# SEEDLESS AND CATALYST- FREE GROWTH OF ZINC OXIDE NANOSTRUCTURES ON GRAPHENE BY THERMAL EVAPORATION

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To my beloved late father and mother,
*Ahmad Mohd Noor*
*Bariah Mat Ali*

My lovely siblings,
*Salwa Ahmad*
*Busra Ahmad*
*Nurul Farhana Ahmad*
*Najiha Ahmad*
*Suhaidal Ismail*

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# ABSTRACT

Metal-oxide, namely zinc oxide (ZnO) nanostructures and thin films on graphene is interesting because these structures can offer additional functionality to graphene for realizing advanced electronic and optoelectronic applications. Graphene has a great potential for novel electronic devices because of its extraordinary electrical mobility exceeding $10^4$ $cm^2/Vs$ and a thermal conductivity of $10^3$ W/mK. Therefore, with the excellent electrical and thermal characteristics of graphene layers, the hybrid ZnO/graphene structure is expected to offer many sophisticated device applications such as sensing devices. In this study, the seed/catalyst-free growth of ZnO on single layer (SL) and multilayer (ML) graphene by thermal evaporation of Zn in the presence of oxygen ($O_2$) gas was performed. The effects of substrate temperatures, substrate positions and graphene thicknesses on the morphological, structural, and optical properties were found to be very pronounced. The grown ZnO structures exhibit three different structures, i.e., nanoclusters, nanorods, and thin films at 600°C, 800°C, and 1,000°C, respectively. By setting the substrate to be inclined at 90°, the growth of ZnO nanostructures, namely nanoclusters and nanorods, on SL graphene was successfully realized at temperatures of 600°C and 800°C, respectively. However, no growth was achieved at 1,000°C due to the possible severe oxidation of graphene. For the growth on ML graphene at 600°C with an inclination angle of 90°, the grown structures show extremely thick and continuous cluster structures as compared to the growth with substrate's inclination angle of 45°. Moreover, the base of nanorod structures grown at 800°C with an inclination angle of 90° also become thicker as compared to 45°, even though their densities and aspect ratios were almost unchanged. The morphologies of grown structures at 1,000°C with an inclination angle of 90° do not show significant difference with 45°. The intensity ratio of UV emission ($I_{UV}$) and visible emission ($I_{VIS}$) was changed, depending on the temperature. The structures grown at a low temperature of 600°C show the highest value of $I_{UV}/I_{VIS}$ of 16.2, which is almost two times higher than the structures grown on SL graphene, indicating fewer structural defects. From the results obtained, the temperature below 800°C, substrate position inclined at 90° towards the gas flow, and ML graphene seems to be preferable parameters for the growth of ZnO structures by thermal evaporation because these factors can overcome the problem of graphene's oxidation that takes place during the growth.

# ABSTRAK

Logam-oksida, iaitu zink oksida (ZnO) berstruktur nano dan filem nipis di atas grafin amat menarik kerana ia boleh menawarkan fungsi tambahan kepada grafin untuk merealisasikan aplikasi elektronik dan optoelektronik maju. Grafin mempunyai potensi besar untuk peranti elektronik novel kerana mobiliti elektrik yang luar biasa melebihi $10^4$ $cm^2/Vs$ dan kekonduksian terma $10^3$ W/mK. Oleh itu, dengan ciri-ciri elektrik dan haba yang sangat baik dari lapisan grafin, struktur hibrid ZnO/grafin dijangka menawarkan banyak aplikasi peranti canggih. Dalam kajian ini, pertumbuhan ZnO bebas pemangkin/benih di atas grafin berlapisan tunggal (SL) dan lapisan berganda (ML) menggunakan penyejatan haba Zn dalam kehadiran oksigen ($O_2$) gas telah dilakukan. Kesan suhu substrat, kedudukan dan ketebalan substrat grafin pada sifat-sifat morfologi, struktur, dan optik didapati sangat ketara. Pada dasarnya, struktur ZnO yang tumbuh menunjukkan tiga struktur berbeza, iaitu nanokluster, nanorod, dan filem nipis masing-masing pada 600°C, 800°C, dan 1,000°C. Dengan menetapkan substrat condong pada 90°, pertumbuhan struktur-struktur nano ZnO iaitu nanokluster dan nanorod pada SL grafin telah berjaya direalisasikan pada suhu 600°C dan 800°C. Walau bagaimanapun, tiada pertumbuhan dicapai pada 1,000°C berkemungkinan akibat daripada pengoksidaan grafin yang teruk. Untuk pertumbuhan di atas ML grafin pada 600°C dengan sudut kecondongan 90°, pertumbuhan menunjukkan struktur kelompok yang sangat tebal dan berterusan berbanding dengan pertumbuhan pada sudut kecondongan substrat 45°. Selain itu, tapak struktur nanorod yang tumbuh pada 800°C dengan sudut kecondongan 90° juga menjadi lebih tebal berbanding 45°, walaupun kepadatan dan nisbah aspek hampir tidak berubah. Struktur morfologi pada 1,000°C dengan sudut kecondongan 90° tidak menunjukkan perbezaan yang signifikan berbanding 45°. Nisbah keamatan UV ($I_{UV}$) dan sinar nampak ($I_{VIS}$) berubah bergantung kepada suhu. Struktur yang tumbuh pada suhu rendah daripada 600°C menunjukkan nilai tertinggi $I_{UV}$ / $I_{VIS}$ iaitu 16.2, yang hampir dua kali lebih tinggi daripada struktur yang tumbuh di atas SL grafin, menunjukkan sedikit kecacatan struktur. Daripada keputusan yang diperolehi, dapat disimpulkan bahawa suhu di bawah 800°C, kedudukan substrat condong pada 90 ° arah aliran gas, dan ML grafin seolah-olah menjadi parameter terbaik untuk pertumbuhan struktur ZnO oleh penyejatan haba kerana faktor-faktor ini boleh digunakan untuk mengatasi masalah pengoksidaan grafin yang berlaku semasa pertumbuhan.

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# LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| 1D | - | One dimensional |
| 2D | - | Two dimensional |
| Ar | - | Argon |
| Au | - | Gold |
| CdO | - | Cadmium Oxide |
| CMOS | - | Complementary metal-oxide semiconductor |
| CNT | - | Carbon nanotube |
| CVD | - | Chemical Vapor Deposition |
| EDS | - | Energy dispersive X-ray spectrometer |
| FESEM | - | Field emission scanning electron microscopy |
| FET | - | Field effect transistor |
| GaAs | - | Gallium Arsenide |
| GaN | - | Gallium Nitride |
| Ge | - | Germanium |
| i-SoC | - | Intelligent system-on-chip |
| MgO | - | Magnesium oxide |
| ML | - | Multi layer |
| MOVPE | - | Metalorganic vapour phase epitaxy |
| $O_2$ | - | Oxygen |
| PL | - | Photoluminscence |
| RT | - | Room temperature |
| Si | - | Silicon |

| | | |
|---|---|---|
| $Si_3N_4$ | - | Silicon Nitride |
| SiC | - | Silicon Carbide |
| $SiO_2$ | - | Silicon Dioxide |
| Si-ULSI | - | Silicon ultra-large-scale integrated circuits |
| SL | - | Single Layer |
| ST | - | Set temperature |
| TEM | - | Transmission electron microscopy |
| UV | - | Ultra violet |
| XRD | - | X-ray diffractometer |
| Zn | - | Zinc |
| ZnO | - | Zinc Oxide |

# LIST OF SYMBOLS

*Å* - Angstroms, 1Å = $1\text{x}10^{-10}$ m

*°C* - Degree celcius

*μm* - Micrometer

*cm* - Centimeter

$cm^2/Vs$ - carrier mobility

*d* - Inter-plane distance in angstroms

*eV* - Electron volt

$I_{UV}/I_{VIS}$ - Intensity ratio

*kV* - Kilo Volt

*n* - An integer 1,2,3

*nm* - nanometer

*meV* - Millielectron volt

$O_i$ - Oxygen interstitial

*sccm* - Standard cubic centimeter per minute

$V_o$ - Oxygen vacancy

$V_{Zn}$ - Zinc vacancy

$\theta$ - Diffraction angle in degree

$\lambda$ - Wavelength

*W/mK* - Thermal conductivity

$m^2/g$ - Surface area

$TPa$ - Young’s modulus

$Z_{ni}$ - Zn interstitial

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# CHAPTER 1

# INTRODUCTION

## 1.1 Research background

We are currently living in the age of silicon nanotechnology. Silicon (Si) based transistor drives a modern computing revolution year by year. The size of a transistor has been reduced consistently which allows more transistors to be packed onto a single chip, thereby increasing a computer power. This follows the Moore's Law, according to which the number of transistors on a chip is doubling approximately once every 2 years. Principally, the miniaturization of a transistor is known to be very helpful in increasing an overall efficiency of the silicon ultra-large-scale integrated circuits (Si-ULSIs). However, this unceasing miniaturization of transistors is becoming increasingly difficult owing to the several limitations such as short channel effect and gate leakage current etc.

In recent years, a concept of the advanced heterogeneous integration of the Si platform has attracted much attention towards the recognition of a ‘More than Moore’ technology [1]. To realize such technology, a growth of the high-quality elements (i.e., germanium (Ge) [2]) compound semiconductors (i.e., gallium arsenide (GaAs) [3], gallium nitride (GaN) [4], silicon carbide (SiC) [5]), metal oxides (i.e., zinc oxide (ZnO) [6]), and carbon-based materials (i.e., graphene [7], carbon nanotube (CNT) [8]) on the Si platform is highly required. The co-integration of these materials enables the present ultra-large-scale integrated circuits (ULSIs) to be facilitated not only with ultra-high speed complementary metal-oxide semiconductor

(CMOS) transistors and novel transistors, [9] but also with the various kinds of functional devices, such as optical devices [10], photodetectors [11], solar batteries [12], and sensors [13,14]. An Intelligent system-on-chip (i-SoC) on the Si is considered as a practical and promising direction. However, for the fabrication of electronic devices, an electronic insulation of these materials and the Si substrate by insulator such as silicon dioxide ($SiO_2$) is necessary. It is worth noting that, the growth of highly crystalline and highly oriented material on insulator is a challenging task towards the realization of such advanced hybrid integration on the Si platform. There is a need of some great development in the field of growth technologies. Hence, this study is going to initiate an innovative technique to grow ZnO on an insulator by utilizing graphene as a template layer. **Figure 1.1** illustrates an evolution of the Si based nano-electronics devices.

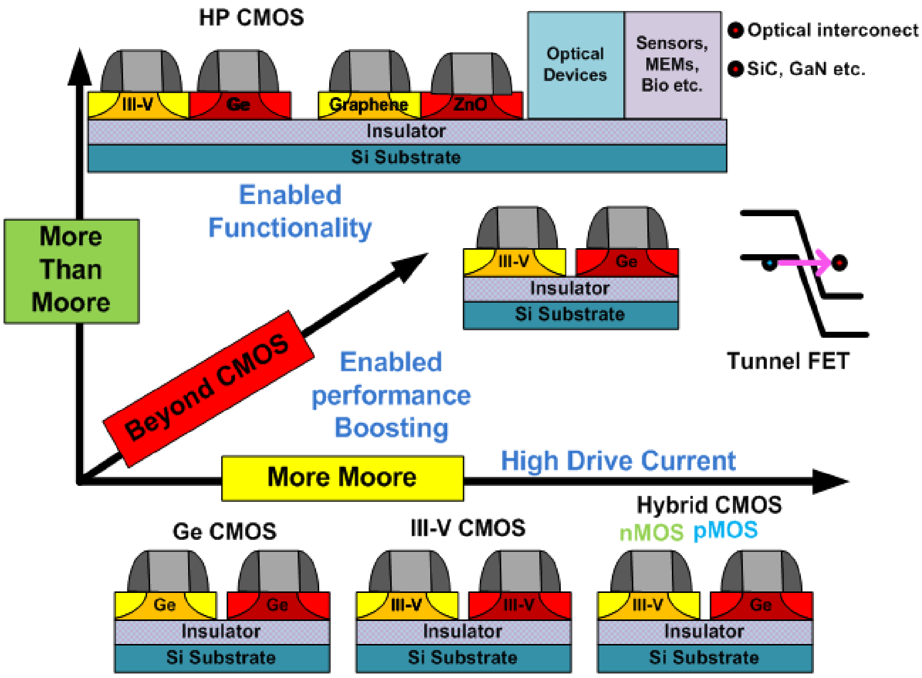

**Figure 1.1** Evolution of Si-based nano electronics [15]

## 1.2 Research motivation

Since a decade ago, intensive researches have been focused on fabricating one-dimensional (1D) zinc oxide (ZnO) semiconducting nanostructures because it can provide a variety of important applications due to their unique morphologies, compositions, and chemical/physical properties [6,16]. Besides that, ZnO possesses wide band gap and large exciton energy and it is considered to be a promising candidate for the fabrication of several kinds of devices.

Meanwhile, at a room temperature (RT), the graphene retains a high carrier mobility of up to 200,000 $cm^2/Vs$ [17], which can provide a long mean free path of 1.2 μm at a carrier concentration of 2 × 1011 $cm^{-2}$. At RT also, the quantum Hall effect exist in graphene, owing to ballistic transport of electrons and holes [18]. This ability makes graphene suitable for use in various ballistic device applications. It has also been observed that the graphene possesses a very high thermal conductivity (~5000 W/mK) [19-23]. Since graphene is an excellent conductor and transparent material, the hybrid structure of ZnO/graphene shall lead to several device applications not only on Si substrate but also on other insulating substrates such as transparent glass and transparent flexible plastic

As mention previously, the growth of semiconductor materials on an insulator such as $SiO_2$ is challenging due to its amorphous structure. Therefore, an introduction of graphene as a template layer is a promising candidate to overcome this issue. The feasibility of growing, highly oriented single crystalline ZnO is one of the main reasons of utilizing graphene as a buffer layer. The graphene consists of a two-dimensional hexagonal network of carbon atoms which is formed by making strong triangular σ-bonds of the sp2 hybridized orbitals. It is worth noting that the atomic arrangement of graphene is similar to the (111) plane of zinc blence structure and *c*-plane of a hexagonal crystalline structure which makes the growth of semiconductor nanostructures and thin film on graphene feasible.

In this study, the formation of ZnO nanostructures on the graphene/$SiO_2$/Si substrates by the thermal evaporation process without any assist form catalyst is carried out. The growth of ZnO nanostructures on the graphene is carried out by using a simple thermal evaporation of Zn powder and oxygen ($O_2$) gas under the atmospheric pressure. The optimization of growth parameter such as substrate temperature, oxygen flow rate, substrate inclination angles and graphene thickness were investigated. Finally, the morphological, compositional, crystallographic and optical properties of the as-grown ZnO nanostructures are systematically characterised. The possible growth mechanism for the different geometrical morphologies of the nanostructure is also proposed.

## 1.3 Objectives

To synthesize high density ZnO nanostructures on the graphene/$SiO_2$/Si substrate by thermal evaporation technique

i) To investigate the effect of oxygen flow rates, substrate temperatures substrate inclination angles and graphene thicknesses on the surface morphology, compositional crystallographic and optical properties of ZnO nanostructures.

ii) To propose the reasonable growth mechanism based on the obtained results.

## 1.4 Research activities

The implementation of this study has been summarized into a flowchart as shown in **Figure 1.2**. This study is focused on the growth of one dimensional (1D) ZnO nanostructures on the graphene by the thermal evaporation process without any assistance of a catalyst. The growth was carried out by the thermal evaporation technique in a dual zone furnace. Firstly, the growth of ZnO nanostructures was investigated by varying the growth parameters (i.e. substrate temperatures, oxygen flow rates, and substrate inclination angles and graphene thickness). The morphological and elemental analysis of the grown ZnO nanostructures were performed by using the field emission scanning electron microscopy (FESEM) equipped with the electron dispersive spectrometer EDS. The crystallographic properties were investigated using x-ray diffractometer (XRD) and the optical properties are characterized using photoluminescence (PL) spectrometer. The possible growth mechanism was proposed based on the obtained morphology.

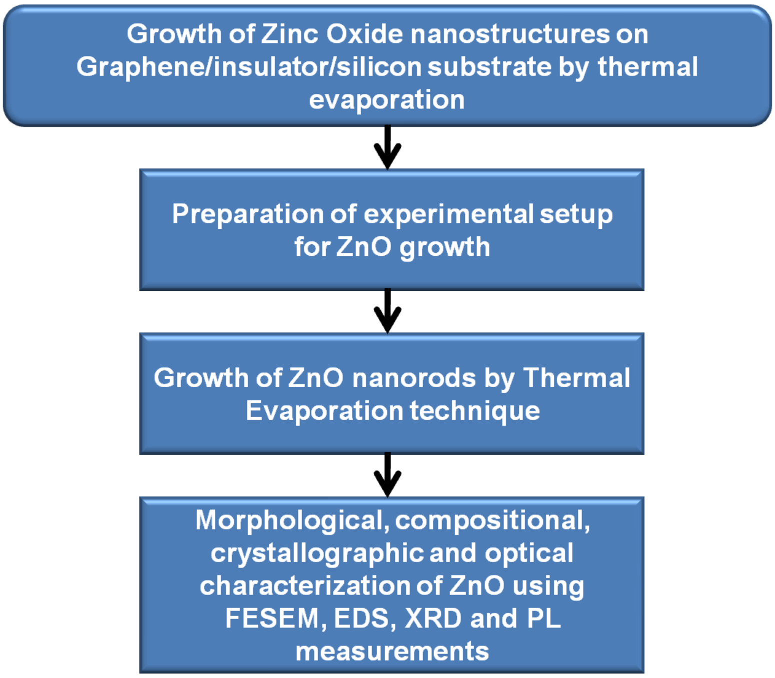

**Figure 1.2** Research activities

## 1.5 Thesis organization

This thesis has been organized into 5 chapters. Chapter 1 gives an overview of the research background and motivation of the study. The objectives and research activities of the present work are also presented in this chapter.

In chapter 2, an overview of the basic material properties of ZnO is presented. The structural, electronic and optical properties of ZnO are described in order to provide an in-depth knowledge of the ZnO materials. This chapter also explains the material properties of graphene and its structural properties. The hybrid integration of the ZnO and graphene and its possible applications are also presented. Besides that, a brief description of the methods that is widely used to grow the ZnO nanostructures, as well as its potential applications in the optoelectronic devices were described.

In chapter 3, the properties of graphene substrate that have been used in this work are described. The growth of ZnO nanostructures on the substrates by a simple thermal evaporation process without any catalyst and the characterization techniques employed are also described in detail.

In chapter 4, the growth of ZnO nanostructure on the graphene by a thermally evaporated Zn powder in the presence of $O_2$ gas is presented. The optimization of the growth parameter such as substrate temperature, oxygen flow rate, substrate inclination angles and graphene thickness were investigated. A basic study of the morphological, compositional, and crystallographic and photoluminescence properties of the grown ZnO are performed and the possible growth mechanism is also proposed in this chapter.

Finally, chapter 5 concludes the contributions of the present work and discusses the future research directions.

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