COMPARISON OF THE STRUCTURAL PROPERTIES OF SILICON CARBIDE USING VERY HIGH FREQUENCY PLASMA ENHANCED CHEMICAL VAPOUR DEPOSITION WITH MAGNETRON SPUTTERING TECHNIQUES

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DEDICATION

IN THE NAME OF ALLAH, ALMIGHTY GOD

To my lovely father and mother, Azali bin Ab Rahaman and Ramlah binti Ramli who always give me unconditional support, love, motivation and prayers for over the years. To my family and my dearest Hamizah, who gives me inspirations and relentlessly to remind me things are possible to complete and achievable. Thank you for all your love and support. This thesis is dedicated to all of you.

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ABSTRACT

In this study, the feasibility of using very high frequency (100 MHz to 200 MHz) Plasma Enhanced Chemical Vapour Deposition (VHF-PECVD) technique to deposit crystalline silicon carbide (SiC) thin films was investigated. High quality crystalline SiC film is very challenging to be produced since high temperature was always involved in the conventional deposition technique such as PECVD and Magnetron Sputtering. The VHF-PECVD technique with frequency up to 200 MHz was used to deposit the SiC thin films since only few attempts has been made using frequency higher than 150 MHz with silane (SiH4) and methane (CH4) as precursor gasses. The deposition time, Radio Frequency (RF) power and temperature were fixed at 900 seconds, 30 W and 180 ^{o}C, respectively. For comparison purpose, the well-established RF-Magnetron Sputtering technique using deposition parameters which can produce similar crystalline silicon carbide thin films was also used at RF power of about 20 W, 30 W and 150 W. Attenuated Total Reflectance Fourier Transform Infrared (ATR-FTIR) spectroscopy and Raman spectroscopy were used to determine the phase transition of films structure from amorphous to crystal. Further analysis using X-Ray Diffraction (XRD) was performed to determine the type of crystallites in the films for both deposition techniques. SiC (109) and SiC (205) were found in both techniques, but in contrast SiC (104) and SiC (103) were only formed in RF-Magnetron Sputtering and VHF-PECVD, respectively. Field Emission Scanning Electron Microscope (FESEM) and Atomic Force Microscope (AFM) were used to study the morphology of the films. Samples synthesized by VHF-PECVD were found to produce higher root-mean-square surface roughness as compared to RF-Magnetron Sputtering with a value of 12 nm and 0.70 nm, respectively. For the same 900 s deposition time, VHF-PECVD gives a better deposition rate which produced thicker films of about 276 nm as compared to 67 nm obtained with the RF-Magnetron Sputtering.

ABSTRAK

Dalam kajian ini, kebolehlaksanaan menggunakan teknik Pemendapan Wap Kimia Peneguhan Plasma (VHF-PECVD) berfrekuensi sangat tinggi (100 MHz hingga 200 MHz) untuk menghasilkan filem tipis kristal silikon karbida (SiC) telah dikaji. Penghasilan kristal filem SiC yang berkualiti tinggi adalah sangat mencabar kerana suhu yang tinggi sentiasa terlibat dalam teknik pemendapan konvensional seperti PECVD dan Percikan Magnetron. Teknik VHF-PECVD dengan frekuensi sehingga 200 MHz telah digunakan untuk memendap filem tipis SiC kerana penggunaan frekuensi lebih daripada 150 MHz bersama gas silana (SiH4) dan metana (CH4) sebagai gas precursor jarang dicuba. Tempoh masa mendapan, kuasa frekuensi radio (RF) dan suhu masing-masing telah ditetapkan masing-masing pada 900 saat, 30 W dan 180 ^{o}C. Untuk tujuan perbandingan, teknik mapan Percikan RF-Magnetron telah juga digunakan untuk memendap filem tipis hablur silikon karbida yang sama dengan kuasa RF pada 20 W, 30 W dan 150 W. Spektroskopi Inframerah Transformasian Fourier – Pantulan Penuh Dikecilkan (ATR-FTIR) dan spektroskopi Raman telah digunakan untuk menentukan peralihan fasa struktur filem dari amorfus kepada hablur. Analisis seterusnya menggunakan Pembelauan Sinar-X (XRD) digunakan untuk menentukan jenis hablur dalam filem untuk kedua-dua teknik pemendapan. SiC (109) dan SiC (205) telah terbentuk dikedua-kedua teknik, sebaliknya hanya SiC (103) dan SiC (104) sahaja telah terbentuk masing-masing untuk teknik Percikan RF-Magnetron dan VHF-PECVD. Mikroskopi Elektron Pengimbasan Pancaran Medan (FESEM) dan Mikroskopi Daya Atom (AFM) digunakan untuk mengkaji morfologi filem. Sampel yang dihasilkan oleh teknik VHF-PECVD menghasilkan kekasaran permukaan RMS yang lebih tinggi sekitar 12 nm berbanding sampel yang dihasilkan oleh Percikan RF-Magnetron sekitar 0.70 nm. Untuk masa pemendapan 900 saat yang sama, VHF-PECVD menghasilkan kadar pemendapan yang lebih baik dengan ketebalan filem tipis lebih kurang 276 nm berbanding dengan 67 nm yang diperolehi dengan teknik Percikan RF-Magnetron.

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LIST OF ABBREVIATIONS

AFM - Atomic Force microscopy
a-SiC:H - Amorphous Hydrogenated Silicon Carbide
ATR - Attenuated Total Reflectance
BSE - Back Scattered Electron
c-SiC:H - Crystalline Hydrogenated Silicon Carbide
Cz - Czochralski
FCC - Face Centered Cubic
FESEM - Field Emission Scanning Electron Microscope
FTIR - Fourier Transform Infrared
FWHM - Full Width Half Maximum
IR - Infra Red
MEMS - Micro Electro Mechanical System
PECVD - Plasma Enhanced Chemical Vapour Deposition
PVD - Physical Vapour Deposition
RF - Radio Frequency
RMS - Root Mean Square
SE - Secondary Electron
SiC:H - Hydrogenated Silicon Carbide
SK - Stranski and Krastanov
TO - Transverse Optical
VHF - Very High Frequency
VW - Volmer and Weber
XRD - X-ray Diffraction
XRR - X-ray Reflectivity

LIST OF SYMBOLS

a - Amorphous
Å - Angstrom
Ar - Argon
B - Boron
c - Crystal
C_H - Hydrogen Content
CH_4 - Methane
cm - Centimeter
cm^3 - Centimeter Cubic
d - Spacing Between Atomic Planes
D - FWHM XRD peak
E_1 - Original IR Signal
E_2 - Shift of IR Signal
eV - Electronvolt
f - Frequency
F_{Total} - Total Force Between Tip and Sample
H - Hydrogen
HF - Hydrofluoric Acid
k - 1000
k - Constant for XRD measurement
k - Spring Constant
m - Mass of Cantilever
MHz - Megahertz
mTorr - Millitorr
mW - Milliwatt
N_2 - Nitrogen
nm - Nanometer
O_2 - Oxygen
^oC - Degree Celcius
P - Phosphorus

Pa - Pascal

R - Hydrogen Dilution Ratio

R_a - Average Surface Roughness

SiC - Silicon Carbide

SiH_4 - Silane

SiO_2 - Silicon Dioxide

W - Watt

X_c - Raman Crystallinity

Z - Atomic Number

z - Distance Between Tip of Cantilever and Sample

δ - Skin Depth

θ - Angle

λ - Wavelength

$\mu_\circ$ - Permeability of Vacuum

μm - Micrometer

μ_r - Relative Permeability of Sample

ρ - Bulk Resistivity

τ - Grain Size

$\omega_\circ$ - Free Resonance Frequency

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CHAPTER 1

INTRODUCTION

1.1 Research Background

Nowadays, nanostructure material has been a great interest of fabrication due to quantum confinement effect as their size and dimension scale down to nanoscale level. Chemical and physical properties of nanostructure material is significantly different from those bulk and micro-size material [1]. The physical properties enhanced when the material scale down to nanoscale as demonstrated from previous studies [2]–[4]. It makes the nanostructure material received more attention from worldwide researchers. One of the nanostructure material that most popular is the silicon (Si) based material since the availability of semiconductor production processes are mostly corresponding to the technique used to fabricate the material.

Silicon carbide (SiC) is one of the nanomaterial of silicon based which get high interest as it becomes the essential material for micro and nanoelectromechanical system (MEMS and NEMS) sensors [5]–[9] for harsh environments specifically due to its optical, mechanical, excellent electrical properties with wide band gap (3eV), high breakdown field strength that is 10 times higher than Si and low intrinsic carrier concentration [6], [10]. SiC also is a well-known material that has speciality on excellent thermal conductivity and high resistance to shock [11]. In addition, crystalline SiC films most optimal used for optoelectronic [12], [13] and ultra-violet wavelength emissions that operate at high power level, high temperature environments, wide band gap (E_g), great thermal properties and large bonding energy [14]. The wide band gap would minimizing minority carrier effect at high temperature [15].

Many semiconductor devices used hydrogenated amorphous SiC (a-SiC:H) films as their composite material [16]. The examples of semiconductor devices are solar cell [17], [18], light emitting diode, colour sensor [19], photo-modulator devices [20] and metal insulator semiconductor structures [21], [22]. SiC also biocompatible with blood as coating material of artificial joints [11].

Plasma Enhanced Chemical Vapour Deposition (PECVD) was among the popular technique to grow SiC film. Here, Silane (SiH_4) gas is the most preferred source for Si precursor. Majority of previous studies shown that methane (CH_4) gas was used as C precursor for SiC films. Hydrogen and argon are usually used as carrier gas [23]–[25]. The ratio of SiH_4:CH_4 and substrate temperature are important for the growth rate of SiC thin films. PECVD uses higher frequency than the conventional (13.56 MHz) can be considered as a very high frequency PECVD (VHF-PECVD). Recently, the higher excitation frequency (f_{exc}) is used for growth of SiC thin films because of their several advantageous such as grown with high deposition rate, deposit with good quality, higher electron density, lower plasma potential and reduced ion bombarded [26]–[28].

J.Huran et al (2013) reported the growth of a-SiC:H films by PECVD using CH_4 and SiH_4 as reactants with various substrate temperature (200^{o}C, 250^{o}C, 300^{o}C, 350^{o}C). The structural properties were investigated by RBS, ERD, IR and Raman methods. This study found that the concentration of hydrogen decreased with the increased of deposition temperature. The deposition temperature also gives small influence to the refractive index value. However, the frequency used is not mentioned in this study [24].

I. Torres (2011) had produced a-SiC_x:H thin films deposited on a c-Si double-sided polished substrate of a p-type with 300 µm thickness. The chamber pressure used was 375 mTorr with 400^{o}C deposition temperature, 13.56 MHz radio frequency (RF) and 10 mWcm^2 of RF power. The thin films were compared with doped (phosphorous) and undoped. The temperature was increased from 27^{o}C until 900^{o}C. From those parameters, this study concluded that temperature range between 27^{o}C to 800^{o}C exhibit the same behaviour (as amorphous). At 900^{o}C, the thin films shown crystalline orientation as observed by XRD. Although, the phosphorous-doped accelerated the crystallization in the films but the undoped thin films by VHF-PECVD would give less cost [29].

Y.T Kim et al (2005) had synthesized the a-Si:H and a-SiC:H on Si substrate using a capacitively coupled with RF PECVD system. The substrate temperature was kept at 150^{o}C and the RF power was varied from 50 to 400W. For the growth of a-SiC:H films, SiH_4 and CH_4 were utilized as the precursor gases and finally the annealing process was performed at high temperature. It showed that the crystallinity of a-SiC:H films start to form at above 900^{o}C. Thus, the forming of SiC crystallity depends on the annealing process with the low frequency used [23].

C.Summonte et al (2004) used VHF-PECVD to fabricate a-SiC:H alloys with SiH_4 and CH_4 as precursors. The 100 MHz frequency was used with flow rates of SiH_4 and CH_4 set at 8 sscm and 25 sscm respectively. The result of this study shown that, the high amount of SiC bond concentration was found on that sample. However, no structural properties of the material [28] were studied. Lei Liu et al (2011) utilized PECVD at 13.56 MHz frequency to deposit SiC thin films. At 900^{o}C, there were no crystalline peaks shown. However, at 1200^{o}C, there were peaks at 33.75^{o} and 60.16^{o} attributed to SiC oriented crystallization [30]. Hence, with lower frequency, higher temperature is needed to grow crystalline SiC.

Dong S. Kim et al (1994) reported that the hydrogen content of a-SiC:H films decreased as the annealing temperature exceeded 400^{o}C. The a-SiC:H films was deposited by PECVD (13.56MHz). The annealing temperature started from 200^{o}C until 400^{o}C. Guozhi Wen et al (2016) reported that crystalline β-SiC (111) QDs had been growth by 900^{o}C annealed temperature and appeared at 33.9^{o} on the XRD spectra [31]. For sputtering, usually post annealing is needed to change the phase from amorphous to crystalline if the film deposited at lower RF power, otherwise, higher temperature is needed during deposition compared to PECVD [32] [33] [34].

From the previous study, most researchers used the conventional PECVD (13.56MHz) [35]–[38] with SiH_4 and CH_4 as precursor gas to deposit SiC thin film. The highest frequency from the previous research was 100 MHz. Furthermore, correlation of RF power and the crystallinity of the samples also had been studied. For PECVD, P. Pratim et al (2002) have shown that the transition of hydrogenated films to form a crystalline film occurred as the RF power reach to 285 mW/cm^2 [39]. The magnetron sputtering also provide similarities results. Y. Bouizem et al (2013) reported that, the transition can be seen into crystalline silicon film when RF power increased from 180 W to 200 W [40]. In addition, the deposition rate also achieved ranging from 1.0 nm/s to 9 nm/s with deposition power from 80 W and 160 W [41].

Apart from temperature, pressure also plays important role. Different working pressure can provide significant change to the sample. In sputtering, according to G. Lihui et al (1998), at 13.56 MHz, deposition rate improved from 0 Torr until 4 Torr, then decreased when the pressure increased to 8 Torr [42]. Deposition pressure also gives a significant effect to the deposition rate, as chamber pressure will affect the mean free path of adatoms. According to S.B Hashim et al (2012), film deposited by RF sputtering shown decrement of deposition rate as deposition pressure increased from 5 Torr to 8 Torr [43].

In industry, the production of SiC electronic devices depends on the structure of the SiC thin films. It can be crystalline, amorphous and polymorphs. Different SiC polytypes (6H-SiC, 4H-SiC and 3C-SiC) of the piezoresistivity have been studied since these materials are advantageous on thermal stability and chemical inertness which allow the preservation of piezoresistivity at harsh condition [44].

In this research, the feasibility of VHF-PECVD was investigated to grow SiC thin films and follow by the characterization of the morphology, structural and crystallinity of the films. Thus, conventional RF Magnetron sputtering also will be included and discussed together with the VHF-PECVD to compare the structural properties and morphology of the films.

1.2 Problem Statement

Silicon Carbide (SiC) is identified as the great material for high power, high thermal conductivity, wide band gap, high electron mobility and high-saturated electron velocity. SiC have inherent outstanding mechanical and chemical properties that make it a material of great interest for mechanical structures in micro and nanoelectromechanical system (MEMS and NEMS) applications.

Recently, SiC thin films was grown by various method such as hot wire chemical vapour deposition (HWCVD) [45], chemical vapour deposition (CVD), gas-source molecular beam epitaxy (MBE), electron cyclotron resonance (ECR) plasma [46] and many more. Crystalline SiC films usually can be deposited by high temperature of CVD technique (up to 1300 ^{o}C), but the limitation of Si based technology decrease their compatibility [47]. Thus, the VHF-PECVD method is the high interest as it uses lower temperature and higher frequency (above 13.56MHz) to deposit the high quality of SiC compared to other methods.

Previous study used conventional PECVD (13.56MHz) with high annealed temperature to grow a-SiC:H thin films. The higher amount of hydrogen gives less performance of MEMS device since it reduce the Q factor. The lower frequency of PECVD needs higher temperature to grow high crystallinity of SiC thin films. The process of annealing boosts the process of crystallinity of SiC thin films. However, the annealed temperatures until 1200^{o}C is very high and affect the thin films quality and can induce residual stress. The low temperature is very important from the point of view of device integration. The lower deposition temperature with highly conductive SiC thin films has been a goal of many researches [48].

The VHF plasma excitation ranging up to 200 MHz produce unknown film morphology condition and deposition rate. These two conditions are significantly important in the solar cell performance [109-110]. C. Das et al. stated that, the surface roughness is related with the crystallinity in the sample. After all, it will reduce as the deposition temperature increase [111]. Regardless, it is opportunity to determine the effect of VHF plasma excitation toward surface roughness. On the other hand, deposition rate and morphology condition for film grown by RF-Magnetron Sputtering without involvement of high temperature could be known.

In this study, the VHF-PECVD with frequency up to 200 MHz is used to deposit the SiC thin films as no attempt has been made using frequency higher than 150 MHz with SiH_4 and CH_4 as precursor gas. It is predicted that, VHF can produce higher SiC crystallinity at much lower temperature. Meanwhile, RF Magnetron sputtering needs either higher temperature or post annealing process to produce crystalline SiC thin films. Therefore, the deposition of SiC thin films by both methods with lower temperature and similar deposition time will provide useful information in term of morphology and structural properties.

1.3 Objectives

The objectives of the research are:

(a) To determine the crystallite type of the SiC thin films deposited using VHF-PECVD and RF Magnetron sputtering under various growth conditions by using Raman Spectroscopy, FTIR and XRD.
(b) To investigate the structural properties of SiC thin films deposited using VHF-PECVD and RF Magnetron sputtering by using AFM, EDX and FESEM.
(c) To determine the thickness and deposition rate of SiC thin films deposited using VHF-PECVD and RF Magnetron sputtering by using FESEM.

1.4 Scope of the Study

SiC thin films on Si substrate will be grown by VHF-PECVD and RF Magnetron Sputtering techniques. SiH_4 and CH_4 gases are used in VHF-PECVD as precursors and argon as the carrier gas. While Ar and N_2 are used in RF Magnetron Sputtering for plasma generation and venting chamber respectively. In VHF-PECVD, the power density, flow rate of gas, temperature and pressure will be kept constant. In addition, the frequency will be varied for up to 200 MHz. For both techniques the deposition time will be set for 900 seconds. The morphology and structural properties are characterized by Raman Spectroscopy, X-Ray Diffraction and Atomic Force Microscopy. Field Emission Secondary Electron (FESEM) Microscopy was used to observe the cross-sectional view and to measure the thickness of the thin films sample.

1.5 Significance of the Study

This research will give some insight of the important parameters of SiC films deposited using VHF-PECVD with specific crystal orientation. The hydrogenated amorphous SiC (a-SiC:H) is mostly used in semiconductor devices such as limiting emitter diode, low cost production for photovoltaic application [49] and photoelectrode [50]. The crystalline of SiC thin films is important for application in MEMS and NEMS. The elimination of hydrogen is important to increase the performance of MEMS devices. From this research, the understanding of the crystal transition and the polytypes of SiC thin films would be established. On the other hand, this will be beneficial in order to understand the effect of high frequency deposition with low defect densities [51]. All the data also discussed along with RF Magnetron sputtering samples. Thus, the two techniques can be compared and give extra information regarding the SiC thin films deposition.

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