OPTICAL AND PHOTOELECTROCHEMICAL STUDIES OF TUNGSTEN SULPHOSELENIDE (WSSe) THIN FILMS FOR SOLAR PANELS

FOO YEONG HORNG
B051210134

UNIVERSITI TEKNIKAL MALAYSIA MELAKA
2016

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

OPTICAL AND PHOTOELECTROCHEMICAL STUDIES OF TUNGSTEN SULPHOSELENIDE (WSSe) THIN FILMS FOR SOLAR PANELS

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

FOO YEONG HORNG
B051210134
920501075417

FACULTY OF MANUFACTURING ENGINEERING
2016

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

TAJUK: **OPTICAL AND PHOTOELECTROCHEMICAL STUDIES OF TUNGSTEN SULPHOSELENIDE WSSe THIN FILMS FOR SOLAR PANELS**

SESI PENGAJIAN: 2015/16 Semester 2

Saya **FOO YEONG HORNG**

mengaku membenarkan Laporan PSM ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. **Sila tandakan (√)

☐	SULIT	(Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysiasebagaimana yang termaktub dalam AKTA RAHSIA RASMI 1972)
☐	TERHAD	(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)
☐	TIDAK TERHAD	

Disahkan oleh:

_________________________ _________________________

Alamat Tetap:
NO.29, JLN SELAYANG, TMN SELAYANG,
SG. PUYU, 13020 BUTTERWORTH,
PULAU PINANG

Cop Rasmi:

Tarikh: _________________________

Tarikh: _________________________

** Jika Laporan PSM ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan PSM ini perlu dikelaskan sebagai SULIT atau TERHAD.

DECLARATION

I hereby, declared this report entitled 'Optical and Photoelectrochemical Studies of Tungsten Sulphoselenide (WSSe) Thin Films For Solar Panels' is the results of my own research except as cited in references.

Signature	:	___________________
Author's Name	:	FOO YEONG HORNG
Date	:	22^{TH} JUNE 2016

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 members of the supervisory committee are as follow:

……………….…………………..……………………..

(Dr. Zaleha Binti Mustafa)

……………….………………...……………………..…..

(Prof. Madya Dr. T. Joseph Sahaya Anand)

ABSTRAK

Filem nipis tungsten sulfoselenide WSSe telah berjaya dielektromendapkan ke atas permukaan substrat kaca bersalut indium-timah-oxida (ITO). Tempoh masa pemendapan telah ditentukan selama 10 hingga 30 minit dengan selang 5 minit. Teknik voltammetri berkitar telah digunakan untuk menilai potensi optimum kepada pemendapan filem nipis WSSe. Ketebalan filem nipis diukur melalui kaedah berat badan. Filem-filem nipis tersebut telah dicirikan untuk ciri-ciri struktur dan morfologi permukaan dengan menggunakan XRD and SEM. Ciri-ciri optik dan parameter-parameter semikonduktor filem nipis tersebut telah dilakukan untuk mengkaji kesesuaian filem tersebut sebagai bahan photoelektrokimia (PEC) / sel solar. Filem nipis WSSe didapati memendap secara baik kepada substrat dengan optimum potensi -0.46V dan mencapai ketebalan sehingga 1.44 µm. Analisis struktur melalui pembelauan sinar-X (XRD) mendedahkan bahawa filem-filem tersebut adalah bersifat polihablur dengan intensiti puncak XRD yang semakin meningkat dalam filem-filem yang lebih tebal. Morfologi permukaan filem-filem yang ditentukan oleh imbasan mikroskop elektron (SEM) menunjukkan pertumbuhan filem-filem yang seragam dan merangkumi seluruh permukaan substrat untuk filem-filem yang tebal. Nilai jurang tenaga optik langsung dalam semua jenis filem dinilaikan 2.14 eV sesuai sebagai bahan PEC / sel solar dan nilai yang semakin menurun untuk filem yang lebih tebal. Nilai jurang tenaga optik WSSe filem nipis yang dikaji didapati berada pada 1.5 eV kepada 30 minit masa pemendapan. Keputusan analisis parameter semikonduksi filem nipis menunjukkan bahawa sifat plot Mott-Schottky yang diperolehi adalah daripada bahan jenis-p. Nilai untuk lebar susutan filem-filem tersebut yang berkurangan dari 8.55 hingga 4.34 Å untuk filem nipis WSSe juga disokong kuat dengan nilai-nilai jurang tenaga yang diperolehi daripada kajian optik. Nilai-nilai yang diperoleh dalam keputusan analisis parameter semikonduktor filem nipis WSSe didapati sesuai dan disokong seperti mana nilai-nilai dalam pelbagai logam peralihan kalkogenida yang lain. Ini telah membuktikan bahawa WSSe filem nipis mampu berfungsi sebagai bahan PEC / sel solar yang baik.

ABSTRACT

Transition metal dichalcogenide thin films has greatly interest in the investigation as solar panel material. Tungsten sulphoselenide WSSe thin films were investigated in this research and it were successfully electrodeposited on indium-tin-oxide (ITO)-coated glass substrates. The deposition time for the thin films were set at 10 to 30 minutes with an interval of 5 minutes. Cyclic voltammetry technique was used to determine the optimum potential for deposition of WSSe thin films. Thickness of thin films were measured by using weight gain method. The thin films were characterized for their structural and surface morphological characteristics by XRD and SEM. Their optical and semiconducting parameters were also analyzed in order to determine the suitability of the thin films for photoelectrochemical (PEC) / solar cell applications. WSSe thin films were well adherent to the substrates with optimum potential of -0.46V and grew up to thickness of 1.44 μm. Structural analysis via X-ray diffraction (XRD) analysis reveals that the films are polycrystalline with increasing intensity of XRD peaks in thicker films. 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 thicker films. The optical bandgap energy of the films measured as 2.14 eV for PEC / solar cell materials which decreased as the deposition time of the film increased. Bandgap energy of 1.5 eV was obtained for WSSe thin films deposited at 30 minutes. Results on the semiconducting parameters analysis of the films showed that the nature of the Mott-Schottky plots indicated that the films obtained was of p-type material. The decreasing values for the depletion width of the films from 8.55 to 4.34 Å in WSSe thin films are in good agreement with the decreasing energy gap values retrieved from optical studies. All values come in the range of many other transition metal chalcogenide and this has proven that WSSe thin films is capable as a solar / PEC cell material.

DEDICATION

To my beloved father, Foo Jiin Ming,
mother, Ong Ah Lian,
and my three sisters, Foo Ann Gie, Foo Li Ying and Foo Li May
for giving me moral support, encouragement and understandings.
Your love is my driving force.

To my supervisors, Dr. Zaleha Binti Mustafa
and Prof.Madya Dr. T. Joseph Sahaya Anand
for all the helps, supports and guidance.

ACKNOWLEDGEMENT

Firstly, I am grateful to have P.M. Dr. T. Joseph Sahaya Anand as my supervisor and Dr. Zaleha Binti Mustafa as my official supervisor for my final year project who have been guiding me throughout the period of the final year project. I would like to thank for their patience and motivation while guiding me to a better track of conducting this project. Their guidance on the report has helped me a lot as they have the willingness to sacrifice their time on checking and giving advice on my report.

Besides that, not to forget to express my sincere appreciation to my friends who are under the supervision of P.M. Dr. T. Joseph Sahaya Anand. Throughout the period of final year project, I am able to learn extra knowledge via discussion among each other. They always provide me with cheers, comments and cooperation.

I am also thankful to the dean, deputy deans, all the lecturers and staffs in the Faculty of Manufacturing Engineering, UTeM for their contribution in providing support and space for the successful completion of this project. I thank the technicians for the stimulating discussions and ideas on lab sessions, sample testing, sample analysis, and for all the help they had offered.

Last but not least, I would like to express my deepest thank my family who always giving me support and encouragement throughout the whole research of my final year project. To those involved directly or indirectly for their assistance in completing this research, thank you.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

2D	-	Two Dimensional
aB	-	Bohr Radius of Exciton
CA	-	Chronoamperometry
CE	-	Counter Electrode
CV	-	Cyclic Voltammetry
DBTT	-	Ductile to Brittle Transition Temperature
E_c	-	Conduction Band Edge
E_v	-	Valance Band Edge
H_2WO_4	-	Tungstic acid
HCL	-	Hydrochloric
Hg	-	Mercury
HgS	-	Mercury Sulphate
HgSe	-	Mercury Selenide
ITO	-	Indium Tin Oxide
JCPDS	-	Joint Committee of Powder Diffraction Standards
MoS_2	-	Molybdenum Disulphate
$MoSe_2$	-	Molybdenum Diselenides
MoSSe	-	Molybdenum Sulphoselenide
$Na_2S_2O_3$	-	SodiumTthiosulphate Pentahydrate,
N_c	-	Density of States in Cond. Band
N_D	-	Doping Density
PEC	-	Photoelectrochemical
QSE	-	Quantum Size Effect
SCE	-	Saturated Calomel Electrode
SEM	-	Scanning Electron Microscope
SeO_2	-	Selenium Dioxide
TiO_2	-	Titanium Dioxide
TMC	-	Transition MetalCchalcogenides

TMDs	-	Transition Metal Dichalcogenides
UV/Vis/NIR	-	Ultraviolet/Visible/Near Infrared
V_b	-	Band Bending
V_{FB}	-	Flat Band Potential
W	-	Depletion Layer Width
WE	-	Working Electrode
WSSe	-	Tungsten Sulphoselenide
XPS		X-ray photoelectron spectroscopy
XRD	-	X-ray Diffraction
X-SEM	-	Cross-sectional Scanning Electron Microscope

LIST OF EQUATIONS

CHAPTER 1

INTRODUCTION

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

1.1 Research Background

In this modern era, people's lifestyle has been greatly depends on fossil fuels. However, fossil fuel reserves are being depleted faster than those currently being developed. Furthermore, the combustion of fossil fuels has caused critical environmental issues such as the acid rain, greenhouse effect and global warming. Therefore, the evolution of a clean and renewable energy carrier has become an urgent priority. In various promising hydrogen technology, Fujishima and Honda was the first to report the photocatalytic water-splitting reaction on TiO_2 in 1972 (Fujishima and Honda, 1972). Since from that time, optimal solar energy conversion by the water-splitting reaction on photo-semiconductor catalysts becomes one of the most promising hydrogen technologies. However, the wide band gap (3.0-3.2 eV) of TiO_2 has caused photocatalytic activity produced low quantum efficiency under the visible-light irritation. Therefore, many efforts have been made to improve the photocatalytic efficiency of solar cell (Pan et al., 2011).

The main attribute of each solar cell is its ability of efficiently absorption of solar radiation (photocatalytic efficiency) which consists of wide light spectrum of photons when achieve solar cell surface. The absorption efficiency depends on the optical and electronic properties of absorber layer (semiconductor material) in the solar cell. It defines the wavelength dependent absorption coefficient values and the parameters which are exactly linked to the band gap of the semiconductor and the energy band structure.

Theoretically, if energy of photons less than the minimum energy of absorber band gap, photons cannot be absorbed thus do not contribute to energy conversion. In contrast, an energy band gap value exceeds doubled photon never create more than a single electron-hole pair, which dissipating all the excess energy as heat in the cell.

When choosing wide band semiconductor optical absorption becomes constrained only to high energy photons while neglect for the sub-band gap photons solar cell. This leads to the low photocurrent solar cell but in this case, the advantages are more efficient solar energy spectrum absorption. This is because more photons energy is being converted into electricity thus the solar cell produce higher output voltage.

Contrary to that, even though narrow band semiconductors solar cells have high effectiveness of absorbing wide range of light spectrum and hence produce higher photocurrent values. However, these solar cells have lower energy conversion efficiency in the range of high energy photons and exhibit lower output voltage. The above discussion bring about clear decision that to reach maximum conversion efficiency for a specified solar spectrum absorber material with optimum band gap should be used for any solar cell manufacture (Zdanowicz et al., 2005).

In order to investigate the alternative material used as solar panel, the unique properties of layered form semiconducting transition metal dichalcogenides (TMDs) make them of excessive interest for both academia and industry. They propose an amazing alternative to graphene, as they have a band gap, which is a key for applications in electronics and photonics. These materials have the general formula MX_2 with M represent a transition metal (commonly, but not limited to, Mo, W, Nb, Ta, Ti) and X represent a chalcogen (S, Se, Te). This compound can form thin films composed of a plane of metal atoms covalently bonded to chalcogen atoms (Gatensby et al., 2014). Concurrently, the fabrication of transition metal dichalcogenides thin films have been carried out widely for its large potential use in the non-conventional energy resources industries. In recent years, TMD thin films such as HgSSe, $ZrSe_2$, CdSeTe, WSSe, and CdSSe have been dynamically investigated for photoelectrochemical (PEC) and solar cell applications.

The synthesis of TMD thin films materials enlarges the possibility of observing the development of physical properties of the materials with sizes. The decreasing of sizes, in terms of thickness of the thin film provide the opportunity of observing new behaviours such as size dependent structural, electrical and optical properties. TMD thin films can be form by numerous methods, containing sputtering, chemical bath deposition, selenization, solid state reaction, sulphurization and electrolytic reduction. However, they are costly and occasionally present unusual problems for the fabrication of transition metal dichalcogenide thin films. On the other hand, the electrodeposition technique presents simplicity and economic, control of film thickness, uniformity and possibility of deposition of thin films onto substrates of large and complex shape (Shariza and Anand, 2011).

Cyclic voltammetry technique is applied in this research to obtain the optimum deposition potential for thin films formation. Cyclic Voltammetry is an electrochemical method which generally consists of the application of a potential to an electrode and the measured of the resulting current flowing. The CV measurement is developed with a three electrodes cell system. The three electrodes are working electrode (the electrode that electrochemical reaction happens and thin films are to be deposited), counter electrode (generally used graphite as the electrode) and reference electrode (saturated calomel electrode).

Nowadays, there are a lot of techniques had been developed which function to analyze the microstructure and the properties of the material. In this research, scanning electron microscope (SEM) is conducted to study the surface morphology of thin films and X-ray diffraction technique (XRD) is used to perform the structural analysis of thin films. Optical properties and optical absorption spectra effectiveness of WSSe thin films is measured by using Ultraviolet-Visible-Near Infrared spectrophotometer (UV/Vis/NIR spectrophotometer). Semiconducting parameters such as type of semiconductor, flat band potential (V_{FB}), doping density (N_D), depletion layer width (W), band bending (V_b) of thin films is measured by applying Mott-Schottky plots.

1.2 Problem Statement

The most common world's supply energy source nowadays is fossil fuel (oil, coal and natural gas) which cause critical pollution issue such as acid rain, global warming and greenhouse effects during used to generate energy. Moreover, consumption of fossil fuel progressively increasing due to soaring in global human population. This results in depleting trend of present energy source. Thus, there is an imperative need to overcome the environment deterioration and energy crisis (Tripathi et al., 2007).The most effective way is to develop a clean and renewable energy to replace the fossil fuel. Interest is growing in the use of photoelectrochemical solar cell which is a device of energy conversion. Photoelectrochemical cell able to absorb light spectrum in the form of photons therefore can convert light energy to electrical energy. Light energy is an inexhaustible also environmental friendly energy.

The majority of solar cells on the market are made with silicon. This is because silicon is an elemental semiconductor with good stability and well-balanced set of electronic, physical, and chemical properties. However, silicon is considered as a high cost material and it involves a complicated design and alignment in manufacturing field (Turmezei, 2004). Hence, extensive research is necessary to investigate a suitable material used as solar panel. This alternative material must low cost in price, easy to fabricate, outstanding semiconductor properties, excellent visible light absorption and an efficient charge separation of electrons and holes during the photocatalytic reaction (Pan et al., 2011).

In last decade, transition metal chalcogenides (TMC) thin films have been frequently investigated for photoelectrochemical (PEC) and solar cell applications. TMC thin films are semiconductors with considerable characteristics by optical and semiconducting properties, which can be used as an efficient photovoltaic material. Furthermore, transition metal chalcogenide thin films can be prepared by a safe, non-toxic, cost-efficient and absolutely simple method which is electrodeposition technique.

In this report, a ternary transition metal dichalcogenide compound, WSSe in thin film form which able to prepare with low cost material is investigated to be the alternative material for photoelectrochemical (PEC) and solar cell panels.

1.3 Objectives

This present research is specifically for the purposes as following:

i. To synthesize stoichiometric Tungsten Sulphoselenide (WSSe) thin films by using an electrodeposition method.

ii. To analyze the optical characterization of Tungsten Sulphoselenide (WSSe) thin films by using UV/Vis/NIR spectrophotometer.

iii. To measure the semiconductor parameters of Tungsten Sulphoselenide (WSSe) thin films by using Mott-Schottky plot.

1.4 Scope of Project

This study will be focusing on the properties of Tungsten Sulphoselenide (WSSe) thin films 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, morphological analysis, optical and photoelectrochemical studies.

This research will begin with a cyclic voltammetry measurement to obtain the range of deposition potentials where the films form on electrode. After synthesis of WSSe thin films, the structural properties of the films will be determined by X-ray Diffraction (XRD) technique; whereas, the morphological properties will be examined by using Scanning Electron Microscope (SEM). To analyze the optical characterization of Tungsten Sulphoselenide (WSSe) thin films, UV/Visible/Near Infrared spectrophotometer is used. Lastly, Mott-Schottky plot is used to measure the semiconductor parameters of Tungsten Sulphoselenide (WSSe) thin films.

In brief, the main intention of the research is to investigate the optical characterization and photoelectrochemical studies of Tungsten Sulphoselenide (WSSe) thin films.

1.5 Outline of Project

This report will be divided into five chapters which consist of introduction, literature review, methodology, results and discussion, and conclusions and recommendations.

Chapter one, is the introductory of the project which comprising the research background, problem statement, objectives, and scope of project, 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 discusses 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 discusses about the experimental results and the findings in this research. Data gained through the methods that declared in the previous chapter is analyzed. Brief description and discussion are made through the analyzed data. Some technical opinions and professional perspectives are included in this chapter to enhance the obtained results and support the discussion made.

Chapter five includes the conclusions, recommendations and sustainable elements of the research. Conclusions give a brief statement based upon the objectives. Recommendations for the solution of problem faced in the research are addressed. Sustainable elements indicate the contribution of this research to various relatively fields.