THE MORPHOLOGY AND STRUCTURAL PROPERTIES OF SILICON CARBIDE QUANTUM DOTS GROWN BY VERY HIGH FREQUENCY PLASMA ENHANCED CHEMICAL VAPOUR DEPOSITION

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This thesis dedicated to
My precious parents Abd Karim and Hasnah.
My beloved husband Mohd Syafiq.
My cutie pie Luthfiyaa Eryna.
Thank you...

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ABSTRACT

The growth and characterization of silicon carbide (SiC) quantum dots (QDs) are reported in this work. The SiC QDs were grown using plasma enhanced chemical vapour deposition (PECVD) at 150 MHz radio frequency (RF). A mixture of silane (SiH_4) and methane (CH_4) with a ratio of 1:4 and diluted in hydrogen (H_2) was used as precursor gaseous. By manipulating the growth parameters such as hydrogen flow rate, growth temperature, growth time and RF power, the morphological and structural properties of SiC QDs were studied. The surface morphology of samples was observed through atomic force microscopy (AFM) and field emission scanning electron microscopy (FESEM). The structural properties of the sample were determined using Fourier transform infra-red (FTIR), emission dispersive x-ray (EDX) and Raman spectroscopy. It was found that a combination of dots formed islands on the substrate and the size of each dot was below 50 nm. By observing the cross-section view of the substrate, this self-assembled quantum dots followed Stranski-Krastanow (S-K) mode in which a combination of both islands and layer mode was formed. FTIR result showed absorption peaks located at 500 to 1000 cm^{-1} which proved the presence of SiC bonding. Further elemental mapping by EDX confirmed that the island and the layer underneath were formed from silicon, carbon and oxygen. This is in agreement with the observed SiC QDs cross-sectional FESEM image. The Raman spectra revealed three SiC polytypes which were 3C-SiC, 4H-SiC and 6H-SiC, and therefore in agreement with XRD results. All polytypes were considered as crystal due to small full width half maximum (FWHM) with crystallite size greater than 1 nm and sharp peaks were formed on Raman spectra. In conclusion, the SiC QDs growth parameters have shown a good impact to the morphological and structural properties of the grown quantum dots.

ABSTRAK

Pertumbuhan dan pencirian titik-titik kuantum (QDs) silikon karbida (SiC) dilaporkan dalam kerja ini. Titik-titik kuantum SiC telah ditumbuh menggunakan pemendapan wap kimia diperkuat plasma (PECVD) pada frekuensi radio (RF) 150 MHz. Campuran silana (SiH_4) dan metana (CH_4) dengan nisbah 1:4 dan dicairkan dalam hidrogen (H_2) telah digunakan sebagai gas pelopor. Dengan memanipulasi parameter pertumbuhan, seperti kadar aliran hidrogen, suhu pertumbuhan, masa pertumbuhan dan kuasa RF, ciri-ciri morfologi dan struktur titik-titik kuantum SiC telah dikaji. Morfologi permukaan sampel dicerap melalui mikroskopi daya atom (AFM) dan mikroskopi elektron pengimbasan pancaran medan (FESEM). Ciri-ciri struktur sampel ditentukan menggunakan transformasi Fourier infra-merah (FTIR), sebaran pancaran sinar-X (EDX) dan spektroskopi Raman. Didapati bahawa kombinasi titik-titik membentuk pulau-pulau pada substrat dan saiz setiap titik adalah di bawah 50 nm. Dengan mencerap pandangan keratan rentas substrat, titik kuantum yang terhimpun-sendiri ini mengikuti mod Stranski-Krastanow (S-K) di mana satu gabungan kedua-dua pulau dan mod lapisan dibentuk. Keputusan FTIR menunjukkan puncak penyerapan terletak pada 500 hingga 1000 cm^{-1} yang membuktikan kehadiran ikatan SiC. Pemetaan unsur seterusnya menggunakan EDX mengesahkan bahawa pulau dan lapisan di bawahnya adalah dibentuk daripada silikon, karbon dan oksigen. Ini adalah sepadan dengan cerapan imej keratan rentas FESEM titik-titik kuantum SiC. Spektra Raman menunjukkan tiga polijenis SiC iaitu 3C-SiC, 4H-SiC dan 6H-SiC, dan oleh itu selaras dengan keputusan XRD. Kesemua polijenis ini dianggap sebagai hablur kerana lebar penuh setengah maksimum (FWHM) yang kecil dengan saiz hablur lebih besar daripada 1 nm dan puncak yang tajam terhasil pada spektra Raman. Kesimpulannya, parameter pertumbuhan titik-titik SiC telah menunjukkan impak baik kepada morfologi dan struktur titik-titik kuantum yang ditumbuhkan.

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μs	- strain energy
0D	- zero-dimensional
1D	- one dimensional
2D	- two-dimensional
3D	- three-dimensional
a_e	- lattice constant of epitaxial layer
a_s	- lattice constant of substrate
d	- lattice distance
DOS	- density of state
E_i	- energy quantized state
f	- lattice mismatch
γ_e	- surface energy
γ_{es}	- interface energy
γ_s	- surface energy of the substrate
m^*	- electron mass
n	- integer
q_B	- maximum wave vector of the Brillouin Zone of 3C SiC
t_c	- critical thickness
δ	- function at descrete energy
λ	- wavelength of incident x-ray
λ_{db}	- de Broglie’s wavelength
ρ	- Momentum
χ	- reduce wave vector
AFM	- atomic force microscopy
Ar	- Argon gas

ATR - attenuated total reflection
CBL - Cluster-Bethe-lattice
CH_4 - methane
CH_4 - Methane gas
CVD - Chemical vapor deposition
D - crystallite size
DI-H_2O - deionized water
EDX - energy dispersive x-ray
FESEM - field emission scanning electron microscopy
FLA - folded longitudinal acoustic
F-M - Frank-van der Merwe
FTIR - fourier transform infrared
FWHM - full width half maximum
H_2 - hydrogen gas
HF - hydrofluoric acid
ICDD - International Center for Diffraction Data
IPA - isopropanol
LA - longitudinal acoustic
LED - Light emitting diode
LO - longitudinal optical phonon
LO - longitudinal optical phonon
MBE - molecular beam epitaxy
MFC - mass flow controller
N_2 - nitrogen gas
NEMS - Nanoelectromechanical system
PDF - powder diffraction file
PECVD - plasma enhanced chemical vapor deposition
RAMAN - Micro-raman spectrometry

RF - radio frequency

R_{rms} - Surface roughness

Si - Silicon

SiC - Silicon Carbide

SiH_4 - Silane gas

SiO_2 - silicon oxide

S-K - Stranski-Krastanow

TEC - Thermoelectric cooling

TEM - Transmission electron microscopy

TMP - turbo molecular pump

TO - transverse optical phonon

T_g - growth temperature

T_s - Growth time

VHF-PECVD - Very high frequency plasma enhanced chemical vapor deposition

V-W - Volmer-Weber

XRD - X-ray diffraction

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CHAPTER 1

INTRODUCTION

1.1 Overview

This chapter gives an overview about background, problem statement, objectives, scope, significant and contribution of this research. Finally, the thesis outline is presented toward the end of this chapter.

1.2 Background of Research

In the past two decades, there has been a great interest of nanostructures materials fabrication. The materials comprising of submicron or nanoscale size with at least at one dimension and exhibit the size effect, had received much attention by researchers due to its interesting characteristics and properties compared to bulk materials (Tiwari *et al.*, 2012). Silicon (Si) based materials are the most popular candidate since the techniques used for fabrication are largely correspond with available semiconductor production processes. However, its crucial limitation where it is not well-matched to handle large current densities and high voltage open up an opportunity to new materials of wide band gap such as silicon carbide (SiC) (Mukherjee, 2011).

Silicon carbide (SiC) is regarded as a promising substitute for silicon especially in high power, high temperature and high frequency devices. This emerging semiconductor material has received a great deal of attention due to its unique properties which fit for application in optoelectronics and microelectronics (Fan *et al.*, 2006; Cheng *et al.*, 2007) such as light emitting diode, electroluminescent devices, nanoelectromechanical system (NEMS) sensors fabrication and also thermoelectric cooling (TEC) devices for deployment in extreme environments (Saddaw, 2012). The most recent application of nanostructures SiC is quantum dots structures in optical device fabrication such as blue LED (Willander *et al.*, 2006).

Generally, thin film deposition method can be used to grow quantum dots structures. Si quantum dots structures are known to be grown with various methods. There are three type of quantum dots synthesis method which are solution-based chemical methods, chemical vapour methods and physical vapour deposition (Fan *et al.*, 2012). Now, quantum dots can be synthesized by chemical colloidal method, self-assembly method, lithography and etching, and split-gate approach.

Through chemical colloidal method, multilayered quantum dots can be obtained and this process is suitable for mass production. In self-assembly method, chemical vapour deposition process (CVD) or molecular beam epitaxy (MBE) is utilized. In this method, the principle of lattice mismatch is applied to ensure the growth of quantum dots can undergo self-polymerization in a particular substrate. This technique is applicable for regularly arranged quantum dots mass production.

By electron beam lithography and etching, quantum dots can be fabricated by direct write of the electron beam to the photoresist on the substrate and followed by etching the substrate. However, it is time consuming and not suitable for mass production. The two-dimensional plane of the quantum well is generated from two-dimensional confinement when external voltage is applied is called split-gate approach. This approach is fit for academic research but not for mass production (Shi *et al.*, 2015).

Cheng *et al* (2007) had synthesized self-assemble SiC quantum dots grown on Si substrate by low-frequency inductively coupled plasma assisted RF magnetron sputtering. The effects of SiC target power and gas pressure on surface morphology and structural properties of SiC quantum dots were investigated. They discovered the growth dynamics of quantum dots obeys cubic root-law behavior. This law state that when an object undergoes a proportional increase in size, its new surface area is proportional to the square of multiplier and its new volume is proportional to the cube of multiplier. Thus, as the dimension increased, the volume will continue grow faster than the surface area. Moreover, the SiC quantum dots morphology is highly uniform and the average size increases as the pressure increase, below 1 Pa. Yet, the morphology became non-uniform and decreases in sized as pressure exceed 1 Pa. These behaviors attributed to scattering effect and surface mobility of the sputtered atoms (Cheng *et al.*, 2007).

In 2012, a group of researchers, Fan *et al* had fabricated SiC quantum dots from three different type of silicon carbide which are 3C, 6H and 4H SiC polytypes by electrochemical method and studied the dots based on its photoluminenscence. They noticed those polytypes show unexpected quite-similar photoluminescence, photoluminescence excitation, and transient photoluminescence properties which can be explained by polytypic transformations of the colloidal SiC quantum dots driven by ultrasonic waves. Although this method can produce SiC quantum dots but the size of the dots is varied from 1 nm to 8 nm. (Fan *et al.*, 2012). Both research done by Cheng *et al* in 2007 and Fan *et al* in 2012 did not discuss the type of quantum dots obtained whether it is amorphous or crystalline.

Mwania *et al* (2013) had produced quantum sized cluster of β-SiC by photo-assisted electrochemical corrosion of bulk powders. Transmission electron microscopy (TEM) result shows that β-SiC quantum dots are single crystalline. The process which is the amount of synthesized quantum dots and its size can be controlled via regulating the deposition time. However, indirect chemical etching through some of the in situ generated oxidizing species which cannot be excluded completely. Also, it requires external bias potential in order to control the size of the dots (Mwania *et al.*, 2013).

3C-SiC quantum dots fabricated via pulsed laser ablation method was carried out by Zhu *et al* in 2014. They successfully produced bulk quantity of crystalline 3C-SiC quantum dots with uniform diameter of about 2 nm. Even though they had fabricated crystalline quantum dots with uniform diameter, but the distribution of the quantum dots obtained is not being observed and discussed. Moreover, the 6H-SiC target used in their experiment to fabricate the dots is quite expensive (Zhu *et al*., 2014).

This research aims to grow SiC quantum dots film over large areas (substrate) which has been predicted to have a crystalline quantum dots structure. Very high frequency plasma enhanced chemical vapour deposition, 150MHz (VHF-PECVD) technique will be utilized to deposit SiC quantum dots film in which variation in chamber pressure, substrate temperature, RF power and precursor gases flow rate will be investigated in order to get a crystalline quantum dots with uniform size and distribution. To the best of our knowledge, the report on SiC quantum dots structure growth by VHF-PECVD is not widely discovered.

Characterization of the growth quantum dots will be carried out to study the surface morphology, structural information and elemental composition using various techniques that serve the required characterization purposes. SiC quantum dots are expected to deposit with uniform size and distribution. Moreover, it is predicted to possess better structural as well as preserving the superior crystalline properties.

1.3 Problem Statement

Silicon carbide has many superior properties which make it a good candidate for semiconductor applications such as power transistor, thermoelectric, optoelectronics and as coating materials.

Many synthesis methods have been done by researchers in order to produce SiC quantum dots such as electrochemical technique, laser ablation, RF magnetron sputtering and electrochemical corrosion. Even though the size of quantum dots fabricated via RF magnetron sputtering has uniform and average size but it needs high power which is above 100 W and longer deposition time between 20 to 60 minute (Cheng *et al*., 2007).

An ultrasmall quantum dots whose diameter below 1 nm had fruitfully obtained by electrochemical etching technique. The ultrasmall quantum dot was stem from bulk of SiC. However, only one type of SiC that can be produced from one bulk SiC. A different type of bulk SiC is needed in order to have various SiC polytypes (Fan *et al*., 2012). Similar to the electrochemical technique, photo-assisted technique also produce one polytype at once depend on the SiC polytype powder used. Moreover, there is formation of oxidizing species which cannot be excluded which affected the properties of quantum dots produced. This method also needs an external bias potential to control the dots size (Mwania *et al*., 2013).

Pulsed laser ablation technique had successfully produced 3C-SiC crystalline quantum dots with a uniform size of 2 nm. This technique needs high temperature and pressure during laser ablation which is analogous to thermal evaporation for SiC polytype from one (target) to another polytypes. For this technique, only one type of SiC polytype can be transformed at a time. In addition, the target used for this technique is expensive (Zhu *et al.*, 2014).

Based on previous research, the quantum dots formations are strongly dependent on the growth technique and the selected growth parameter. High temperature, pressure and power are needed to produce quantum dots. Some of the techniques can only produce one polytype at a time and depend on the polytypes of bulk or target used. In addition, the structural properties of SiC quantum dots obtained are not widely discussed and most researchers only focus on the fabrication technique and photoluminescence of the SiC quantum dots.

Therefore, in this research various polytypes of SiC quantum dots will be grown via VHF- PECVD at low temperature and limited growth time. The morphology and structural properties of the dots will be analyzed and discussed.

1.4 Research Objectives

i. To grow silicon carbide quantum dots on silicon substrate using very high frequency chemical vapour deposition method.

ii. To optimize the growth parameters (growth temperature, RF power, hydrogen flow rate and growth time) of silicon carbide quantum dots.

iii. To characterize the morphology and structural properties of the deposited silicon carbide quantum dots.

1.5 Scope of Research

The fabrication of self-assembled SiC quantum dots was carried out using VHF-PECVD technique at 150 MHz in order to produce a crystalline quantum dots at low growth temperature and limited growth time. The Si substrate was used to grow the quantum dot. The structure was grown only limited to zero dimensions. The effect of varying growth temperature from 24 to 400˚C, RF power from 10 to 25 W, hydrogen flow rate from 60 to 140 sccm and growth time from 1 to 5 minutes were observed and investigated. The morphology and structural properties of the SiC quantum dots grown were characterized by Atomic Force Microscopy, Field Emission Scanning Electron Microscopy, Fourier Transform Infra-Red spectroscopy, X-ray

Diffraction Spectroscopy, Electron Dispersive X-ray, and Scanning/Transmission Electron Microscopy.

1.6 Significant of Research

Based on this research, the uniformity of dots size and distribution was controlled by 150MHz VHF-PECVD method. The different in polytypes and the size of the quantum dots are important for application in optoelectronics and microelectronics especially in extreme environment. This research would contribute to the understanding of the morphological and structural properties of SiC-quantum dots.

1.7 Thesis Outline

Chapter 1 presents some previous works that are related to this study. The problem statement, objectives, scope and significant of the research are also presented. In Chapter 2, literature reviews related with this research were discussed. This chapter covers the growth mechanism of quantum dots, Si and SiC as semiconductor materials, fundamental of PECVD method and the precursor gases used in SiC quantum dots growth. Chapter 3 described the methodology which included the fabrication of SiC quantum dots, operation procedure for VHF-PECVD system and followed by characterization techniques in order to observe the morphological and structural properties of SiC quantum dots. In Chapter 4, the results from each characterization were analysed. The influenced of each parameter upon quantum dots growth were critically studied. The conclusions and suggestions are stated in Chapter 5.

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