CYCLIC VOLTAMMETRY ANALYSIS OF TUNGSTEN SULPHO SELENIDE (WSSe) THIN FILMS BY ELECTROCHEMICAL ROUTE

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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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BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

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SESI PENGAJIAN: **2015/16 Semester 2**

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I hereby, declared this report entitled "Cyclic Voltammetry Analysis Of Tungsten Sulpho Selenide (WSSe) Thin Films By Electrochemical Route" is the results of my own research except as cited in references.

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Author's Name : LEE WEN LONG

Date : 21/6/2016

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Material) (Hons.). The members of the supervisory committee are as follow:

……

(Principal Supervisor)

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(Co-Supervisor)

ABSTRAK

Salah satu teknologi yang melibatkan pembangunan sel fotovolta adalah teknologi filem nipis. Logam peralihan chalcogenides merupakan semikonduktor yang telah menarik minat pengkaji untuk dibuat menjadi sel fotovolta disebabkan sifat-sifat unik yang ia mempunyai. Penyelidian ini bertujuan untuk menghasilkan filem nipis dengan cara selamat, tidak bertosik dan murah iaitu dengan cara teknik pengenapan . Antara peralihan logam chalcogenides, Tungsten Sulphoselenide telah dikaji dalam kajian ini. Tujuan-tujaun kajian ini adalah mengkaji potensi pemendapan bagi pemendapan WSSe, mensintesis filem nipis WSSe yang stoikiometri melalui teknik pengenapan dan mencicirkan sifat-sifat struktur, komposisi dan morfologi permukaan filem nipis. Dengan kitaran voltammetry analysis, potensi pemendapan bagi pemendapan WSSe filem nipis telah didapati dan pemendapan telah dijalankan dengan masa 15 minit. Selepas pemendapan, kewujudan WSSe telah dipastikan dengan menggunakan X-Ray Diffraction (XRD). Morfologi permukaan filem telah ditentukan dengan mengimbas mikroskop elecktron (SEM), dan ia telah menunjukkan kewujudan retak pada permukaan filem nipis. Ketebalan filem nipis telah ditentukan dengan teknik penambahan keberatan. Daripada keputusan Energy Dispersive X-ray Spectroscopy (EDX), stoikiometri WSSe thin film iaitu nisbah W:S:Se dalam nisbah 1:1:1 tidak didapati . Walaubagaimanapun, potensi yang optimum untuk pemendapan WSSe filem nipis dalam ITO bersalut kaca substrat dan aluminum substrat telah didapati iaitu -0.46V dan -0.78V masing-masing. Untuk mendapati filem nipis yang stoikiometri, rawatan haba bagi filem nipis telah dicadangkan bagi penyelidikan masa depan.

ABSTRACT

One of the most developing technologies that involved in the development of solar cell is the thin film technology. Among the material for the thin film technology, the ternary transition metal chalcogenides (TMCs) are semiconductor which attracts the interest of researcher as the efficient photovoltaic material due to its unique properties. This research is intent to investigate safe, non-toxic, and simple method for the synthesizing of ternary transition metal chalcogenide thin films. Among the TMCs, the tungsten sulphoselenide, WSSe, is one of the TMCs that can be applied in thin film technology. The purposes of this research is to synthesize the stoichiometry WSSe thin film by electrochemical route, to determine the optimum potential for the deposition of the WSSe thin film via cyclic voltammetry analysis, to characterize the thin film that had been deposited via electrodeposition technique for its composition, surface morphological and structural properties. The deposition potential for the WSSe thin film had been determined by using cyclic voltammetry analysis. The presence of the WSSe material on the thin film had been confirmed by the X-ray Diffraction (XRD). The thickness of the thin film had been calculated using the weight gain method. From the surface morphological study by Scanning Electron Microscopy (SEM), it shown that cracking are presence on all the film as the time for the deposition is only 15 minutes. The Energy Dispersive X-Ray Spectroscopy (EDX) had shown that the ratio of W: S: Se is not in the ratio of 1:1:1. The stoichiometry of WSSe thin film is not obtained as there is presence of contaminant on the thin film surface. From the result, it had been shown that the WSSe thin film had successfully deposited on ITO coated glass substrate and aluminum substrate by using optimum potential of -0.46V and -0.78V respectively with the electrodeposition technique. The annealing is suggested for further improvement for the deposition of WSSe thin film.

DEDICATION

To my beloved father, Lee Siang,
mother, Tan Siew Choo,
and my sisters, Lee Lan Yin and Lee Yu Qin,
for giving me moral support, encouragement and understandings.
Your love is my driving force.

To my supervisors, Prof. Madya Dr. Mohd Asyadi Azam Bin Mohd Abid
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LIST OF ABBREVIATIONS

Cd – Cadmium

CdSSe – Cadmium Sulphoselenide

CdTe - Cadmium Telluride

CIGS - Copper Indium Gallium Selenide

Cu - Copper

CV - Cyclic Voltammetry

CVD - Chemical Vapor Deposition

EDX – Energy Dispersive X-ray Spectroscopy

Er - Reflected Energy

Es - Substrate Energy

Ev - Vapor Energy

HCl - Hydrochloric Acid

ITO - Indium Tin Oxide

JCPDS - Joint Committee on Powder Diffraction Standards

Mn - Manganese

MoSSe - Molybdenum Sulphoselenide

$Na_2S_2O_3$ - Sodium thiosulphate pentahydrate

NH_3 - Ammonia

PEC - Photoelectrochemical

PSM - Projek Sarjana Muda

PV - Photovoltaic

PVD - Physical Vapor Deposition

S - Sulphur

SCE - Saturated Calomel electrode

Se - Selenium

SEM - Scanning Electron Microscope

SeO2 - Selenium Dioxide

SiO2 - Silicon dioxide

Te - Tellurium

TMCs - Transition Metal Chalcogenides

W – Tungsten

WS – Tungsten Sulphide

WSe – Tungsten Selenide

WSSe – Tungsten Sulphoselenide

XRD - X-ray Diffraction

$ZnIn_2S_4$ – Zinc Indium Sulphide

ZnO – Zinc Oxide

CHAPTER 1

INTRODUCTION

1.1 Research Background

Thin film solar cells had becoming increasingly popular in recent year. Many researches had been done for the study on different material for the effective solar energy conversion. As an example, Anand & Shariza (2012) had study on the molybdenum sulphoselenide thin films for the development of Photoelectrochemical (PEC) purpose.

The study on the solar energy conversion is important for solving the problem of energy availability. The energy that produces from the current energy sources is not sufficient to compensate the usage of energy today and this had makes the human being to face an energy availability problem. Moreover the solar energy can effectively solve the pollution that cause by the energy source that in the world today as the solar energy are environmental friendly source of energy which does not bring harm to our environment (Mane et al., 2014).

Many of the researchers are focusing on the study of ternary transition metal chalcogenide (TMC) thin films for the solar cell application. This is because the ternary material had some of the advantages. Bhuse (2007) had mentioned the advantages of the ternary material in his study. The main advantage is that over a photon energies range, the ternary material can have a better absorptivity and spectral sensitivity. Therefore, in this research, Tungsten Sulphoselenide (WSSe) that is a new ternary transition metal chalcogenide was study.

The deposition method for the thin film formation can be mainly classified into two types that are Chemical Vapor Deposition (CVD) and Physical Vapor Deposition. The PVD can produce a high quality film, however the thin film that form using the PVD is hard to scale up (Lai et al., 2009). This is because the scale up of the thin film using the PVD process may result in affecting film thickness uniformity and the uniformity of the composition across the substrate (Kapil Mukati et. al., 2009). Therefore the CVD method is introduce for the deposition of the WSSe thin film.

The electrodeposition method is one of the CVD methods for the deposition of the thin film. It was used for the deposition of the WSSe thin film as it had some advantages over other deposition method. the electrodeposition method is a method that is simple, cheap, allow the thickness of the thin film to be adjust, high deposition speed and it does not involve toxic gaseous precursors (Ebrahim et al., 2011; Darmadasa, 2013; Liu et al., 2015)

The cyclic voltammetry (CV) analysis is employ in this project to obtain the suitable potential for the deposition of the WSSe thin film. It is an efficient tool in determining the potential range for the electrodeposition process (Saoniemi, 2000). The suitable potential range can be determined from the cyclic voltammograms that obtain from the cyclic voltammetry analysis. The cyclic voltammetry analysis can be done with the three electrode system and the scanning using different sweep rates was done to obtain the cyclic voltammogram.

There are various types of method that can be employed to analyze the microstructure and properties of material. There are various properties that can be study for certain material, these properties includes the optical properties, electrical properties, mechanical properties, magnetic properties, thermal properties and physical properties. These properties can be study using the material characterization techniques.

In this project, the material characterization method had been carried out for the study of the properties of the WSSe thin films. The material characterization method that is used in this project is the Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) method. The major crystalline phase that present on the thin film was studied using the XRD method. As for the SEM method, it was used to determine the surface morphological properties of thin films. The film thickness can be obtained by using the weight gain method.

1.2 Problem Statement

The main energy source such as fossil fuel and coal that used by the human being had becoming lesser and lesser. The amount of energy usage cannot be compensated with the amount of energy produced as these energy sources need few hundred millions of years to form. Also these energy sources can cause pollution to our environment. Therefore, the solar energy that is a renewable, lower in cost and environmental friendly energy source has becoming increasingly popular in the research recently (Mane et al., 2014).

The solar panel is very expensive in the market. The manufacturing cost of the monocrystalline silicon solar panel with module efficiency of 15% conversion efficiency panel cost USD 47/m^2 (Alan et., al. 2012). Therefore, the price of the solar panel in the market will be higher than the manufacturing cost. This is one of the reasons that the solar panels are not widely used as the main energy sources of the

world. For this reason the development and the research on the solar cell had become a major concern of the world. Therefore, a ternary metal chalcogenide, WSSe thin film with cheaper price was suggested to be used as the material for the solar panel.

There are many research had been done for researching the material for the solar panel. However, many of the research are focusing on the binary TMC such as tungsten disulfide. For example, the research on the tungsten disulfide had been done by Tonti et. al., 1997. The research had shown that the tungsten disulfide is suitable for the material for photovoltaic cells, however it still does not have a high efficiency in converting the solar energy to the electrical energy as the material have an indirect band gap (Brunken et. al., 2009). To obtain the material that have high efficiency in converting solar energy, many research had been done by the researcher. Due to the interesting structural chemistry, unusual electronic properties and rich intercalation chemistry, the TMCs have attracted wide interest in the few decades in the past (Tremel et al, 1995). Therefore, the WSSe thin film had been suggested to be used as the material for the solar panel.

To produce a high quality film for the purpose of the solar cell, the material must deposited uniformly in term of thickness and composition onto the substrate by using the optimum potential. The deposition potential may be vary with the different type of material used. Therefore, the cyclic voltammetry analysis was done to determine the range of potential for the deposition of the WSSe thin film.

1.3 Objectives

In this project, the objectives that are achieved in the research are:

a) To synthesize the stoichiometric WSSe thin films by electrochemical route.
b) To obtain the suitable potential for the growth of thin films using Cyclic Voltammetry Analysis
c) To characterize the composition, crystallography, surface morphological properties of WSSe thin films by using X-ray Diffraction (XRD), Energy Dispersive X-ray Spectroscopy (EDX) and Scanning Electron Microscopy (SEM).

1.4 Scope of Project

The research is focusing on the properties of Tungsten Sulphoselenide (WSSe) thin films for the application of the Photoelectrochemical (PEC) solar cells. In the research, the characterization techniques and experimental procedures for the WSSe thin films was discussed. These including the eletrodeposition of thin films, structural and morphological analysis on the WSSe thin films.

The research is started with the cyclic voltammetry analysis to get the range of deposition potentials for the growth of films. After that, the structural analysis had been done to determine the structural properties of the thin film by using the X-ray Diffraction (XRD) techniques. The morphological properties of the thin films is also be determined by using the Scanning Electron Microscope (SEM). Also, the stoichiometry of the WSSe thin film is confirmed with Energy Dispersive X-ray Spectroscopy (EDX).

As a summary, the main purpose of the research is to obtain the most suitable potential for the deposition of the WSSe onto the substrate and obtain the WSSe thin films.

1.5 Outline of Project

The report is divided into five chapters that is introduction, literature review, methodology, results and discussion, and lastly is the conclusion and recommendation.

Chapter one, is the introduction for the project. It including the research background, problem statement, objective, scope of project and also the outline of the project. Each of these sections can help people to gain the understanding for the project.

Chapter two that is the literature review of the project. In this section, the information which related to the title of this research and also had been published was discussed. This chapter can provide guidance for the research and give an overview for the relevant title. The investigation that had been done by other researcher had given a clear background for the research and also the improvement can be done based on the previous work that had been done by other researcher.

Chapter three is the methodology of the research. In this section, the methods which used in the research had been discussed. The method that used to conduct the research, the material that used for the research and the material characterization method that used for the research to be carried out were discussed in this chapter.

Chapter four is the result and discussion of the research. In this section, the result for the research is presented. The discussion had been done based on the result that obtained from the experimental procedure and characterization technique. The result