UNIVERSITI TEKNIKAL MALAYSIA MELAKA

ELECTROSYNTHESISED $NiSe_2$ THIN FILMS FOR PHOTOELECTROCHEMICAL (PEC) APPLICATIONS

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

by

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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 Materials) (Hons.). The member of the supervisory committee is as follow:

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ABSTRAK

Teknologi filem nipis adalah salah satu teknologi yang membangun pada hari ini yang melibatkan pembangunan sel fotovolta. Nikel selenida, $NiSe_2$ adalah salah satu daripada bahan-bahan yang terlibat dalam teknologi filem nipis. Laporan ini menghuraikan teknologi sel foto kimia yang menggunakan semikonduktor nikel selenida, $NiSe_2$ yang disintesiskan menggunakan elektrik; sebagai bahan penyerap. Mengandungi gambaran keseluruhan mengenai sintesis nikel selenida filem nipis yang baru dibangunkan, latar belakang dan aplikasi sel foto kimia atau solar, projek tahun akhir ini juga menjelaskan secara terperinci kaedah dalam menghasilkan bahan semikonduktor ini daripada bahan-bahan mentah. Teknik sintesis menggunakan elektrik merupakan kaedah yang dipilih untuk menghasilkan filem nipis nikel selenida disebabkan oleh kelebihan teknik ini seperti kemungkinan pengeluaran secara besar-besaran, sisa komponen yang minima, pemantauan proses pemendapan yang mudah dan pemendapan kawasan yang besar. Sampel telah didepositkan dalam tiga masa pemendapan iaitu 20, 25 dan 30 minit bergantung kepada nilai voltan yang diperoleh daripada eksperimen *cyclic voltammetry*. Setelah mendapat keputusan, analisis dilakukan, melibatkan ukuran ketebalan filem nipis oleh kaedah perbezaan berat gravimetrik, kajian struktur oleh pembelau sinar-X (XRD), kajian optik oleh spektrometer UV-Vis, analisis morfologi dan komposisi oleh mikroskop imbasan elektron (SEM) dan spektroskopi serakan tenaga X-ray (EDX) serta Mott-Schottky plot oleh meter LCR (kearuhan (L), kapasitan (C) dan rintangan (R)) untuk mengkaji sifat semikonduktor daripada filem yang didepositkan.

ABSTRACT

Thin film technology is one of the developing technologies nowadays that involves the development in photovoltaic cell. Nickel selenide, $NiSe_2$ is one of the materials involving in thin film technology. This report elaborates the photoelectrochemical technology that utilises electrosynthesised nickel selenide, $NiSe_2$ semiconductor as the absorbent material. Containing the overview regarding the synthesis of newly developed nickel selenide thin film, background and applications of photoelectrochemical or solar cell, this final year project also explains in details the methodology in producing such semiconductor material from the raw materials. Electrodeposition technique is a preferred method to produce nickel selenide thin film due to its advantages such as the possibility of large scale production, minimum waste of components, easy monitoring of deposition process and large area deposition. The samples were deposited within three deposition times namely 20, 25 and 25 minutes with respect to the potentials acquired from cyclic voltammetry. Upon obtaining the result, the analysis was done, involving thin film thickness measurements by gravimetric weight difference method, structural studies by X-ray diffractometer (XRD), optical studies by UV-Vis spectrometer, morphological and compositional analyses by scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDX) as well as Mott-Schottky plot by LCR (inductance (L), capacitance (C) and resistance (R)) meter to study the semiconducting nature of deposited thin film.

DEDICATION

To my beloved parents, supervisor, IMC research group members, siblings, lecturers and friends.

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Å	-	Angstrom
A	-	Optical absorption, angle between solar ray and vertical line
Ag	-	Silver
AgCl	-	Silver chloride
AM	-	Air mass
Al	-	Aluminum
AlSb	-	Aluminum antimonide
α-Si	-	Amorphous silicon
a-Si:Ge:H	-	Hydrogenated amorphous silicon-germanium
a-Si:H	-	Hydrogenated amorphous silicon
CdS	-	Cadmium sulphide
CdTe	-	Cadmium telluride
CIGS	-	Copper indium gallium selenide
CIS	-	Copper indium diselenide
cm	-	Centimeter
C_{sc}	-	Space charge capacitance
c-Si	-	Crystalline silicon
CSS	-	Close-spaced sublimation
Cu	-	Copper
$CuGaSe_2$	-	Copper gallium diselenide
$Cu(In,Ga)Se_2$	-	Copper indium gallium diselenide
$Cu(In,Ga)(S,Se)_2$	-	Copper indium gallium disulphide diselenide
$CuInS_2$	-	Copper indium disulphide
$CuInSe_2$	-	Copper indium diselenide
Cu_2O	-	Copper oxide
Cu_2S	-	Copper sulfide
CV	-	Cyclic voltammetry
CVD	-	Chemical vapour deposition

D_p	-	Crystallite size
€	-	Euro
E_c	-	Conduction band
ECD	-	Electrochemical deposition
EDTA	-	Ethylenediaminetetraacetic acid
EDX	-	Energy disperse analysis of X-rays
E_g	-	Bandgap
E_{ph}	-	Photon energy
eV	-	Electron volt
E_v	-	Valence band
EVA	-	Ethylene vinyl acetate
g	-	Gram
GaAs	-	Gallium arsenide
GaP	-	Gallium phosphide
Ge	-	Germanium
h	-	Planck's constant ($6.62606957 \times 10^{-34}$ J·s)
H_2SO_4	-	Sulphuric acid
InP	-	Indium phosphide
ITO	-	Indium tin oxide
JCPDS	-	Joint Committee on Powder Diffraction Standards
k_{pn}	-	Momentum
LCR	-	Inductance, capacitance and resistance
L	-	Litre
LSPV	-	Linear sweep photovoltammetry
μc-Si:H	-	Hydrogenated microcrystalline silicon
m	-	meter
M	-	Mol
μ	-	micro
mL	-	millilitre
Mo	-	Molybdenum
NaOH	-	Sodium hydroxide
Na_2SeO_3	-	Sodium selenite
Ni^{2+}	-	Nickel ion

$NiSe_2$	-	Nickel selenide
$NiSO_4$	-	Nickel sulphate
NH_4	-	Ammonia
PEC	-	Photoelectrochemical
p-i-n	-	Positive-intrinsic-negative
P-N	-	Positive-negative
PV	-	Photovoltaic
PVD	-	Physical vapour deposition
Se	-	Selenium
SeO_2	-	Selenium dioxide
SEM	-	Scanning electron microscopy
Si	-	Silicon
SiO_2	-	Silicon dioxide
SnO_2	-	Tin dioxide
TCO	-	Transparent conductive oxide
TEA	-	Triethanolamine
v	-	Frequency of light (in Hz)
V_{fb}	-	Flat band potential
V_{SCE}	-	Saturated calomel electron potential
Wp	-	Watts peak
XRD	-	X-ray diffractometry
ZnO	-	Zinc oxide
Zn_3P_2	-	Zinc phosphide
θ	-	Angle of diffraction
$\beta_{1/2}$	-	Broadening of diffraction line measured at the half of its maximum intensity
%	-	Percentage
λ	-	Wavelength

CHAPTER 1

INTRODUCTION

1.1 Research Background

Researchers have been making their efforts in finding new materials for solar energy conversion. There are two important factors that should be taken into consideration in producing these materials which are the band gap energy matching solar spectrum and the competitiveness of production cost. The transition metal chalcogenides and their mixtures have shown the attractive and useful systems for solar energy conversions studies for photoelectrochemical (PEC) cell applications.

Nowadays, thin film technology is being continuously developed as a good approach of substantially reducing the cost of photovoltaic (PV) or photoelectrochemical systems. The rationale for this is that thin-film solar panels are expected to be economical to be manufactured due to their reduced costs of material, energy, handling as well as capital. The minimisation of the amount of material used, the use of cost effective materials, processing methods and mounting arrays contribute to the achievement of reduction cost of thin film cells.

Electrodeposition is the preferred technique in thin film preparation owing to its advantages such as the possibility for large scale production, minimum waste of components and easy monitoring of the deposition process. This technique generally cost effective rather than those physically prepared methods. The quality of the films deposited depends on the composition of the electrolytes throughout the electrodeposition process (Zainal *et al.*, 2005). The preparatory parameters such as growth rate, deposition temperature and the structural, compositional, morphological, optical, semiconductor properties are among the matters to be studied on the produced thin films.

1.2 Problem Statement

As a renewable energy, the solar power is capable to provide green and clean electrical source, but there are obstacles that prevent this source to be widely used worldwide. One of the major concerns regarding the photovoltaic application is the cost of its implementation. Solar panels including their components are indeed very expensive. They must be constantly be maintained and often replace as they are constructed from fragile materials such as semiconductors and glass. Furthermore, each single photovoltaic panel has low conversion efficiency, resulting in deficiency of power produced. The action of using a greater number of solar panels does overcome this problem, provided the sufficiently large land area is available, but the overall cost will increase by the amount and size of solar panels used. There is a higher barrier for market entry for thin film technologies of solar cell due to higher capital cost per unit output for thin film manufacturing facilities. Also, as solar cells require the sunlight to perform, only areas of the world with a lot of sunlight are suitable for solar power generation.

1.3 Objectives

(a) To electrically synthesise the exact stoichiometric $NiSe_2$ thin film by elecdrodeposition method.

(b) To study the structural, optical and semiconductor properties of $NiSe_2$ thin films in comparing with existing materials.

1.4 Scope

The scope of this project lies on the nickel selenide thin film itself in term of its experimental procedures, characterisation techniques as well as its semiconducting and optical properties. Since, this project involve the tiny thin films, ranging from fractions of a nanometre (monolayer) to several micrometers, the microstuctural analysis is to be carried out by using scanning electron microscope and X-ray diffractometer that are capable of analysing the samples at such scale. Lastly, the observation regarding the performance of nickel selenide thin film with respect to solar energy conversion efficiency will be carried out after performing its microstructural analysis.

1.5 Outline of Project

This final year project is divided into five chapters comprising of introduction, literature review, methodology, results and discussion as well as conclusion and future work respectively. The first introductory chapter elaborates briefly the research background, problem statement, objectives, scope of study and the outline of project.

Chapter two, literature review chapter presents the published literatures that are relevant to particular topic of this research, demonstrating the knowledge of any previous work and awareness of related theories, debates and controversies. Also, this chapter provides background to the new research, linking the new research to what has preceded it.

On the other hand, chapter three discusses the review of the methodology carried out in order to produce the desired product or outcome of the project. The most appropriate method was chosen, allowing the sample to be further analysed by suitable material characterization methods.

Chapter four provides the details of the results acquired throughout the experiment as well as the discussion on the results. The discussion consists of the justification and problems that have been undergone. The data have been given in the forms of tables and figures.

The conclusions and recommendation about this study are discussed in Chapter five, concluding all other chapters and recommending the possible betterment to get the more satisfactory outcome in the future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Thin Film

Thin film solar cells are showing a great potential for terrestrial and space photovoltaics and offer a wide variety of choices in terms of the device design and fabrication. A variety of substrate (flexible or rigid, metal or insulator) can be used for deposition of different layers such as contact, buffer, absorber and reflector using different deposition techniques including PVD, CVD, ECD, plasma-based or hybrid. Such versatility allows tailoring and engineering of the layers in order to improve device performance. Thin film device fabrication becomes complex, requiring proper control over the entire process sequence for large area devices. Proper understanding of thin film deposition processes can help in achieving high efficiency devices over large areas as has been demonstrated commercially for different cells. Research and development in new, exotic and simple materials and devices incorporated with innovative, but simple manufacturing process need to be pursued thoroughly in order to get that outstanding performance of thin film solar cells. The features of thin film processes have been shown to be of interest for solar cell technologies as listed below.

(a) The availability of a variety of physical, chemical, electrochemical, plasma based and hybrid techniques for depositing thin films of same material.

(b) Microstructure of the thin films of most materials can be varied from amorphous/nanocrystalline to a highly oriented and/or epitaxial growth, depending on the technique, deposition parameters and substrate.

(c) A wide choice of shapes, sizes, areas and substrates are available.