SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF MOLYBDENUM CHALCOGENIDE THIN FILMS

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Synthesis and Structural Characterization of Molybdenum Chalcogenide Thin Films

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A thesis submitted
in fulfilment of the requirements for the degree of
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DECLARATION

I declare that this Choose an item. entitled " Synthesis And Structural Characterization On Molybdenum Chalcogenide Thin Films" is the result of my own research except as cited in the references. The Choose an item. has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature :

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Date : 19/1/2023

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APPROVAL

I hereby declare that I have checked this thesis, and, in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Manufacturing Engineering Technology (Product Design) with Honours.

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DEDICATION

To my lovely parent

And my fellow friends

Thanks for your long-standing support

You are always my spiritual support

Also

Thanks for everyone who helped me to complete this task

Keep fighting to next stage

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ABSTRACT

With the growing interest in science and technology, significant achievements have been made in various fields. This includes the development of renewable energy sources. This report, therefore, focuses on the characterization of transition metal chalcogenide films for photovoltaic applications. Due to their exceptional structural, optical, and electrical properties, layered transition chalcogenide materials such as Molybdenum Diselenide ($MoSe_2$) have attracted a lot of attention recently. Electrodeposition is used to build low-cost solar cells with direct band gaps and the ability to control the band gap through in-situ doping for the creation of possible heterojunctions. The objectives of this project are to determine the suitable potential to be used using cyclic voltammetry analysis, synthesize molybdenum chalcogenide thin films by using the electrodeposition method and the thickness was investigated using the weight gain method. Investigate the structural characterization of these thin films by using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) reveal information about their grain or molecular structures by analysing their surface morphology. The synthesized films confirm their suitability for being the alternate materials in Solar panel applications.

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ABSTRAK

Dengan minat yang semakin meningkat dalam sains dan teknologi, pencapaian ketara telah dicapai dalam pelbagai bidang. Ini termasuk pembangunan sumber tenaga boleh diperbaharui. Laporan ini, oleh itu, memberi tumpuan kepada pencirian filem kalkogenida logam peralihan untuk aplikasi fotovoltaik. Disebabkan oleh sifat struktur, optik dan elektriknya yang luar biasa, bahan chalcogenide peralihan berlapis seperti Molibdenum Diselenide ($MoSe_2$) telah menarik banyak perhatian baru-baru ini. Electrodeposition digunakan untuk membina sel suria kos rendah dengan jurang jalur langsung dan keupayaan untuk mengawal jurang jalur melalui doping in-situ untuk penciptaan kemungkinan heterojunctions. Objektif projek ini adalah untuk menentukan potensi yang sesuai untuk digunakan menggunakan analisis voltammetri kitaran, dan mensintesis filem nipis molibdenum chalcogenide dengan menggunakan kaedah elektrodeposisi dan ketebalan disiasat menggunakan kaedah penambahan berat. Siasat pencirian struktur filem nipis ini dengan menggunakan Pembelauan sinar-X (XRD) dan Mikroskopi Elektron Pengimbasan (SEM) untuk mendedahkan maklumat tentang butiran atau struktur molekulnya dengan menganalisis morfologi permukaannya. Filem yang disintesis mengesahkan kesesuaiannya untuk menjadi bahan ganti dalam aplikasi panel solar.

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

TABLE OF CONTENTS

PAGE

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

LIST OF TABLES

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

LIST OF FIGURES

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

LIST OF SYMBOLS AND ABBREVIATIONS

CVD	-	Chemical Vapour Deposition
PVD	-	Physical Vapour Deposition
CV	-	Cyclic Voltammogram
XRD	-	X-Ray Diffraction
SEM	-	Scanning Electron Microscope
EDX	-	Energy-Dispersive X-Ray Spectroscopy
TMC	-	Transition Metal Chalcogenide
TMDCs	-	Transition Metal Dichalcogenide
TMTCs	-	Transition Metal Trichalcogenide
PV	-	Photovoltaic
Mo	-	Molybdenum
S	-	Sulphur
Se	-	Selenium
Te	-	Tellurium
Si	-	Silicon
Ge	-	Germanium
P	-	Phosphorus
As	-	Arsenic
Sb	-	Antimony
SCE	-	Saturated Calomel Electrode
Ag/AgCl	-	Silver/ Silver Chloride
MoS_2	-	Molybdenum Disulphide
$MoSe_2$	-	Molybdenum Diselenide
$MoTe_2$	-	Molybdenum Ditellurium
DI	-	De-Ionized
ITO	-	Indium Tin Oxide
°C	-	Celsius
λ	-	Wavelength
TiO_2	-	Titanium Dioxide

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JCPDS	-	Joint Committee on Powder Diffraction Standards
SE	-	Secondary Electrons
BSE	-	Backscattered Electrons
WE	-	Working Electrode
RE	-	Reference Electrode
CE	-	Counter Electrode
CdTe	-	Cadmium Telluride
CIGS	-	Copper Indium Gallium Selenide
PCE	-	Power Conversion Efficiency
CL	-	Contaminant Level
nm	-	Nanometre
cm	-	Centimeter
µm	-	Micrometer
g/cm^3	-	Density

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LIST OF APPENDICES

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CHAPTER 1

INTRODUCTION

1.1 Background

Thin films are layers of material with two dimensional deposited on a large substrate to impart properties not easily achieved by the base material. Its thickness ranges from a fraction of a nanometre for single layer to a micron for multilayers. The act of depositing a thin film of any substance to a surface, such as a substrate or an already existing layer, is known as thin film deposition.

There are some methods to deposit the thin films which includes vacuum evaporation, sputtering, plasma processes and chemical vapour deposition (CVD). The growth mechanisms of thin films show a microstructure and morphological characteristics. Electrodeposition is one of the techniques to produce thin films. According to Sudipto Saha, (2020), eelectrodeposition is an optimum deposition method for forming thin films due to its low cost, ease of scale-up, high composition control, and compatibility with flexible substrates.

X-ray Diffraction is carried out to determine the characteristic and structural properties of thin films. The thickness of thin films can be measured by using Scanning Electron Microscope (SEM). The surface morphological study on thin films is obtained by SEM and Energy-dispersive X-ray spectroscopy (EDX).

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1.2 Problem Statement

It is entirely possible for humankind to move away from dependence on limited energy sources. Thin-film technology is often used in photovoltaic (PV) cells. Photovoltaic cells are used to convert light energy into electricity. Although solar energy is a renewable and green source of energy, the high demand for silicon for solar system applications indirectly leads to increased costs. Silicon currently accounts for more than 90% of the solar cell market.

In addition, photovoltaic cells made from silicon are not very efficient. The energy leaving the sun is different from the energy received on the earth. This is because the radiation must pass through the thick atmosphere that surrounds the earth. Now, different phenomena such as the scattering and refraction of light reduce its intensity. The ozone layer prevents harmful ultraviolet radiation from reaching the Earth. However, these are the waves that can cross the threshold energy, but those that reach the surface are rare, which in turn leads to a reduction in the efficiency of the solar panels, reducing what was 100% energy to less than 10-30% energy conversion. The conversion of solar energy needs to be more efficient to provide electricity on a large scale.

Also, the reflectivity of the silicon surface is quite high. Roughly 30% of the incident sunlight is reflected from the surface of the silicon solar cell. The remaining will be used as electrical energy to ensure internal operation. (J.D et al 2022).

Hence, seeking a new potential material is urgent to replace the inefficient and expensive silicon material. A Molybdenum (Mo) chalcogenide is used for the interest of the material. Mo chalcogenides need to be deposited as thin films and the characteristic with its structural analysis should be carried out.

1.3 Research Objective

i. To determine the suitable potential to be used using cyclic voltammetry analysis.

ii. To synthesize Molybdenum (Mo) chalcogenide thin films by using electrodeposition method.

iii. To investigate the structural characterization of Mo chalcogenide thin films by using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM)/ Energy Dispersive X-ray Spectroscopy (EDX).

1.4 Scope of Research

The task will focus on the stoichiometric, optical, and microstructural depiction of the thin films. An electrodeposition strategy is utilized for synthesizing the Molybdenum (Mo) chalcogenides thin films. The range of size for the thin films is obtained by Scanning Electron Microscopy (SEM) and X-ray Diffraction. In short, seeking the suitable potential material is the main purpose for this project by depositing molybdenum chalcogenides thin films.

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1.5 Project Outlines

There are 4 chapters involve in this project:

i. Introduction

Chapter 1 will briefly introduce the background and purposes for this project, as well as the scope of the research.

ii. Literature review

For Chapter 2, the literature review will be carried out for the overview based on the previous research and journals.

iii. Methodology

In Chapter 3 (methodology) will be explained the method used for the conducting the experiment including the deposition process, structural investigation via X-ray Diffraction (XRD) and morphological properties via Scanning Electron Microscope (SEM) and Electron Dispersive X-Ray Spectroscopy (EDX) for the thin films.

iv. Result and Discussion

Chapter 4 will process data in accordance with the principles of data processing and provide a sound and reliable analysis and understanding of the results.

v. Conclusion and Recommendation

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The conclusion will be made based on the result on Chapter 4. Some recommendation can be listed to improve and overcome the limitation of the research.

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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Based on the prior literature, this chapter briefly explained about the preparation of thin films, Cyclic voltammetry (CV), X-ray Diffraction (XRD) for investigating the structural of thin films, Scanning Electron Microscopy (SEM) with Electron Dispersive Spectroscopy (EDX) for morphological studies, as well as the chemical and physical properties for element used.

2.2 Transition elements

The elements that were half full at d-orbitals can be named as transition elements. It can develop stable cation that do not totally fill the d orbitals (International Union of Pure and Applied Chemistry- IUPAC). Commonly, the orbitals consist of four types including s, p, d, and f (Hayami, 2018). Transition metals have forty elements, which are able to discover in groups 3-12 while 28 elements are belonging to the lanthanide or actinide family too. The elements for this family were thought of as inner transition metals since they lack sufficiently filled electrons at the f-orbital.

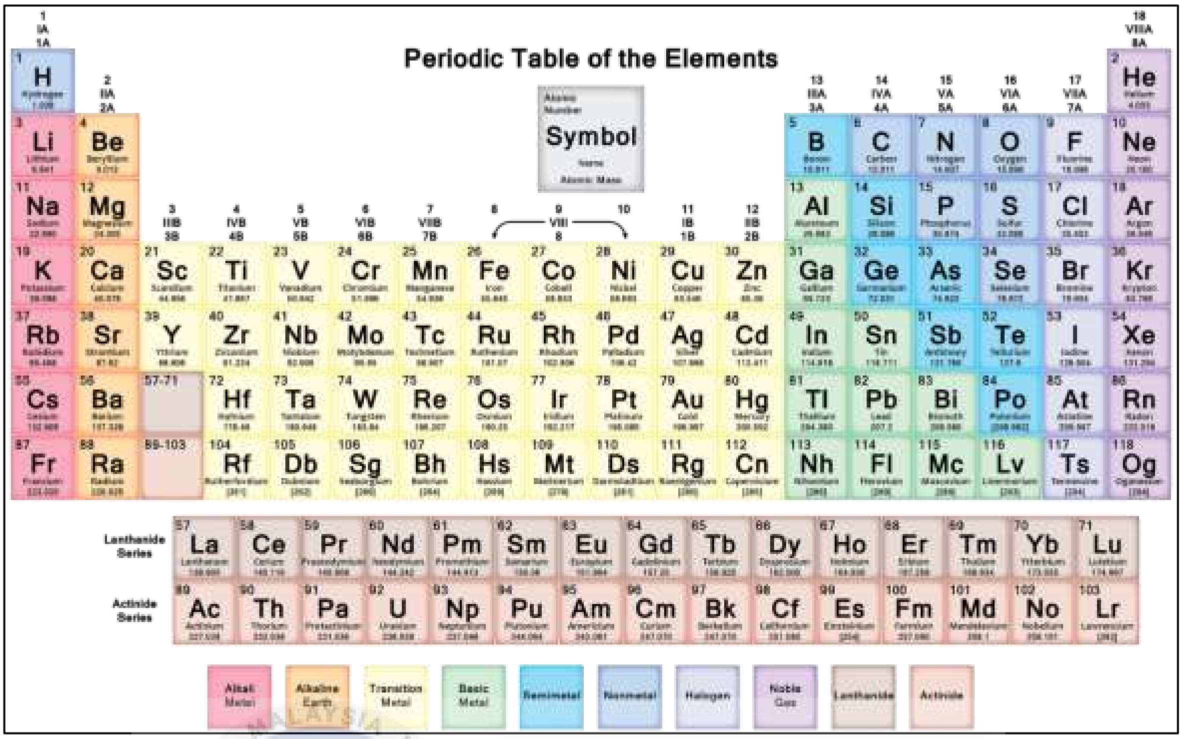

Figure 2.1 Periodic Table (Pngwing.com)

2.2.1 Molybdenum (Mo)

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Molybdenum (Mo) is a transition element as it is in Group VI with Period II, which fulfil the position requirement as the transition metal. 2, 8, 18, 13, 1 is the electron configuration for Mo element. Additionally, it contains forty-two electrons and has six valence electrons for its outer shell. Even though the Mo element is often known as a ‘heavy metal’, its characteristics differ significantly from those of thallium, and lead. Due to low toxicity, the Mo element is safer than other heavy metals and is treated as an appealing alternative to harmful compounds.

Since oxidation state with numbers of coordination for Mo element is prohibitive (from -2 to 6 and from 4 until 8 respectively). Mo element can be reacted with a variety of ligands, including organic as well as inorganic. Because of their versatility, their compounds have a broad range of practicable and prospective uses. Molybdenum is the very first