

Synthesis and Characterization of Electrodeposited Bi₂Te₃-Graphene Nanocomposite Film



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DECLARATION

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.



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



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



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

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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

Bi2Te3	-	Bismuth-Telluride
CV	-	Cyclic Voltammetry Analysis
CVD	-	Coating Vapor Deposition
DOS	-	Density of States Locally
eg	-	Example
etc	-	Et Cetera
HNO3	-	Nitric Acid
IoT	-	Internet of Thing
NMP	WALAYSIA MA	N-Methyl-2-Pyrrolidone
NPs	- X	Nanoparticles
PVD	- <u></u>	Physical Vapor Deposition
SEM	E	Scanning Electron Microscope
TE	A BAING	Thermoelectric
TEC	chi T I	Thermoelectric Cooling
TEG	مايسيا ملاك	Thermoelectric Generator
WSNs		Wireless Sensor Networks
XRD	UNIVERSITIE	X-Ray Diffraction Analysis
Bi2O3	-	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
Т	-	Absolute Temperature
(V/K)	_	Volt Per Kelvin
wt%	-	Weight Percentage
V	ANLAYS/A	Voltage
кВ	St - ME	Boltzmann Constant
D	- T	Resistivity
' μe	۳	Electron Mobility
uh	Fax -	Hole Mobility
n	"PAININ	Carrier Density of Electron
n	1 alunda Va	Carrier Density of Hole
r ul		Mobility of Ionised Impurities
<i>ш</i> .	UNIVERSITI TEI	Mobility of Lattice Vibration Effect
D	_	Thermal Diffusivity
Cn		Specific Heat
⊂p σ/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.

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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.
- To synthesis of pristine bismuth telluride (Bi₂Te₃) film and bismuth telluride (Bi₂Te₃) 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.

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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

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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.

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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

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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/Bi2Te3) characterization and properties, fabrication, preparation, characterization and evaluation of Bi2Te3 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).



The Peltier Effect, a second substantially similar phenomenon discovered by French scientist Jean Charles Athanase Peltier in 1834, is today known as the Peltier Effect. Figure 2.2 shows demonstration of Peltier effect, where Peltier conducted an experiment where he varied the voltage between two metal conductors and observed that the temperature at each junction changed proportionately to the voltage variation. The German scientist Heinrich Lenz further developed the finding of Peltier in 1839, who characterized heat transfer at intersections as a function of the direction in which the current runs through the circuit. Although these both studies were concentrated on distinct portions of the circuit and different TE effects, are sometimes stated to as the Seebeck-Peltier Effect in a single sentence (Richmond 1966).



Figure 2.2: Demonstration of Peltier effect (Zhou et al., 2021)

2.3 Application of Thermoelectric

The use of thermoelectric nanogenerators (TEGs) to capture thermal energy from the surrounding environment is a promising method of realizing powered by itself and functioning of electronics and alleviating global energy crisis and also cause very huge damage to the environmental. With excellent thermoelectric performance, TEGs have emerged as very important for the energy associated technologies. TEGs also played progressively significant part in the gathering and converting heat into electric energy, and their study is becoming one of the active research fields (GOLDSMID 1955). It is discussed and described here how TEGs have progressed through the stages of materials optimization, structural designs, future applications, and the possibilities, problems, and future development direction. The optimization of materials and the development of flexible structural techniques for possible applications in wearable electronics are both addressed in detail in this paper (Yang et al., 2020). The flexible advancement and wearable electronic equipment were resulted in flexible TEGs having more promising future applications include artificial intelligence, self-powered sensing systems, and other fields.

2.3.1 Thermoelectric Generators (TEG)

Seebeck generators are thermoelectric generators that directly transform heat energy (temperature difference) into electrical power without needing a revolving element or mechanism. Thermoelectric generators are also known as Seebeck generators. Described the use of TEG in power plants for in process recovering waste heat and vehicles to boost fuel economy, among other benefits. (Zheng et al., 2014) have documented several TEG applications in the automotive, aerospace, most advance electrical and electronic industrial, home, and thin film industries. This TEG creates electrical energy from waste thermal energy derived from the automobile, aerospace, and industrial industries, among other sources.

2.3.2 Thermoelectric Cooling (TEC)

Electronics equipment come with high-power, such as microprocessors, power amplifiers, and computers, which are utilized in servers, are continually operational and give service to their clients. A significant quantity of heat is generated inside the system during the run, and this heat must be dissipated since this system prevent hardware malfunction. Consequently, cooling is more essential to improve the performance and longevity of electronic equipment. Conventional cooling systems have difficulty keeping up with the demands of modern technological gadgets (Cai et al., 2017). Because there are not small and need a large amount of area for installation. TE coolers provide several benefits over conventional cooling systems, such as being smaller in size, being vibration-free according to the absence of parts motions, requiring less maintenance, and operational on a DC power supply.

2.4 **Performance of Thermoelectric**

2.4.1 Figure of Merit

Thermoelectric efficiency is broken down into thermoelectric material efficiency and thermoelectric device efficiency (TEG). In addition to structural design, thermoelectric material is an essential aspect in creating high-performance thermoelectric devices. The figure of merit evaluates the total performance of a thermoelectric (TE) material, where includes the Seebeck coefficient (S), electrical conductivity (σ), thermal conductivity (κ), and absolute temperature (T). The seebeck square, average absolute temperature, and electrical conductivity rise. Thermal conductivity diminishes as the temperature rises. Both factors influence the temperature. Calculating the ZT value becomes more complicated as a result:



2.4.2 Seeback Coefficient

It is possible to measure the Seebeck coefficient for the material (referred to as thermoelectric power, thermoelectric sensitivity, or thermoelectric sensitivity) by measuring the direction of an induced voltage of TE in response for the temperature variation among the material, as generated by the Seebeck effect, in that material. The Seebeck coefficient measured in volts per kelvin (V/K), the SI unit of measurement.

When it comes to the efficient behavior of thermoelectric generators, the materials selection come with huge Seebeck coefficient is only an example of crucial aspects to consider. The Seebeck effect is utilized to detect temperatures in thermocouples, it also preferable for utilize the materials by using Seebeck coefficient that remains constant over time to achieve high accuracy. If think about the physical, the size of Seebeck coefficient may be approximated as transpiring determined by entropy amount and charge per unit carried by electrical currents in to a material's electrical conductivity. For negatively charged carriers in conductors that may be viewed as independently moving as nearly-free charge carriers in the Seebeck coefficient when it in state negative (Snyder, 2008).

One way of defining the coefficient of Seebeck is to build up voltage while a slight temperature differential utilised to a material and when the material is in stable condition, with the current density being zero everywhere. If the temperature difference temperature between the two ends of a material is small, and V is the thermoelectric voltage visible at the terminals, the Seebeck coefficient of the material is defined as follows (Erickson et al., 2015):

$$S = \Delta V / \Delta T \tag{2.2}$$

Influence in charge carrier concentration, n in substance where the interaction is comparatively proportional $(S \propto 1n/)$, is one of the factors influencing the Seebeck Coefficient. Previous experiments have shown that the charge is related to Seebeck Coefficient experimentally and in theory. The effect of carrier concentration, n can ideally be clarified by using the theory of nearly free electron and simplified.

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$$S = \frac{8 \pi^2 k_B^2}{3eh^2} m^* T \left(\frac{\pi}{3n}\right)^{\frac{2}{3}}$$
(2.3)

Where (T) is the temperature, (κB) is Boltzmann constant, (n) which is concentration of carrier and (m*) the effective carrier mass. In 2008, two mechanisms have been discussed that can increase the Seebeck coefficient based on Mott expression (Cutler et al., 1969).

$$S = \frac{\pi^{2}.KB}{3q} KB.T. \left(\frac{1}{n} \cdot \frac{dn(E)}{dE} + \frac{1}{\mu} \cdot \frac{dn(E)}{dE}\right) E = EF$$
(2.4)

Where n(E) is the energy carrier density, (E) is the energy mobility, EF is Fermi energy, and q is the electronic charge. The first mechanism to disperse load carrier that relies heavily on the μ decrease. Second, to lower the carrier density by increasing the density of states locally (DOS). Both equations conclude that the value of the Seebeck coefficient would increase if the charge carrier density is reduced by n.

2.4.3 Electrical Conductivity

As the temperature of a metallic conductor is decreased, the electrical conductivity steadily rises. Resistance in superconductors reduces to zero below a threshold temperature, allowing an electrical current to flow around a loop of superconducting wire with no input power. Conduction occurs in various materials due to band electrons or holes. In electrolytes, whole ions flow, carrying their net electrical charge. Ionic concentration species on the electrolyte suspensions is a significant element in the material's conductivity.

The value of electrical conductivity, σ can be accomplished by resistivity, ρ material value. Since the resistivity equation is inverse to the electrical conductivity, the basic equation can be expressed in the following way;

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$$\sigma \neq 11/\rho L$$
 MALAYSIA MELAKA (2.5)

In general, the material's electrical resistivity characterises the potential of the material to disrupt the flow of power. Therefore, the statistics representing the electron-hole movement in a substance are closely connected. Those two measurement factors in electron and hole mobility in a material affected the importance of electrical conductivity, as seen below (Gayner, 2016):

$$\sigma = e \left(\mu e.n + \mu n.P\right) \tag{2.6}$$

where μe , n, μh , and p symbolize electron mobility, carrier density of electron, hole mobility and carrier density of hole respectively.

The correlation between the vibration of the lattice and several electron mobility can be defined as follows, where μI and μL are the mobility numbers induced by the vibrational effect of the ionize and the lattice vibration effect.

$$\frac{1}{\mu e} = \frac{1}{\mu I} + \frac{1}{\mu L} \tag{2.7}$$

By inserting a temperature change factor in a substance, it is simpler to understand the effect of the lattice vibration by above equation. As higher temperatures are applied the lattice vibration scattering in a material is increased. The scattering effect will interrupt the electron flow and lead to low electron mobility for the lattice vibration effect (μL). In comparison, ionised impurity due to high temperatures would be less dispersed into the material. Low degree of dispersion of ionised impurity contributes to a high electron mobility of ionised impurities (μI)

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2.4.4 Thermal Conductivity

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Thermal conductivity is a measurement of a substance's ability to transmit heat. Temperature is a common way to describe it as kappa.

High-conductivity materials transfer heat more quickly, whereas low-conductivity materials take longer to do so. Insulating materials like Styrofoam, on the other hand, have a low thermal conductivity and are ineffective in transferring heat. Heat sinks often utilise materials with high thermal conductivity, while thermal insulation typically uses materials with low thermal conductivity. Thermal conductivity's inverse is thermal resistance.

The conductivity of thermal relies upon the carriers of charge and the movement of the phonons for a semiconductor substance. The diffusion of the phonons contributes to the strengthened resonance. Therefore, the lattice of vibration greatly exaggerates the conductivity of thermal and is known as lattice thermal conductivity (κ lattice) (CHASMAR et al., 1959). The thermal conductivity of the material varies accordingly with the major effects of charge carriers and can be calculated by applying the law of WiedemannFranz (Krumhansl, 1959). By considering these two variables, charge carriers and lattice as the next equation, the overall thermal conductivity can be expressed where κe is charge-carriers thermal conductivity:

$$K = Ke + Klattice \tag{2.8}$$

In turn, the following relation D, Cp and ρ signify as thermal diffusivity, specific heat, and material density respectively can be referred to for the equation of lattice thermal conductivity.

$$Klattice = DCpP$$
(2.9)

In general, the overall thermal conductivity of metal is enhanced when electrical conductivity is high, since the electrical conductivity is directly connected to the conductivity of thermal to the carrier-charge. Best way to minimise thermal conductivity is by analysing the importance of the thermal conductivity grid. Lower thermal conductivity grid implies lower overall thermal conductivity value. Introduction of nanoparticles to the metal could reduce the conductivity of thermal to the lattice by blocking a lattice-vibrating excitation stream, also called phonons. The disrupted phonon flow increases the scattering of the phonon and the wavelength of the phonon. The time required to pass the heat is thus increased.

2.5 Types of Film Fabrication Technology

Process of the thin film deposition is depositing a very nano size film to material ranging thickness from the nanometers to around 100 micrometers, or few little atoms, a "substrate" which are coated on surface or a before formed coating to create layers. Manufacturing technologies for the thin film deposition at core of current semiconductor manufacturing. Thin Film Deposition a typically classified as two groups. Chemical Vapor Deposition (CVD) Coating and Physical Vapor Deposition Coating (PVD) are also used (Zamfirescu et al., 2007). A chemically deposited coating is formed when a volatile fluid precursor produces a chemical change on a surface. For example, CVD is utilized to create today's high-purity, very high-performance solid materials at semiconductor manufacturing. PVD known as various methods that use mechanical, electromechanical, or thermodynamic processes to release material from a source and deposit it on a substrate. Thermal evaporation and sputtering are two most prevalent PVD method.

2.5.1 Chemical Vapor Deposition (CVD)

CVD is a method that is utilized in various manufacturing process for hightemperature shield, protection of erosion, and a combination of two. A chemical reaction involving a phase of gas or phase of vapour precursor is deposited on the substrate's surface. This technique necessitates the use energy activation. A variety of gases are entered into the vacuum chamber through the intake, and the newly created chemical molecules are deposited on the heated substrate after the species have been dissociated (Madhuri, 2020).



Figure 2.3: Illustration of Chemical Vapor Deposition (CVD) (Madhuri, 2020)

Figure 2.3 illustrated CVD process which are categories as plasma-enhanced CVD, Atmospheric pressure CVD, low-pressure CVD, laser CVD, photochemical vapor deposition, chemical vapor infiltration, and chemical beam epitaxy. The parameters of operating, modes, and conditions be able to choose depending material and application. Researchers developed technology to create a barrier of thermal coating with great operating parameters on the CVD development. The mechanical, thermal, and phase stability of a material, like melting point and conductivity of conductivity, are all significant when depositing it as thermal coatings. Lightweight materials with high specific strength are coated with high-temperature protective coatings.

2.5.2 Thermal Evaporation

Thermal evaporation known as process of coating the thin layer. The base material evaporates on vacuum because of high-temperature heating, accepting the vapor particles to let go more quickly to a substrate, when it becomes solid again. Resistive coil into shape of a sloid bar or powder is utilized in the procedure. The resistive coil is exposing to a substantial DC to achieve a high-melting temperature required for metals. The high vacuum (below 104 Pa) aids in the metal evaporation and subsequent transport for the substrate. The

method is very beneficial for materials with low melting points. Figure 2.4 shows a schematic of the thermal evaporation system.



Thermal evaporation methods may be used to apply pure atomic elements, including metals and nonmetals, as well as compounds like oxides and nitrides. The substrate is the item that will be coated, and it may be anything from semiconductor wafers to solar cells to ocular parts, among numerous another options. (Bashir et al., 2020) Evaporation of thermal is a process to heat-up a solid substance to temperature which creates pressure in a vapor form within a high vacuum chamber. When vapor pressure within vacuum is sufficient to producing vapor clouding in the inner part of chamber. Films are created when the material is heated and evaporated in the chamber, which moves through the chamber and onto the substrate.

2.5.3 Electron Beam Evaporation

As illustrated in figure 2.5 electron beam evaporation is a way that employs highspeed electrons for blast the focus basis. The electron beam's kinetic energy is generated by the electron cannon, which uses magnetic fields and electric for shooting it target and evaporate the surround vacuum. Atoms from surface will have sufficient energy to depart from the substrate after being heated by the radiation heating device. Meanwhile, the substrate will be coated when the thermal energy is much less than 1 eV and the working displacement somewhere between 0.3 and 1 m (Rased et al., 2021).



Figure 2.5: Illustration of Electron Beam Evaporation (Rased et al., 2021)

Due to limited evaporation, the material escapes from target location and migrates to deposition site. Whole over process is carried out from vacuum, and high-pressure nucleation allows elements to be deposited as thin films. This approach has a much greater deposition rate than sputtering. The e-beam is focused using condensers, resulting in localized heat more significant at the boiling point from its target materials. EBE may be used to make nanoparticle assemblies in both 2D and 3D.

2.5.4 Sputtering & Magnetron Sputtering

In sputtering process, a target to the cathode plate is blasted by intense ions created in the glow plasma discharge located at front of target. Bombardment is a technique allows target atoms to be removed or sputtered and condensed on a substrate as a thin layer. The consequence of bombardment ions, secondary electron discharge at the targeted surface, and the electron play crucial role at the plasma maintenance. Most of the materials were successfully deposit by using the fundamental method of sputtering, known for many years (Kelly et al., 2000). However, poor deposition speeds, low plasma ionization efficiency, and strong substrate heating effects restrict the technique. Since magnetron sputtering and unbalanced magnetron sputtering were developed, these limitations have been eliminated.



Figure 2.6: Illustration of the Sputtering & Magnetron Sputtering (Kelly et al., 2000)

Magnetrons take advantage of the fact that a magnetic field aligned with the target surface may constrain secondary electron mobility to the target's vicinity. The magnets are structured so that one pole is positioned at the target's central axis, while the second pole is formed by a ring of magnets around the mark's outer edge. The risk of an ionizing electron– atom collision increases considerably when electrons are confined in this form. A magnetron's improved ionization efficiency produces a concentrated plasma in the target area. The target is bombarded with more ions, resulting in greater sputtering rates and, as a result, greater rate of deposition rate on the substrate. Furthermore, the magnetron mode's
higher ionization efficiency allows the discharge for maintained at lesser operating pressures (Kakaei et al., 2019).

2.5.5 Electrodeposition

An electrochemical process, such as electro-polymerization of a polymer or graphene composite on a working electrode, is used in electrodeposition. A conductive substance is used as the working electrode. When a certain potential is attained, the deposition continues until the stated charge is deposited. Although there are many methods for making graphene-based nanocomposites with polymer matrices, it is essential to remember that each approach produces a distinct nanocomposite. The generated graphene nanocomposites have varying properties depending on the process used. As a result, these nanocomposites will have different characteristics from those created using conventional methods (Singh et al., 2019).

Electroplating is a flexible method that expands the breadth of coatings and thin films to millimeter heights, ranging from less than one micron to dense mechanical components. Goods are placed near environmental conditions without the need for expensive vacuum equipment. The deposition rate is substantially faster than that of vapors, and the thickness of the coatings may be up to one millimeter. An anode, an electrode, or an electrolyte with a reducible metal ion, and the cathode, or the workpiece to be put, make up the process (Losey et al., 2017). Electrodeposition is a technique of coating by electrolysis that works in the opposite direction of a galvanic cell. Reaction deposition, co-deposition, and two-step deposition are the most often used electrodepositions. It's ideal for making TE films because of its speed, low cost, and room-temperature method. Even though the electrodeposition technique is simple, the influencing elements are. Current, voltage, temperature, solvents, pH, and concentration solution are all factors that affect film efficiency, as are ionic strength, surface condition, and other variables. It's tough to keep track of the composition and thickness of films, especially when making the best and intricate composition films.

As illustrated in figure 2.7, in a typical three-electrode cell, three distinct electrodes (working, Counter, and reference) are put at the same electrolyte solution. During three-electrode tests, charge flow (current) predominantly occurs between the working electrode.

In this setup, the Reference lead is detached at Counter and linked from third electrode. This kind of electrode is typically positioned such that is monitoring a place exceptionally near at working electrode. Three-electrode operations provide a clear experimental benefit over the two-electrode operation; the quantity just the half cell. No matter what happens at the counter electrode, the working electrode's potential changes are tracked and recorded.



2.6 Device of Characterization

2.6.1 Cyclic Voltammetry Analysis

The electroanalytical technique of cyclic voltammetry (CV) has gained prominence and wide use in several fields of chemistry. A wide range of redox processes can be studied using this technique, including the stability of reaction products, the presence of intermediates in redox reactions, electron transfer kinetics, and the reversibility of reactions. An analyte's diffusion coefficient, formal reduction potential, and electron stoichiometry can all be established using the CV, which can be used to identify the analyte. Because an unknown solution's attention is proportional to its concentration in a reversible Nernstian system, a calibration curve plotting concentration vs current may be used to assess it.

Potentiodynamic electrochemical measurements, such as CV, are included in this category. The working electrode potential is scaled linearly with time in a cyclic voltammetry experiment. In contrast to linear sweep voltammetry, CV experiments ramp the potential of the working electrode in the opposite direction to return to the starting potential. You can repeat these potential-ramping cycles as many times as you like. A graph of the current flowing through the working electrode versus the applied voltage is used to generate the cyclic voltammogram trace. Cyclic voltammetry is used to study the electrochemical properties of a solution or a molecule that has been attached to the electrode.

2.6.2 Energy Dispersive X-Ray (EDX) analysis

X-ray diffraction (XRD) has been extensively handled throughtout the project to study crystallinity of the nanostructures. In short, the XRD theory derives from the bombardment of an X-ray radiation sample that is elastically distributed in the sample by the atoms. The crystallinity of the material can be analysed from this scattering (low crystallinity contributes to the loss peaks reflections because of irregular direction of these X-rays). Regarding crystal structures, the scattered x-rays are interacting with 2 types: there will be disruptive in certain directions, however the regularity of crystals within exact circumstances, would be constructive interfering relatively implies optimum strength presence for those situations. That's related to the rule of Bragg's law (Joseph et al., 2018):

$$2dsin(\theta)n\lambda \tag{2.10}$$

When d is the spacing between the atomic layers of a crystal and the wavelength of the X-ray. The information from an XRD study helps to explain the phases (space group) of the sample. The Bragg Brentano configuration was used for that reason. Moreover, it could derive crystallite size from the shape/size of the peaks by using the Debye-Scherrer formula (Cutler et al., 1964):

$$\tau = \frac{\kappa\lambda}{\beta\cos\theta} \tag{2.11}$$

Mean size of the crystallite (K), is a definite form factor which has a value near to unity, is the wavelength of the X-ray, line expanding at half utmost intensity and is the Bragg angle. Finally, the volume of the unit cell could be extracted using fullprof software, namely Le bail suit. The measurement system is totally non-destructive and owns great penetration depths, which offer clear insight of the characteristics of the sample rather than just the surface which may vary considerably from the remaining material being studied (Rodríguez-Carvajal, 1993)

2.7 Bismuth Telluride-based Nanocomposite Film

There are various of strategies that can be defined for enhancing the thermoelectric performance of material. The strategies include doping, nanostructuring, superlattices and nanocomposites or nanoinclusion. This section focusses on discussing the bismuth telluride (Bi₂Te₃)-based nanocomposite in film condition. The pristine Bi₂Te₃ film will be briefly discussed first to present certain thermoelectric properties in purpose of comparison with the nanocomposites. Based on previous findings, the *ZT* values of pristine Bi₂T₃ films were not as high as the bulk. According to (Lee et al., 2016) has reported that their pristine Bi₂T₃ film can gain *ZT* value around 0.56 near room temperature. The film was synthesized using chemically bottom-up method and then sintered with spark plasma sintering process. The *ZT* value can be considered as the best overall thermoelectric performance among the Bi₂T₃ films. However, the value still lower as compared to the bulk Bi₂Te₃.

There are a lot of studies have been published on the analysis of electrodeposited Bi2Te3. However, the studies were dominant on the investigation of the synthesis process which included the details of electrochemical reactions without considering the effect on thermoelectric properties. A few studies on the thermoelectric performance (up to ZT calculation) of the electrodeposited Bi2Te3 film have been reported in the last ten years.

(Manzano et al., 2016) published the results in which they reported that the maximum ZT value of electrodeposited Bi₂Te₃ film was 0.1 in the out-of-plain direction (also known as cross-plane), and the in-plane investigation recorded ZT up to 0.056. Higher ZT value had been presented by (Zhou et al., 2015) which reached 0.16. The study proposed that, by optimizing the ratio of pulse off-to-on in the pulse electrodeposition process, it can enhance the Seebeck coefficient and reduce the thermal conductivity of the electrodeposited Bi2T3 film.

Since the Bi2Te3 films suffer with lower *ZT* value, there were various of approaches to enhance the film performance especially in nanocomposite method. (Wang et al., 2015) demonstrated the significant effects of nano-silicon carbide (SiC) particles in the thermoelectric enhancement of electrodeposited SiC/Bi2Te3 nanocomposite film. (Jianhui Li et al., 2013) did study on the nano-SiC particles inclusion produced better coherent interfaces between the nanoparticles and the Bi2Te3 matrix that lead to the enhancement of electrical conductivity and power factor. The power factor improvement based on SiC nanoparticles also has been proved by the study with bulk bismuth antimony telluride (BiSbTe) composite. Research finding by (Han Xu and Wei Wang et al., 2013) reported the improvement of electrical conductivity by synthesizing the multiwalled carbon nanotubes/Bi2Te3 film through the electrodeposited film performance by significantly reduced the resistivity up to 38% compared to the electrodeposited pure Bi2Te3 film.

(Lee et al., 2016) synthesized the gold (Au) nanoparticles-Bi2Te3 nanocomposite by a chemical solution-based bottom-up method at low temperature and successfully improved the *ZT* up to 0.95 at 450K. The improvement was largely contributed by the increment of carrier concentration which improved the electrical conductivity and the reduction of lattice thermal conductivity. With the same route of synthetization, the nano-silver (Ag) particles incorporated with Bi2Te3 matrix also increased the carrier concentration and lowered the thermal conductivity compared to pure Bi2Te3 at 470K (Qihau zhang et al., 2015). However, the thermoelectric performances of both the Au and Ag nanoparticles-Bi2Te3 nanocomposites only significantly increased at higher temperature than the room temperature. At 300K, the power factor of the Au/Bi2Te3 nanocomposite did not changed significantly and the ZT value was slightly lower than pristine Bi2Te3. Meanwhile the Ag/Bi2Te3 nanocomposite power factor less than pure Bi2Te3 by almost half at room temperature (300K) due to lower value of Seebeck coefficient and as a result, lessened the ZT value.

(Hyun Ju et al., 2016) reported that graphene decorated in Bi2Te3 matrix synthesized by two fabrication methods which are wet chemical synthetic route and subsequent sintering can obtain higher *ZT* value at room temperature. Study also done between with two form of bismuth telluride which made of Bi2Te3 powder and nanowires. Power factors of the both form graphene inclusion bismuth telluride increased uniformly by increased the graphene content in to it and the graphene content obtained up to 0.5wt%. Inclusion graphene cause the increased in seebeck values and reduce the electric conductive of the sample. Then the research also proven that the k values of the graphene/Bi2Te3-powder more than the graphene/Bi2Te3-nanowires. Because the nanowires bismuth telluride based sample have large homo-interfaces when compare to the powder based. It improvised the phonons scattering. By improved the power factors and reduced thermal conductive (k) values, made outcome as maximized the ZT value graphene/Bi2Te3-nanowires composite of 0.4 at 300

K.

2.8 Summary

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In this chapter, researcher will be found that, literature review provides a comprehensive background and supportive materials for this research. Each of the subtopics in this chapter is crucial to complete this project. The research methodology and development of this research will be depending on the literature review in the next chapter.

From the study of research papers and journals, get lot of ideas and knowledge for this project such as thermoelectric concepts and working mechanism, performance of thermoelectric, film fabrication method, and film characterization method. The study of the literature is provide a support for the further research on the project.

CHAPTER 3 METHODOLOGY

3.0 INTRODUCTION

This chapter explains the various steps involved in the planning and execution of the study. Before done with the triple electrodeposition process, it is necessary to establish a definite plan for the project. Having a plan for the experimental is very important for a successful project. So, in this chapter will discuss about the research methodology that were applied to conduct the study. It includes the planning of study, flow chart, film fabrication, film preparation, film characterization, film evaluation and all the materials, procedures and parameters of each method to conduct this research. Furthermore, this chapter will guide the author in preparing the entire report content including the graphical illustration. The approach to be used must be in line with the study's objectives to produce accurate results. Designing flowcharts and displaying the relationship between the objectives and the approaches chosen will improve the methodology. The approaches were chosen based on the publications of prior studies and their compliance with the study's goal.

3.1 Overview of process flow

The flowchart used to overview of the process flow used to execute this project. The main goal of this project is to deliver pristine Bi2Te3 and graphene/Bi2Te3 nanocomposite films by using electrodeposition process (Potentiostatic deposition). For the expected result, the inclusions of graphene with Bi2Te3 that will be deposited as nanocomposite films and pristine Bi2Te3 nanocomposite film by electrodeposition process. The improved stability of graphene dispersion and suspension electrolyte solution is prepared for electrodeposition process. The film that will be deposited then will be characterized and evaluated in this research by XRD. The methodology begins with the mixing process of graphene, electrolyte and electrodeposition process and lastly the deposited film will be characterized. All the process will briefly be explained in this chapter.



GANTT CHART

Table 3.1: Project Planning PSM 1

	PROJECT PLANNING PSM 1														
	~Duration: 4 October 2021 to 01 January 2022~														
Lis	List major activities involved in the proposed project. Indicate duration of each activity to the related week(s).														
Project Activities	W1	W2	W3	W4	W5	-W6	-W7	W8	W9	W10	W11	W12	W13	W14	W15
Project Title Selection	TER		•		(A)										
Determination of problem statement, objective, and scope	1118								5						
Discussion on introduction		ALM.	1												
Discussion on literature review	5	No	un	Joh,	\geq	Rin		zů,	5	"	ويتو				
Research of articles and journals	-	13.7771	DOIT		12510	2.6.1	54 A I	A.V.(
Discussion on methodology	NU	IVE	KOLL	IIE	NNI	AL	MAI	LAT	JIA I	VIEL.	ANA				
Prepare draft report															
Presentation															
Submission of PSM 1 report															

Table 3.2: Project Planning PSM 2

					PROJ	ECT PL	ANNIN	G PSM	2						
	~Duration: 7 March 2022 to 30 June 2022~														
Lis	List major activities involved in the proposed project. Indicate duration of each activity to the related week(s).														
Project Activities	W1	W2	W3	W4	W5	W6	W7	W8	W9	W10	W11	W12	W13	W14	W15
Checked schedule for lab appointment	4	×		1											
Study & answer FYP chemical safety SOP quiz. Handel chemicals correct method	AL TEKI				KA.										
Prepared electrolyte solution for electrodeposition process	42	Sam.		١.			_								
Electrodeposition process		, <u>, , , , , , , , , , , , , , , , , , </u>	*	. (5	-1		~ (5	5	29				
Film Characterization by SEM and EDX	UN	IVE	RSIT	I TE	KNI	KAL	MA	AY:	SIA I	MEL	AKA				
SEM images and EDX results analysis															
Thesis Writing															
Presentation															
Report submission															

Project Activities



Figure 3.1: Project 1 Activities for Each Chapter

FLOWCHART



Figure 3.2: Flow Chart

3.2 Experimental

3.2.1 Film fabrication Bismuth Telluride Nanocomposite with Inclusion of graphene nanoparticles

Figure 3.3 and 3.4 shows the process flow of synthesis process of graphene/Bi2Te3 nanocomposite film. It started with Step 1 which is get the graphene dispersion with N-Methyl-2-pyrrolidone (NMP). In this condition the graphene no need to do dispersion and suspension process for the graphene and NMP solvent, the graphene itself already dispersed with NMP solvent. The dispersion graphene with NMP is established one of the greatest solvents to generating the most stable graphene dispersion and NMP itself boasts the electrical conductivity of the graphene. Then, in preparation of electrolyte solution, the 1.0 M nitric acid (HNO3) was used to dissolve the bismuth oxide (Bi2O3) and tellurium dioxide (TeO2) powders. The mixing process was done in ultrasonic bath and magnetic stirring to enhance the process of dissolving the powders. The electrolyte solution that consisted of 3.2 mM Bi3+ and 7.2 mM HTeO2+ in HNO3. The mixing process should take place for one and half hour to ensure the bismuth telluride powder fully dissolved in the Nitric Acid solution.

After that, the electrolyte and electrodeposition process are prepared as Step 2 and the detailed process are shows at Table 3.3. The composition of the electrolyte is shows at Table 3.4. Lastly, the electrodeposition process is conducted as Step 3 and Figure 3.5 shows the setup of the electrodeposition process. The parameters of the electrodeposition process are shows at Table 3.5. Graphene/Bi2Te3 nanocomposite films was synthesized by using potentiostatic electrodeposition system with a three-electrode cell. The auxiliary and reference electrodes that were used in this research Pt strip and silver/silver chloride (Ag/AgCl), respectively. A glass substrate with a seed layer of chromium-gold (Cr-Au) as a working electrode was vertically submerged in the electrolyte and the distance between the substrate and the counter electrode was set at 4 cm.

Furthermore, in the step 2 electrolyte solution also consisted the desired concentration of graphene dispersion in NMP at 0.25 up to 1.25 g/L. A further mixing procedure was applied for 1 hour of stirring after nearly 2 hours of sonication of the mixed electrolyte. The commercial synthesized of nanoparticles of graphene (>99% purity) is usually hydrophobic with the predicted size of \leq 100 nm. It was very difficult to suspend the

graphene except momentarily in the aqueous solution. The issue of suspension is the stability of dispersed graphene. Due to the attraction of Van der Waals between particles, stability is impractical. The particles are attracted as the force of attraction dominates and the dispersion eventually threatens. To prevent this attraction, NMP must then be coated in graphene as a comparable surround repulsion force to cause stability. After prepare the electrolyte solution it need undergoes the three-electrode cell electrodeposition process to fabricate the film. In the electrodeposition process need ensure all the parameter of the process shown in Table 3.5 such as applied potential, time taken for film synthesis, and others.



Figure 3.3: Preparation Electrolyte Solution for the Electrodeposition process UNIVERSITI TEKNIKAL MALAYSIA MELAKA

In (step3 of the Figure 3.4), electrochemical co-depositions process is conducted without applying heat on the electrolyte solution and potentiostatically performed at - 60mV in a pulse deposition process. At 3 to 4 µm. was monitored the target thickness of the deposited films. The duty cycle (ton/(ton+toff)) was maintained at 33% for all depositions. Nanocomposite films were synthesized by a conventional three-electrode cell of electrochemical deposition system as illustrated in Figure 3.4. A pristine Bi2Te3 film was synthesized through the same method of deposition as a reference to compare the thermoelectric performance of the nanocomposite films. In the step 3 also the cyclic voltammetry on both Bi2Te3 and graphene/Bi2Te3 were carried out to study the reaction mechanisms of deposition especially on the working electrode and to determine the appropriate range of potential for electrodeposition. Figure 3.5 depicts the cyclic

voltammograms (CVs) for electrolytes that contained only bismuth-telluride ions (Solution I) and with added graphene nanoparticles (Solutions II and IV).



Procedures of Film Synthesis

Figure 3.4: Procedures of Film Synthesis

Step 1: Preparing the electrolyte solution

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Table 3.3: Preparing the electrolyte solution

Gris pames Bir Porsión Tribup 50,-4	Get graphene dispersion with N-Methyl-2- pyrrolidone (NMP) with 1 weight% for the first test and it gradually increase for each test.
Ser in the series of the serie	For the bismuth-telluride, 1.0 M nitric acid (HNO3) was used to dissolve the bismuth oxide (Bi2O3) and tellurium dioxide (TeO2) powders. The mixed powder produces bismuth-telluride acidic solution.
	AL MALAY SIA MELAKA Then, the bismuth-telluride solution undergoes ultrasonic bath to enhance the process of dissolving the powders. The electrolyte solution that consisted of 3.2 mM Bi3+ and 7.2 mM HTeO2+. The mixing process need conduct for one and half hour.

Step 2: Electrolyte and electrodeposition preparation

Table 3.4: Electrolyte and electrodeposition preparation



Electrolyte	HNO3 (mol	[Bi] ^(+3)	[[HTeO]] ^(+2)	Graphene (g
solution	L^(-1))	mol L^(-1)	$(mol \ L^{(-1)})$	L^(-1))
1	1.00	0.0032	0.0072	0.00
2	1.00	0.0032	0.0072	0.25
3	1.00	0.0032	0.0072	0.75
4	1.00	0.0032	0.0072	1.25

Table 3.5: Composition of electrolyte solution



Table 3.6: Electrodeposition process parameter

Electrolyte	Applied	Eon	Eoff	Ton	Toff	Stirring	Deposition
solution	potential	(mV)	(mV)	(ms)	(ms)	speed	time (min)
	(mV)					(rpm)	range
	range						
1	-20 to -100	180	-90	200	100	100	60 to 150
2	-20 to -100	180	-90	200	100	100	60 to150
3	-20 to -100	180	-90	200	100	100	60 to 150
4	-20 to -100	180	-90	200	100	100	60 to150

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3.2.3 Film characterization

Through scanning electron microscopy, the surface morphology is analyzed (SEM). Meanwhile, using energy dispersive X-ray spectroscopy, the composition of the deposited film was examined. EDX shows a compositional analysis with very high accuracy.

3.3 Expected Result

The inclusions of graphene with Bi2Te3 that will be deposited as nanocomposite films and pristine Bi2Te3 nanocomposite film by electrodeposition process. The improved stability of graphene dispersion and suspension electrolyte solution is prepared for electrodeposition process. The film that will be deposited then will be characterize and evaluate in this research by SEM & EDX. Thus, it will conclude the expected results in this research.

The expected result for the SEM analysis should in the structure of the electrodeposited pure Bi2Te3 surface-crystals structure must be different when compare to the inclusion of graphene/Bi2Te3 structure. The graphene/Bi2Te3 structure should be much smaller size than the pure Bi2Te3 structure. Inclusion of graphene in Bi2Te3 affecting the size of crystals structure. A finer scale is represented in the smaller image for the graphene/Bi2Te3 structure, which shows visibly a much smaller needle-like crystals of the nanocomposite compared to pure Bi2Te3. The smaller structure growth tends to produce lower porosity size and denser surface structure. Faster formation of Bi2Te3 crystals during electrodeposition supposedly happened due to the effect of deposited graphene nanoparticles that acted as catalyzer which enhance the electron transfer to the Bi/Te ions through them.

3.4 Summary

As a conclusion, there are several methods to accomplish this study which are the project planning itself, the way of how the data was taken and the Gantt Chart of this study showing how the study is running weekly. Project planning important for this study which helps the step by step to get the data. The sample of table showing that how the data is taken in the production line. Gantt Chart to ensure that this study is accomplish smoothly in every week. Methodology also a main element in a research project. In the research methods, the researcher will define all the procedures and parameters to stimulate the research objective and expected results.



CHAPTER 4

RESULT AND DISCUSSION

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.

4.1 Preparation of the electrolyte solution of graphene/Bismuth-Telluride for the electrodeposition process. IKAL MALAYSIA MELAKA

To prepare the pristine bismuth-telluride and graphene/ Bi2Te3 needs five materials. The materials are telluride oxide powder, bismuth oxide powder, graphene dispersion with N-Methyl-2-pyrrolidone (NMP), 1.0M nitric acid (HNO3), and distilled water. Table 4.1 shows the composition of the electrolyte solution used for this study and the molarity of graphene in the electrolyte sample (1, 3, and 5)wt%. Based on the methodology first procedure was both 1.15g of telluride oxide powder, and 1.5g of bismuth oxide powder should dilute the 1.0 M nitric acid (HNO3). Then, the acid was added with 1000ml of distilled water, and this solution was known as pristine bismuth-telluride. Add graphene solvent with desired weight percentage from 250ml of the pristine bismuth-telluride solution. Now the graphene inclusion bismuth telluride undergoes magnetic stirrer and sonification for one and half hours at room temperature until there is no graphene aggregation in the

electrolyte solution. The rapid magnetic stirrer and sonification process for one and half hours made electrolyte solution achieve good dispersion and suspension throughout the beaker. Figure 4.1 a and b shows differentiation between aggregated graphene in bismuthtelluride and stable condition of graphene in the bismuth-telluride. When the graphene nanoparticles achieved good dispersion and suspension, it reduced the effect of the Van de Waals attraction force between the nanoparticles and improved electrolyte had greater stability.

Electrolyte	HNO3 (mol	[Bi] ^(+3)	【HTeO】 ^(+2)	Graphene (g
solution	L^(-1))	mol L^(-1)	(mol L^(-1))	L^(-1))
1	1.00	0.0032	0.0072	0.00
2	1.00	0.0032	0.0072	0.25
3	1.00	0.0032	0.0072	0.75
4	1.00	0.0032	0.0072	1.25

Table 4.1: Composition of electrolyte solution



Figure 4.1: Electrolyte solution condition for electrodeposit process, (a) aggregated graphene in electrolyte solution without magnetic stirrer and sonification process. B shows good dispersion and suspension of graphene in electrolyte solution after the magnetic stirrer and sonification process.

4.2 Effect of Cyclic Voltammetry in the Presence of Graphene Nanoparticles

The cyclic voltammetry on both Bi2Te3 and Graphene/Bi2Te3 were carried out to study the reaction mechanisms of deposition especially on the working electrode and to determine the appropriate range of potential for electrodeposition. Figure 4.2 depicts the cyclic voltammograms (CVs) for electrolytes that contained pristine bismuth-telluride ions (Solution I) and with added graphene nanoparticles (Solutions II, III and IV). Various graphene contains added in each solution.



Figure 4.2: Cyclic voltammograms analysis of Bi3+ (3.2mM) and HTeO2+ (7.2mM) in HNO3 (1.0M) solution without graphene nanoparticles and with graphene nanoparticles (0.25 to 1.25) g l-1.

Sample	Reduction	Peak	Fixed	Deposition
	Range (-mV)	reduction	deposition	Time
		Voltage (-mV)	potential (mV)	(min)
Bi2Te3	-1 to -82	-5	-60	120
1%	-20 to -145	-50	-60	120
Graphene/Bi2Te3				
3%	-28 to -128	-55	-60	120
Graphene/Bi2Te3				
5%	-40 to -150	-70	-60	120
Graphene/Bi2Te3				

Table 4.2: Result of Cyclic Voltammetry Analysis

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The existence of positively charged surface of N-Methyl-2-pyrrolidone (NMP) dispersed graphene nanoparticles promoted stable condition of electrolytic co-deposition of the nanoparticle in parallel with the reduction of Bi3+ and HTeO2+ (S.Spanou et al., 2009). CVs analysis was also performed on other solutions (solution II, solution III and solution IV). The trends and the values of reduction peak change significantly. The deposition of Bi2Te3 occurred when electrons were transferred to Bi3+ and HTeO2+ at the working electrode surface. This process implicated the reduction of positive charges on both ions and eventually the deposition process happened.

The formation of Bi2Te3 involved the reduction of Bi3+ over the co-depositing of Te (Kumawat, A. & Sarkar., 2019). The possible half reactions involved is according to the following chemical reactions:

$$HTeO_{2+} + 3H_{+} + 4e \rightarrow Te(s) + 2H_2O$$
(4.1)

$$3\text{Te} + 2\text{Bi}_{3+} + 6\text{e}_{-} \rightarrow \text{Bi}_2\text{Te}_3(s) \tag{4.2}$$

With graphene introduction in the electrolyte solution of graphene content 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 bismuth-telluride have peak reduction potential at -5mV. The reduction peak potential of the graphene

added electrolyte also have pattern significantly increase with rate of the graphene added. As compared to the electrolyte without graphene, there is no second reduction in CV. The formation of Bi2Te3 was still occurred in the reduction range; however, the deposition mechanism was different compared to graphene added electrolyte. The reduction process tends to produce H₂Te, which was a direct reduction from HTeO₂+ at the first place and then straightly formed Bi2Te3 due to the instability of H2Te in the presence of Bi3+.

$$HTeO_{2+} + 5H_{+} + 6e \rightarrow H_2Te(aq) + 2H_2O$$

$$(4.3)$$

$$3H_2Te + 2Bi_{3+} + 6e \rightarrow Bi_2Te_3(s) + 6H_+$$

$$(4.4)$$

The studied CVs were brought to appropriate decision of applied potential in electrodeposition of pristine Bi2Te3 and graphene/Bi2Te3. The value of -60 mV was used as a fixed parameter for all electrodepositions of the nanocomposite instead of the maximum rate potential supply due to better morphological growth of the deposited films. Both of electrodeposited graphene/Bi2Te3 and Bi2Te3 films identically experienced faster crystals growth when more cathodic potentials (< -100mV) was applied. Higher cathodic potentials in electrodeposition of graphene/Bi2Te3 resulted in larger reduction current, reflecting that more electrons were transferred during Bi/Te ions diffusion. Due to faster crystals growth, the films surface became rougher and the color turned black instead of the typical grey color. This phenomenon was in line with the previous studies of Bi2Te3 analysed by potentiostatic and galvanostatic electrochemical deposition process (Tseng et al., 2017). Meanwhile, for this experiment the pristine Bi2Te3 film and graphene added film was deposited at -60 mV due to the optimized condition of Bi2Te3 atomic ratio for 120 minutes.

4.3 Morphological Analysis (Scanning Electron Microscope)

The morphological analysis conducted to study the microstructure of the pristine bismuth-telluride and graphene inclusion bismuth-telluride. The inclusion of the graphene were effect the microstructure of the pristine Bi2Te2. For conduct this analysis, scanning electron microscope used with magnificent rate of 15.00kx.



(c) 3% graphene/Bi2Te3

(d) 5% graphene/Bi2Te3

Figure 4.3: SEM micrograph images of electrodeposited films surface (a) Bi2Te3; (b) 1% graphene/Bi2Te3; 3% graphene/Bi2Te3; and 5% graphene/Bi2Te3. The inset demonstrates magnified SEM image of the pristine Bi2Te3 and Pt/Bi2Te3 films surface.

The first picture (a) shows a typical growth of electrodeposited pristine Bi₂Te₃ microstructure named as plate-like structure or also called as needle-like structure. The microstructure of the nanocomposite films with various amount of graphene shown in above figure come with in the typical needle-like shape but in a much smaller microstructure size and it also look like the needle-like shape. The change of the size characterized the inclusion of graphene in Bi₂Te₃ affecting the size of microstructure. The microstructure size of inclusion of graphene gradually become smaller when compere to each other. When the microstructure decreases the gap between it cause increase in porosity. It evidently can compete with figure (a) and (b). In the figure (a) the needle-like structure pack very closely and there is low porosity when compare to the smaller structure size in figure (b, c, and d). According to (Hosseini et al., 2021) porosity in the thermoelectric (TE) material will increasing the ZT value, by decrease the thermal conductivity. But in a side effect the porosity will unfavourable on TE power factor in the numerator of figure of merit, ZT. But the effect can cover by huge number of electron density in graphene

That porosity should not affect performance since it should not modify the electrical to thermal conductance ratio if neither electricity nor heat is transported within the pores. Lowering both conductances by the same percentage may have a benefit.

4.3 Composition of Graphene/Bi2Te3

Table 4.3 summarizes the composition of the elements in the deposited Graphene/Bi2Te3 nanocomposite films with the respective electrolyte used in the study. There are four electrolyte solutions that were distinguished by the amount of graphene that were blended. These included an electrolyte that did not include any graphene at all, which was used to produce the pristine Bi2Te3 film. Another three electrolytes comprised of the inclusion of graphene up to 1.25 g/L in the electrolyte solution. Using average values from an EDX measurement system, the amount of co-deposited graphene in the nanocomposite film was evaluated as C-wt.%. Up to 2.7% graphene has been successfully deposited in the nanocomposite film with reduced problems on the aggregated deposition. There is a pattern where the graphene increase in electrolyte, the composition carbon rate in the deposited film also increases. The nanomaterial content in bismuth telluride, synthesis nanocomposites deposited films will optimized and increased the electrical conductivity and reduced the effect of Seeback coefficient reduction.

Electrolyte	Graphene	Electrodeposited	Composition in the deposited film				
	content in	film	Graphene	Bi:Te	Atomic		
	electrolyte		(wt. %)	(at%)	percentage error		
	(g/L)				due to Bi2Te3		
					phase ratio (%)		
1	0.00	Bi2Te3	0.00	40:60	0		
2	0.25	Graphene/ Bi2Te3	1.17	42:58	± 2		
3	0.75	Graphene/ Bi2Te3	1.61	37:63	± 3		
4	1.25	Graphene/ Bi2Te3	2.69	42:58	± 2		

Table 4.3: Result of Energy Dispersive X-Ray

4.4 Summary

In this chapter, the pure bismuth telluride nanocomposite films and graphene incorporated with bismuth telluride nanocomposite films that were deposited were studied and analyzed from its film characterization. The morphological and composition of deposited films were analyzed in this chapter by using SEM and EDX.

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CHAPTER 5 CONCLUSION AND RECOMMENDATION

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.

5.1 Conclusion

In conclusion, all the objectives in this study have been achieved as the first objective is to prepare the electrolyte solution of bismuth telluride for the electrodeposition process with good dispersion and suspension of graphene. The both pristine bismuth-telluride and graphene inclusion bismuth-telluride electrolyte solution successfully prepared with good dispersion and suspension of graphene. The second objective is to synthesis of pristine Bismuth Telluride film and Bismuth Telluride nanocomposite film with inclusion of graphene up to 3 wt% electrochemical deposition process. According to the second objective up to 2.7% graphene has been successfully deposited in the nanocomposite film as C-wt.% recorded from average values of EDX. For achieve third objective the sample undergoes composition and surface morphology analysis. The SEM method used to identify the porosity between pristine Bi2Te3 and Graphene/Bi2Te3 and the EDX method able show atomic percentage of Bi2Te3 in each sample as the ratio which used in the electrolyte solution. All the objective were successfully achieved which stated on the table 5.1.

Table 5.1: Summary of achievements

Objectives	Expected	Achievements
Prepare the electrolyte	Prepare the electrolyte	The both pristine Bi2Te3 and
solution of Bi2Te3 and	without any aggregated	Graphene/ Bi2Te3 electrolyte
Graphene/ Bi2Te3 for the	particles form.	solution prepared with good
with good dispersion and		dispersion and suspension of
suspension.		graphene
Synthesis of Graphene/	Upto 3.0 wt% of	Up to 2.7% graphene has been
Bi2Te3 nanocomposite film	graphene deposit in the	successfully deposited in the
with graphene weight	synthesis film.	nanocomposite film as C-
percentage up to 3% in		wt.%.
electrochemical deposition		
process.	361	
Analyze surface morphology	• Finer microstructure in	• Graphene/Bi2Te3 have finer
and chemical composition of	Graphene/ Bi2Te3 film.	microstructure than pristine
the pristine Bi2Te3 film and	• Larger microstructure	Bi2Te3.
Graphene/Bi2Te3	in pristine Bi2Te3 film.	• All the four samples have
nanocomposite film.	•Obtain Bi2Te3 at%	at% error under ± 5 .
	error ± 5 in each sample	

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5.2 Recommendations

The study of graphene/Bi2Te3 can be further extended by studying the film evaluation of Seeback coefficient, electrical conductivity and thermal conductivity. The future works for this study can conducted by annealed all the sample at 250°C for 2 hours before proceeding to the sample preparation process. After the nano-film spread from the working electrode it transferring to solidified epoxy that acted as an insulator layer was crucial to avoid any electrical shortage during the further study.

The Seebeck coefficient and the electrical conductivity of the samples were measured at room temperature by steady state method using a Seebeck measurement system This measurement conducted by DC four-probe system. Then, carrier concentration and mobility have been measured using van der Pauw Hall measurement method and thermal conductivity of deposited films is measured by a differential 3-omega (3ω) method. Finally, after done all this study the figure of merit will be obtain for the pristine bismuth-telluride and graphene inclusion bismuth-telluride.

5.3 Sustainable Design and Development

Regarding key global challenges like resource scarcity and climate change, sustainability looks at how processes and activities could last longer while having less detrimental consequences on ecological systems. By concerning the sustainable issues the remaining used chemicals disposed properly. Disposing of chemical waste has huge environmental impacts and can cause serious problems left on the environment. Specially for this study acetone and ethanol were used for rinse the beaker before conduct an experiment. After rinse the chemical will stored in a used chemical storage instead of disposed in the laboratory sink.

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5.4 Complexity VERSITI TEKNIKAL MALAYSIA MELAKA

Defects and disorders in materials are viewed by complexity as modifiable design parameters, just as element bonding structure, electronic tools, and composition. Disorder and faults frequently influence general behaviour, for example, voids that encourage mobility or charged substitutes that regulate conductivity. This calls for advancements in characterization to comprehend how to manage the concentration of faults and degree of disorder, as well as computer simulation to simulate faulty crystals and glasses with no longrange order. Design of defective and disordered materials to achieve specific performance results and guided synthesis to stabilize targeted concentrations.

5.5 Life Long Learning

The idea of continuing one's education and developing new talents after completing their official or required education is known as lifelong learning. Lifelong learning is often voluntary and self-motivated, driven by a desire to learn more, acquire new abilities, or advance one's career. From conduct this study able to learn the material characterization techniques and it's parameter for the sample which suitable to conduct the studies. Other than that, research regrading materials specially nanomaterial gave a huge knowledge on the related field.



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APPENDIX

Appendix A: Energy Dispersive X-Ray Result





Graphene 5%





The Business of Science*

Graphene 3%



Te La1



Graphene 3%





64

Graphene 1%



Te La1



Graphene 1%





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