy the electrical properties . Thus essentially these effects namely, the grain boundary and surface scattering, modify the mobility. The mobility can be written as [14].

$$\frac{1}{\mu_f} = \frac{1}{\mu_g} + \frac{1}{\mu_s}$$ where μ_s (due to surface scattering)

$$= \mu_b \left[1 + \left(\frac{2\lambda}{t}\right) \left\{ 1 - \frac{1}{2}(p + q)\right\}\right]^{-1}$$

and μ_g (grain boundary scattering)

$$= \mu_1 \left[1 + \left\{\frac{L_2}{L_1} \exp (q\phi_b/KT)\right\}^{-1} + \frac{L_2}{L_1}\right]$$

where λ is the mean free path of the carriers, p and q are the specular (elastic) scattering coefficients from the upper and lower film surface, μ_1 is the bulk grain mobility, L_1 is the grain size, L_2 is boundary width and ϕ_b is the barrier potential related to the grain boundary domains.

1.7 Reflectance and Transmittance of Thin Films

Considerable theoretical and experimental investigations on the optical behaviour of thin films deal primarily with optical reflection, transmission and absorption properties. The reflection, transmission and interference properties of thin films have made it possible to determine the optical constants conveniently. The absorption studies have led to a variety of interesting thin film optical phenomena which have thrown considerable light on the electronic structure of solids [5]

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For a parallel - sided, isotropic film of refractive index n and thickness (t) between media of indices n_o and n_s, the amplitude of reflectance and transmittance are readily obtained by summing the multiply reflected beams and these are given by the expression [3]

$$R\ (A) = \frac{r_2 + r_1 \exp(-2i\delta_1)}{1 + r_2 r_1 \exp(-2i\delta_1)}$$

$$\text{and } T\ (A) = \frac{t_1 t_2 \exp(-i\delta_1)}{1 + r_2 r_1 \exp(-2i\delta_1)}$$

where $\delta_1 = \frac{2\pi}{\lambda}$ nt $Cos\phi_1$, and r_1, r_2, t_1 and t_2 are the Fresnel coefficients. If the medium of the film is absorptive, then n in Fresnel coefficients is replaced by n-ik, where k is the extinction coefficient of the film. The reflectance (R) and transmittance (T) of the absorbing film (n) on transparent substrate are readily obtained by

$$R = |R(A)|^2 \quad \text{and} \quad T = \frac{n_s}{n_o}|T(A)|^2$$

The expressions of R and T, and their relationship with optical constants (n and k) are given in the Appendix - II which are utilized for the determination of optical constants.

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1.8 Absorption Phenomena in Semiconductor Films

Study of the optical absorption of thin films at different wavelengths gives information about the nature of the band to band (fundamental absorption) transitions as well as the optical absorption related to the impurities and associated defects.

For a semiconductor in which the minimum of the conduction band and the maximum of the valence band occur at the same value of k, absorption begins at the photon energy $h\nu - E_g$. The optical absorption in this case, considering simple parabolic energy bands, is given by [8,10&16]

$$\alpha h\nu = A (h\nu - E_g)^n, \text{ for } h\nu > E_g,$$

where $n = \frac{1}{2}$ for direct allowed and $\frac{3}{2}$ for direct forbidden transitions respectively, and A is a constant.

In the case where the conduction band minimum and the valence band maximum occur at different k values, the band to band opitcal transitions require the participation of phonons. The absorption or emission of phonons in this case involves indirect transitions and α is given by [8,10 & 16].

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$$\alpha h\nu = \frac{B(h\nu + E_p - E_g)^2}{\exp(E_p/KT) - 1} + \frac{B(h\nu - E_p - E_g)^2}{1 - \exp(-E_p/KT)}$$

where the first term represents the contribution of phonon absorption, the second term represents the contribution of phonon emission, E_p is the phonon energy and B is a constant. Indirect transitions give rise to an absorption edge that is less steep than for direct transitions.

On the low energy side of the absorption edge, the excess absorption (usually an exponential tail) occurs between the energy states developed near the band edges due to the presence of impurities or defects. The variation of absorption coefficient with $h\nu$ in this low energy region often follows Urbach's rule of the form [16],

$$\alpha = \alpha_o \exp\left[-\frac{b}{KT}(h\nu_o - h\nu)\right]$$

where α_o and b are constants, and, $h\nu_o$ is the threshold energy.

1.9 Modulation Spectroscopy

Information regarding various critical points or singularities associated with the energy band structure can be obtained from modulation (derivative) spectra. The optical transitions related to these singularities are not generally observed clearly in the conventional optical

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absorption spectra (record of α versus λ). Structure which appears only as a change of slope in this conventional optical spectra, appears as a step in the first derivative and as sharp peaks in the second derivative spectra[6]. Thus, resolved peaks in the second order derivative spectra furnish the details of the V-band splitting due to spin-orbit coupling and crystal field interaction. The band splitting parameters can be estimated from the energy differences between these peaks.

1.10 Photoluminescence

If excess electron-hole pairs are generated in a semiconductor by photon absorption, the electromagnetic radiation resulting from the recombination between them is called photoluminescence (PL). Transitions related to the impurities and native defects even if their concentration is as low as 10^{15} cm^{-3}, can be observed in a PL spectrum when it is recorded at very low temperatures [9]. There are many radiative recombination mechanisms (extrinsic and intrinsic) proposed to explain the PL spectra. Transitions that are generally, considered, are. (i) band to band - the radiative recombination between the electrons in the conduction band and the holes in the valence band which isalso called intrinsic/or recombination luminescence. (ii) band to acceptor/donor energy states - an electron in the conduction band can recombine with either a trapped hole in an acceptor

centre, or with the hole in the valence band involving the donor state as an intermediary step. These transitions will help to estimate the ionization energies of the acceptor/donor and (iii) donor-acceptor (DA) pair the recombination between the electrons and positive holes at the centre formed due to the pairing of oppositely charged defects or ionized donors and ionized acceptros in the semiconductors [2 & 9]. A neutral DA pair can be approximated by a model similar to hydrogen molecule, consisting of two oppositly charged ion cores separated by a distance r. As a consequence of pairing the energy levels of donor and acceptor are perturbed towards their respective band edges [4]. The related emission ($h\nu$) is given by

$$h\nu = E_g - (E_A + E_D) + \frac{e^2}{Kr}$$

where E_g is the band gap, K is the dielectric constant and, E_A and E_D are the ionization energies of acceptor and donor levels respectively. The nature of native defects and their association with the impurities can be understood by considering the DA pair spectra in the PL spectrum particularly recorded at low temperatures.

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