CYCLIC VOLTAMMETRY ANALYSIS OF TRANSITION METAL CHALCOGENIDE TUNGSTEN DITELLURIDE (WTe_2) THIN FILMS

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

by

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Tajuk: **CYCLIC VOLTAMMETRY ANALYSIS OF TRANSITION METAL CHALCOGENIDE TUNGSTEN DITELLURIDE (WTe_2) THIN FILMS**

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Signature : ……………………………………
Author's Name : FAN KERRY
Date :

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee are as follow:

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(Principal Supervisor)

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(Co-Supervisor)

ABSTRAK

Perkembangan teknologi filem nipis untuk aplikasi panel suria meningkat disebabkan usaha untuk mencari alternatif bagi menggantikan wafer silikon mahal dan meningkatkan kecekapan panel solar. Logam peralihan chalcogenide (TMCs) adalah semikonduktor yang mempunyai ciri-ciri yang unik dengan mempunyai jurang jalur antara 1.0 hingga 2.0 eV. Ciri-ciri TMC ini menjadi kepentingan penyelidik untuk membangunkan bahan solar yang efisien menggunakan filem nipis TMC. Tungsten ditelluride (WTe_2) adalah salah satu TMC yang boleh digunakan sebagai teknologi filem nipis. Tujuan kajian ini adalah untuk mensintesiskan filem-filem tipis stoikiometri WTe_2 melalui kaedah elektrodeposisi, untuk menentukan parameter pemendapan seperti potensi pemendapan dan masa pemendapan bagi pertumbuhan filem-filem nipis melalui analisa voltametri kitaran, dan untuk mencirikan komposisi dan kristalografi menggunakan Pembelauan Sinar-X (XRD) dan sifat morfologi permukaan filem nipis WTe_2 dengan menggunakan Mikroskopi Pengimbasan Elektron (SEM). Potensi yang sesuai untuk pemendapan WTe_2 telah ditentukan dengan menggunakan voltametri kitaran. Tiga penyelesaian prekursor tanpa aditif dan dengan aditif, asid atelindiamintetrasetik (EDTA) dan trietanolamin (TEA) dipelajari. Adalah didapati bahawa EDTA sesuai digunakan untuk meningkatkan lekatan filem nipis pada substrat kaca oksida timah indium (ITO). Potensi yang sesuai untuk pemendapan filem nipis ialah antara -0.85V hingga -1.35V. Kaedah elektrodeposisi digunakan untuk menghasilkan filem tipis WTe_2. Ketebalan filem tipis WTe_2 dikira dengan menggunakan kaedah perbezaan berat gravimetrik. Dari XRD, kehadiran filem nipis WTe_2 disahkan dengan kehadiran puncak tajam yang menunjukkan struktur polikristalin. Kajian dari SEM menunjukkan bahawa 20 minit masa pemendapan memberikan pembentukan filem tipis WTe_2 yang baik. Dari hasil yang diperolehi, filem-filem tipis WTe_2 berjaya disimpan di substrat kaca ITO di bawah keadaan optimum potensi pemendapan -0.95V selama tempoh 20 minit pemendapan dengan kehadiran EDTA dan komposisi prekursor elektrolit 0.0038M TeO_2 + 0.0024M H_2WO_4 + 0.021M EDTA.

ABSTRACT

The development of thin films technology for solar panel application is rising due to the effort of researchers to find an alternative to replace expensive silicon wafer and to improve the efficiency of solar panel. Transition metal chalcogenide (TMCs) are semiconductor that has unique properties from having ideal bandgap between 1.0 to 2.0eV. This characteristic of TMCs becomes the interest of researchers to develop an efficient photovoltaic material using transition metal chalcogenide thin films. Tungsten ditelluride (WTe_2) is one of the TMCs that can be applied as thin film technology for solar panel application. The purpose of this research is to synthesize the stoichiometry WTe_2 thin films by electrodeposition method, to determine the deposition parameters such as deposition potential and deposition time for the growth of thin films via cyclic voltammetry analysis, and to characterize the composition and crystallography using X-Ray Diffraction (XRD) and surface morphological properties of WTe_2 thin films by using Scanning Electron Microscopy (SEM). The suitable potential for the deposition of WTe_2 had been determined by using cyclic voltammetry. Three precursors solutions without additive and with additive, EDTA and TEA was studied. It is found that EDTA is suitable to be used for the deposition of WTe_2 thin films to improve the adhesion of thin films on ITO glass substrates. The range of potential suitable for the deposition is -0.85V to -1.35V. Electrodeposition method is used to deposit WTe_2 thin films. The thickness of WTe_2 thin films are calculated by using gravimetric weight difference method where -0.95V is found to be the most suitable deposition potential in giving a dark uniform black film. From XRD, the presence of WTe_2 thin films are confirmed with the presence of sharp peak indicating polycrystalline structure. Surface morphological study by SEM shows that 20 minutes deposition time gives uniform WTe_2 thin films formation with uniform film distribution. From the results obtained, WTe_2 thin films is successfully deposited on ITO glass substrates under the optimum condition of deposition potential -0.95V for duration of 20 minutes deposition time with the presence of EDTA and electrolyte precursors composition of 0.0038M TeO_2 + 0.0024M H_2WO_4 + 0.021M EDTA.

DEDICATION

This project is dedicated to
my beloved father, Fan Yin Seng,
my loving mother, Kee Lai Cheng,
and my supportive sister, Lee Soong Ai,
for giving me never ending of moral support,
encouragement and understandings.
Your undeniable love is my driving force.

To my supervisors,
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LIST OF ABBREVIATIONS

Ag	-	Silver
AgCl	-	Silver Chloride
C	-	Carbon
CBD	-	Chemical Bath Deposition
CdSe	-	Cadmium Selenide
CdTe	-	Cadmium Telluride
CIGS	-	Copper Indium Gallium Sulphide
CV	-	Cyclic Voltammetry
EDTA	-	Etylenediaminetetraacetic acid
H_2SO_4	-	Sulphuric acid
HCl	-	Hydrochloric acid
ITO	-	Indium Tin Oxide
MoS_2	-	Molybdenum Disulphide
$MoSe_2$	-	Molybdenum Diselenide
$MoTe_2$	-	Molybdenum Ditelluride
MW_p	-	Mega Watt per peak
$NiTe_2$	-	Nickel Ditelluride
PSM	-	Projek Sarjana Muda
Pt	-	Platinum
PV	-	Photovoltaic
PVD	-	Physical Vapour Deposition

S	-	Sulphur
SCE	-	Saturated Calomel Electrode
Se	-	Selenium
SEM	-	Scanning Electron Microscopy
Te	-	Tellurium
TEA	-	Triethanolamine
TMC	-	Transition Metal Chalcogenide
TWh	-	Terawatt per hour
VdW	-	Van der Waals
WS_2	-	Tungsten Disulphide
WSe_2	-	Tungsten Diselenide
WTe_2	-	Tungsten Ditelluride
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

$^{\circ}$		degree
eV	-	electron Volts
nm	-	nanometre
mm	-	millimetre
µm	-	micrometre
i_{pa}	-	Anodic peak current
i_{pc}	-	Cathodic peak current
E_{pa}	-	Anodic peak potential
E_{pc}	-	Cathodic peak potential
MW_p	-	Megawatt per peak
W_p	-	Watt per peak

CHAPTER 1

INTRODUCTION

1.1 Research Background

This research is the study on alternative supply of energy such as solar energy which is essential to solve energy deficiency. As the world population and development is rising exponentially, the energy resources are depleting and is unable to meet global needs. Solar energy is obtained from the sun, so the energy is replenished naturally throughout the year providing continuous supply of energy.

As solar market is expanding, high portion of sales is dominated by silicon wafers whereby silicon wafer costs over 50% of the total module costs (Green, 2007). Thin film materials become the potential candidate to reduce the use of expensive silicon wafer. Many researchers had done extensive study on thin films development for solar panel. Paetzold et al., (2017) has recently studied the efficiency of copper indium gallium selenide (CIGS) and perovskite thin films to converse energy for solar panel.

The development of various materials by researchers lead to the findings of transition metal chalcogenide (TMC) thin films as suitable candidates for solar panel. One significant example is cadmium telluride (CdTe) whereby Britt and Ferekides (1993) had done a research on the efficiency of this material. The research shown that cadmium telluride is a suitable candidate whereby it has near ideal bandgap of 1.45eV and high absorption ability. Even until the millennium, cadmium telluride continues becoming the research prospective for the development of cost effective and reliable solar material (Morales-Acevedo, 2006).

Recently, TMCs becomes the attention of several researchers due to its adjustable crystal structures under various condition, unique morphological structure with admirable optical, electrical, chemical and physical properties allowing them to suit in various application. TMCs also shows remarkable interactive properties with visible lights due to its bandgap properties (Gao et al, 2016). Various TMCs thin films has been studied by other researchers. Lin et al., (2014) had studied the contact of $MoSe_2$ layer with CIGS thin films using sputtering to enhance the performance of solar cell. The characteristics of TMCs has sparks the interest to study other TMCs thin films material for solar panel application. One of them are the research of this project which is Tungsten Ditelluride (WTe_2).

To study the properties of WTe_2, synthesizing and characterizing of this material is the main focus. Thin films can be extracted from different techniques which are primarily categorize into two which are physical vapour deposition (PVD) and chemical method of deposition (Deo et al., 2015). Chemical method of deposition is preferred by most researchers due to its low-cost set ups, fast deposition and good deposition quality compared to PVD. And thus, chemical method of deposition is chosen to be used in the synthesizing of WTe_2.

In chemical methods, there are several techniques categorize under this which are electrodeposition, chemical bath deposition, spray pyrolysis and others. In this research project, electrodeposition method is chosen to deposit WTe_2 thin films. Electrodeposition technique require basic tools set-up at lower cost compared to other deposition methods. Besides that, electrodeposition set-up can be utilised in cyclic voltammetry analysis (Deo et al., 2015).

Cyclic voltammetry (CV) analysis is used in this research to determine optimal potential for the deposition of WTe_2 thin films. As stated by Lee et al., (2014), cyclic voltammetry helps to get important information regarding the electrochemical reaction that occurs where the peak of cyclic voltammograms shows whether a reaction occurred or not at that particular potential. Several other researchers have used CV analysis to determine the redox potential of thin films. Kim et al., (2010) has used a three electrodes system to study the deposition of CdTe and CdSe thin films under various concentration and analyse the electrochemical reaction at different potential. In CV, the standard potential range used is -2 to +2V to accomplish both oxidized and redox potential.

After synthesizing these thin films, the characterization will be carried out using various techniques to determine the properties of WTe_2 thin films. To determine the surface morphology properties of thin films, SEM technique can be used (Vernon, 2000). XRD is used to study the crystallography or crystalline phase that is present in the thin film.

1.2 Problem statement

The global energy demand is increasing rapidly due to world's rising population and advancement in technology (Kannan and Vakeesan, 2016). As the consumption of energy overshoot the available conventional energy supply, other alternatives sources like renewable energy to generate energy supply that is needed to prevent energy crisis.

As there are several renewable energies to be considered from resources such as wind, biomass, geothermal, solar and hydro, solar energy or known as photovoltaic energy is the most suitable option due to its availability all year long. Based on the Technology Roadmap on International Energy Agency by Philibert et al., (2014), solar energy is the most abundant energy available with about 885 million terawatt hours (TWh).

Crystalline silicon becomes the most viable material in the production of solar modules whereby silicon wafers take at least 80% of shipped modules globally with volume of 3400 to 4600 tonnes of silicon consumed by PV industry yearly (Sarti and Einhaus, 2002). But the deficiency of silicon has elevated the market price of raw silicon which in turn increasing the price of solar panel in the market. Saga (2010) states that the demand and supply balance for PV cell and production of semiconductor affects the market price of silicon. Such that, the cost of silicon rises from 30% to more than 50% due to high market demand of silicon. This condition is not favourable as PV industry is looking for various ways to reduce the cost of PV cells to ensure that it can compete with the electricity prices worldwide.

Various research has been done to reduce the costs and increase efficiency of solar cells. One of the focus is the use of thin films as solar panel's absorber to replace the expensive silicon. The development of various materials by researchers lead to the findings of TMCs as suitable candidates for solar panel. One significant example is cadmium telluride whereby Britt and Ferekides (1993) had done a research on the efficiency of this material. The research shown that cadmium telluride is a suitable candidate whereby it has near ideal bandgap of 1.45eV and high absorption ability.

On the other hand, TMCs such as molybdenum diselenide ($MoSe_2$), tungsten disulphide (WS_2), tungsten diselenide (WSe_2) and others have also gained the interest of researcher due to its unique characteristics for wide range of application including for the use of energy storage and conversion efficiency (Tan et al., 2017). Although, Srivastava and Avasti (1985) had studied the properties and preparation method for tungsten dichalcogenides series, the research concludes that WS_2 and WSe_2 has been proved to be showing potential in converting sunlight to electrical or chemical energy. Therefore, WTe_2 has been suggested to be the research material for this project to test for its suitability to become one of the solar panel material as part of the tungsten dichalcogenides series.

1.3 Objective

The objectives to be achieved in this research project are:

a) To synthesize the stoichiometric tungsten ditelluride thin films by using electrodeposition method.
b) To analyze the deposition parameters such as deposition potential and deposition time for the growth of thin films by using Cyclic Voltammetry Analysis.
c) To characterize the composition and crystallography using X-Ray Diffraction (XRD) and surface morphological properties of tungsten ditelluride thin films by using Scanning Electron Microscopy (SEM).

1.4 Scope of Project

In this project, Tungsten Ditelluride (WTe_2) is the focus material of research whereby its properties will be determined for the application of solar panels. The experimental procedures and characterization techniques will be discussed in this research which include the synthesizing method, synthesizing parameters determination and method of characterization of WTe_2 thin films.

The research starts off with cyclic voltammetry analysis to determine the suitable range of deposition potentials and parameters for the growth of thin films. The synthesizing method that will be used in the analysis is electrodeposition. After that, structural analysis by using X-ray Diffraction (XRD) technique is done to determine the structural properties of WTe_2. The surface morphological of WTe_2 will be determined by using Scanning Electron Microscope (SEM).

1.5 Outline of Project

There are three chapters in this report which includes introduction, literature review, methodology with possible expected results.

Chapter one is the introduction of the project. In this chapter, research background, problem statement of research topic, objective of this research, scope and the outline of the project is stated. The aim of this chapter acts as introductory to research topic which is Cyclic Voltammetry Analysis of Transition Metal Dichalcogenide Tungsten Ditelluride Thin Films to help readers gain relevant initial understanding regarding the research.

Chapter two covers the literature review of the project. In this section, information about previous research published by other researchers on this topic or similar topic was discussed. All this information act as guidance for this research and give us an overview to how our research should be done. Based on the works of previous researcher, we can gain clear ideas for the research and improvements that should be made based on previous research.

Chapter three states the methodology of the research. In this chapter, the methods and technique used in this research is discussed. The synthesizing method and parameters and characterization techniques is outlined in this section to give readers understanding of how the research is carried out. The expected results will be summarized.

Chapter four shows the results and discussion of the research. In this chapter, the results obtained from cyclic voltammetry analysis, electrodeposition of thin films at different voltages and times and the characterization of thin films were shown and discussed. This will help to determine the important findings of this research.

Chapter five highlights the conclusion and recommendation of the research. The conclusion of the research is done based on the findings gained from this research. Some recommendation had been done to make improvement for further studies. Sustainable elements of this research were discussed to identify any sustainable elements present for future works.

CHAPTER 2

LITERATURE REVIEW

2.1 Thin Films

Thin film technology is both conventional and modern rising technology whereby it is fabricated by depositing atoms onto a substrate as a thin layer with the thickness of less than several micron-metre. Thin film can also be defined as a quasi-three-dimensional material created by condensing the atomic or molecular species of matter (Adachi and Wasa, 2012). Where numerous application relies on thin film characteristics, the basic property of thin films which covers the film thickness, structural properties and chemical composition are controlled by its deposition condition.

Thin films configuration can be categorised into three types which are film or layer, line or wire and island or dot as shown in Figure 2.1 (Freund and Suresh, 2003). The categories of constraint can be divided into unconfined, partially confined and fully confined. A small structure is said to be unconfined if the boundaries associated with its thin dimensions can be displaced freely. If the dimensions are constrained against deformation, it is said to be fully confined. When the boundary constraint is caused by another material that shares the same boundary, it is classified as partially confined.