THERMAL EVAPORATION SYNTHESIZED ZINC OXIDE NANOWIRES FOR IMPROVED MORPHOLOGICAL AND STRUCTURAL PROPERTIES

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DEDICATION

This thesis is dedicated to my father, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my mother, who taught me that even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

Considering the immense fundamental and applied interests of the zinc oxide nanowires (ZnONWs), this study produced these nanowires (NWs) on the glass substrates by the standard thermal evaporation technique. The as-deposited samples were characterized systematically at the room temperature via diverse analytical instruments. The main aim was to optimize the growth parameters to achieve vertically aligned ZnONWs that have not been fully explored yet. The influence of different deposition or growth parameters (such as the growth temperatures, growth times, oxygen gas flow rates and substrate positions) on the structures and morphologies of the proposed ZnONWs were determined. The longer growth time was utilized to obtain highly aligned ZnONWs that are useful for various practical applications. To deposit the ZnONWs, 1 gram of high purity (99.99%) zinc (Zn) powder was spread in an alumina boat, placed on the alumina boat holder which was then put in the glass tube of the thermal evaporator. The lengths and diameters of the NWs were found to be significantly affected by the variations of the growth temperatures and times. The growth temperature of 500°C was shown to be optimum to produce the strong vertical alignments of the NWs on the substrate surface. The transmission electron microscopy (TEM) images of the as-deposited samples revealed the growth of long ZnONWs with uniform morphological distribution and crystalline structures. In addition, the lengths of the NWs were first increased with the increase in the growth duration and then broken, scattered as well as bent on the substrate surface at longer time. The growth time of 20 min was observed to be ideal to attain the vertically aligned NWs on the substrate surface. Moreover, an increase in the oxygen gas flow rate caused an imbalance in the NWs growth directions with strong misalignment. Besides that, the higher oxygen gas levels led to a reduction in the Zn contents in the deposited NWs. The changes in the substrate positions in the thermal evaporation glass chamber (near the gas inlet, in the centre of the furnace and near the gas outlet) were found to influence the NWs growth, wherein the boat placement at the central region of the evaporator tube produced the optimum vertical alignments of the ZnONWs. It was concluded that by carefully adjusting the growth parameters of the thermal evaporation process the structures and morphologies of the vertically aligned ZnONWs can be tailored. The current findings may be useful for the progress of the ZnONWs production essential for different applications.

ABSTRAK

Mempertimbangkan Mempertimbangkan kepentingan penggunaan wayar nano zink oksida (ZnONWs), kajian ini menghasilkan wayar nano (NWs) pada substrat kaca dengan kaedah penyejatan terma. Sampel yang ditumbuhkan dikaji secara sistematik pada suhu bilik melalui pelbagai peralatan analisis. Tujuan utamanya adalah untuk mengenalpasti parameter optimum pertumbuhan untuk mencapai ZnONWs sejajar secara menegak yang belum diterokai sepenuhnya. Pengaruh parameter pertumbuhan yang berbeza (seperti suhu pertumbuhan, masa pertumbuhan, kadar aliran gas oksigen dan kedudukan substrat) terhadap struktur dan morfologi ZnONWs yang dicadangkan telah ditentukan. Masa pertumbuhan yang lebih lama digunakan untuk mendapatkan ZnONWs yang lebih sejajar dan dapat digunakan untuk pelbagai aplikasi praktikal. 1 gram serbuk zink (Zn) yang tinggi tahap ketulenen (99.99%) ditabur di dalam perahu alumina yang diletak diatas pemegang perahu alumina dan kemudian dimasukkan kedalam tiub kaca unit penyejat termal untuk proses pertumbuhan NWs. Panjang dan ukur lilit NWs dipengaruhi secara ketara oleh variasi suhu dan masa pertumbuhan. Suhu pertumbuhan 500°C terbukti suhu optimum untuk menghasilkan NWs sejajar dan menegak serta kukuh pada permukaan substrat. Imej mikroskopi penghantaran elektron (TEM) menunjukkan sampel yang disintesis adalah pertumbuhan ZnONWs yang panjang dengan taburan morfologi dan struktur kristal yang seragam. Di samping itu, panjang NWs bertambah dengan peningkatan dalam tempoh pertumbuhan dan kemudian patah, tersebar serta membengkok di atas permukaan substrat pada masa pertumbuhan yang lebih lama. Masa pertumbuhan 20 min diperhatikan sebagai ideal untuk mencapai NWs yang sejajar secara menegak pada permukaan substrat. Lebih-lebih lagi, peningkatan kadar aliran gas oksigen menyebabkan ketidakseimbangan terhadap arah pertumbuhan NWs. Selain itu, tahap gas oksigen yang lebih tinggi menyebabkan pengurangan kandungan Zn dalam NWs yang ditumbuhkan. Perubahan kedudukan substrat (berhampiran saluran masuk gas, di tengah relau dan berhampiran saluran keluar gas di dalam tiub kaca) didapati mempengaruhi pertumbuhan NWs, di mana penempatan perahu di tengah tiub penyejat menghasilkan penjajaran menegak ZnONWs yang optimum. Disimpulkan bahawa dengan menyesuaikan parameter pertumbuhan proses penyejatan termal dengan teliti, struktur dan morfologi ZnONWs yang sejajar secara menegak dapat dihasilkan. Penemuan terkini ini mungkin berguna untuk kemajuan pengeluaran ZnONWs yang penting bagi aplikasi yang berbeza.

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LIST OF ABBREVIATIONS

ZnO	-	Zinc Oxide
Zn	-	Zinc
ZnONWs	-	Zinc Oxide Nanowires
nm	-	Nanometre
VLS	-	Vapor Liquid Solid
PLD	-	Pulsed Laser Deposition
CVD	-	Chemical Vapor Deposition
MBE	-	Molecular Beam Epitaxy
AFM	-	Atomic Force Microscopy
FESEM	-	Field Emission Scanning Electron Microscope
XRD	-	X-Ray Diffraction
TEM	-	Transmission Electron Microscope
0 – D	-	Zero Dimension
1 – D	-	One Dimension
2 – D	-	Two Dimension
MEMS	-	Micro-electro Mechanical System
PVD	-	Physical Vapor Deposition
VS	-	Vapor Solid
O_2	-	Oxygen gas
Au	-	Gold
EDX	-	Energy-Dispersive X-Ray Spectroscopy
UV-Vis	-	UV-visible Spectroscopy
sccm	-	Standard cubic centimetres per minutes
Ar	-	Argon

LIST OF SYMBOLS

nm	-	nanometer
d	-	distance between atomic layers in a crystal
λ	-	wavelength of the incident X-ray beam
θ	-	angles of incident ray
r	-	Aspect ratio
°C	-	Degree celcius
g	-	grams
nm	-	nanometer

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CHAPTER 1

INTRODUCTION

1.1 Background of The Study

Research on structures and materials at nanometre scales are known as nanoscience (Whitesides, 2005). The terms “nano” which based on metric prefix 10^{-9} is the unit measure that refers to one billionth of a certain material. Nanometre size range structures can make up fascinating and functional properties either in nature or science. Such nanoscale structures are 2 nm wide DNA strand which exists as building blocks in living things (Seeman, 2003), protein carrying oxygen through the body which is known as haemoglobin with diameter 5.5 nm (Erickson, 2009), a million nanometres wide pinhead and 75000 nm thick single sheet of paper (Stanford & Pte, 2010). For a better understanding of the world, nanoscience is studied in versatile manner which includes scientist from different fields of biology, chemistry, physics, engineering and material science.

The evolution of nanoscience has led to many potential and comprehensive advancement in nanotechnology. Design, manufacture and implementation of nanostructures and nanomaterials with a basic knowledge of the correlation between physical properties and material dimensions has led to several division of nanotechnology such as nanostructured material, nanotools and nanodevices. Nanotechnology has been applied in many industries such as health, food industry, mobile and web services, communication, aerospace and defence, energy, transportation and environment. Due to wide range of potential applications in nanotechnology, a step by step approach are crucial on synthesizing and processing of nanomaterials and nanostructures and learning physical properties associated with nanometre scale. Innovative technologies that have been supported and developing previously are image processing (Dhandapani *et al.*, 2019), power transmission (Z. Zhang *et al.*, 2018) and mechatronics (Kim *et al.*, 2019) in mechanical components, lithium batteries in energy storage (Niu *et al.,* 2019), conservation of DNA (Dika *et al.,* 2019), cold plasma in surface treatment (Bekeschus *et al.,* 2019), and materials such as special alloys (Kalantar-Zadeh *et al.,* 2019), elastomers (Fomenko *et al.,* 2019), ceramics (Petit *et al.,* 2018), polymers (Spahiu *et al.,* 2018) and composites (Zhou *et al.,* 2019).

Nanomaterials are materials that are created from a very small scale of chemical substances in the nanoscale size range about 1 – 100 nm (Sudha *et al.,* 2018) that can vary in the form of particles (Yan *et al.,* 2019), rods (Kasim *et al.,* 2018), tubes (Loise *et al.,* 2019) or fibres (Jones *et al.,* 2019) and produced by nanotechnology. Nanomaterials are of interest because at this scale unique optical, magnetic, electrical, and other properties emerge. These emergent properties have the potential for great impacts in electronics, medicine, and other fields. The particular interest are engineered nanomaterials (Hochella *et al.,* 2019), which are designed for, and already being used in many commercial products and processes. Engineered nanomaterials are designed at the molecular (nanometre) level to take advantage of their small size and novel properties which are generally not seen in their conventional, bulk counterparts. The two main reasons why materials at the nanoscale can have different properties are increased relative surface area (B. Yao *et al.,* 2019) and new quantum effects (Shi *et al.,* 2019). Nanomaterials have a much greater surface area to volume ratio than their conventional forms, which can lead to greater chemical reactivity and affect their strength (J. Liu *et al.,* 2019). Also at the nanoscale, quantum effects can become much more important in determining the materials properties and characteristics, leading to novel optical (M. H. Huang, 2019), electrical (Ikramova & Filippov, 2018) and magnetic behaviour (Y. F. Wang *et al.,* 2018).

The number of products produced by nanotechnology or containing nanomaterials entering the market is increasing. Current applications include healthcare (Muhammad *et al.,* 2018), electronics (Contreras *et al.,* 2017), cosmetics (Hameed *et al.,* 2019), textiles (Yetisen *et al.,* 2016), information technology (Nayyar *et al.,* 2017) and environmental protection (C. H. Lee *et al.,* 2017). For example, nanosilver (Khaksar *et al.,* 2019) is appearing in a range of products, including washing machines, socks, food packaging, wound dressings and food supplements.

A considerable amount of literature has been published on semiconductor nanostructures. Intense amount of research on zinc oxide (ZnO) semiconductor has been studied due to existence of large amount of nanostructures with unique structures and properties that can be used for various applications such as sensors (Zubair and Akhtar, 2019) and transducers (Chianese *et al.,* 2019). Different ZnO fabrication techniques produce different nanostructures of nanocombs (J. X. Wang *et al.,* 2006), nanorings (Wu *et al.,* 2009), nanohelixes/nanosprings (Choi *et al.,* 2019), nanobows (Hughes & Wang, 2004), nanobelts (Pan, 2001), nanowires (Chen *et al.,* 2019), nanorods (Karim *et al.,* 2019) and nanocages (Y. Song *et al.,* 2017).

A few techniques have been used to grow ZnONWs. The first technique is known as vapor liquid solid (VLS) technique (Roso *et al.,* 2016). Small nano size gold (Au) droplet is put on the surface of the substrate. The dimensions of the gold determine the diameter of the wire and other locations so, this can be used to pattern and achieved wires in specific arrays or points using nanolithography technique and by changing the size, the width or the diameter of the nanowire can be determined (Malik *et al.,* 2018). The second technique is the technique which does not require the use of any catalysts and the fact that the ZnO start to form large crystal initially in growth of different surface planes is utilized (Sekar *et al.,* 2005). Basically, Zn is used as the source powder and heated to high temperature to reduce the Zn and that reduced gas float over the substrate and thus the ZnONWs grow on the substrate. By varying the growth conditions, abundance of different structures and densities of particles and size of particles can be achieved. Combinations of parameters are very unique to furnace setup done throughout the experiments.

A great deal of previous research into ZnO has focused on vertically aligned ZnONWs. These structures have been intensively investigated recently due to its importance in many applications such as nanowire lasers (Vanmaekelbergh & Van Vugt, 2011), solar energy cells (H. Guo *et al.,* 2019) and field emitters (Materials, 2019). Lately, these wires have been grown by chemical vapor deposition (CVD)

(Maitra *et al.,* 2019), pulsed laser deposition (PLD) (Karnati *et al.,* 2018), spin-coating deposition of a sol-gel (Demes *et al.,* 2016) and thermal evaporation methods (Saha *et al.,* 2016). However, it was later found that the most conventional methods to grow vertically aligned ZnONWs are using thermal evaporation method without using any catalyst under specific growth conditions.

Thermal evaporation technique is a physical vapor deposition method which is superior to other technique because it is a simple method to synthesized large-scale of ZnONWs compare to other method such as laser assisted molecular beam epitaxy (MBE) (Kennedy *et al.,* 2019). It was also proven to produce crystalline ZnONWs with an average diameter of 30 - 100 nm according to structural analysis of TEM. The crystalline ZnO shows to have hexagonal structure of (a = 0.325 nm and c = 0.521 nm) crystals (Rai *et al.,* 2013). Furthermore, high quality monocrystalline nanowires can be achieved with low density of structural defects such as stacking faults and dislocations using thermal evaporation method (B. D. Yao *et al.,* 2002). In this research, vertically aligned ZnONWs are being studied by their morphological and structural properties. A brief discussion of the growth mechanism involved in synthesizing ZnO by using thermal evaporation method on glass substrate without the use of any catalysts is discussed.

1.2 Problem Statement

Vertically aligned ZnONWs have received great attention among researchers due to importance in many recent technological applications of semiconductor nanostructure. Hydrothermal method have been successfully used to synthesized vertical aligned ZnONWs, however this technique requires longer growth time (Podrezova *et al.,* 2013). Besides, there are challenges in constructing a much more durable vertical aligned ZnONWs structure due to its high growth time which may be disadvantageous for nanowires properties. To overcome this problem, other method to synthesize vertical aligned ZnONWs is being proposed for stronger integrated nanowire structure which uses low growth time during the process. Thus, it is necessary to deposit ZnONWs and characterize their structural and morphological properties to achieve optimum nanowires useful for various applications such as electronic (D. Wang *et al.,* 2020) and medical field (Z. Wang *et al.,* 2016).

Despite these challenges, scientists have come up with different approach to use thermal evaporation method with intermediate growth temperature during the process to improve the performance of the nanowires. Thermal evaporation method which also known as vapor transport method using a horizontal furnace have been used in this research to grow ZnONWs with quality structures. This method is a simple growth technique which uses glass as a substrate and without any catalysts. Besides using an intermediate growth temperature during the process this method also requires shorter growth time to grow vertical aligned ZnONWs.

1.3 Objectives of The Study

This research aims to synthesize ZnONWs as potential materials for high performance applications. The objectives are as follows:

1. To synthesize ZnONWs using thermal evaporation method in terms of growth temperature, growth time, gas flow rate and different substrate position.

2. To determine the optimum growth parameter of ZnONWs based on growth temperature (450 - 550°C), growth time (10 - 60 minutes), gas flow rates (160 – 240 sccm) and different substrate positions.

3. To characterize the morphological and structural properties of ZnONWs.

1.4 Scope of The Study

To achieve the objectives for this study, the scope and limitations of the work performed are as follows. 99.99 % purity zinc powder was used as the source material. ZnONWs were synthesized by using thermal evaporation technique using horizontal quartz tube furnace. Then, the deposition process of ZnO on glass substrate with the working parameters by thermal evaporation method was performed. The growth parameters of ZnONWs are growth temperature (450 - 550°C), growth time (10 - 60 minutes), gas flow rate (160 – 240 sccm) and substrate position (near gas inlet, centre of tube furnace and far from gas inlet). Samples are characterized by atomic force microscopy (AFM), field-emission scanning electron microscopy (FESEM), x-ray diffraction (XRD) and transmission electron microscopy (TEM). The samples were analysed based on their morphological and structural properties.

1.5 Significance of The Study

The findings of this study will contribute to the benefit in science and technologies that semiconductor nanostructure plays an important role. The greater demand for high performance applications justifies the need for effective and much more integrated ZnONWs with shorter growth time that can be an advantage to nanowire properties. Using thermal evaporation method as the recommended approach to produce optimum nanowires is vital for various applications. The study will improve the performance of nanowires using intermediate growth temperature.

1.6 Outline of Thesis

The thesis consists of 5 chapters. Chapter 1 summarizes general information on nanomaterials and nanostructures as well as introduction to ZnONWs. Moreover, details of the problem statements, objectives and scope of study have also been stated comprehensively. Chapter 2 discusses the background information related to the ZnO materials, nanowires structure and the growth technique. Furthermore, the basic theory and fundamental study related to the research are also discussed. Chapter 3 describes the experimental approaches and characterization techniques that were applied during the study. The analysis of sample is also briefly elaborated. Chapter 4 presents in detail the results and discussion of the research findings. The effect of various operating conditions applied during the experiment and detailed explanations related to the nanowires are discussed in this chapter. Chapter 5 concludes the research and some recommendation for future works.

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