UNIVERSITI TEKNIKAL MALAYSIA MELAKA

SYNTHESIS AND OPTICAL CHARACTERIZATION OF MERCURY DISELENIDE ($HgSe_2$) THIN FILMS

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

by

ANIS NABILA BINTI MOHD ISMAIL

B 051110041

920307-14-6322

FACULTY OF MANUFACTURING ENGINEERING

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Date	:	26th 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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ABSTRACT

The stoichiometric mercury diselenide ($HgSe_2$) thin films were prepared by using an electrodepositions technique. The mercury diselenide ($HgSe_2$) thin films were deposited on both the ITO- glass substrates for optical characterization and the stainless steel substrates for the structural and surface morphological characterization. The cyclic voltammetry analysis was done in order to select the best deposition potential which then followed by deposition of thin films process. The selected deposition potential are -1.2, -1.4, -1.5 and -1.6 V. The bath temperature was maintained at 40 ± 2°C. The time taken for each deposition was set at 30 minutes. The thickness of the thin films was measured using weight gain method. The thickness of the thin films was in the range of 1.2651 μm to 2.1084 μm deposited on stainless steel substrates. Based on the X-ray Diffraction analysis, the intense peaks were at deposition potential of -1.6 V. The thin film is confirmed to be polycrystalline in nature and possessed cubic structure with lattice parameters values a = b = c = 6.08 Å. From Scanning Electron Microscope (SEM) analysis, it was proven that when the potential deposition is increase; the surface of the thin film will become non-uniform. The highest deposition potential which is -1.6 V been applied shows a crack on the film's surface. However, the data from UV-Vis-NIR Spectrophotometric measurement revealed that the optical band gap values of the $HgSe_2$ thin film decreased with the increasing of potential voltage which is between 2.91 eV for -1.2 V and 2.37 eV for -1.6 V. The positive slope of the graph plotted from UV-Vis-NIR Spectrophotometric measurement shows that the band gap of $HgSe_2$ thin film is direct.

ABSTRAK

Filem nipis stoikiometri merkuri diselenide ($HgSe_2$) telah disediakan dengan menggunakan teknik pengenapan. Filem nipis merkuri diselenide ($HgSe_2$) telah didapati di kedua-dua jenis substrat iaitu substrat gelas untuk pencirian optik dan substrat keluli tahan karat untuk yang struktur serta pencirian permukaan mofologikal. Analisis voltammetri berkitar telah dilakukan bagi memilih keupayaan endapan terbaik yang kemudiannya diikuti oleh proses pemendapan filem nipis. Keupayaan endapan yang dipilih ialah -1.2, -1.4, -1.5 -1.6 V. Suhu mandi telah dikekalkan pada suhu 40 ± 2°C. Masa bagi setiap pemendapan ditetapkan pada 30 minit. Ketebalan filem nipis diukur menggunakan kaedah tambahan berat. Ketebalan filem nipis di atas substrat keluli tahan karat didapati berada dalam julat 1.2651 μm kepada 2.1084 μm. Berdasarkan sinar X analisis pembelauan, puncak kuat berada di keupayaan endapan -1.6 V. Filem nipis disahkan polihabluran secara semulajadi dan mempunyai struktur padu dengan parameter kekisi nilai satu = b = c = 6.08 Å. Dari penilitian terhadap analisis mikroskop elektron, ia terbukti bahawa apabila pemendapan berpotensi bertambah; permukaan filem nipis akan menjadi tidak seragam. Apabila keupayaan endapan tertinggi; -1.6 V digunakan, terdapat satu rekahan di permukaan filem. Bagaimanapun, data dari UV ukuran Vis NIR Spektrofotometrik menunujukkan bahawa nilai-nilai jurang jalur yang optik filem nipis $HgSe_2$ semakin berkurangan apabila ada penambahan voltan berpotensi yang mana antara 2.91 eV untuk -1.2 V manakala 2.37 eV untuk -1.6 V. Cerun positif graf yang telah diplot berdasarkan UV ukuran Vis NIR Spektrofotometrik menunjukkan bahawa jurang jalur filem nipis $HgSe_2$ adalah langsung.

DEDICATION

To my father; Mohd Ismail bin Hori, my mother; Mahani binti Mohamed Nor, siblings and friends. Your love is my driving force.

To my supervisor, Prof. Madya Dr. T. Joseph Sahaya Anand and all staffs in UTeM.

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

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LIST OF ABBREVIATIONS, SYMBOLS AND SPECIALIZED NOMENCLATURES

°C	-	Degree Celsius
α	-	Alpha
μ	-	Micron
Å	-	Amstrong
nm	-	Nanometer
m	-	Meter
V	-	Voltage
XRD	-	X-Ray Diffractometer
SEM	-	Scanning Electron Microscope
UV-Vis-NIR	-	Ultraviolet Visible-Near-Infrared spectrophotometer
CVD	-	Chemical vapour Deposition
SCE	-	Standard Calomel Electrode
PEC	-	Photoelectrochemical
MoS_2	-	Molybdenum Disulphide
$MoSe_2$	-	Molybdenum Diselenide
$MoTe_2$	-	Molybdenum Ditelluride
Si	-	Silicon
$HgSe_2$	-	Mercury Diselenide
CV	-	Cyclic Voltammetry

Se	-	Selenium
$HgCl_2$	-	Mercury Chloride
$CdSO_4$	-	Cadmium Sulphate
SnO_2	-	Tin Oxide
TeO_2	-	Tellurium Oxide
MCT	-	Mercury Cadmium Telluride
H_2SO_4	-	Sulphuric Acid
CdTe	-	Cadmium Telluride
Ag	-	Silver
AgCl	-	Silver Chloride
NiS_2	-	Nickel Diselenide
TMC	-	Transition Metal Chalcogenide
HCL	-	Hydrochloric acid
Na_2SeSO_3	-	Sodium Selenosulphate
H_2HgO_3	-	Mercuric Acid
SeO_2	-	Selenium Dioxide
JCPDS	-	Joint Committee of Powder Diffraction Standards
ITO	-	Indium Titanate Oxide

CHAPTER 1

INTRODUCTION

1.1 Research Background

Energy has becoming one of the most vital problems at a global scale. The rising price of oil is threatening World's economies whereas the pollution caused by fossil fuels, climate change, is becoming a major source of concern worldwide. The World Energy Outlook published in 2007 by the International Energy Agency (WEO 2007), estimated a continuous growth in demand of energy that would, by 2030, require 55% more energy that today (Mediavilla et al., 2011). Amy Galland, PhD, Research Director at As You Sow said that "Although most of the manufacturing of solar panels use toxic compound; but still the generation of electricity from solar panels is rather safer to both the environment and workers compared to production of electricity from coal, natural gas or nuclear" (Samuels, 2013). On top of that, over 60% of the world's electrical energy used today is generated by steam turbine generators burning fossil fuels as their source of energy. In fact, fossil fuelled plants need some non-renewable sources such as coal, oil and gas in order to produce steam in the combustion chambers. All these non-renewable sources will probably exhaust. Moreover, the combustion process has its potential to release greenhouse gases (Woodbank Communications Ltd, 2005).

Recently, the layered semiconducting compounds which consist of group VIA transition metal dichalcogenides MX_2(M= Mo, W, Sn, Cd, Zn etc. and X= S, Se, Te) has been received a great interest (Anand, 2009).

The development of solar selective coatings, solar cells, photoconductors, sensors, narrowband filters; IR detectors etc. probably use nanocrystalline thin films (Ubale and Ibrahim, 2014). The X-ray diffraction (XRD) studies confirmed the crystallographic nature of these films. Thin films are likely being chosen in solar cells applications due to small thickness that can lead to high absorption; small diffusion length as well as very low weight GHT per unit power (Chopra, n.d.).

The transition metal chalcogenides have received considerable attention because of their ultimate properties. These materials, in thin-film form, are often important candidates for photovoltaic conversion. This is due to a matchable band gap of 1-3 eV with solar spectrum, high optical absorption band gaps and better electrical conductivity. Similarly, they show interesting electric and magnetic properties (Anand et al., 2013). Due to the much larger exciton binding energies than these in III-V compound semiconductor; II-IV semiconductor superlattices, excitonic properties are expected to play a prominent role in optical transitions (Khairnar, 2012). The well-known semiconductor material, Cadmium Selenide (CdSe) is considered as a photovoltaic material due to its high absorption coefficient and almost optimum band gap energy for the efficient absorption of light and conversion into electrical power (Ubale and Ibrahim, 2014).

Present project will adopt the use of Mercury Diselenide ($HgSe_2$) thin film for the photoelectrochemical (PEC) solar cell. Semiconductor is the main component of the PEC cell. The semiconductor will convert the incident photons to electron-hole pairs. Due to the presence of an electric field inside the semiconductor, origin of the space charge layer; these electrons and holes are spatially separated from each other (Krol, 2012).

This project will focus on the Electrodeposition technique for preparing the Mercury Diselenide ($HgSe_2$) thin film. Electrodeposition is an electrochemical liquid phase thin-film and/or powder preparation method where the reactions, either reduction or oxidation, are accomplished using an external current source. The deposition is carried out in an electrochemical cell consisting of a reaction vessel and two or three electrodes (Anand et al., 2013). The thin film processing by electrodeposition technique is more flexible as it can be deposited on various shape of substrate and more simple than other techniques.

Furthermore, it is also relatively lower cost, less time consuming and environmental friendly because it produces no pollution to the surrounding. Hence, no vacuum facility needed. Films deposits obtained by electrodeposition show individual advantage like improved deposit distribution, minimized edge effect, pinhole free deposit, less hydrogen uptake, uniform thickness, better adhesion and low impurity content compared to conventional methods (Anand, 2009).

Cyclic voltammetry (CV) test will be conducted before the deposition process to derive a deposition potential for the Mercury Diselenide $HgSe_2$thin film. Voltammetric measurements are carried out using an electrochemical cell made up of three electrode immersed in a solution containing the analyte and an excess of a non-reactive electrolyte. The three electrodes are graphite electrode, coated conducting glass substrates metal (as a cathode for depositing the thin films) and the standard calomel electrode (SCE).

1.2 Problem Statement

Researchers and engineers have been looking for the method to convert light energy to the electric power. One of the things that can be done is to capture the energy that is freely available from sunlight sources and convert it into the electric power. Increase in awareness that the Earth's oil reserves would run out during the century; then there is a development of the new type of solar cells such as photovoltaics. However, in the photovoltaics mechanism; the photons that fall on a semiconductor will create electron-hole pairs. Whereas at a junction between two different materials; this effect can set up an electric potential difference across the interface (Gratzel, Photoelectrochemical cells, 2001). For the sake of reducing cost; the best material such as elements of Mercury Diselenide ($HgSe_2$) is needed to replace the current material like Silicon which is much expensive. In this issue, cost effective material is highly demand. The current material used in solar panels, crystalline silicon, is more expensive (Anand et al., 2013).

Mercury may come in form of powder which is non-toxic. Selenium also has higher availability in market. On the other hand, they also encounter difficulties in controlling a sophisticated or even complicated deposition process. The sophisticated process might need a critical sample preparation in order to be successful. Since, solar panels have a long production line; need to be fabricated in mass production; so the best approach is to use an electrodeposition technique to synthesize a thin film. One of the suitable methods for producing thin films, owing to the possibility of large area deposition at low cost is the electrodeposition method (Anand & Shariza, 2012). The main concern for electrochemical solar cell technology is to find new materials that have suitable properties for electrochemical energy conversion as well as to have a lower cost energy conversion (Anand et al., 2013). In this report, an attempt is made to prepare Mercury Diselenide ($HgSe_2$) thin films through electrodeposition method and enables the film to be used for characterization studies and thus deciding whether the materials is suitable for photoelectrochemical solar cells application.

1.3 Objectives of the project

This present research is specifically for the purposes as following:

i. To synthesis stoichiometric Mercury Diselenide ($HgSe_2$) thin films by using an electrodeposition method.

ii. To analyse the optical characterization of Mercury Diselenide ($HgSe_2$) thin films by using UV-Visible spectrophotometer.

1.4 Scope of Project

The preliminary experiment will be conducted on synthesizing of $HgSe_2$ thin films by using an electrodeposition technique. Firstly, the best deposition potential of $HgSe_2$ thin films is derived by using a cyclic voltammetry. Next, the $HgSe_2$ thin films need to be confirmed by using X-ray Diffraction and Scanning Electron Microscope methods for its structural and morphological properties. Later, an optical characterization will be conducted by using an UV-visible spectrophotometer.