PREPARATION, CHARACTERIZATION AND PHOTOELECTROCHEMICAL BEHAVIOUR OF $CuInS_2$ AND $CuInSe_2$ THIN FILMS AND PHOTOELECTROCHEMICAL INVESTIGATIONS ON POLYPYRROLE COATED CdTe

by

AKHLESH GUPTA

Department of Chemistry

SUBMITTED

IN FULFILMENT OF THE REQUIREMENTS

OF THE DEGREE OF

DOCTOR OF PHILOSOPHY

to the

INDIAN INSTITUTE OF TECHNOLOGY, DELHI

JUNE, 1990

CERTIFICATE

This is to certify that the thesis entitled, "**Preparation, Characterization and Photoelectrochemical Behaviour of $CuInS_2$ and $CuInSe_2$ Thin Films; and Photoelectrochemical Investigations on Polypyrrole Coated CdTe**" being submitted by **Akhlesh Gupta** to the Indian Institute of Technology, Delhi, for the award of the degree of "**Doctor of Philosophy**" in Chemistry, is a record of bonafide research work carried out by him. Mr. Akhlesh Gupta has worked under our guidance and supervision and has fulfilled the requirements for the submission of the thesis, which to our knowledge, has reached the requisite standard.

The results contained in this thesis have not been submitted, in part or in full to any University or Institute for the award of any degree or diploma.

(**Prof. A.S.N. Murthy**)
Head, Department of Chemistry
Indian Institute of Technology
Hauz Khas, New Delhi - 110 016
INDIA

(**Prof. G.N. Rao**)
Department of Chemistry
Indian Institute of Technolog
Hauz Khas, New Delhi - 110 01
INDIA

ACKNOWLEDGEMENTS

I take this opportunity to express my sincere gratitude to Prof. A.S.N. Murthy and Prof. G.N. Rao for their inspiring guidance, critical evaluation and continuous encouragement, which have greatly contributed towards the completion of this work.

I am grateful to Department of Non-Conventional Energy Sources (New Delhi) and IIT Delhi for financial assistance and Head of the Chemistry Department, for providing the necessary facilities to carry out the research work. Thanks are also due to Head, Material Science Research Centre, IIT Madras for providing some facilities.

The cooperation and help extended by Drs. M.P. Singh, S.K. Jain, N. Homendra, S.K. Sharma, R.S. Rastogi and R. Thangraj is gratefully acknowledged. I am thankful to my friends and colleagues Arun, Gurpreet, Sunil, Surendra, Indra Kumar, Jha, Rao, Vinod, Anita, Vibha, Tanuja, Kiran, Nandini, Balvinder, Anil, Himanshu and Girish for providing a pleasant company and extending their full cooperation whenever needed.

I wish to thank Drs. G.K. Padam, S.P.Singh and A.N.Tiwari for intensive discussions related to various aspects of my work. I am indebted to Dr. Bhatnagar for helping me in XPS/AES measurements, Mr. Sharma for SEM measurements, Ms. Rama Saxena for XRD measurements and Mr.

G.L. Malhotra for SEM/TED measurements. I am thankful to technical and supporting staff of Chemistry Department for providing necessary help.

I am grateful to Mr. Jagdish and Mr. Gaur for their meticulous typing, Mr. Saxena for drafting the figures.

Care has been taken to give proper credit for the work of other authors in the literature. The author would like to apologize for any unintentional omission which might have occurred by oversight.

Last but not the least, I would like to record my deep sense of gratitute to my parents, uncle, aunt, brothers and sisters for their moral support in achieving the present objective.

JUNE 8, 1990
IIT, DELHI

(AKHLESH GUPTA)

ABSTRACT

The thesis reports investigations on the preparation of thin films of CuIn alloy, $CuInS_2$ and $CuInSe_2$. All the materials have been characterized by X-ray diffraction, Auger electron spectroscopy, scanning and transmission electron microscopy and X-ray photoelectron spectroscopy. $CuInS_2$ and $CuInSe_2$ have also been examined for their photoelectrochemical behaviour. In addition, a novel method has been employed to prevent photocorrosion of CdTe in aqueous electrolytes.

Thin films of CuIn alloy have been prepared by a simple electroless technique which involves short circuiting a Ti substrate with an easily oxidizable metal like Al in an acidic bath containing $CuCl_2$, $InCl_3$, triethanolamine and ammonia. Conditions have been optimized after a detailed examination of the effect of different variables to obtain a uniform film.

$CuInS_2$ films have been prepared by annealing the CuIn alloy films (as above) in H_2S atmosphere at high temperatures. The films have been well characterized and the conditions established for obtaining a uniform photoactive film. The PEC characteristics of such films have been examined in detail in polysulphide electrolyte and various device parameters evaluated.

$CuInSe_2$ films have been prepared by electroless technique from an acidic bath containing $CuCl_2$, $InCl_3$, SeO_2, triethanolamine and ammonia. The structural, compositional and morphological characteristics of the films have been determined. Results on the PEC characterization of the films are critically examined.

Photoassisted electropolymerization of pyrrole on CdTe single crystal has been found to be effective in preventing photocorrosion of CdTe in PEC cells containing acidic polyiodide solutions. Stability and power characteristics have been investigated.

CONTENTS