UNIVERSITI TEKNIKAL MALAYSIA MELAKA

STRUCTURAL CHARACTERIZATION OF $HgSe_2$ THIN FILMS BY ELECTROCHEMICAL ROUTE

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

by

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FACULTY OF MANUFACTURING ENGINEERING

2015

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

TAJUK: **Structural Characterization of Mercury Diselenide ($HgSe_2$) Thin Films by Electrochemical Route**

SESI PENGAJIAN: 2015/16 Semester 2

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I hereby, declared this report entitled "Structural Characterization of Mercury Diselenide ($HgSe_2$) Thin Films by Electrochemical Route" is the results of my own research except as cited in references.

Signature	:	……………………………………..
Author's Name	:	AINI RAHAYU BINTI MD KHIROM
Date	:	5th JUNE 2015

APPROVAL

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

………………..………………………

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ABSTRAK

Filem nipis Merkuri Diselinida ($HgSe_2$) berjaya dielektromendapkan ke atas permukaan substrat kaca bersalut indium-timah-oxida (ITO) dan substrat besi tahan karat. Tempoh masa pemendapan telah ditetapkan 30 minit. Sebab utama adalah kerana ion memerlukan masa untuk bertindak balas pada dengan kimia. Tiada pemendapan berlaku atas permukaan substrat sehingga ke 20 minit. Filem nipis telah disediakan dengan menggunaka tindak balas kimia melibatkan merkuri asid (H_2HgO_3) dan selenia oksida (SeO_2) menggunakan formula $H_2HgO_3 + 2SeO_2 \rightarrow HgSe_2 + H_2O + 3O_2$. Voltan pemendapan menggunakan -1.2 V, -1.4 V, -1.5 V dan -1.6 V dengan berpandukan pintasan oxidasi dan reduksi kitaran voltametri. Filem-filem nipis tersebut telah dicirikan untuk ciri-ciri struktur dan morfologi permukaan. Ciri-ciri struktur menunjukkan yang $HgSe_2$ setuju mempunyai struktur kubik padu serta mempunyai persamaan dengan JCPDS, a= b= c = 0.60 nm. Analisis struktur menggunakan pembaisan X-ray mendedahkan polihabluran filem dengan penaikan pengamatan puncak XRD dengan penaikan voltan pemendapan. Puncak tertinggi dalam struktur analisis menunjukkan orientasi pilihan bagi filem-filem tersebut ialah orientasi (2 2 0) dan (4 2 0) bagi filem $HgSe_2$. Morfologi permukaan filem-filem yang ditentukan oleh imbasan mikroskop electron (SEM) menunjukkan pertumbuhan filem-filem yang seragam dan merangkumi seluruh permukaan substrat untuk filem-filem yang nipis. Walau bagaimanapun, pada voltan pemendapan yang lebih tinggi, struktur filem mula berpecah membentuk kepingan-kepingan akibat daripada kesemua ion telah dimendapkan pada substrat.

ABSTRACT

Mercury diselenide ($HgSe_2$) thin films were successfully electrodeposited on indium-tin-oxide; (ITO)-coated glass substrates and stainless steel substrates in which the deposition time for the thin films were fixed at 30 minutes. The reason is that the ion needs time to react within the chemical. There is no deposition on the substrate up to 20 minutes. The thin film is prepared by using chemical reaction of mercuric acid (H_2HgO_3) and selenium oxide (SeO_2) given by formula $H_2HgO_3 + 2SeO_2 \rightarrow {}_{HgSe2} + H_2O + 3O_2$. Electrodeposition voltages is set to -1.2 V, -1.4 V, -1.5 V and -1.6 V referring to the interception of forward and reverse scan of cyclic voltammetry. Thin films were characterized for their structural and surface morphological. The structural studies indicate that the $HgSe_2$ agreed to be a simple cubic structure with a lattice parameters match the JCPDS, a= b= c= 0.60nm. Structural analysis via X-ray diffraction (XRD) analysis reveals that the films are polycrystalline with increasing intensity of XRD peaks in increasing electrodeposition voltages. Preferred orientation of (2 2 0) plane and (4 2 0) in $HgSe_2$ was observed as the highest peak in the spectrum. The surface morphology of the films determined by scanning electron microscope (SEM) showed the growth of the films to be uniform and well covered for thinner films. However, at higher electrodeposition voltages, the structure of the films starts to break into grains due to the all of the ion has been deposited on the substrate.

DEDICATION

To my father;Md Khirom bin Ab Rahman, my mother;Sa'ah binti Januri, siblings and friends. Your love and support gives me the strength in completed my research.

To my supervisor, Prof. Madya Dr. T. Joseph Sahaya Anand and all the staffs especially in Faculty of Manufacturing Engineering, UTeM and UTeM.

Thank you for all your supports, guidance, helps and co-operation, directly or indirectly.

ACKNOWLEDGEMENTS

A big thanks and appreciation I would like to give to my supervisor, Prof. Madya Dr. T. Joseph Sahaya Anand for his guidance, concerns, patience and moral support in leading me the way to perfecting the research. I could not imagine the hardship that I am going through without his guidance. Also, I would like to thanking the to the technicians and staffs in Faculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka (UTeM) who had given their full commitment to lend their hand to help me doing the research. Lastly, thank you very much towards all my friends for the helps and supports given to completed my research.

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LIST OF ABBREVIATIONS, SYMBOLS AND SPECIALIZED NOMENCLATURES

TMC	-	Transition Metal Chalcogenides
PEC	-	Photoelectrochemical cell
α	-	Alpha
%	-	Percentage
λ	-	Wavelength
ºC	-	Degree Celsius
µm	-	Micrometer
C_B	-	Conduction Band
cm	-	Centimeter
C_{SC}	-	Space Charge Capacitance
CV	-	Cyclic Voltammetry
CVD	-	Chemical Vapor Deposition
d	-	Interplanar Distance
EDS	-	Energy Dispersive X-ray Spectroscopy
E_g	-	Band Gap Energy
eV	-	Electron Volt
g	-	Gram
GaAs	-	Gallium Arsenide
h	-	Planck's Constant (6.636 x 10^{-34} J)
$HgSe_2$	-	Mercury diselenide
H_2HgO_3	-	Mercuric Acid
SeO_2	-	Selenium dioxide
ITO	-	Indium Tin Oxide
m	-	Meter
M	-	Molarity
NH_3	-	Ammonia
nm	-	Nanometer
O	-	Oxygen

PEC	-	Photoelectrochemical
PVD	-	Physical Vapor Deposition
SEM	-	Scanning Electron Microscope
Si	-	Silicon
t	-	Time
V	-	Potential voltage / Volt
V_B	-	Valence Band
XRD	-	X-Ray Diffractometer
ZnS	-	Zinc blende
Cu_2O	-	Cuprite
PbS	-	Galena
Ge	-	Germanium
P	-	Phosphorus
S	-	Sulphur
Se	-	Selenium
Te	-	Tellurium
PbI2	-	Lead iodide
C60	-	fullerene
Eu	-	Europium
Mn	-	Manganese
TEM	-	Transmission Electron Microscope

CHAPTER 1
INTRODUCTION

1.1 Research background

In recent years there is a lot of attention due to its application in the fabrication on the thin films for the transition metal chalcogenides. (Chopra et al., 2004) Researchers are realising on the application of this transition metal chalcogenides into the efficiency of its conversion of sunlight to electricity. The semiconductor chalcogenides in thin film technology has been developing in the photoelectrochemical (PEC) cells applications.

There are two considerations in producing the semiconducting chalcogenides which is minimization of the production cost and the band gap energy able to produce to match the solar spectrum. The mixtures of the transition metal chalcogenides not only provide better efficiencies but it will minimize the cost to manufacture (material, energy and handling). It is important in producing a low cost of PEC cells and to increase the efficiency of the photovoltaic.

Nowadays, there are several techniques in producing the thin film such as vacuum evaporation, chemical vapour deposition (CVD), sputtering, electrodeposition and solution growth technique. Electrodeposition is most preferred techniques in the preparation of the thin film since it is are the most economical ways for the forming of thin films than the other techniques. The quality will depend on the composition of the electrolytes through the electrodeposition techniques. It is going to be studied on the surface morphology, optical, compositional, temperature of the deposition, and deposition time and semiconductor properties throughout these techniques.

1.2 Problem statement

The solar power is one of the renewable energy which converts the sunlight energy to electrical energy used to generate power in our daily life. By this, it is confirms that the solar energy is infinite. The existing material that was used in solar panel such as crystalline silicon (Si), multi crystalline Si, amorphous Si, and etc. has a lower conversion of efficiencies for generating power. (Choubey et al., 2012). A new alternative material for the replacing the existing Si material in the solar panel is needed to overcome the problem. Transition metal chalcogenides (TMC) has been very popular for the application for the solar panel. (Anand et al, 2011). Energy is needed for the daily life. A lot of sources energy has been run out. Furthermore, it is expected that earth will run out of the oil reserves in the future. Even though that the solar energy is free but the application of solar energy required large area of land to capture the sun that will affectedly increase the overall cost of amount and size of solar panel. Besides, it is considered that for each single photovoltaic cell is one of the costly materials and also has low conversion of efficiency. (Heal, 2009). Solar energy does not cause pollution but in certain cases, solar collectors and other associated equipment or machines that are manufactured in factories will turnout causing some pollution. Besides that, the solar energy will only efficiently applicable to a certain areas since its only can be applied with the presence of sunlight. The used of the electrochemical technique which is used to deposit the thin film on the substrate will gives the overall solar cell fabrication cost. It is also can be produced in large area scale and the process itself can be control which will reduce the cost of the material. (Becker et al., 2013).

1.3 Objectives

a) To synthesized stoichiometric of $HgSe_2$ thin film prepared by using the electrodeposition technique.

b) To analyse the structural characterisation of $HgSe_2$ thin films by X-ray diffraction (XRD) and scanning electron microscope (SEM).

1.4 Scope

This project will focus only on the mercury diselenide, $HgSe_2$ thin film itself. The thin film will be experimentally to characterise the microstructural, optical and electrical properties of the material used. The ranging of the thin films is about in nanometre to several micrometres scale that will be carried out using the scanning electron microscope and X-ray diffractometer that is suitable for that kind of scale. The final methods are to observe energy conversion efficiency based on the performance of the mercury diselenide thin film.

1.5 Outline Project

This project is divided into five chapters that will include:

a) Introduction
b) Literature review
c) Methodology

This report will only cover the introduction, literature review and methodology part. The first chapter will briefly explain the background of this project and objective that are going to be achieved besides scope and outline of the project.

In Chapter 2 which is the literature review that are going to elaborate the understanding from previous reading related to journal, books and other medias related to this project from previous research. In addition, this topic will provide background to the new research that link previous research.

For Chapter 3, methodology, will discuss on the experimental procedures which is carried out in producing the thin film and the outcome of the project. The most appropriate method is choosing and will let the sample for further analysed by a suitable material characterisation.

CHAPTER 2

LITERATURE REVIEW

2.1 Semiconductor

Semiconductor is usually defined as a material with electrical resistivity lying in the range of 10^{-2}-$10^{9}\Omega$cm. It can be defined as a material whose energy gap for electronic excitations lies between zero and about 4 electron volts (eV). As shown in Figure 2.1, the conduction band of the metal overlap with the valence band whiles the insulator conduction band and valence band are separated by the energy band gap, E_g. The semiconductor shows a moving of electron from the valence band that excite to the conduction band between the band gap energy.

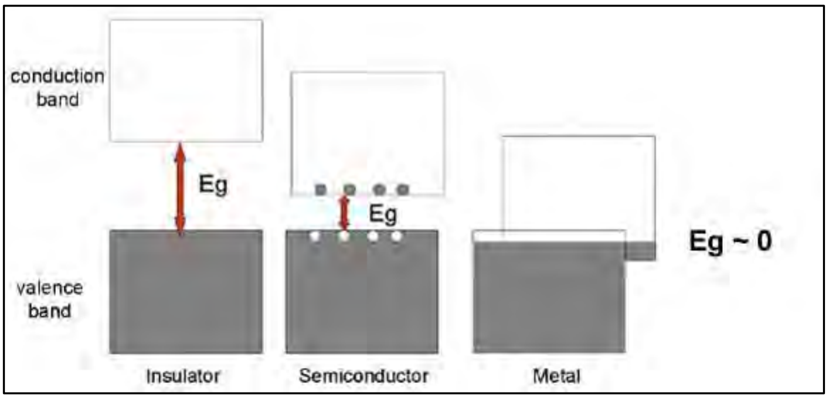

Figure 2.1 Schematic band diagrams for an insulator, semiconductor and metal (Adapted from http://education.mrsec.wisc.edu/background/LED/images/band_theory.JPG)

Semiconductor is widely used in the electronic circuits. The name itself shows that the semiconductor is a material that can conduct a part of current. The conductivity of the semiconductor is between the insulator and a conductor since it only can conduct a current partially.

Moreover, the common material used in the semiconductor is made up of a crystal which undoubtedly is silicon. The movement of the electrons that organized are closely related with the semiconductors behaviour. The electrons in an atom will be organized layer by layer and this layer is called a shells. The outermost shell is called the valence shell. The outermost shell of the electron will share electrons from the neighbouring atom to become a semiconductor. The sharing of the electrons in atom is called a covalent bond. However, there are other semiconductors beside silicon. In fact, there are many minerals found in nature such as zinc-blende (ZnS) cuprite (Cu_2O) and galena (PbS). Semiconductors occur in much different chemical composition with a large variety of crystal structures. (Yu & Cardona, 2001)

2.1.1 Elemental semiconductors

The well-known semiconductors are of course the element silicon (Si). Other than silicon, the same element with the same crystal structure of a large class semiconductors are the germanium (Ge). As shown in Figure 2.2, the elemental silicon which is in a Group IV tended to chemically bonded to their element itself tp achieve a stability as a semiconductor. In this structure, each of the atom are surrounded by the four nearest neighbouring atom which then forming a tetrahedron. Some elements in the group V and VI in the periodic tables such as phosphorus (P), sulphur (S), selenium (Se) and tellurium (Te) also are the semiconductors. This element can exist in different crystal structure depend in where they are coordinated. (Yu & Cardona, 2001)

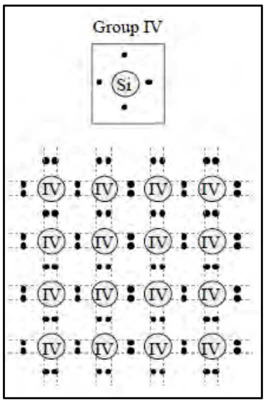

Figure 2.2 Elemental Silicon

2.1.1 Binary compounds

Binary compounds consist of more than one element in a formula. The atom will tend to chemically bonded to each other by sharing their electron to each other to achieve an octet state. As shown in Figure 2.3, the element of the binary compound usually is made up from group III-V of periodic table which also similar properties as group IV counterparts. From group IV elements to the III-V compound, the bonding partly become ionic due to a transfer electronic charge from the group III atom and group V atom. The ionicity in an molecule causes significant changes in semiconductor properties.

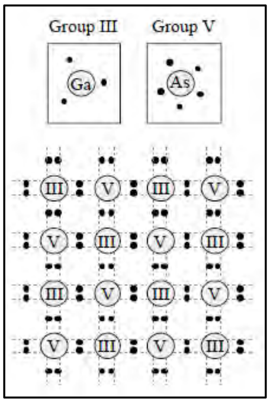

Figure 2.3 Group III-V semiconductor compounds as GaAs

The ionicity becomes even larger and more important in the II-VI compound such as CdSe. The positions of this element in the periodic table are important due to its