STRUCTURAL AND SURFACE MORPHOLOGY OF NANOCRYSTALLINE BISMUTH TELLURIDE THIN FILMS DEPOSITED USING RADIO FREQUENCY MAGNETRON SPUTTERING

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To my beloved family and friends.

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ABSTRACT

Nanocrystalline Bi_2Te_3 thin film is a highly potential material to be used in semiconductor thermoelectric because of its refined and enhanced properties. The deposition and characterization of Bi_2Te_3 thin films are reported in this work. Films were deposited with substrate temperature ranging from 50 ºC to 150 ºC, argon flow rate from 5 $cm^3 min^{-1}$ to 25 $cm^3 min^{-1}$, deposition time from 300 s to 900 s and RF power from 50 W to 250 W. In-situ annealing and rapid cooling for thin film deposited under 100 ºC substrate temperature was performed. AFM topography images shows that Bi_2Te_3 thin films follow the Frank de Merwe deposition mode. The lowest surface roughnesses obtained were 0.35 nm, 0.02 nm, 0.11 nm and 0.06 nm, at 150 ºC substrate temperature, 20 $cm^3 min^{-1}$ argon flow rate, 450 s deposition time and 50 W RF power, respectively. The smallest grain sizes obtained were 33.9 nm, 19.91 nm, 37.99 nm and 14.27 nm which were deposited at 150 ºC substrate temperature, 25 $cm^3 min^{-1}$ argon flow rate, 450 s deposition time and at 50 W RF power, respectively. XRD analyses revealed that the thin films were in the form of polycrystalline structure. The largest nanocrystalline size was obtained at 900 s deposition time while the smallest size was obtained at 125 ºC substrate temperature. In-situ annealing showed an improved grain size and surface roughness as annealing temperature increased. Rapid cooling successfully eliminated the worm-like dimer on the surface, improved grain size and area grain density. Great significant of structural and surface morphology was found as a function of deposition parameters, namely the substrate temperature, argon flow rate, deposition time and RF power.

ABSTRAK

Nanohablur saput tipis Bi_2Te_3 adalah satu bahan yang berpotensi tinggi untuk digunakan dalam semikonduktor termoelektrik kerana sifatnya yang diperhalusi dan dipertingkatkan. Pemendapan dan pencirian saput tipis Bi_2Te_3 dilaporkan dalam kajian ini. Saput tipis telah dimendapkan pada suhu substrat bermula daripada 50 ºC hingga 150 ºC, kadar aliran argon daripada 5 $cm^3 min^{-1}$ hingga 25 $cm^3 min^{-1}$, masa pemendapan dari 300 s hingga 900 s dan kuasa RF daripada 50 W hingga 250 W. Penyepuhlindapan setempat dan penyejukan pantas untuk saput tipis yang dimendapkan pada suhu substrat 100 ºC telah dijalankan. Imej topografi AFM menunjukkan bahawa pemendapan saput tipis Bi_2Te_3 mengikuti mod pemendapan Frank de Merwe. Kekasaran permukaan terendah yang diperoleh ialah 0.35 nm, 0.02 nm, 0.11 nm dan 0.06 nm bagi masing-masing parameter iaitu suhu substrat 150 ºC, kadar aliran argon 20 $cm^3 min^{-1}$, masa pemendapan 450 s dan kuasa RF 50 W. Saiz butiran terkecil bagi setiap parameter ialah 33.9 nm, 19.91 nm, 37.99 nm dan 14.27 nm masing-masing dimendapkan pada suhu substrat 150 ºC, kadar aliran argon 25 $cm^3 min^{-1}$, masa pemendapan 450 s dan kuasa RF 50 W. Analisis XRD menunjukkan bahawa saput tipis adalah dalam bentuk struktur polihablur. Saiz nanohablur terbesar terhasil pada masa pemendapan 900 s manakala saiz nanohablur terkecil pada suhu pemendapan 125 ºC. Penyepuhlindapan setempat telah berjaya memperbaiki saiz butiran dan kekasaran permukaan dengan peningkatan suhu penyepuhlindapan. Penyejukan pantas berjaya menghapuskan dua butiran yang serupa dan bersambung pada permukaan, memperbaiki saiz butiran dan kepadatan butiran. Hubungan yang ketara ditemui antara struktur morfologi dan permukaan dengan parameter pemendapan, iaitu suhu substrat, kadar aliran argon, masa pemendapan dan kuasa RF.

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α	-	Seebeck coefficient
AFM	-	Atomic force microscopy
$\boldsymbol{B}$	-	Magnetic field
Cu	-	Copper
d	-	Interplanar spacing
$\boldsymbol{E}$	-	Electrical field
EDX	-	Energy dispersion x-ray
F	-	Force
FESEM	-	Field emission scanning electron microscopy
FWHM	-	Full width high maximum
HVC	-	High vacuum coater
κ	-	Thermal conductivity
κ_E	-	Electrical thermal conductivity
κ_L	-	Lattice thermal conductivity
λ	-	Wavelength
MOCVD	-	Metal-organic chemical vapor deposition
σ	-	Electrical conductivity
RF	-	Radio frequency
SEM	-	Scanning electron microscopy
SPM	-	Scanning probe microscopy
T	-	Temperature
TEM	-	Transmission electron microscopy
XRD	-	X-ray diffraction
ZT	-	Thermoelectric figure of merit

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CHAPTER I

INTRODUCTION

1.0 General Introduction

Thin film fabrication for semiconductor material faces revolution of various methods throughout the last century. Thin film is a growing and continuously expanding subject of interest in electronics area as well as being competitive form of material in industry. In recent years, a particular thermoelectric device was developed based on low-dimensionality concepts of material fabrication. Co-sputtering and co-evaporation have been the classic approach, which requires much effort in controlling the composition ratio of at least two elements. Metal-organic chemical vapour deposition (MOCVD) is an advance technique for producing thin films however demands high costs and extra precautions on chemical procedure. Therefore, cost-effective technique suitable for mass production of thin film fabrication is required to obtain thin film with controllable structure and grain size. Radio frequency (RF) magnetron sputtering is not a new method in thin films technology, however parameters manipulation play the main role in obtaining controlled structure and grain size of fabricated thin films.

1.1 Research Background

Different approaches have been introduced throughout the past century in fabrication of thin film semiconductor materials. In a remarkable experiment by Ventakasubramanian *et al.* superlattice of Bi_2Te_3/Sb_2Te_3 (Ventakasubramanian et al., 2001) gives 2.4 figure-of-merit value that successfully reduced lattice thermal conductivity which is 2.2 times smaller compared to $Bi_{0.5}Sb_{1.5}Te_3$ alloy (Hamachiyo et al., 2009). Currently there has not been any new report of reproduction of this value (Shakouri, 2011). As-deposited $(Bi_{0.15}Sb_{0.85})_2Te_3$ thin film leads to enhancement of power factor (Rothe, 2010) with real time measurement of electrical conductivity and Hall effect was measured in room temperature, $Ce_{0.9}CoFe_3Sb_{12}$ thin film deposition using RF magnetron sputtering gives increasing of resistivity with respect to temperature (Lopera, 2005), 25%Bi_2Te_3-75%Sb_2Te_3 compounds ball mill gives maximum power factor and figure-of-merit at room temperature 3.10 x 10^{-3} Wm^{-1} K^{-2} and 2.85 x 10^{-3} K^{-1} respectively (J Y Yang, 2006), and elemental bismuth, antimony and tellurium powder co-evaporation get different composition of superlattices which increases Seebeck coefficient with the rising of annealing temperature (L.M. Goncalves, Schubert). Boukai *et al.* (Heath et al., 2008) and Hochbaum *et al.* (Hochbaum et al., 2008) reported that silicon nanowires could have ZT about 0.6 at room temperature and about 1 at lower temperature.

Due to its particularly remarkable quality as semiconductor material, Bi_2Te_3 has been consistently used and manipulated in various ways to transfer into nanostructure level (Huang et al., 1999, Lu et al., 2005, Silva et al., 2005, Dheepa et al., 2007, Li et al., 2008, Scheelr et al., 2009) since it was first introduced in 1950s. It is not an unusual method to fabricate Bi_2Te_3-based thermoelectric material using co-evaporation(Zou et al., 2001, Goncalves et al., 2008, Goncalves et al., 2010), flash-evaporation method on glass substrate (Dheepa et al., 2007), ligand-assisted solvothermal method (He et al., 2012) and sputtering (Tomozeiu, 2002, Christie et al., 2003, Kim et al., 2006, Huang et al., 2009, Zhi-Wei et al., 2011, Arina et al., 2012). Spark plasma sintering of bulk Bi_2Te_3 Marcus et al. (Scheelr et al., 2009) has improved the power factor but only decrease the thermal conductivity by one order smaller that the bulk. Fabrication using metal organic chemical vapour deposition was conducted by Kwon et al (Kwon et al., 2009) but unable to get the maximum output power of the fabricated device. Liao et al. (Liao and She, 2007) successfully deposited Bi_2Te_3 thin films using sequential sputter deposition of Bi and Te elements followed by annealing treatment. Though high Seebeck coefficient of 221 μ V/K achieved yet the electrical resistivity of Bi/Te multilayer structure has to be improved. New method also has been attempted such as synthesizing Bi_2Te_3 nanostructure by refluxing method by Gupta et al. (Gupta et al., 2012), high-pressure torsion by Aside et al. (Ashida et al., 2009), ultrarapid quenching process route by Friety et al. (Friety, 2000) claiming that chemical concentration, reaction timing, additives sequence are playing the main role in synthesizing nanostructure Bi_2Te_3 to get more controlled size and shape.

Focusing onto sputtering method that is obviously trending high in the past few years (Christie et al., 2003, Kim et al., 2006, Kim and Lee, 2006, Huang et al., 2009, Arina et al., 2012) researchers are more excited in fabricating thin films by combining the two elements namely Bi and Te (Huang et al., 2009) as well as studying the effect of temperature to deposited thin film (Kim et al., 2006, Arina et al., 2012). Zhang et al. (Zhi-Wei et al., 2011) has been using hot-pressed Bi_2Te_3 single target in fabricating Bi_2Te_3 thin film by varying deposition temperature from 250°C to 400°C and gained thick layers of large poor oriented structures. Large grain of thin films may not suitable for micro-devices fabrication due to integration problem (Liao and She, 2007). Yuan *et al* demonstrated that RF magnetron sputtering is able to produce oriented Bi_2Te_3 films by varying substrate temperature and deposition pressure (Deng et al., 2011). Though nanolayers was obtained but the great film thickness may not suitable for micro devices as predicted by Liao. Much attempts has been carried out utilizing RF magnetron sputtering but limiting manipulation of parameter only to substrate temperature (Tan et al., 2007, Huang et al., 2009, Zhi-Wei et al., 2011).

1.3 Problem Statement

The phenomenon stated previously indicates that optimizing experimental parameters is vital in fabricating Bi_2Te_3 thin film deposited using radio frequency magnetron sputtering. It is important to minimize the work procedure in producing nanostructure of Bi_2Te_3 thin film with high efficiency utilizing radio frequency magnetron sputtering. Feasible technique such as using single target of Bi_2Te_3 for sputtering in order to produce thin film with uniform morphology, smooth surface and good crystal structure. There are also challenges in determining the deposition parameters in order to analyze the dependence of structural and surface morphology of Bi_2Te_3 thin film to argon flow rate rather limiting to deposition temperature, RF power and deposition time.

1.3 Objectives

This work focuses on the study of structural properties and surface morphology of deposited single layer Bi_2Te_3 thin film on Si (111) substrates as well as to carry out the following objectives.

i. To characterize the surface morphology of the deposited Bi_2Te_3 thin films using atomic force microscopy (AFM)
ii. To analyse the structural properties of deposited Bi_2Te_3 thin films using AFM, X-ray Diffraction (XRD).
iii. To determine the dependent of deposition parameters to surface morphology and structural properties.

1.4 Research Scope

In order to achieve the objectives, this study Bi_2Te_3 thin film were deposited using RF magnetron sputtering on Si (111) substrates. Thin films were deposited under various conditions by the manipulation of substrate temperature, gas flow rate, deposition time and RF power. This study is focusing on structural and morphology of the deposited thin film. The behaviour of deposited Bi_2Te_3 films structure dependent on substrate temperature, gas flow rate, deposition time and RF power were studied. Grain sizes, roughness area and grain density as a function of deposition parameters were also investigated. Crystallite size and structures of deposited films were also studied using XRD.

1.5 Significant of Study

i. Structural and surface morphology of Bi_2Te_3 thin film dependence on deposition parameters using radio frequency magnetron sputtering were deeply investigated
ii. Appropriate deposition parameters obtained and can be used to deposit Bi_2Te_3 thin film.
iii. Nanocrystalline Bi_2Te_3 thin film with high grain density can produced using only one sputtering target.
iv. AFM can be used for thin film heat treatment and rapid cooling to enhance surface morphology quality.

1.5 Thesis overview

The study of structural and surface morphology of Bi_2Te_3 thin film deposited using RF magnetron sputtering is thoroughly reported in this thesis. This report classified in five chapters.

The first chapter precisely specifies the general introduction, research background, the objectives, research scopes and thesis overview.

In chapter two, theoretical background and concepts of thin films are explained. The material used in this study, Bi_2Te_3 was also described associated with RF magnetron sputtering trends review concerning fabrication of the same material. The description of structural and surface morphology of semiconductor thin films and RF magnetron sputtering basic concepts also included.

Chapter three consists of description on sample preparation, thin films deposition procedure using RF magnetron sputtering as well as the characterization and measurement method. The characterization instruments namely atomic force microscopy (AFM), X-ray Diffraction (XRD), scanning electron microscopy (SEM).

Chapter four presents the results and analysis of entire work in this study, associated with detailed discussions. Optimization of parameters is also included. Substrate temperature, argon flow rate, deposition time and RF power dependent of surface morphology and structural properties were explained chronologically. Conclusions and suggestions are given in the final section, chapter 5.

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