FABRICATION OF HEXAGONAL ZINC OXIDE NANORODS ON SEEDED SUBSTRATE VIA HYDROTHERMAL METHOD

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To my beloved parents and family members,
thank you for the endless love, support and encouragement throughout this journey.

Maarof Abd Razak
Zainon Jayos
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ABSTRACT

Zinc oxide (ZnO) has been studied due to have a direct wide bandgap (~3.7 eV), large exciton binding energy (~60 meV), non-toxic material. In this study, two step hydrothermal process was introduced to fabricate seed layer in a form of ZnO nanoparticles (ZnO-NPs) and ZnO nanorods (ZnO-NRs). The seed layer solution concentration is 0.3 M and consists of zinc acetate dihydrate (ZAD) and diethanolamine (DEA). Besides that, 5 layers of coated seed layer was deposited using spin coating method. Subsequently, the high quality of aligned hexagonal ZnO-NRs were fabricated using hydrothermal method which consists of an equimolar nutrient solution (0.05 M) synthesized using zinc nitrate hexahydrate (ZNH) and hexamethylenetetramine (HMTA). The hydrothermal reaction times were varied from 1 hour to 9 hours. The structural, morphological, topological, optical and electrical properties of the ZnO-NRs were studied using X-ray diffraction (XRD), Field-Emission Scanning Electron Microscopy (FESEM), Atomic Force Microscopy (AFM), Ultraviolet-visible (UV-Vis) spectroscopy and four-point probe, respectively. Based on the research, the deposition of ZnO-NPs indicated a significant improvement in the well-developed and aligned hexagonal of ZnO-NRs. As a result, ZnO-NRs synthesized with 6 hours of hydrothermal reaction time had an average diameter of 185 nm and rod lengths of 2.04 µm. However, recorded the lowest value of resistivity (0.83×10^{-3} Ω.cm) thus, exhibited the highest value of conductivity (12.11×10^{2} $\Omega.cm^{-1}$). As higher conductivity materials can transfer more electrons, thereby increasing the rate of electron-hole generation. This will lead to greater current flows. Therefore, ZnO-NRs can be widely implemented in electronic devices application such as ultraviolet sensor and thermoelectric application.

ABSTRAK

Zink oksida (ZnO) telah dikaji kerana mempunyai jalur lebar langsung (~3.7 eV), tenaga pengikatan *exciton* yang besar (~60 meV) dan bahan tidak bertoksik. Dalam kajian ini, proses hidroterma dua langkah diperkenalkan untuk membuat lapisan biji benih dalam bentuk ZnO nanopartikel (ZnO-NPs) dan ZnO nanorod (ZnO-NRs). Kepekatan larutan lapisan biji benih adalah 0.3 M dan terdiri daripada zink asetat dihidrat (ZAD) dan dietanolamina (DEA). Selain daripada itu, 5 lapisan biji benih yang disalut didepositkan menggunakan kaedah salutan pusingan. Seterusnya, ZnO-NRs yang berkualiti tinggi dan mempunyai bentuk heksagonal yang sejajar dibuat menggunakan kaedah hidroterma yang terdiri daripada larutan nutrien setara (0.05 M) yang disintesis menggunakan zink nitrat heksahidrat (ZNH) dan heksametilenetramina (HMTA). Masa tindak balas hidroterma adalah berbeza-beza dari 1 jam hingga 9 jam. Sifat struktur, morfologi, topologi, optik dan elektrik bagi ZnO-NRs dikaji menggunakan difraksi sinar-X (XRD), Mikroskopi Elektronik Pengimbasan Pancaran Medan (FESEM), Mikroskopi Daya *Atom* (AFM), spektroskopi tampak *Ultraviolet* (UV-Vis) dan kuar empat titik. Berdasarkan penyelidikan, pemendapan ZnO-NPs menunjukkan peningkatan yang ketara dalam ZnO-NRs yang berbentuk heksagon yang dibangunkan dengan baik dan sejajar. Hasilnya, ZnO-NRs yang disintesis dengan 6 jam masa tindak balas hidroterma mempunyai diameter purata 185 nm dan panjang nanorod 2.04 µm. Walau bagaimanapun, mencatatkan nilai resistiviti yang paling rendah (0.83 $x10^{-3}$ Ω.cm) dan dengan itu, menunjukkan nilai kekonduksian yang paling tinggi (12.11 $x10^{2}$ $\Omega.cm^{-1}$). Oleh kerana bahan kekonduksian yang lebih tinggi boleh memindahkan lebih banyak elektron, dengan itu meningkatkan kadar penjanaan electron-*hole*. Ini akan membawa kepada aliran arus eektrik yang lebih besar. Oleh itu, ZnO-NRs boleh dilaksanakan secara meluas dalam peranti aplikasi elektronik seperti penderia ultraungu dan aplikasi termoelektrik.

CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS AND ABBREVIATIONS

C	-	Molar Concentration
D	-	Average Crystallite Size
K	-	Scherrer Constant, K=0.94Å.
α	-	Absorption Coefficient
δ	-	Dislocation Density
E_g	-	Bandgap Energy
E_w	-	Lower Work Function
$h\nu$	-	Photon Energy
k_BT	-	Room Temperature Thermal Energy
R_a	-	Average Surface Roughness
t	-	Thickness of The Material
v	-	Volume
λ	-	Wavelength
ρ	-	Resistivity
σ	-	Conductivity
θ	-	Incident Angle
ΔV	-	Change In Voltage
1D	-	One-Dimensional
2D	-	Two-Dimensional
3D	-	Three-Dimensional
AAO	-	Anodic Aluminium Oxide
AFM	-	Atomic Force Microscopy
CBD	-	Chemical Bath Deposition
CdTe	-	Cadmium Telluride

DEA	-	Diethanolamine
DI	-	Deionized Water
DSSC	-	Dye-Sensitized Solar Cells
ED	-	Electrochemical Deposition
FESEM	-	Field-Emission Scanning Electron Microscopy
FTO	-	Fluorine Doped Tin Oxide
FWHM	-	Full Width At Half Maximum
HMTA	-	Hexamethylenetetramine
HMTA	-	Hexamethylenetetramine
In_2O_3	-	Indium Trioxide
ITO	-	Indium Tin Oxide
KCl	-	Potassium Chloride
KOH	-	Potassium Hydroxide
L	-	Number Of Coated Seed Layer
MBE	-	Molecular Beam Epitaxy
MgO	-	Magnesium Oxide
MW	-	Molecular Weight
NaCl	-	Sodium Chloride
NaOH	-	Sodium Hydroxide
O	-	Oxygen
PA-MBE	-	Plasma-Assisted Molecular Beam Epitaxy
PEI	-	Polyethylenimine
PLD	-	Pulsed Laser Deposition
SEM	-	Scanning Electron Microscope
SnO_2	-	Tin Dioxide
TiO_2	-	Titanium Dioxide
UV	-	Ultraviolet
UV-Vis	-	Ultraviolet-Visible
VDP	-	Van Der Pauw
WO_3	-	Tungsten Trioxide
XRD	-	X-Ray Diffraction
ZAD	-	Zinc Acetate Dihydrate

Zn	-	Zinc
ZNH	-	Zinc Nitrate Hexahydrate
ZnO	-	Zinc Oxide
ZnO-NPs	-	Zinc Oxide Nanoparticles
ZnO-NRs	-	Zinc Oxide Nanorods
ZnS	-	Zinc Sulphide
$ZnSO_4$	-	Zinc Sulfate
VDP	-	Van Der Pauw
WO_3	–	Tungsten Trioxide
XRD	-	X-Ray Diffraction
ZAD	-	Zinc Acetate Dihydrate
ZNH	-	Zinc Nitrate Hexahydrate
ZnO	–	Zinc Oxide
ZnO-NPs	-	ZnO Nanoparticles
ZnO-NRs	-	ZnO Nanorods
ZnS	-	Zinc Sulphide

LIST OF PUBLICATIONS

Journal /Proceedings:

1. Shazleen Ahmad Ramli, Fariza Mohamad, A.G.A Anizam, M.K. Ahmad, Norazlina Ahmad, Anis Zafirah Mohd Ismail, Nurliyana Mohamad Arifin, **Nurul Amiera Shahida Maarof**, A.M.S Nurhaziqah, D.G Saputri, Nik Hisyamudin Muhd Nor and Izaki Masanobu, "Properties enhancement of TiO_2 nanorod thin film using hydrochloric acid etching treatment method", *J Mater Sci: Mater Electron* 33, 16348–16356 (2022).

2. Nyaanalechumi Vijayan, **Nurul Amiera Shahida Maarof**, Nurul Huda Abdul Rahman, Mohd Khairul Ahmad, "Study of Zinc Oxide Nanoflower for Photocatalytic Applications Prepared via Hydrothermal Method", *Evolution in Electrical and Electronic Engineering* Vol. 2No. 2, p. 10-17 (2021).

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LIST OF AWARD

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CHAPTER 1

INTRODUCTION

1.1 Research background

Zinc oxide (ZnO) is one of the metal oxide semiconductor materials that has been extensively studied due to its unique properties. Other metal oxide semiconductor materials are indium trioxide (In_2O_3), tin dioxide (SnO_2), titanium dioxide (TiO_2), tungsten trioxide (WO_3) and zinc oxide (ZnO), which has special catalytic, anti-bacterial, as well as good optical and electrical properties [1],[2]. ZnO also known as an Earth abundant material which is non-toxic, chemically and thermally stable semiconductor material that has wide direct band gap energy (~3.37 eV). Thus, make it more preferable over the other wide-band-gap materials due to its high energy radiation stability and amenability to wet chemical etching [3]. This makes ZnO as a suitable candidate for space applications.

Meanwhile, large excitation binding energy of ~60 m*eV* paves the way for an intense near-band-edge excitonic emission at room and high temperature [4], [5], [6]. ZnO has been considered as the most promising nanostructures material that has a wide range of application such as solar cells [7], photocatalyst [8], UV photodetector [9] and thermoelectric devices such as energy generators or cooling devices [10]. Moreover, ZnO exists in three crystal structures which are cubic rocksalt, cubic zinc blende, and hexagonal wurtzite, as shown in Figure 1.1 (a), 1.1 (b), and 1.1 (c), respectively were shaded with light green refers to zinc atom while shaded in dark green represents oxygen atom [11]. From these three structures, the wurtzite structure is the most stable structure under normal conditions while rocksalt structures can only fabricated at high pressure and zinc blende structures can only synthesized on cubic

substrates such as zinc sulphide (ZnS), cadmium telluride (CdTe), magnesium oxide (MgO), potassium chloride (KCl) and sodium chloride (NaCl) [3], [12]. The wurtzite structure is a hexagonal crystal system that belongs to the space group of $P6_3mc$ (No. 186), possesses two lattice parameters which are; a=3.2495Å and c=5.2069Å with a ratio of c/a=1.63 [3].

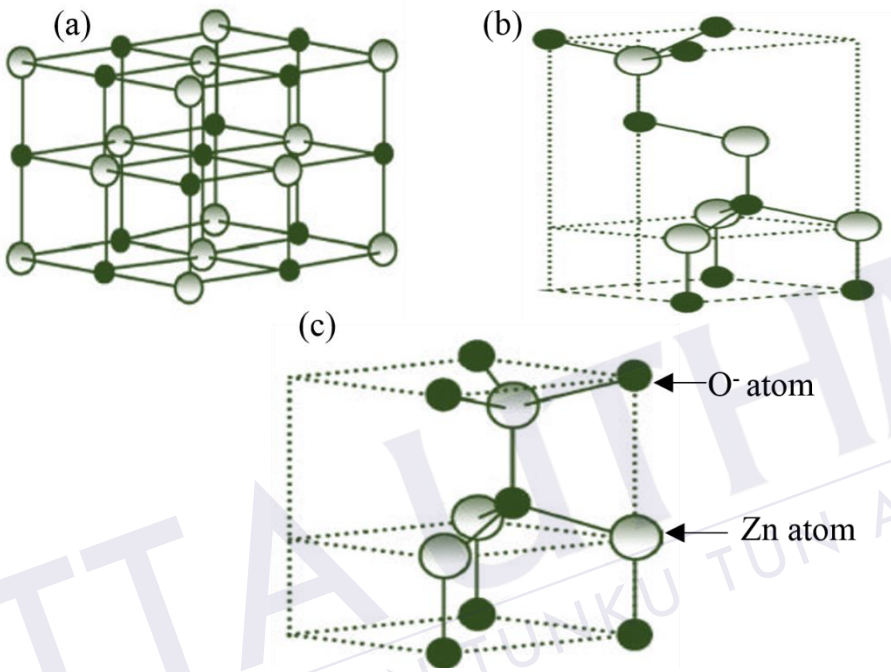

Figure 1.1:

Figure 1.1: The crystal structure of ZnO: (a) Cubic rocksalt, (b) Cubic zinc blende and (c) Hexagonal wurtzite atom [3].

A schematic representation of the wurtzite ZnO structure is shown in Figure 1.2. This shows that wurtzite crystal structure correlates with the ratio of an ideal crystal. Despite the fact that the ionic nature of Zn-O is quite strong, ZnO displays a little covalent bonding. These can be compared to the basal plane, which it has facets that exhibit massive surface reconstruction. In this structure, ZnO has high electron mobility and thermal conductivity in this form because of the sp^3 hybrid valence electrons in its wurtzite lattice structure [13]. Besides that, single crystal ZnO has high electrons mobility (200-300 $cm^2V^{-1}s^{-1}$) at room temperature, which the great advantage in its electrical characteristics [13]. However, ZnO has high thermal conductivity and it is the main disadvantage for thermoelectric applications. The thermal conductivity

of pure ZnO at 300 K is around 49 $Wm^{-1}K^{-1}$ and drops to 10 $Wm^{-1}K^{-1}$ at 1000 K [17]. As a result, in addition to enhance the electrical conductivity, most attempts to improve the thermoelectric performance of pure ZnO focus on lowering thermal conductivity.

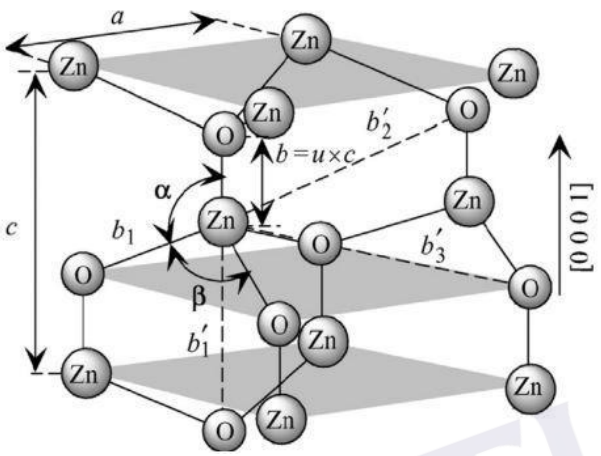

Figure 1.2: Schematic representation of ZnO wurtzite structure [3].

Besides that, ZnO can be synthesized by low-cost production, easy to produce, and allows a good perspective in large scale assembly for the fabrication of thermoelectric modules. There have been numerous reports regarding the fabrication method of ZnO nanostructures either using physical or chemical methods [14]. In this study, ZnO nanostructures will be synthesized by using hydrothermal method. Hence, more details of hydrothermal method will be explained on the next subchapter 2.3.

1.2 Problem statement

The existing zinc oxide nanorods (ZnO-NRs) fabricated using hydrothermal method growth at random orientation on foreign surface that produce non uniform diameter of nanostructures [14]–[17]. These are due to the pure nutrient solution for the hydrothermal method consisting of zinc nitrate hexahydrate (ZNH) and hexamethylenetetramine (HMTA). This process does not include any additive that acts as the catalyst to enhance the growth of ZnO-NRs during the heating process. In the meantime, Y. F. Hsu et al. discovered that using the same material as the seed layer is vital to promote the growth, structure, and orientation of deposited crystals [18]. As ZnO has higher electron mobility (200-300 $cm^2V^{-1}s^{-1}$) [19]–[22], alignment is essential to promotes higher electron movement. Hence, seed layer is introduced to initiates the growth of well aligned ZnO nanorods. Furthermore, high resistivity material will resist

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