CHARACTERIZATION OF ALUMINIUM DOPED ZINC OXIDE NANOSTRUCTURES SYNTHESIZED BY THERMAL EVAPORATION METHOD

NORAFIZAH BINTI SALIHIN

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To my late father (Allahyarham Salihin bin Hj.Asri)
to my beloved mother (Hjh. Noor Seah binti Mohamed)
and to my siblings

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ABSTRACT

This study focuses on the synthesis and characterization of undoped and aluminium (Al) doped ZnO nanostructures for examples nanoflowers, nanorods, nanowires and nanopetals grown by thermal evaporation method. Samples were grown on silicon (100) substrate. The silicon substrate was placed at 17 cm away from zinc target and aluminium powder mixture mounted on horizontal quartz tube under controlled oxygen. The aluminium concentration dependent morphology, crystalline structure and optical properties of these prepared nanostructures were determined. Samples were characterized using field emission scanning electron microscopy, energy dispersive X-ray analysis (EDX), X-ray diffraction (XRD), Raman spectroscopy, ultraviolet-visible (UV-Vis) spectroscopy and photoluminescence spectroscopy. As the Al dopant concentrations increased, the morphology of ZnO changed from uniform nanoflowers to randomly oriented nanostructures. The flower-like ZnO:Al nanorods have the length of about 333 nm and diameter of about 117 nm. The optimum dopant concentration which can produce uniform size, length and diameter was found to be 0.5 at% of Al. EDX analyses revealed the presence of Zn, O, and Al in the samples. From XRD patterns, the samples had high degree of crystallization with crystallite sizes of about 24.66 nm to 46.98 nm. The ZnO:Al nanoflowers also exhibited a strong ultra-violet emission at 380 nm. Additionally, the band gap energy of ZnO:Al was not significantly changed as found from UV-Vis analyses at 3.24 eV. The concentration of Al plays a significant important role in controlling structural, morphological and optical properties of ZnO nanostructures. The ZnO:Al nanostructures are expected for future technological application due to its impact on optical properties.

ABSTRAK

Kajian ini memberi tumpuan kepada sintesis dan pencirian nanostruktur contohnya nanobunga, nanorod, nanowayar dan nanokelopak ZnO yang tidak didop dan didop dengan aluminium (Al) yang dihasilkan menggunakan kaedah penyejatan haba. Sampel disintesis pada substrat silikon (100). Substrat silikon diletakkan pada jarak 17 cm dari sasaran zink dan campuran serbuk aluminium yang diletakkan pada tiub kuarza mendatar di bawah oksigen terkawal. Morfologi bersandar kepada kepekatan aluminium, struktur kristal dan sifat optik nanostruktur yang disediakan telah ditentukan. Sampel dicirikan menggunakan mikroskop pengimbasan elektron medan terpancar, analisis penyebaran tenaga X-ray (EDX), pembelauan sinar X-ray (XRD), spektroskopi Raman, spektroskopi ultraungu-nampak (UV-Vis) dan spektroskopi fotoluminasi. Apabila kepekatan dopan Al meningkat, morfologi ZnO berubah dari nanobunga ke nanostruktur berorientasikan secara rawak. Nanorod ZnO:Al berbentuk seperti bunga mempunyai panjang kira-kira 333 nm dan diameter kira-kira 117 nm. Kepekatan dopan optimum yang boleh menghasilkan saiz, panjang dan diameter seragam adalah didapati pada 0.5 at% Al. Analisis EDX mendedahkan kehadiran Zn, O, dan Al dalam sampel. Dari corak XRD, sampel mempunyai penghabluran tinggi dengan saiz kristal sekitar 24.66 nm hingga 46.98 nm. Nanobunga ZnO:Al juga mempamerkan sinaran ultraviolet kuat pada 380 nm. Tambahan lagi, tenaga jurang ZnO:Al tidak banyak berubah seperti yang didapati daripada analisis UV-Vis pada 3.24 eV. Kepekatan Al memainkan peranan penting dalam mengawal sifat-sifat struktur, morfologi dan optik nanostruktur ZnO. Nanostruktur ZnO yang didop dengan Al dijangka untuk aplikasi teknologi masa depan disebabkan kesannya terhadap sifat optik.

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θ	-	Angle
γ-AuZn	-	Solid Gold-Zinc
Å	-	Angstrom
a_0	-	Lattice constant
AED	-	Alloying Evaporation Deposition
AFM	-	Atomic Force Microscopy
Al	-	Aluminium
Al^{3+}	-	Aluminium ion
AlCl	-	Aluminium Chloride
Al_2O_3	-	Aluminium Oxide
Ar	-	Argon
Au	-	Aurum/Gold
Au-Si	-	Gold-Silicon
c_0	-	Lattice constant
°C	-	Degree celcius
Cr	-	Chromium
1D	-	One-dimensional
DOS	-	Density of States
EDX	-	Energy Dispersive X-ray
en	-	Ethylenediamine
eV	-	Electron-volt

F-E	-	Field-emission
FESEM	-	Field Emission Electron Microscopy
FEs	-	Field-emitters
g	-	Gram
g/cm^3	-	Gram per cubic centimetre
$\hbar$	-	Photon
HCP	-	Hexagonal closed packing
HR-TEM	-	High-Resolution Transmission Electron Microscopy
JCPDS	-	Joint Committee on Powder Diffraction Standards
K	-	Kelvin
kV	-	Kilovolt
M	-	Mole
mm	-	Milimetre
nA	-	Nanoampere
NAPLD	-	Nanoparticle assisted pulsed laser deposition
NFs	-	Nanoflowers
nm	-	Nanometre
N/mm^2	-	Newton per square milimetre
NPs	-	Nanoplates
NRs	-	Nanorods
NSs	-	Nanostructures
NWs	-	Nanowires
O_2	-	Oxygen
O^{2-}	-	Oxygen ion
PA	-	Photodecomposition Activity
PEI	-	Polyethyleneimine
PL	-	Photoluminescence
PLD	-	Pulsed Laser Deposition
sccm	-	Standard cubic centimeter per minute
SEM	-	Scanning Electron Microscopy
Si	-	Silicon

SILAR - Successive Immersion Layer Adsorption Reaction
T-Ag - Triangular silver
UV - Ultraviolet
UV-Vis - Ultraviolet-visible
V_0 - Oxygen vacancies
VLS - Vapour-Liquid-Solid
VS - Vapour-Solid
VSS - Vapour-Solid-Solid
XRD - X-ray Diffraction
Zn^{2+} - Zinc ion
Zn_i - Zinc intersitials
ZnO - Zinc Oxide
ZnO:C - Carbon doped Zinc Oxide
ZnO:Al - Aluminium doped Zinc Oxide
ZnS - Zinc sulfide
ZONFs - Zinc Oxide Nanoflowers

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Materials can be divided in three groups which are conductors, semiconductors and insulators based on their electrical properties. In this new era, researchers give tremendous attention on semiconductor materials due to its potential technological application. Semiconductors such as InP, GaAs, ZnO and SiO_2.ZnO are being used in optoelectronic, electronic, biomedical sciences and sensor device. Among these semiconductors, ZnO is one of the most studied materials for their electronic and optical properties for application prospects.

Zinc oxide (ZnO) is a combination of II-VI semiconductor that is widely used in optoelectronic devices. ZnO has a wide band gap of 3.37 eV and large exciton binding energy of 60 meV at room temperature. Recently, nanostructure materials have been of interest because of their behaviour which is expected to be superior in tiny dimensions. One-dimensional nanostructures comprise nanobelts, nanorods, nanowires and nanotubes are applied in many devices such as gas sensors (Tang *et al.*, 2016), light emitting diodes, solar cells and lasers (Bu, 2014b). Besides that, ZnO has been proposed in humidity sensors and nanogenerators due to its moisture sensitivity and piezoelectricity.

ZnO has a stable wurtzite structure with lattice parameters a = 0.325 nm and c = 0.521 nm (Fan and Lu, 2005). The non-central symmetry in wurtzite structure contributes to piezoelectric and pyroelectric effect. A tetrahedral unit of Zn^{2+} and O^{2-} form polar surface which could give a unique growth morphologies; nanowires, nanosprings, nanorings, nanocombs, nanohelixes, nanorods and nanocages. Therefore, ZnO could give a huge impact for technological applications such as ultrasensitive nano-sized gas sensors, nanolasers, field-emitters (FEs) and nanoresonators.

There are several growth mechanisms to synthesize nanostrutures (NSs) including Vapour-Liquid-Solid (VLS), Vapour-Solid-Solid (VSS) and Vapour-Solid (VS). Temperature and other parameters can directly affect the growth mechanism. In VLS mechanism, a mixture of ZnO (vapour) and Au (catalyst) will form alloy (liquid Au-ZnO) and then ZnO NFs with temperature greater than 400 °C. For VSS mechanism, a mixture of ZnO (vapour) and Au (catalyst) will form Alloy (solid γ-AuZn) and then ZnO NFs with temperature smaller than 400 °C or greater 400 °C depending on catalyst or materials. VS mechanism involves a mixture of Zn (catalyst free) and O_2 to form ZnO (solid).

Although it is possible to synthesize high quality of nanoflowers (NFs) through various methods such as chemical bath deposition (Shi and Walker, 2016), sol-gel (Zhou *et al.*, 2016) and hydrothermal (Cunha and Souza, 2013; Adhyapak *et al.,* 2014; Saleem *et al.*, 2017), such methods required a long deposition time. Another alternative method NFs was thermal evaporation (Umar *et al.*, 2016)**.** The metallic powder was evaporated in the furnace and then oxidized in the presence of argon or helium and oxygen.

Abdulgafour *et al.* (2010) have revealed that the growth of low density and non-uniformity of undoped ZnO NFs on Silicon (Si) substrate is by thermal evaporation. Some defects were also found in the crystal structure as presented in PL analysis. In the experiment, the Si substrate was placed at the top of alumina boat with the face towards the Zn powder and heated from 400 °C to 850 °C for one hour.

Currrently, doped ZnO NFs have a wide range of application such as piezoelectric devices, transparent conducting electrode devices, gas sensor devices and photo voltaic devices. Aluminium doped zinc oxide (ZnO:Al) has advantages of low-production cost, non-toxicity and thermal stability. It is one of the materials using in photocatalysis, photosensors and optoelectronic devices. Most researchers now pay much attention on ZnO:Al especially in the field of material science and technology.

The morphology of Al doped ZnO thin films changed to stacking of nanowires when the Al concentration increases (Chandramohan *et al*., 2012). The quality of crystalline structure and optical transmission of thin films strongly depended on the concentration of Al dopants. Moreover, the samples had potential in constructing gas sensors due to high ratio between surface and volume of nanowires.

Recent literatures also showed that complex growth morphologies were observed by using microscopes. Mamat and his co-workers (2011) prepared Al doped ZnO NSs which the films consisted of nanorod-nanoflake networks. Zhang *et al.* (2013), Tashi (2013) and Kumar *et al.* (2014) concluded that the morphology of ZnO:Al NSs varies with different Al concentrations and Al does not affect the hexagonal structural of ZnO. Most of the previous studies on ZnO:Al NSs claimed that the samples exhibited in ultraviolet (UV) and visible region upon in photoluminescence (PL) analysis (Rajan *et al.*, 2014; Bu, 2014b and Tang *et al.,* 2016).

In terms of energy band gap, Kumar *et al.* (2014) found that it was increased with increasing Al concentration. However, Dhas *et al.* (2017) has contradicted the finding about the energy band gap. This implies that different materials and methods may generate different results.

1.2 Problem Statement

Nanotechnology has become an intensively and extensively pursued topic in this new era because it provides a deeper understanding of functional materials for advanced applications. This leads the researchers who work in nanomaterial fields to construct advanced nanoscale devices and optoelectronic devices. ZnO is considered to be a promising candidate for optoelectronic devices, UV sensors and gas sensors.

The formation of nanorods, nanowires and nanosheets which in microscopic view resemble flowers is known as nanoflowers (NFs). Non-uniformity of undoped ZnO NFs on Silicon (Si) substrate by thermal evaporation were observed through scanning electron microscopy (SEM) (Abdulgafour *et al.*, 2010). Some oxygen vacancies were detected in PL analysis which is related to visible emissions. Moreover, a few studies have been conducted on the doping of ZnO NFs by simple technique which is thermal evaporation method.

So far, many reports have focused on synthesis of ZnO:Al NSs via various methods including sol-gel, pulsed-laser deposition, precipitation, chemical bath, evaporation-deposition and thermal evaporation. However, Tashi (2013) have revealed that the growth of low density and non-uniformity size of ZnO:Al nanowires (NWs) on Silicon (Si) substrate by thermal evaporation. The morphology of ZnO varies with different concentrations of Al (Mamat *et al.*, 2011; Zhang *et al.*, 2013 and Kumar *et al.*, 2014).

Some defects were also found in the crystal structure based on PL analysis (Tashi, 2013; Rajan *et al.*, 2014 and Tang *et al.*, 2016). A broad of blue to violet region was observed which indicate that there is few oxygen vacancies and Zn intersitials.

The influence of dopants on the formation of ZnO nanostructures would keen to a better understanding of their growing mechanisms. Since the growth parameters affect the structural, magnetic and electrical properties of ZnO, thus the main focus here is to synthesize aluminium (Al) doped ZnO NFs and discovers the influence of dopant concentrations on the structural and optical properties. Furthermore, the structural and optical of ZnO:Al NFs are expected to be improved. In this work, the reactions were carried out in an electric furnace as Zn powder was evaporated directly. The process did not use metal catalyst to avoid catalyst contamination. During the growth of ZnO:Al NFs, it is expected that more than one mechanism involve in the process. In this study, the formation of ZnO:Al NFs involves VS mechanism as the absence of the use of catalyst in the reaction. Undoubtedly, Zn nanoparticle acts as seed in the nucleation base process which is believed that VLS mechanism also occured.

1.3 Research Objectives

The objectives of this study are:

i) To synthesize aluminium doped zinc oxide (ZnO:Al) nanoflowers by thermal evaporation method.

ii) To characterize the grown nanostructures for structural, morphology and optical properties.

iii) To determine the aluminium concentration dependent morphological, structural and optical properties for optimization.

1.4 Scope of Study

Recently, there has been a drastic increase of the literature of NFs. However, most of the previous works studied on undoped ZnO. Doping is the best tool to manipulate the structural, optical and electrical properties of ZnO. So, it is an opportunity to explore a further detail the doped counterparts. Group III elements are close lattice matching with ZnO and it can be considered as a dopant in this work. This research based on aluminium doped ZnO NFs with dopant concentrations ranging from 0.5 at% to 6.0 at%. A p-type ZnO was formed when doped with Al because it behaves as an acceptor in ZnO with its energy level located at 0.1 eV below the bottom of the conduction band (Kanai, 1991). Additionally, when doped with Al, the various concentrations of Al may affect the structural and optical properties of ZnO:Al NFs. This process was completed without a catalyst unlike the other methods. FESEM, EDX, RAMAN and XRD were used to investigate the surface features of the sample and its content and the optical properties of the sample were analyzed by using PL and UV-Vis spectrophotometer.

1.5 Significances Study

The development of nanotechnology gives impact in the quality of human life including computer, textile, medicine, communication and economy. NFs have unique morphology which are expected to show different magnetic, optical and electrical properties from their bulk 3D structures. Thus, the study on structural and optical of Al doped ZnO NFs with different dopant concentrations will contribute a new knowledge in the gas sensing and electronic industry for better live of mankind.

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