It is believed that we will seriously deplete our natural gas and petroleum supplied within the next two decades. Regardless of these supply estimates, the negative environmental impact due to excessive CO₂ production is of paramount concern.

Renewable technologies cover an extensive range of disciplines, including bio-fuels, ocean geothermal, hydrogen, wind energy and photovoltaics. Photovoltaics is the conversion of solar radiation into
electrical power through photon absorption by semiconductor materials. In addition, photoelectrochemical conversion of solar radiation has also recently received considerable attention as an attractive alternative to solid state photovoltaic conversion.

The elemental and compound material systems most widely used in optoelectronic, photovoltaic and photoelectrochemical applications can be produced in a variety of crystalline and non-crystalline forms.

Several compounds in the crystalline and polycrystalline family of materials have recently gained much attention and interest due to their enhanced performance, potential for low cost manufacturability and exhibited stability and reliability. Included among the successful ones are CdTe, CuInSe₂, MoSe₂ and WSe₂. These materials have exhibited considerable conversion efficiencies in small area heterojunction thin film devices and photoelectrochemical solar cells.

CuInSe₂ (CIS) materials belong to larger family of I-III-VI₂ ternary semiconductor compounds. The opto-electronic structural and morphological properties of CuInSe₂ and the corresponding device performance, are
highly dependent on film composition, defect chemistry and growth parameters, such as substrate temperature. It was therefore, thought worthwhile to study the fabrication and characterization of the ternary chalcopyrite CuInSe₂ thin film semiconductor material systems.

Among the layered compounds of transition metal dichalcogenides, all research activities of solid state photovoltaic cells have been limited to WSe₂. WS₂ which is used as a lubricating agent at high temperature and in UHV studies is also suitable in photoconversion studies. Both these compounds have been subjected to intensive studies as photoelectrochemical cells.

It is seen that the band gap of WSe₂ is 1.35 eV whereas that of WS₂ is 2.1 eV. By suitable adjustment of the sulphur and selenium content in WSₓSe₂₋ₓ the band gap can be altered and a material suitable for solar energy conversion can be prepared. By taking equal portions of sulphur and selenium in WSₓSe₂₋ₓ, single crystals of tungsten sulpho-selenide (WSSe) can be grown and their suitability in photoelectrochemical studies can be studied.
Following the above introduction, the thesis is divided into four sections. Section I, deals with the growth and characterization of CuInSe$_2$ both in the form of thin film and single crystals. Section II, describes the growth and characterization of tungsten sulphoselenide crystals, while section III is devoted to the photoelectrochemical studies on CuInSe$_2$ and WSSe. Section IV summarises the general conclusions drawn from the entire work.

Chapter 1.1 emphasises the importance of CuInSe$_2$. A review of the existing information and the scope for the present work has been described.

Chapter 1.2 deals with the growth and structural analysis of CuInSe$_2$ thin film. It reports the detailed study on the role of deposition parameters on the crystallinity and homogeneity of the films.

Chapter 2.1 gives an account of the available work on WSSe single crystals and points out the importance of doing the work on this compound.

A description of vapour transport technique is given in chapter 2.2. The salient features of chemical
vapour transport method are pointed out in the earlier part of this chapter, whereas the details of the direct vapour transport method is given in the later part. Details of experimental set up, temperature controlling system, construction of the furnace etc. have also been thoroughly described.

Chapter 2.3 deals with the growth of WSe$_2$ single crystals using chemical vapour transport method with iodine as a transporting agent as well as by direct vapour transport. Lattice parameter, cell volume and X-ray densities have been determined. The composition of the grown crystals has been examined by EDAX.

Chapter 2.4 describes the stoichiometric dependence of electrical characteristics in WSe$_2$ single crystals grown by direct and chemical vapour transport techniques. The implications of the observations have been discussed.

A necessary introduction to photoelectrochemical solar cell has been presented in chapter 3.1. Different types of PEC solar cells have been described and discussed by giving their classification. The advantages and disadvantages of PEC solar cells over
the solid state photovoltaic cells have also been discussed. The basic electrode processes and mechanism of charge transfer process into the electrolyte have also been presented.

As molybdenum disulphide (MoS₂) single crystals are available abundantly in nature, it was decided to investigate them thoroughly as a test case study for photoelectrochemical properties. The fabrication of PEC solar cells with these single crystals is given in chapter 3.2. A complete characterization of the cell has been made.

Chapter 3.3 deals with the study of PEC solar cells with WSSe single crystals grown by chemical vapour transport as well as direct vapour transport method. The effect of light intensity and the nature of the surfaces used in the fabrication of PEC solar cells have been thoroughly investigated. All these results are elegantly presented in this chapter.

Chapter 3.4 describes the feasibility of copper indium diselenide single crystals in the fabrication of PEC solar cells. Semiconductor electrolyte interface characterization in terms of location of
valence band and conduction band edges and fermi levels has been carried out. All this work is thoroughly presented in this chapter.

The last chapter 4.1 describes the conclusions drawn from the entire work and the scope for the future work.