



Faculty of Manufacturing Engineering

**ELECTROSYNTHESISED NiTe₂ THIN FILMS FOR
PHOTOELECTROCHEMICAL (PEC) CELL**

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Master of Science in Manufacturing Engineering

2014

**ELECTROSYNTHESIS AND CHARACTERIZATIONS OF NiTe₂ THIN FILMS
FOR PHOTOELECTROCHEMICAL (PEC) CELL**

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**A thesis submitted
in fulfillment of the requirements for the degree of Master of Science
in Manufacturing Engineering**

Faculty of Manufacturing Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2015

DECLARATION

I declare that this thesis entitled “Electrosynthesis and Characterizations of NiTe₂ Thin Films for Photoelectrochemical (PEC) Cell” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :

Name :

Date :

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering.

Signature :

Supervisor Name :

Date :

DEDICATION

To my beloved family, teachers, lecturers and friends.

ABSTRACT

This project emphasises the synthesis of the stoichiometric nickel telluride, NiTe₂ thin films as the solar or photoelectrochemical (PEC) cells absorbent. Nickel telluride thin film in the form of transition metal chalcogenide, MX₂ (M = transition metal, X = chalcogenide [S, Se, Te]) offers promising properties in such application. Electrodeposition has been chosen to deposit the film onto the substrate due to its advantages such as possibility of large scale deposition, minimum waste of components, easy monitoring of deposition process and large area deposition. Using this technique, nickel telluride thin films were cathodically deposited onto indium tin oxide (ITO) glass substrates. By changing the deposition parameters such as deposition potential, additive concentration and deposition time throughout the film synthesis, high quality films having good adhesion, smooth surface and uniform distribution were acquired. It was found that the optimal films parameters are in the presence of 0.1 M triethanolamine (TEA), 20 min deposition time and -1.0 V potential based on a few electrodeposition experiments. Structural characterisation through X-ray diffraction studies revealed the presence of hexagonal structure of nickel telluride, NiTe₂ thin film with lattice parameters $a = b = 0.3843$ nm and $c = 0.5265$ nm. Scanning electron micrographs exposed that the films was pinhole-free, compact and smooth, showing a granular structure having almost spherical shape with well-defined grains. On the other hand, the film composition was confirmed to present both nickel and tellurium complying the correct stoichiometry by using Energy-dispersive X-ray (EDX). The optical absorption analysis employed by Shimadzu 1700 UV-Vis Spectrophotometer has confirmed that the energy bandgaps of NiTe₂ thin film lie within the semiconductor range (1 - 1.2 eV) with indirect nature. The positive Mott-Schottky plots indicates that NiTe₂ thin film is negatively charged (n-type conductivity), having more electrons (e⁻) than holes (h⁺). The derivation of semiconductor parameters like doping density, N_D, built in voltage, V_b (band bending) and flat band potential, V_{fb} obtained from semiconductor studies play significant part in determining the conversion efficiency of photoelectrochemical (PEC) cell. The results attained from these characterisations have verified the compatibility of NiTe₂ as solar cell green alternative materials.

ABSTRAK

'Elektro-sintesis dan Pencirian Filem Nipis NiTe₂ untuk Sel Fotoelektrokimia'. Projek ini menekankan sintesis filem nipis nikel telurida, NiTe₂ yang stoikiometri sebagai bahan penyerap sel fotoelektrokimia atau sel solar. Filem nipis nikel telurida dalam bentuk logam peralihan kalkogenida, MX₂ (M = logam peralihan, X = kalkogenida [S, Se, Te]) menawarkan ciri-ciri yang meyakinkan dalam aplikasi tersebut. Elektroenapan telah dipilih untuk mendepositkan filem ke atas substrat kerana kelebihan seperti kemungkinan pemendapan berskala besar, pembaziran komponen yang minimum, pemantauan proses pemendapan yang mudah dan kawasan pemendapan yang luas. Dengan menggunakan teknik ini, filem nipis nikel telurida telah didepositkan secara katodik ke atas kaca substrat yang bersalut indium timah oksida. Dengan mengubah parameter pemendapan seperti voltan pemendapan, konsentrasi bahan tambahan dan masa pemendapan di sepanjang proses sintesis filem, filem-filem yang berkualiti tinggi yang mempunyai kelekatan yang baik, permukaan licin dan penyebaran yang sekata telah diperolehi. Didapati bahawa parameter filem yang dipilih telah diperolehi melalui kehadiran 0.1 M Triethanolamine (TEA), masa pemendapan 20 minit dan voltan -1.0 V berdasarkan beberapa eksperimen elektroenapan. Kajian struktur melalui belauan sinar-X mendedahkan kehadiran struktur heksagon filem nipis nikel telurida, NiTe₂ dengan parameter kekisi $a = b = 0.3843$ nm and $c = 0.5265$ nm. Mikroskop pengimbasan elektron (SEM) mendedahkan bahawa filem adalah bebas daripada liang halus, padat dan rata, menunjukkan struktur bergranul yang mempunyai bentuk hampir sfera dengan butiran yang jelas. Selain itu, komposisi filem disahkan bagi memaparkan kedua-dua nikel dan telurium yang mematuhi stoikiometri yang betul dengan menggunakan Serakan-Tenaga Sinar-X (EDX). Analisis penyerapan optik dijalankan menggunakan Shimadzu 1700 UV-Vis Spektrofotometer telah mengesahkan bahawa jurang jalur tenaga filem nipis NiTe₂ terletak di dalam lingkungan semikonduktor (1 – 1.12 eV) dengan sifat tidak langsung. Plot Mott Schottky yang positif menunjukkan bahawa filem nipis NiTe₂ bercas negatif (konduktiviti jenis-n), mempunyai lebih banyak elektron (e^-) daripada lubang (h^+). Pemerolehan semikonduktor parameter seperti ketumpatan pendopan, N_D , voltan terbina dalam, V_b dan pinggir jalur valens, V_{fb} yang diperolehi dari kajian semikonduktor memainkan peranan penting dalam menentukan kecekapan penukaran sel fotoelektrokimia. Keputusan yang dicapai daripada pencirian ini telah mengesahkan keserasian NiTe₂ sebagai bahan alternatif hijau sel solar.

ACKNOWLEDGEMENTS

First and foremost, I would like to express my heartiest gratitude to Almighty God for bringing me up, giving me opportunity to further study. To my beloved family, thanks for the continuous support from you. Also, I would like to thank my supervisor, Associate Professor Dr. T. Joseph Sahaya Anand for his countless guidance and advice that give me vigorous spirit to finish my Master's degree on time.

To the Fundamental Research Grant Scheme (FRGS), Ministry of Energy, Green Technology and Water (KeTTHA) and Zamalah Scholarship, millions of thanks for sponsoring me in terms of facilities and allowance. Particularly, I would like to express my deepest gratitude to library staffs and laboratory technicians for their assistance and efforts in the entire lab and analysis works. Lastly, special thanks to all my peers and everyone who had been contributed to the crucial parts of realization of this project.

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LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURES

α	-	Absorbance
Ag	-	Silver
Ag ₂ Te	-	Silver telluride
AgCl	-	Silver chloride
a-Si:H	-	Hydrogenated amorphous silicon
$\beta_{1/2}$	-	Broadening of diffraction line measured at the half of its maximum intensity
Bi ₂ Te ₃	-	Bismuth telluride
c	-	Speed of light
CBD	-	Chemical bath deposition
CdS	-	Cadmium sulphide
CdTe	-	Cadmium telluride
CGS	-	Copper gallium selenide
CIGS	-	Copper indium gallium selenide
cm	-	Centimetre
CoTe	-	Cobalt telluride
CSS	-	Closed space sublimation
Cu	-	Copper
CV	-	Cyclic voltammetry
CVD	-	Chemical vapour deposition
DC	-	Direct current
D_p	-	Crystallite size
DSSC	-	Dye-sensitized solar cell
E	-	Photon energy
ϵ	-	Dielectric constant
ϵ_0	-	Dielectric constant of free space

EDA	-	Ethylenediamine
EDTA	-	Ethylenediaminetetraacetic acid
EDX	-	Energy dispersive X-ray
E_g	-	Bandgap
$e-h$	-	Electron-hole
eV	-	Electron volt
g	-	Gram
Ga	-	Gallium
GaAs	-	Gallium arsenide
GaInP	-	Gallium indium phosphide
GaN	-	Gallium nitride
GaP	-	Gallium phosphide
Ge	-	Germanium
h	-	Planck's constant
H ₂ O ₂	-	Hydrogen peroxide
HCl	-	Hydrochloric acid
Hg _{1-x} Cd _x Te	-	Mercury cadmium telluride
Hz	-	Hertz
I	-	Current
I_F	-	Forward-bias current
I_L	-	Photocurrent
In	-	Indium
InGaAs	-	Indium gallium arsenide
InP	-	Indium Phosphide
I_{sc}	-	Maximum possible current
ITO	-	Indium tin oxide
JCPDS	-	Joint Committee on Powder Diffraction Standards
k_B	-	Boltzmann's constant
KCl	-	Potassium chloride
LCR	-	Inductance, Capacitance, Resistance
LED	-	Light-emitting diodes
m_e^*	-	Effective electron mass in the conduction band
μF	-	Microfarad

μm	-	Micrometer
MOCVD	-	Metal organic vapour chemical deposition
MoO_3	-	Molybdenum oxide
NaOH	-	Sodium hydroxide
N_c	-	Density states in conduction band
N_D	-	Doping density
nF	-	Nanofarad
$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$	-	Nickel sulphate hexahydrate
NiTe_2	-	Nickel telluride
OPV	-	Organic photovoltaics
P3HT	-	Poly(3-hexyothiophene)
Pb	-	Lead
PbTe	-	Lead telluride
PCBM	-	[6,6]-phenyl C61-butyric acid methylester
PEC	-	Photoelectrochemical
PECVD	-	Plasma enhanced chemical vapour deposition
PEDOT	-	Poly(3,4-ethylenedioxythiophene)
PET	-	Polyethylene terephthalate
P_{in}	-	Power input
PIN	-	Doped pn-junction
PLD	-	Pulsed laser deposition technique
PSS	-	Poly(styrenesulfonate)
PV	-	Photovoltaic
PVC	-	Polyvinyl chloride
QDSC	-	Quantum dot solar cells
RF	-	Radio frequency
S	-	Sulphur
s	-	Second
Sb_2S_3	-	Antimony sulphide
Sb_2Se_3	-	Antimony selenide
SbCl_3	-	Antimony chloride
SCE	-	Saturated calomel electrode
Se	-	Selenium

SEM	-	Scanning electron microscope
SHE	-	Standard hydrogen electrode
Si	-	Silicon
SiC	-	Silicon carbide
SiO	-	Silicon monoxide
SnO	-	Tin oxide
T	-	Temperature
Ta ₂ O ₅	-	Tantalum oxide
TCO	-	Transparent conducting oxide
Te	-	Tellurium
TEA	-	Triethanolamine
TeO ₂	-	Tellurium dioxide
TiO ₂	-	Titanium dioxide
TMC	-	Transition metal chalcogenides
UV-Vis	-	Ultra violet-visible
ν	-	Frequency
V	-	Potential/Voltage
V ₂ O ₅	-	Vanadium oxide
V_b	-	Built-in voltage
$V_{F,redox}$	-	Redox potential
V_{fb}	-	Flatband potential
V_m	-	Voltage that produces the maximum power
V_{oc}	-	Maximum possible voltage
wt%	-	Weight percentage
XRD	-	X-ray diffraction
ZnS	-	Zinc sulphide
λ	-	Wavelength
θ	-	Angle of diffraction
°C	-	Degree Celcius
Ω	-	Ohm

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Concerning the environmental sustainability, the energy supplies from renewable sources such as solar, thermal, wind, hydro, biofuels and geothermal have been every nation's energy strategy (Twidell and Weir, 2006). The interaction of light and semiconductors has been studied for a long time, including the photoelectric effect, which proved that light acted as a particle in many cases (Poortmans and Arkhipov, 2006). Solar cell is one of the semiconductor devices that can be designed and fabricated to detect and generate optical signals. Solar cells and photodetectors convert optical power into electrical power whereas light-emitting diodes (LED) and laser diodes convert electrical power into optical power. The characteristics of solar cells and photodetectors are a function of optical energy that is absorbed in the semiconductor, generating the excess electron-hole pairs, producing photocurrents. Solar cells have gained tremendous importance in the area of renewable energy sources.

Thin film is a material created from the beginning by the random nucleation and growth processes of individually condensing/reacting atomic/ionic/molecular species on a substrate. Its structural, chemical, metallurgical and physical properties are energetically dependent on a large number of deposition parameters and may be thickness dependent as well. Thin films may encompass a considerable thickness range, differing from a few nanometres to tens of micrometres (Poortmans and Arkhipov, 2006). Apart from the widespread use of polycrystalline silicon solar cell, thin film technology has been