CHAPTER 6

SUMMARY AND SUGGESTIONS FOR FUTURE WORK

6.1 SUMMARY

Cadmium Selenide (CdSe), Lead Selenide (PbSe) and Lead sulphide (PbS) thin films were deposited by chemical and physical methods. In the chemical method, chemical bath and photochemical deposition techniques were used to deposit the n - and p - type thin films. The source materials were synthesized via simple chemical method and used for deposition by thermal evaporation technique. The thermal stability of the synthesized nanoparticles was analysed using TG/DTA curves. The deposited thin films were characterized using XRD to know the structural properties and UV-Visible, photoluminescence to find the optical properties. Raman spectral studies show the fundamental LO and 2LO modes. The Z-scan study with open aperture was carried out at 532 nm using 5 ns laser pulse on the deposited films. High resolution scanning electron microscopy and atomic force microscopy studies were employed to investigate the surface morphology and surface roughness of the thin films. Electrical parameters were measured with the help of Hall measurements in van der Pauw configuration (ECOPIA HMS-3000) at room temperature and the results are compared with the reported values.

6.2 RESULTS OBTAINED IN THE PRESENT INVESTIGATION

(i) CADMIUM SELENIDE (CdSe)

In the chemical method, the importance of chemical bath deposition and the structural properties of CdSe thin films for various concentrations of the EDTA were discussed. EDTA was used as a chelating agent to deposit CdSe films. By the addition of EDTA in solutions (metal ions), growth rate of film can be controlled which assist the uniform deposition of film. The thickness of the film is found to be 0.6 μm using interferometery technique. The as-deposited film exhibits the cubic structure and the 350 °C annealed film changes from cubic to hexagonal. Annealing of the film increases the crystallite size and decrease the dislocation density. The optical study reveals the bandgap for as-deposited CdSe thin film and is found to be 2.1 eV and 1.84 eV for annealed film. The optical transmittance of the film gets reduced after annealing at 350°C. From the photoluminescence analysis, it is inferred that the deposited samples have direct band transition and also red shift is observed after annealing. This shows CdSe is a potential candidate for solar cell fabrication. The homogeneous formations of crystalline grains were observed from SEM as well as AFM images. The as-deposited and annealed CdSe films have negative Hall coefficient which confirms the n - type conductivity. The Hall mobility of the material, resistivity, carrier concentration and conductivity were also estimated. Hence it is concluded that Chemical bath deposition is a suitable method for the preparation of various metal chalcogenide thin films.

The CdSe nanocrystalline particles were synthesized using solvothermal method and used for deposition in physical methods. The 450ºC vacuum annealing improves the properties and gives the stable phase of CdSe. The synthesized and annealed materials were studied by XRD, SEM, EDX and DTA. The XRD patterns of the thermally deposited CdSe samples show the stable hexagonal phase, which will be useful in solar energy conversion due to high photo absorption. From the scanning electron microscope study it is inferred that the thin films have smooth surface and the particles are agglomerated on the surface and the composition is ~1:1 ratio. The thickness of the films is found to be 60-85 nm using thickness profilometer. The optical bandgap values slightly increase with increasing the substrate temperature, owing to variation in the crystallite size of the material. The observed bands in the Raman spectra correspond to the longitudinal optical modes. Photoluminescence studies show the emission in the visible region (534 nm). The negative Hall coefficient obtained confirms the n - type conductivity of the films. The Hall mobility, resistivity, carrier concentration and conductivity were also estimated. Hence it is concluded that the thermal evaporation technique is suitable for the preparation of compound semiconductor and various metal chalcogenide thin films from the synthesized nanoparticles.

(ii) LEAD SELENIDE (PbSe)

Lead selenide thin films have been deposited via simple chemical bath deposition technique at room temperature. The different concentrations of ethylene diamine tetra acetic acid 0.05 and 0.1 M were added into the solution to enhance the crystalline perfection of the deposited films and studied their properties. X-ray diffraction shows the deposited film exhibits cubic phase. When the 0.1 mol of EDTA was added to the reacting solution, the deposited film shows an additional peak in the XRD pattern. The optical study reveals that the absorbance band shifts towards the red region after annealing at 450°C. In HRSEM micrograph, it is found that rod shaped particles after annealing gives rise to a flower like structure. EDX spectrum reveals that the percentage composition of Pb:Se ~1:2, due to large capturing of Pb ions by the chelating agent which creates excess of Se ions in the depositing solution. 3D view of AFM shows that spherical grains get agglomerated and the surface roughness of the deposited film reduces and it entirely spreads over the surface of the substrate.

In the physical method, the substrate temperature induced changes in physical properties of thermal evaporated PbSe thin films were studied. Nanoparticle of lead selenide semiconducting material with stoichiometry was synthesised using simple chemical method which exhibits cubic phase. The chelating agent EDTA enhances the growth of the lead selenide particle and controls the shape of the crystalline powder. XRD measurements reveal the substrate temperature induced grain growth and decrease in the dislocation density. A gradual reduction in optical bandgap is observed with increasing substrate temperature, which is associated with the crystallization of the films. The scanning electron microscopic analysis of the synthesized nano particle shows cubic shape with regular morphology. The AFM images of room temperature and 450°C substrate temperature deposited PbSe indicate the well covered granular surface of the film. From the observation, it is concluded that the 450°C substrate temperature is the best condition to prepare good quality lead selenide thin films. The positive Hall coefficient obtained for the film confirms the p-type conductivity.

(iii) LEAD SULPHIDE (PbS)

The PbS thin films were deposited by simple chemical methods (Chemical bath and photochemical). The effects of deposition parameters on structural, optical, morphological and electrical properties of PbS thin films by chemical bath and photochemical deposition techniques were studied. X-ray diffraction study enumerates that the chemical bath deposited PbS polycrystalline thin films belong to cubic phase and the photochemical deposited PbS thin films crystallize in tetragonal system. The crystallite size and lattice constant of the films were determined from the high intense XRD peak. The nanocrystalline nature of PbS (i.e. size variation) was confirmed from the shift in the optical absorption into the visible region of the spectrum. The HRSEM images indicate the homogeneous formation. The plate like and oval shape morphologies were observed for chemical bath and photochemical deposited samples. The as-deposited PbS films have positive Hall coefficient, which confirms the p-type conductivity. From the Raman spectral studies the fundamental LO modes were observed for the films deposited by both the methods. The Raman shift in the Photochemical deposited thin films may be due to the variation in the crystallite size.

The nanoparticle PbS was synthesized via simple chemical route and deposited by the physical method-thermal evaporation technique at different substrate temperatures. TG/DTA curves show the thermal stability of the synthesized nanoparticle PbS. The deposited films show the cubic phase, while increasing the substrate temperature the crystalline nature of the films gets improved. The % transmittance is found to be reduced on increasing the substrate temperature. Negative nonlinear absorption (i.e. saturated absorption) was found in Z-scan studies, which is strongly influenced by the particle size. The LO and 2LO phonon modes are revealed from the Raman shift. SEM micrograph of the synthesized nanoparticle PbS shows cubical morphology, while increasing the substrate temperature the particles are orderly arranged and forms a needle like network. EDX pattern shows the composition of deposited PbS thin film is ~1:1. AFM analysis indicates that the grain size increases with increase in the substrate temperature. The Hall measurement confirms the presence of p-type charge carrier. The chemical stability of the material makes it a promising candidate for several photonic applications. It can be concluded that the substrate temperature plays a vital role on the properties of the PbS thin films.

6.3 SUGGESTIONS FOR FUTURE WORK

(i) The suitable dopants can be added in the films to improve the electrical properties and also different complexing agents can be added to improve the properties of the films

(ii) The thin films of title compounds at various concentrations can be deposited to make the ternary compounds at different substrates.

(iii) In physical method the effects on the films at various substrate temperatures and on different substrates (Silicon, Corning glass plates and ITO) can be studied and also their effects on the properties of the films.

(iv) The films may be deposited at different atmospheres like inert gases, to investigate film properties at different substrate temperatures.

(v) Deposited films can be irradiated by different ions with different energy and fluence to study the effect of irradiation on the modification of physical properties.

(vi) The film may be deposited at high temperatures to study their phase transitions.