CYCLIC VOLTAMMETRY ANALYSIS OF HgSSe THIN FILMS

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This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.)

by

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DECLARATION

I hereby, declared this report entitled 'Cyclic Voltammetry Analysis of HgSSe Thin Films' is the results of my own research except as cited in references.

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfilment of the requirements for the Degree of Manufacturing Engineering (Engineering Materials) (Hons.). The member of the supervisory committee is as follow:

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(Official Stamp of Principal Supervisor)

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(Official Stamp of CoSupervisor)

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SESI PENGAJIAN: 2014/15 Semester 2

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ABSTRACT

Thin film technology is one of the most developing technologies nowadays that involves in the development of solar cell. Ternary Transition Metal Chalcogenides (TMCs) are semiconductors which can be used as an efficient photovoltaic material. These materials have covered many areas of technological interest due to its outstanding properties. This research intent to predict safe, non-toxic, cost-efficient and relative simple method for synthesize of ternary transition metal chalcogenide thin films. Among the Ternary TMCs, Mercury Sulphoselenide, HgSSe is one of the materials that can be applied in thin film technology. The objectives of this project are to determine the deposition potential using cyclic voltammetry analysis, synthesize the HgSSe thin film via electrodeposition technique and characterize the structural and morphological properties. Mercury sulphoselenide, HgSSe thin films were successfully electrodeposited on stainless steel substrates with deposition time of 30 minutes. Cyclic voltammetry analysis confirmed the reduction range of HgSSe occurred between -1.3V to -1.8V. Films were observed by optical microscope found to be well adherent to the substrates and grew up to thickness of 1.22µm. Structural analysis via X-ray Diffraction (XRD) analysis reveals that the films are polycrystalline with increasing intensity of XRD peaks in thicker films. (2 2 0) plane for $HgSe_2$, (2 0 0) plane for HgS_2, and (0 1 3) plane for HgSSe were observed to be the most preferred orientation as they were the highest peak in the spectrum. The surface morphology of the films determined by scanning electron microscope (SEM) showed that the growth of the films were uniform and well adhered for thinner films. At the potential of -1.5V, HgSSe films showed the most optimum potential to be used for electrodeposition.

ABSTRAK

Teknologi filem nipis adalah salah satu teknologi yang mambangun pada hari ini yang melibatkan pembangunan sel fotovolta. Logam peralihan chalcogenides merupakan semikonduktor yang boleh digunakan sebagai bahan fotovolta dengan cekap disebabkan ia mempunyai sifat-sifat yang unik. Penyelidikan ini berniat untuk menghasilkan filem nipis yang selamat, tidak bertosik, mudah dan kos rendah. Antara bahan-bahan logam peralihan chalcogenides, Mercury Sulphoselenide, HgSSe telah dikaji dalam penyelidikan ini. Tujuan penyelidikan ini adalah mengaji potensi pemendapan dengan menggunakan kitaran voltammetry analysis, mensintesis filem nipis HgSSe melalui teknik pengenapan dan mencirikan sifat-sifat struktur dan morfologi. Mercury Sulphoselenide, HgSSe filem nipis telah berjaya elektrodeposisi pada substrat keluli tahan karat dengan masa 30 minit. Analisis cyclic voltammetry mengesahkan penurunan berlaku antara -1.3V dan -1.8V. Filem diperhatikan menjadi baik melekat kepada substrat dengan ketebalan 1.22 μm. Analisis struktur melalui XRD mendedahkan bahawa filem-filem yang polihabluran dengan peningkatan keamatan puncak XRD dalam filem tebal. (2 2 0) untuk $HgSe_2$, (2 0 0) untuk HgS_2 dan (0 1 3) untuk HgSSe diperhatikan untuk menjadi orientasi yang paling digemari kerana ia adalah puncak tertinggi dalam spektrum. Morfologi permukaan filem dianalisis melalui imbasan mikroskop elektron (SEM) telah menunjukkan pertumbuhan filem adalah seragam dalam bentuk filem nipis. Filem HgSSe menunjukkan pengenapan yang terbaik pada potensi -1.5V.

DEDICATION

To my beloved father, Ho Wooi Min,
mother, Oh Guat Liang,
and my brother, Ho Zhao Yong
for giving me moral support, encouragement and understandings.
Your love is my driving force.

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LIST OF ABBREVIATIONS

a-Si	-	Amorphous Silicon
Cd	-	Cadmium
CdTe	-	Cadmium Telluride
CIGS	-	Copper Indium Gallium Selenide
CV	-	Cyclic Voltammetry
CVD	-	Chemical Vapor Deposition
ED	-	Electrodeposition
E_r	-	Reflected Energy
E_s	-	Substrate Energy
E_v	-	Vapor Energy
HCl	-	Hydrochloric Acid
Hg	-	Mercury
HgSe	-	Mercury Selenide
HgSSe	-	Mercury Sulphoselenide
ITO	-	Indium Tin Oxide
JCPDS	-	Joint Committee on Powder Diffraction Standards
M	-	Metal
Mo	-	Molybdenum
MoSSe	-	Molybdenum Sulphoselenide
$Na_2S_2O_3$	-	Sodium thiosulphate pentahydrate
NH_3	-	Ammonia
PEC	-	Photoelectrochemical
PV	-	Photovoltaic
PVD	-	Physical Vapor Deposition
S	-	Sulphur
SCE	-	Saturated Calomel electrode
Se	-	Selenium
SEM	-	Scanning Electron Microscope

SeO_2	-	Selenium Dioxide
SiO_2	-	Silicon dioxide
Te	-	Tellurium
TMCs	-	Transition Metal Chalcogenides
X	-	Chalcogenide
XRD	-	X-ray Diffraction
X-SEM	-	Cross-sectional Scanning Electron Microscope
Zn	-	Zinc

LIST OF SYMBOLS

-	-	Negative
%	-	Percentage
:	-	Ratio
±	-	Plus-Minus
×	-	Multiplication
↔	-	Material Equivalence
°	-	Degree
μm	-	Micrometre
A	-	Ampere
Å	-	Angstrom
cm	-	Centimetre
cm^2	-	Centimetre square
eV	-	Electron Voltage
g	-	Gram
g/cm^3	-	Gram per centimetre cube
g/mol	-	Gram per mole
M	-	Mole
mm	-	Millimetre
^oC	-	Degree Celsius
USD	-	United State Dollar
V	-	Voltage
θ	-	Theta
λ	-	Lambda

CHAPTER 1
INTRODUCTION

1.1 Research Background

Recently, the application of thin film on solar cell (photo-electrochemical cell) has become a great intention and increasingly popular in the solar cell development and research. Solar energy that produced by solar cells are well-known as an eco-friendly source of energy which brings no harms to the environment (Mane *et al.*, 2014).

There are number of researchers studied on the new material thin film to be used in solar cell applications in the last ten years. Bhuse (2007) has studied the photoelectrochemical properties of a ternary material. The material would have a better adsorption and sensitivity if the material varies between specified limits within the photon energies. Therefore, a new material of ternary transition metal with a lower cost and comparable conversion efficiency is introduced in this research.

There are mainly two deposition methods which are Chemical Vapor Deposition (CVD) and Physical Vapor Deposition (PVD). The method chosen to be used in this research is electro-deposition (ED) method which is categorized as one of the CVD methods. Electro-deposition method has increasingly applied to semiconductor synthesis in recent years. ED method is consumed due to its numerous advantages. ED method provides simple and easy deposition, low temperature processing, arbitrary substrate shapes, controlled film thickness, morphology, composition and the width band gap, and potential low cost technique that gives good quality of film deposited (Anand, 2001; Ebrahim *et al*., 2010; Naghavi, 2011).

Cyclic voltammetric analysis is used to obtain the most suitable potential to be used by referring to the cyclic voltammograms. Cyclic Voltammetry (CV) will be done using electrochemical analyzer to fix the deposition potential. Voltammograms will be collected for different aqueous alkaline solutions in various sweep rates. Then, a conventional three electrode system is employed.

CV analysis was implemented to find the suitable region of growth potential to be used for the PEC cells. Kassim *et al.* (2009) has successfully synthesized the thin films by using cyclic voltammetry technique. By using this technique, the suitable potential application in PEC solar cell can be observed and serves a better absorption properties and solar cell conversion.

Today, there is a vast array of techniques used to analyze the microstructure and the properties of the material. Material characterization is crucial in analyzing material properties. The properties of material that can be examined include mechanical properties, physical properties, thermal properties, optical properties, electrical properties and magnetic properties.

The film thickness can be obtained by using weight gain method and Cross-sectional Scanning Electron Microscope (X-SEM) analysis. X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) were carried out to characterize the thin films. The structural properties of the thin films can be revealed by using X-ray diffraction technique, whereas the surface morphology properties of the thin films can be determined using Scanning Electron Microscope.

1.2 Problem Statement

Solar energy which is a green energy sources has been widely used as preventing pollution to the environment has grown more important with each passing day. Rajalakshmi *et al.* (2013) studied that transition metal chalcogenides are the salient material in application for solar energy conversion in recent years.

Solar panels are indeed very expensive in the market. Most of the solar panels nowadays are made of silicon dioxide (SiO_2) where Silicon material is considered as an expensive material. Pure silicon cost around USD5.4 per 100g. This has become the major concern in the solar cell development and research. Therefore, a ternary transition metal chalcogenide thin film with a lower price was suggested to be used, which is mercury sulphoselenide (HgSSe).

There is an essential task to identify the conversion efficiency of an appropriate material. An attempt has to be made to confirm that the thin film has obtained the optimum deposition potential in order to produce a high quality thin film.

A cyclic voltammetry study is undergoing to analyze the corresponding chemical reactions. This is to ultimate the efficiency of energy conversion or photovoltaic activities. This action is essential to ensure the performance of the materials in the devices.

1.3 Objectives

There are several objectives that need to be achieved in this project.
These objectives are:

1. To determine the suitable potential to be used using cyclic voltammetry analysis.
2. To deposit stoichiometric HgSSe thin film by electrodeposition technique.
3. To characterize the crystallography structure and morphological properties of HgSSe thin films by using X-ray diffraction (XRD) and scanning electron microscopy (SEM).

1.4 Scope of Project

This study is focusing on the properties of mercury sulphoselenide (HgSSe) thin flims for the application in photoelectrochemical (PEC) solar cells. The project includes the experimental procedures, characterization techniques which involve the electrodeposition of thin films, structural analysis and morphological analysis.

The research was first started with a cyclic voltammetry study to obtain the range of deposition potentials where the films grow. Then, the structural properties of the films were determined by X-ray Diffraction (XRD) technique; whereas, the morphological properties were examined by using Scanning Electron Microscope (SEM).

In short, the main intention of the research is to find out to most suitable potential to be used to deposit the mercury sulphoselenide on to the thin films.

1.5 Outline of Project

This report was divided into five chapters which consist of introduction, literature review, methodology, results and discussion, and conclusion and recommendation.

Chapter one, is the introductory of the project which comprising the research background, problem statement, objectives, and scope of study, as well as the outline of the project.

Chapter two is the literature review of the project that discusses published information that related to the title of this research. It is important to provide a handy guide to the topic of research and gives an overview on the relevant title. It also provides a solid background for the research's investigation to know the previous work that have done by the other researchers and make improvement towards the limitations.

Chapter three discussed the methodology of the research. This chapter made up of the methods to carry out the project and comprises the theoretical analysis of the body of the methods by using material characterization methods.

Chapter four discussed on the results obtained. This chapter is the summary of the entire project where the results were analyzed with some discussions. This chapter has covered the whole of the work and all of its phases. The discussion presents a deeper enriched by the knowledge gained during the process of compiling this report.

Chapter five is the conclusion of the project which includes the conclusion and recommendation for future studies.

CHAPTER 2
LITERATURE REVIEW

2.1 Thin Film

Thin film is defined as a thin layer of deposited material with tenth of nanometer to several micrometers thickness on a substrate. It requires substrates to be adhered to where it cannot stand alone by itself.

Thin films have several applications in various kinds of field especially in material science and engineering. Thin film studies have advanced in a lot of new areas of research due to its unique and astounding characteristics which have the ability to tailor the desired properties by variation of the compositional parameter (Bhuse, 2007).

Owing to the environmental energy conservation and protection, a clean, cost effective and renewable energy source has been an essential intention. Solar energy is a energy which is safe, clean, renewable and environmental friendly source of energy amid the energy resources. Thin film solar cells are finding increasing interest in solar cell development and research (Mane *et al.,* 2014).

The effective conversion of solar energy is a viable approach to overcome the problem for energy crisis as the valuable resources of the earth such as natural gas and fossil oil are limited and might be used up one day in the future (Rajalakshmi *et al.*, 2013). Moreover, these earth resources may produce pollution to the environment which will bring harm to human health. Therefore, a new material is needed to be