Synthesis and Characterization of Electrodeposited Bi_2Te_3-Graphene Nanocomposite Film

This report submitted in accordance with requirement of the University Technical Malaysia Melaka (UTeM) for bachelor's degree of Manufacturing Engineering

By

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I hereby, declared this report entitled "Synthesis and Characterization of Electrodeposited Bi2Te3-Graphene Nanocomposite Film" is the result of my own research except as cited in references.

Signature : Eswaran

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Date : 29 JUNE 2022

APPROVAL

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

………………………………………………………..

(DR. KHAIRUL FADZLI BIN SAMAT)

ABSTRACT

The project titled "Synthesis and Characterization of Electrodeposited Bi2Te3-Graphene Nanocomposite Film" has been carried out. The inclusion of graphene is needed to increase electrical conductivity and reduce the effect of Seebeck coefficient reduction. The Bi2Te3 film incorporated with high percentage of graphene has been successfully synthesized. The Graphene/Bi2Te3 nanocomposite films were synthesized by using electrodeposition process with pulsed potentiostatic deposition. Prior to electrodeposition process, good dispersion and suspension of graphene in the electrolyte solution has been successfully prepared. The electrolyte solution went through magnetic stirrer and sonification process approximately for one and half hour to obtain good dispersion and suspension of Graphene/Bi2Te3 electrolyte solution without aggregation form in electrolyte. The CV analysis shows, with graphene introduction in the electrolyte solution of 1wt%, 3wt%, and 5wt%, have a maximum peak reduction of potential value started hitting -50 mV and became maximum at -70 mV at the 5wt% graphene content. While, the pristine Bi2Te3 have peak reduction potential at -5mV. The characterization of the films was conducted by using scanning electron microscope (SEM) and energy dispersive X-ray (EDX). In SEM the microstructure size of inclusion of graphene gradually become smaller when compere to pristine Bi2Te3. Reduce size of microstructure cause increased the porosity. Used average values from an EDX measurement system, up to 2.7% graphene has been successfully deposited in the nanocomposite film.

ABSTRAK

Projek bertajuk "Sintesis and Characterization of Electrodeposited Bi2Te3-Grapene Nanocomposite Film" telah dijalankan. Kemasukan graphene diperlukan untuk meningkatkan kekonduksian elektrik dan mengurangkan kesan pengurangan pekali Seebeck. Filem Bi2Te3 yang digabungkan dengan peratusan graphene yang tinggi telah berjaya disintesis. Filem nanokomposit Graphene/Bi2Te3 telah disintesis dengan menggunakan proses elektrodeposisi dengan pemendapan potensiostatik berdenyut. Sebelum proses elektrodeposisi, penyebaran dan penggantungan graphene yang baik dalam larutan elektrolit telah berjaya disediakan. Larutan elektrolit telah melalui proses pengacau magnetik dan sonifikasi lebih kurang selama satu setengah jam untuk mendapatkan penyebaran dan penggantungan larutan elektrolit Graphene/Bi2Te3 yang baik tanpa bentuk pengagregatan dalam elektrolit. Analisis CV menunjukkan, dengan pengenalan graphene dalam larutan elektrolit 1wt%, 3wt%, dan 5wt%, mempunyai pengurangan puncak maksimum nilai potensi mula mencecah -50 mV dan menjadi maksimum pada -70 mV pada kandungan graphene 5wt%. Manakala, Bi2Te3 tulen mempunyai potensi pengurangan puncak pada -5mV. Pencirian filem telah dijalankan dengan menggunakan mikroskop elektron pengimbasan (SEM) dan sinar-X penyebaran tenaga (EDX). Dalam SEM saiz struktur mikro kemasukan graphene secara beransur-ansur menjadi lebih kecil apabila dibandingkan dengan Bi2Te3 murni. Mengurangkan saiz struktur mikro menyebabkan keliangan meningkat. Menggunakan nilai purata daripada sistem pengukuran EDX, sehingga 2.7% graphene telah berjaya didepositkan dalam filem nanokomposit.

DEDICATION

TO MY DEAREST PARENTS,

THIAGARAJAN A/L RAGAWAN AND PUVANESVARI A/P RAJAMANICKAM

TO MY HONOURED SUPERVISOR

DR. KHAIRUL FADZLI BIN SAMAT

For his advice, support, motivations, and guidance during accomplishment of this project

TO ALL STAFF & TECHNICIANS

For their direction and advice during completion of this project

TO ALL MY BELOVED FRIEND,

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LIST OF ABBREVIATIONS

Bi_2Te_3	-	Bismuth-Telluride
CV	-	Cyclic Voltammetry Analysis
CVD	-	Coating Vapor Deposition
DOS	-	Density of States Locally
eg	-	Example
etc	-	Et Cetera
HNO_3	-	Nitric Acid
IoT	-	Internet of Thing
NMP	-	N-Methyl-2-Pyrrolidone
NPs	-	Nanoparticles
PVD	-	Physical Vapor Deposition
SEM	-	Scanning Electron Microscope
TE	-	Thermoelectric
TEC	-	Thermoelectric Cooling
TEG	-	Thermoelectric Generator
WSNs	-	Wireless Sensor Networks
XRD	-	X-Ray Diffraction Analysis
Bi_2O_3	-	Bismuth Oxide
TeO_2	-	Telluride Oxide
Ag/AgCl	-	Silver/Silver Chloride
Pt	-	Platinum
Cr-Au	-	Chromium-Gold

LIST OF SYMBOLS

ZT	-	Figure of Merit
S	-	Seebeck Coefficient
σ	-	Electrical Conductivity
κ	-	Thermal Conductivity
T	-	Absolute Temperature
(V/K)	-	Volt Per Kelvin
wt%	-	Weight Percentage
V	-	Voltage
κB	-	Boltzmann Constant
ρ	-	Resistivity
μe	-	Electron Mobility
μh	-	Hole Mobility
n	-	Carrier Density of Electron
p	-	Carrier Density of Hole
μI	-	Mobility of Ionised Impurities
μL	-	Mobility of Lattice Vibration Effect
D	-	Thermal Diffusivity
Cp	-	Specific Heat
g/L	-	Gram Per Litter

CHAPTER 1

INTRODUCTION

1.0 Background of Study

Thermoelectric (TE) energy conversion method consume a huge deal in its term of calm, for the easiness and dependability as compared beside in a conventional power generator. The earlier twenty years witnessed to improved activities of academic and engineering relevance in TE materials. For the main reason to significant motivations for this boost concept of "nano", which would be trace back to pioneer works of Mildred S. Dresselhaus at 1990s. In pioneer passed away, the research around the nano TE materials is still going on. Thermal electric effect is the fundamental principle of the thermoelectric generator (TEG). At the TE effect, the active electrons are moving from one place to another place based on the temperature gradient. This temperature gradient was achieved, when there were a various in temperature levels between two points. Temperature gradient is known as a physical quantity that defines in which direction and change rate of temperature the most rapidly around a particular location. Nano-structuring is one of the effective methods to improve the TE in the materials and it also improve the efficiency of TE in the particular material.

The TEG is related to two mechanisms which are Peltier Effect and Seebeck Effect. The Seebeck effect and the Peltier effect both can be categorised as TE effect. Any TE effect includes converting temperature differences into voltage differences. The effects of Seebeck and Peltier are different forms of the same physical process. In certain cases, the Seebeck-Peltier effect is linked and known. The reasons for separating these two effects are due to two different individuals' independent discoveries. The Seebeck Effect was found by Thomas Johann Seebeck, the Baltic German physicist (Recatala-Gomez et al., 2020). Based on figure 1.1, seebeck effect is a condition where the change on temperature between two different electrical conductors creates a difference in voltage between these two objects. As heat is applied to all conductors or semitrails, the electrons are excited by the heat. Since there is just one side hot, the electrons are going to the cooler side of the two drivers. If all drivers are joined in a circuit, the circuit is flowing by a direct current. The Seebeck effect's voltages are very low. The voltages produced are typically of the order of a few microvolts (1 millionth of a volt) per Kelvin of temperature difference at the intersection. Some instruments will generate a few millivolts if there are sufficiently variations in temperature (which is one-thousandth of a volt). In order to optimise the total supply current, some of those devices can be connected in parallel. There also shown that a large variation in temperature across the junctions is held in place were it provide limited energy.

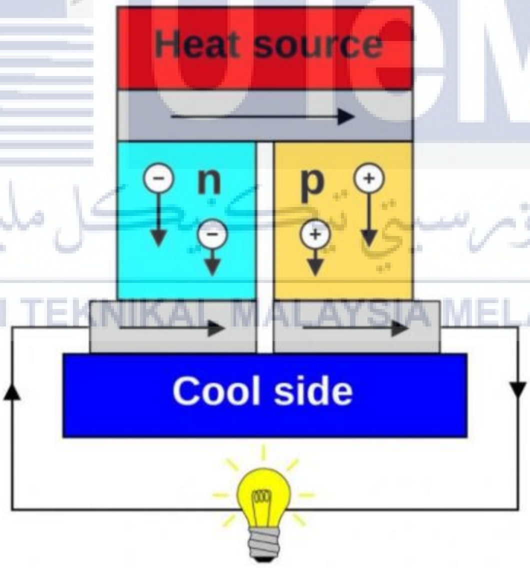

Figure 1.1 Demonstration of Seebeck effect (Lee et al.,2012)

Research by (Ju et al., 2016) state that performance of TE of a material calculated with merit, ZT. As in theory, to increase the ZT value, the TE materials should have huge electrical conductivity, a great Seebeck coefficient, and lower thermal conductivity. The lower thermal conductivity should be obtained through nano-structuring the TE material, so rising of lattice scattering of phonons. In early the nano-structures introduces at bulking TE materials creates many limits that performance more and more efficiently than the electrons in the scattering phonons. There are two principles or approaching can be used to fabricated nano-structure materials, the technique knowns as bottom-up and top-down. Bottom-up technique used molecules to generate blocks of building that are subsequently accumulate inti nano-scale cluster. While, top-down technique leads with solid and nanostructure were formed via the structure composition.

Evaluation of thick films synthesis and calculate the thickness TE films that could be used for the such an application as TEG. There are two electrochemical deposition technique were found, constant and pulsed deposition that advanced methods for both N-type bismuth telluride (Bi2Te3) and P-type antimony telluride (Sb2Te3), were implemented. According to the result, very oriented Bi2Te3 and Sb2Te3 thick film with a bulk such as structure were effectively synthesized with high Seebeck coefficients and lower resistivities (Trung et al., 2017).

In this study synthesis the bismuth telluride film with graphene inclusion through the electrochemical deposition process will conduct. The nanocomposites film of Bismuth Telluride-Graphene will be synthesised by using electrodeposition process. Prior the synthesis process, the cyclic voltammetry analysis will be carried out to study the reduction and oxidation reactions especially on the optimum reduction potential. The optimum reduction potential will be used to synthesis the nanocomposite film based on the variable of time deposition.

1.1 Problem Statement

TE materials, which produce electric current from excess heat or act as solid-state Peltier coolers, might show a key part in a sustainable energy solution through the global. If the progress would necessitate the identification of materials with huge amount of TE effectiveness than those currently accessible, which is difficult because to the competing requirements for material properties. Despite this, a new period of complicated TE materials is emerging, thanks to improved characterization methods, especially the nanoscale materials. (Liu et al., 2017)

As the need for Internet of Things (IoT) with integrated wireless sensor networks (WSNs) develops, power supply and management have become key challenges to address. Thermal energy, such as excess heat or metabolic heat, is a prospective source of power for electronic equipment; for example, thermoelectric power generators, which convert thermal energy into electricity, are a hot topic of research (Ouyang., et al 2019). However, due to the obvious expensive cost of materials and fabrication methods, as well as the low overall performance, it may be limited. Many materials can be prohibitively expensive in many thermoelectrical applications, owing to the widespread use of Tellurium and Germanium in most thermoelectrical applications.

In addition, small thermoelectric power generators can be employed in portable and or self-powered energy sources, such as wearable electronics and IoT systems. The dimensionless merit figure ZT value which manipulated by it dependent thermoelectric properties. The performance of a TE material, where S is the Seebeck coefficient, T is the absolute temperature, conductivity of electrical, and is the thermal conductivity. Because semiconductors have a larger thermoelectric effect than metallic materials, the first feasible thermoelectric material, Bi2Te3, was discovered (Evans, 2018).

There were several purposes to do research on this project such as currently the usage internet of things (IOT) is increasing all over the world which are directly related to the usage of thermoelectric, such as portable or self-powered energy sources. Now days, everyone over the world are access the IoT devices which the components made up with the thermoelectric properties material to convert heat energy into electrical energy. Some more, use of Bi2Te3 film in micro energy harvesters and sensors which detect and measure such as heat, noise, vibration and others.

Then one of the main challenges in this project is Bi2Te3 film has low ZT when compare to the bulk Bi2Te3 and it is hard to enhance the ZT value of Bi2Te3 film (figure of merit) which includes all the three thermoelectric properties (seebeck coefficient, electrical conductivity, thermal conductivity) seem mutually dependent. The low ZT value will reduce the thermoelectric generator efficiency. Which is the ratio between the electrical power produced and the heat flow.

There is also less studies are conducted using the synthesis of pure Bi2Te3 film through the electrodeposition process. Many researchers were studying used doped Bi2Te3 film and bulk Bi2Te3 to enhance the thermoelectric properties. Rather than, doping process there is another way to improve the ZT value of pristine bismuth telluride is to adding nanocomposite elements into it. This nanocomposite material manipulated the pristine bismuth telluride structure which cause huge change in it electrical properties.

1.2 Objective

The objectives of the project are:

1. To prepare the electrolyte solution of bismuth telluride for the electrodeposition process with good dispersion and suspension of graphene.
2. To synthesis of pristine bismuth telluride (Bi_2Te_3) film and bismuth telluride (Bi_2Te_3) nanocomposite film with inclusion of graphene up to 3 wt% electrochemical deposition process.
3. To analyze surface morphology and chemical composition of the pristine bismuth telluride film and the nanocomposite film with inclusion of graphene.

1.3 Scope

This project will be covering:

1. The electrochemical deposition process will be utilized to synthesis the pristine bismuth telluride and bismuth telluride nanocomposite films with inclusion of graphene by the three-electrode cell.
2. The stable suspension of graphene in bismuth telluride solution will be prepared with ultrasonic bath and magnetic stirrer intermittently prior to the deposition.

3. The synthesis film undergoes surface morphology study and chemical composition analysis through scanning electron microscope (SEM) and energy dispersive x-ray (EDX).

1.4 Important of Study

The important of this study to produce nanostructure in bismuth telluride with added of graphene by prepared electrolyte based on graphene/Bi2Te3 and conducting electrochemical process deposition. Then, the restructure nanocomposite will undergo cyclic voltammetry analysis, in this analysis able to study reduction and oxidation reactions on the optimum reduction potential. By scanning the voltage supplied to the working electrode in the direction of a triangle waveform and detecting the current flow that results. Then, also need to study the nanocomposite material characterization through the scanning electron microscope (SEM) and energy dispersive X-ray (EDX).

1.5 Organization of Report

Chapter 1

This chapter explains the study's background information, the location where the study is being done, and the types of material analysis that will be required. Then there are the objectives that must be met during the investigation as well as the project scope, which is centred on the subject matter of the study. This chapter also includes an examination of the findings of the study as a result of the analysis.

Chapter 2

This section of the chapter covers the fundamental theories as well as the knowledge of the theories portion of the study. It is based on legal sources as well as earlier research from articles, journals, and books that have been published on the internet. The current equipment is described in detail. Part of this section also includes the tools that are utilised in the manufacturing industry. Finally, the alternative that is being proposed is described.

Chapter 3

In this chapter, it focuses on the recipe that was employed in this study. It demonstrates the step-by-step procedure used in this study to acquire data from the area under investigation. This section will cover the differences between primary and secondary sources. The process flow chart will be described in detail, as well as the project framework for each of the objectives that have been identified.

Chapter 4

This chapter presents the results obtained in the study in the forms text, tables, and figures to highlight the key information. Discussion were made on each of the presented data for every objective of this study. The data were arranged in section to ease the readers to understand the finding as well as the discussions.

Chapter 5

All of the research findings are discussed in this chapter to achieve the main objective of this study. This chapter covers the result of data analysis and recommendations suggested by researcher. In the final of this chapter, the research findings of this study will conclude.

1.6 Summary

To summarise this chapter, it provides the first layer of understanding of the whole subject matter of this research. This chapter provides a high-level overview of the study and helps the reader understand the point. The objectives that must be attained and the project scopes that must be followed in order for the study to be successful are the most significant aspects of this study.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The literature review of this chapter is carried out on the basis of trustworthy references such as journals, newspapers and conferences prior to relativity of the project. Input on works linked to this, Graphene/Bismuth-Telluride Nanocomposite (Graphene/Bi_2Te_3) characterization and properties, fabrication, preparation, characterization and evaluation of Bi_2Te_3 film are studied with each other's contrast. The assessment of each part, procedure and methodology for comparison is seen below. This segment consists of a study of a variety of analysis or mission comparisons undertaken by other researchers.

2.2 Discovery of Thermoelectric

The thermoelectric effect occurs when temperature differences are directly changed to electric voltage and contrariwise. This is accomplished by using a thermocouple. If there got any difference in temperature among the both side of a TE device, a generation of voltage occurs. If a voltage is used to it, on the other hand, heat is transferred from one side to another, resulting in a temperature differential between the two (Gorai., et al 2017). If the gradient of temperature is provided to a material at the molecules level, the charge will carry and it will diffuse into the hot side to cold side. A variety of applications for this phenomenon include the generation of energy, temperature measurement, and the modification of object temperatures. Because the delivered voltage affects the path of heating and cooling, TE devices may be used as temperature controllers in a variety of applications (Iwasaki et al., 2019).

TE effect is states to three different phenomena discovered separately: the Seebeck effect, the Peltier effect, and the Thomson effect.

2.2.1 Seebeck Effect

According to Thomas Johann Seebeck's discovery in 1821, a circuit consisting of two dissimilar metals may conduct electricity if the two points at which the metals link are kept at different temperatures from one another. A compass was put near the circuit Seebeck had constructed, and the needle deflected due to the proximity. He noticed that the deflection amplitude grew according to the difference in temperature between the two temperatures (Zhou et al., 2021). Furthermore, according to his studies based on figure 2.1, the temperature transfer among the metal rods wouldn't not disturb the compass. When manipulated the sorts of metals used, on the other hand, did affect the amount of deflection of the needle. In electrical engineering, the Coefficient of Seebeck is a numerical representation of voltage generated among two places in a wire when a constant variation in the temperature (one kelvin) between the sites. Metals were used in Seebeck's experimentations reacted to the heats, resulting in a existing loop into the circuit and also affect the magnetic field around the course. Seebeck made the mistake of supposing this was a thermomagnetic effect since he was unaware that there was an electric current at the time (Zimbovskaya, 2016).