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STRUCTURAL AND OPTICAL PROPERTIES OF RF-SPUTTERED GE THIN FILMS USING MAGNETRON SPUTTERING TECHNIQUE

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By

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STRUCTURAL AND OPTICAL PROPERTIES OF RF-SPUTTERED GE THIN FILMS USING MAGNETRON SPUTTERING TECHNIQUE

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Chairman : Suriati Binti Paiman, PhD
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The desire to control germanium (Ge) thin film quality while keeping it cost effective has become one of the biggest challenges. This thesis proposes radio frequency (RF) magnetron sputtering as a technique to deposit Ge thin films (towards nanowires growth) on a glass substrate at room temperature. This research focuses on the structural and optical properties of Ge thin films by varying the pressure and RF power. The structural properties were characterized using atomic force microscopy (AFM), high surface profilometer, and x-ray diffraction (XRD). Meanwhile, the optical properties were investigated using ultraviolet-visible spectroscopy (UV-Vis) and Raman spectroscopy.

Based on the study, at a high pressure of 15 mTorr, the thickness obtained was 114.76 ± 2.89 nm for the as-deposited Ge thin film. This is due to the bombardment of the atom during the sputtering process caused the thickness to decrease as the pressure was increased. Meanwhile, at a higher RF power of 100 Watt, the thickness obtained was found to increase to 232.32 ± 5.67 nm. This was caused by the atoms that gained more kinetic energy to be bombarded onto the glass substrate when the RF power was increased.

The AFM studies show that the lowest root-mean-square (*rms*) surface roughness obtained the in lowest pressure of 5 mTorr was 1.898 nm. On the other hand, at 50 Watt of RF power, the lowest *rms* surface roughness obtained was 10.283 nm. Moreover, based on the band gap energy analysis using UV-Vis, values obtained were in the range of 3.84 to 3.91 eV. Besides, the phase analysis using XRD also shows all the deposited Ge thin films obtained were in an amorphous phase. In addition, Raman analysis also shows second-order Ge phonon modes at the region of 535 to 610 cm^{-1} which tend to shift due to its amorphous behavior.

The heat treatment was applied at a different annealing temperature of 280 ˚C and 450 ˚C in order to recover and alter the microstructure of Ge thin film. The thickness was found to be increased from 40.53 ± 2.026 nm to 126.06 ± 6.378 nm as the pressure was increased when the thin films were annealed at a temperature of 280 ˚C. Meanwhile, at annealing temperature of 450 ˚C, the thickness of thin films decreased from 148.76 ±7.4 nm to 69.83 ± 3.471 nm as the pressure was increased. In comparison, when the annealing process was applied, the thickness increased as the RF power was increased in both of the annealing temperatures of 280 ˚C and 450 ˚C from 102.07 ± 5.12 nm to 137.43 ± 5.471 nm and 76.46 ± 3.387 nm to 177.43 ± 6.832 nm, respectively.

In this study, it is found that the most optimized Ge thin film was from annealed Ge thin film at temperature of 450 ˚C with a thickness of 148.76 ±7.4 nm and the *rms* surface roughness of 1.898 nm, which was deposited at a lower pressure and RF power of 5 mTorr and 25 Watt, respectively. This shows that the deposition parameters influence the surface morphology, phase, band gap energy, and phonon modes of Ge thin films. By controlling these parameters, Ge thin films surface morphology can be optimized, thus producing low *rms* surface roughness. The development of Ge thin films as the high-quality film might be useful in the future especially in the growth of nanowire for solar cell application.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk Ijazah Master Sains

CIRI-CIRI STRUKTUR DAN OPTIKAL SAPUT NIPIS GE MENGGUNAKAN KAEDAH RADIO FREKUENSI PERCIKAN MAGNETRON

Oleh

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Keinginan untuk mengawal kualiti saput nipis germanium (Ge) sambil mengekalkan kos yang efektif menjadi satu cabaran yang terbesar. Tesis ini mencadangkan kaedah frekuensi radio (RF) percikan magnetron sebagai teknik untuk menghasilkan saput nipis Ge yang berkualiti tinggi (ke arah aplikasi pertumbuhan nanowayar) pada substrat kaca pada suhu bilik. Penyelidikan ini memberi tumpuan kepada sifat struktur and sifat optikal saput nipis Ge dengan menvariasikan tekanan dan kuasa RF. Sifat struktur dikaji dengan menggunakan kaedah pencirian mikroskopi daya atom (AFM), profilometri permukaan tinggi, dan belauan sinar-x (XRD). Manakala, sifat optikal pula disiasat menggunakan spektroskopi ultra-lembayung (UV-Vis) dan spektroskopi Raman.

Berdasarkan kajian, pada tekanan yang tinggi iaitu 15 mTorr, ketebalan yang diperolehi adalah 114.76 ± 2.89 nm bagi saput nipis Ge yang termendap. Ini akibat daripada tekanan yang tinggi, pembedilan atom semasa proses percikan menyebabkan ketebalan berkurang sejajar dengan tekanan yang meningkat. Sementara itu, pada kuasa RF yang tinggi iaitu 100 Watt, ketebalan yang diperolehi ialah 232.32 ± 5.67 nm. Atom memperoleh lebih tenaga kinetik untuk membedil pada substak kaca apabila kuasa RF bertambah.

Kajian AFM menunjukkan kekasaran permukaan punca-minimum-persegi (*rms)* yang paling rendah diperolehi pada tekanan 5 mTorr ialah 1.898 Selain itu, pada kuasa RF 50 Watt, kekasaran permukaan *rms* paling rendah ialah 10.283 nm. Tambahan pula, berdasarkan kajian UV-Vis, jurang jalur tenaga yang diperolehi di antara 3.84 ke 3.91 eV. Selain itu, analisis fasa yang menggunakan XRD juga menunjukkan semua saput nipis Ge yang didepositkan adalah fasa amorfus. Sebagai tambahan, analisis Raman juga menunjukkan jelas puncak serakan pada rantau 535 – 610 cm^{-1} di mana mempunyai kecenderungan untuk beralih disebabkan oleh sifat amorfus bahan.

Rawatan haba dikenakan pada suhu penyepuhlindapan yang berbeza iaitu 280 ˚C dan 450 ˚C untuk memulihkan dan mengubah struktur micro saput nipis Ge. Ketebalan didapati bertambah dari 40.53 ± 2.026 nm kepada 126.06 ± 6.378 nm apabila tekanan bertambah untuk penyepuhlindapan pada suhu 280 ˚C. Sementara itu, ketebalan pada suhu penyepuhlindapan 450 ˚C, ketebalan saput tipis berkurang dari 148.76 ±7.4 nm kepada 69.83 ± 3.471 nm apabila tekanan bertambah. Sebagai perbandingan, apabila process penyepuhlindapan dilakukan, ketebalan bertambah apabila kuasa RF meningkat pada kedua-dua keadaan suhu penyepuhlindapan iaitu 280 ˚C dan 450 ˚C dari 102.07 ± 5.12 nm kepada 137.43 ± 5.471 nm dan 76.46 ± 3.387 nm kepada 177.43 ± 6.832 nm, masing-masing.

Kajian ini mendapati saput nipis yang paling optimum adalah saput tipis yang telah dipenyepuhlindapan pada suhu 450 ˚C dengan memiliki ketebalan iaitu 148.76 ±7.4 nm dan kekasaran permukaan *rms* iaitu 1.898 nm dimana ianya telah didepositkan pada tekanan dan kuasa RF yang paling rendah iaitu masing-masing pada 5 mTorr dan 25 Watt. Ini menunjukkan bahawa parameter pemendapan dapat mempengaruhi permukaan morfologi, fasa, jurang jalur, dan mod fonon dalam tipis Ge. Dengan mengawal parameter-parameter ini, saput nipis Ge permukaan morfologi dapat dioptimumkan, sehingga menghasilkan saput nipis Ge yang berkualiti tinggi dengan kekasaran permukaan *rms* yang rendah. Perkembangan saput nipis Ge sebagai saput nipis yang berkualiti tinggi mungkin berguna pada masa akan datang terutamanya dalam pertumbuhan nanowayar untuk aplikasi sel solar.

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LIST OF ABBREVIATIONS

$\bar{Z}$	Mean value of the surface height relative to the center
A	Absorbent
Å	Armstrong
AFM	Atomic Force Microscopy
CVD	Chemical Vapor Deposition
d	Diameter
DC	Direct current
E_g	Band gap energy
GaAs	Gallium arsenide
Ge	Germanium
ICDD	International Centre of Diffraction Data
LA	Longitudinal acoustical mode
LO	Longitudinal optical mode
MOSFET	Metal Oxide Semiconductor Field Effect Transistor
n	Type of transition
PE-CVD	Plasma Enhanced Chemical Vapor Deposition
PVD	Physical Vapor Deposition
RF	Radio Frequency
rms	root-mean-square
RP-CVD	Reduced Pressure Chemical Vapor Deposition
Si	Silicon
t	Sample thickness
TA	Transverse acoustical mode
TO	Transverse optical mode
UHV-CVD	Ultra-High Vacuum Chemical Vapor Deposition
UV-Vis	Ultra-Violet Visible
XRD	X-ray Diffraction
α	Absorption coefficient
γ	Sputtering yield
θ	Angle in degree
λ	Wavelength
N	Number of point in the sample area
Z	Vertical height of the surface

CHAPTER 1

INTRODUCTION

1.1 Background

The development of thin film technology starts around 1965 with the manufacturing of an integral part of the mass manufacturing process in the semiconductor and optical industry (Seshan, 2002). Many studies have been done due to high demand in the thin film technology which can be a fundamental basis of a product development. The demand has been evolved through many fields including; integrated circuits, optoelectronics (Colace *et al.,* 2010), aerospace, biomedicine, and photovoltaic applications (Tsao *et al.,* 2011a; Shahahmadi *et al.,* 2016).

Nowadays, the deposition of germanium (Ge) thin film becomes the centre of attention due to its attractive properties and behaviour. From an optical perspective, due to high refractive index and minimal optical dispersion, Ge thin film is useful for lenses and optical elements for infrared imaging (Cariou *et al.,* 2014). Owing to the small energies band gap and high carrier mobility, Ge can be used as a bottom layer multijunction solar cell application (Goh *et al.,* 2010; Cariou *et al.,* 2014). Further investigation of optical properties of Ge thin film reported by Liu *et al*. and Tsao *et al*., have found out that the energy band gap of Ge thin films can be varied by thermal treatment during the deposition process. Therefore, Ge thin film has become a reliable candidate for future electronic devices (Tsao *et al.,* 2011b; Liu *et al.,* 2015).

For electronic devices, Ge thin film demanding a specific requirement. The most important parameter to obtain high-quality Ge thin film is the film thickness and surface morphology. Nguyen *et al.,* and Zhang *et al.,* have reported that the stress due to lattice mismatch between Si substrate and Ge thin film fabricated using RF magnetron sputtering exhibits low root-mean-square (*rms*) surface roughness of 1.6 nm and thickness of 100 nm (Nguyen *et al.,* 2013; Zhang *et al.,* 2010).

In this study, we aim to deposit low surface roughness and desired thickness of Ge thin film on a glass substrate by using RF magnetron sputtering utilizing ex-situ annealing process. Parameters such as pressure and RF power were optimized in order to achieve the desired Ge thin film quality whereby nanowires can be grown for the solar cell application in the future.

1.2 Motivation and problem statement

Ge thin film usually utilizes as a buffer layer or virtual substrate for the integration and fabrication of GaAs-based optical devices and III-V compound semiconductor metal-oxide-semiconductor-field-effect-transistor for (MOSFET) on Si due to its specific advantages (Choi *et al.,* 2008). The main advantages of Ge thin film are their electrical and structural properties are close to Si (Shah *et al.,* 2011a), provide a good epitaxial structure with near-perfect lattice match to GaAs (Choi *et al.,* 2008), and low deformation of layer (Nguyen *et al.,* 2013). However, further investigation is needed to study the implementation of Ge thin film since large thermal expansion coefficient (Choi *et al.,* 2008), high threading dislocation density at the interface and high surface roughness limiting the performance of the solar cell.

The thermal coefficient expansion is known as a measure of a fractional change in surface, per unit degree of changes in temperature. A thin film with a larger thermal expansion will give higher tensile strain (Shah *et al.,* 2011a). Therefore, it is common to avoid depositing a thin film with a mismatch of thermal expansion, which caused the malfunction devices (Fang and Lo, 2000).

The mismatches between the thin film and the substrate generally refer as the line imperfection in the lattice (Kittel, 2015). The imperfection of lattice structure can be caused by different bonding energy between atoms. A higher density of threading dislocation resulted from large lattice mismatch and high thermal expansion coefficient between thin film and substrates degrade the carrier mobility and increasing the current leakage path in the devices (Wong *et al.,* 2010).

As the film growth, the problem from the dislocation might contribute to the surface roughness irregularity and unevenness on the plane of a thin film. The roughness of thin film on nanostructure such as solar cell will affect the structural properties (Suh *et al.,* 2010). Therefore, low surface roughness will improve the strength and ductility of the thin film.

RF magnetron sputtering was used to improve the strength and ductility of the thin film since it has high deposition rate, good reproducibility and a possibility of using commercially available large area sputtering system (Kurdesau *et al.,* 2006). Several studies reported that the RF magnetron sputtering can produce a lower surface roughness of Ge thin film compared to others typical method (Samavati *et al.,* 2013; Choi *et al.,* 2008). Moreover, low surface roughness may avoid the deposition of a thicker film. The thicker film caused clusters and coarsening effect which leads to the high surface roughness in film deposition.

Hence, the annealing process has been introduced to overcome this problem. The annealing process was reported can produce a smoothing effect and lowers the surface roughness of the thin film (Shah *et al.,* 2011b). The annealing process is a common process used to recover structural quality in materials. In the highly damaged semiconductor materials caused by ion bombardment, annealing allows atoms to move back into their lattice sites, removing structural damage and recrystallize material from an amorphous structure to a crystalline or polycrystalline structure (Kang *et al.,* 2009).

Therefore, this study related to the deposition of the lower surface roughness of Ge thin film using RF magnetron sputtering technique. Parameters such as pressure and RF power were varied throughout the study. The annealing process was applied in order to investigate the effect of thermal treatment on the thin film.

1.3 Research objectives

The interest of this research is to study the properties of Ge thin films by RF magnetron sputtering technique. Thus, this research embarks the following objectives:

i. To deposit Ge thin film using radio-frequency (RF) magnetron sputtering by varying the pressure and RF power.
ii. To investigate the effect of annealing process of deposited Ge thin films.
iii. To investigate the structural properties (thin film thickness, surface morphology, *rms* surface roughness, and crystal phase) of Ge thin film using a high surface profilometer, atomic force microscopy (AFM) and X-ray diffraction (XRD) techniques.
iv. To characterize the optical properties (energy band gap and phonon modes) of Ge thin film using ultra-violet visible (UV-Vis) and Raman spectroscopy techniques.

1.4 Thesis outline

The thesis is divided into five chapters. Chapter 1 introduces the thesis background, problem statement and the objectives of this research. Chapter 2 reviews the literature studies that have been done. Meanwhile, Chapter 3 explains about the methodology used in this study. Chapter 4 discusses the effect of pressure and RF power to the surface morphology of the films. The effects of annealing process also being discussed in this chapter. Finally, Chapter 5 summarizes the results of the study and suggests directions for future work.

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BIODATA OF STUDENT

Miss Nurul Assikin Binti Ariffin was born in Tapah, Perak on 8th August 1989, and stayed in Puchong for more than 25 years. She received her primary education at Sekolah Kebangsaan (SK) Puchong Batu 14, Puchong and Sekolah Kebangsaan Puchong Perdana (SKPP), Puchong, Selangor. She furthered her secondary education at Sekolah Menengah Kebangsaan Puchong Perdana (SMKPP), Puchong, Selangor. She then completed her tertiary education with the matriculation programme and received a matriculation certificate from Kolej Matrikulasi Melaka in 2008. She is then pursued her first degree in Bachelor of Science (Hons.) Majoring in Physics in Universiti Putra Malaysia, Serdang, Selangor.

During her first degree study, she was awarded two awards "*Hadiah Projek Ilmiah Terbaik Peringkat Jabatan Fizik 2012/2013*" and "*Hadiah Projek Ilmiah Terbaik Fakulti Sains 2012/2013*". Her interest in research grew in her final year as an undergraduate, where she was introduced with the research project entitled "*Microwave Extraction of Fennel and Cumin Seeds Essential Oils Using Microwave Extraction Technique (MET) and Conventional Extraction Technique (CET)*". Her interest in research will keep on until she achieved her dreams to pursued Ph.D in the future.

LIST OF PUBLICATIONS

Ariffin, N. A., Mustaffa, S. A., Paiman, S., Tamchek, N., & Shaari, A. H. (2016). Effect of Post-Annealing Treatment on The Structural Properties of RF-Sputtered Germanium Thin Films. *Journal Solid State Science & Technology Letters,17*(2), 191-194.

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