SYNTHESIS AND CHARACTERIZATIONS OF ALUMINUM/GALLIUM CO-DOPED ZINC-OXIDE NANOSTRUCTURES FOR HYDROGEN SENSORS

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ABSTRACT

Transparent electrode materials with the transparency of up to 80% in the visible wavelength region and resistivity as low as 10^{-3} Ω.cm are in high demand for various optoelectronic applications. It is believed that nanostructured Zinc oxide (ZnO) with modified electronic structure properties may be a promising alternative to Indium tin oxide (ITO). This is due to its similarity in band gap energy. This research attempted to improve the structural, morphological, optical and electrical properties of ZnO nanostructures by co-doping it with aluminium (Al) and gallium (Ga) (AGZO) in order to use the material as a hydrogen gas sensor. To achieve this goal, a series of Al/Ga co-doped ZnO nanostructure (NS) films with new morphologies was synthesised on the p-type Si (100) substrate using combined sol-gel and spin coating methods. Samples were annealed at different temperatures, time durations and laser energies to examine their effects on overall properties. The influence of varying Ga content was determined. FESEM images showed two new different morphologies for two different samples with increasing Ga content nanoleaves Ga (4 at. %) and nanopeanuts-like (5 at. %). The optical bandgap energy (3.26 - 3.20 eV) of AGZO nanofilms was discerned to be lower than pure ZnO films (3.37 eV). The resistivity of the prepared AGZO nanofilms at 1 at. % of Ga was found to be 4.571×10^{-3} Ω.cm which was lower than Ga free (AZO) thin films (6.4×10^{-2} Ω.cm). The sensing performance of the AGZO film was evaluated at (100 and 150) ºC under varying H_2 gas concentrations (250 – 1750 ppm). The best sensitivity was achieved at a H_2 gas concentration of 1750 ppm. The improvement of overall properties was attributed to Ga mediated enhanced polycrystalline growth of the films and the production of new two different nanostructures. It was affirmed that the present method of sample preparation is simple and economical. The systematic characterisations might constitute a basis for producing high-quality ZnO nanofilms suitable for assorted applications. The laser annealing is a fast, cheap and better method than the thermal annealing method, but the annealing by the thermal method is the best to make a good hydrogen gas sensor in this study.

ABSTRAK

Bahan elektrod lutsianr dengan kelutsianran melebihi 80% dalam julat panjang gelombang nampak dan kerintangannya serendah 10^{-3} Ω.cm mempunyai permintaan yang tinggi bagi pelbagai aplikasi optoelektronik. Adalah dipercayai Zink oksida (ZnO) nanostruktur dengan sifat-sifat struktur elektronik yang diubahsuai mungkin merupakan salah satu alternatif yang baik kepada indium tin oksida (ITO). Ini adalah disebabkan tenaga jurang jalurnya yang hampir sepadan. Kajian ini bertujuan untuk meningkatkan lagi sifat-sifat ZnO nanostruktur darisegi struktur, morfologi, optik dan elektrik melalui dop ber sama dengan alumium (Al) dan gallium (Ga) (AGZO), membolehkan bahan tersebut digunakan sebagai sensor gas hidrogen. Untuk mencapai matlamat ini, satu siri filem Al/Ga co-dop pada filem ZnO nanostruktur (NS) dengan morfologi baru disintesis pada substrat jenis p Si (100) menggunakan kaedah salutan gabungan larutan gel dan putaran. Sampel telah disepuhlindap pada suhu, tempoh masa dan tenaga laser yang berbeza-beza untuk mengkaji kesan sifat-sifat keseluruhan terhadapnya. Pengaruh kandungan Ga yang berbeza-beza terhadapnya telah ditentukan. Imej FESEM menunjukkan, dua morfologi yang berbeza untuk dua sample berlainan dengan peningkatan Ga mengandungi "nanoleaves" Ga (4 at.%) dan "nanopeanuts-like" (5 at.%). Tenaga jurang jalur optik nanofilem AGZO (3.26 - 3.20 eV) didapati lebih rendah daripada filem ZnO tulen (3.37 eV). Kerintangan nanofilem AGZO yang disediakan pada 1 at.% daripada Ga didapati pada 4.571×10^{-3} Ω.cm lebih rendah daripada filem tipis bebas Ga (AZO) (6.4×10^{-2} Ω.cm). Prestasi penderia filem AGZO dinilai pada 100 °C dan 150 °C di bawah kepekatan gas H_2 yang berbeza-beza (250 - 1750 ppm). Kepekaan terbaik dicapai pada kepekatan gas H_2 1750 ppm. Peningkatan sifat-sifat keseluruhan adalah disebabkan oleh pengantara Ga yang meningkatkan pertumbuhan polikristal bagi filem-filem tersebut dan pengeluaran dua nanosturuktur yang berbeza. Ini mengesahkan bahawa kaedah penyediaan sampel yang sekarang ini adalah mudah dan menjimatkan. Pencirian-pencirian yang sistematik mungkin membentuk asas untuk menghasilkan nanofilem ZnO yang berkualiti tinggi sesuai untuk pelbagai aplikasi. Penyepuh laser adalah kaedah yang cepat, murah dan lebih baik daripada kaedah penyepuh haba, tetapi penyepuhlindapan dengan kaedah termal adalah yang terbaik bagi menghasilkan sensor gas hidrogen pada kajian ini.

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LIST OF ABBREVIATIONS

0D	-	Zero Dimension
1D	-	One Dimension
2D	-	Two Dimensions
3D	-	Three Dimensions
AFM	-	Atomic Force Microscopy
AGZO	-	Aluminum Gallium co-doped Zinc Oxide
CB	-	Conduction Band
COP	-	cyclo-olefin polymer
CVD	-	Chemical Vapor Deposition
DW	-	Distilled Water
EDX	-	Energy Dispersive X-ray Diffraction
FESEM	-	Field Emission Scanning Electron Microscopy
FQ	-	Fused Quartz
FT-IR	-	Fourier Transform-Infra Red
FTO	-	Fluorine-Doped Tin Oxide Coated glass
h	-	Hour
HRTEM	-	High Resolution Transmission Electron Microscopy
IR	-	Infrared
ITO	-	Indium Tin Oxide
JCPDS	-	Joint Committee of Powder Diffraction Standards
MBE	-	Molecular Beam Epitaxy
MOCVD	-	Metal Organic Chemical Vapor Deposition
NBE	-	Near Band Edge Emission
NBs	-	Nanobelts
Nd:YAG	-	Neodymium-Doped Yttrium Aluminum Garnet
NFLs	-	Nanoflakes
NFs	-	Nanoflowers
NFs		Nanoflowers
NIR	-	Near Infrared
NLs	-	Nanoleafs
Nm	-	Nanometer

NPNs - Nanopeanuts
NPs - Nanoparticles
NRs - Nanorods
NSs - Nanostuructures
NWs - Nanowires
PL - Photoluminescence
PLD - Pulsed Laser Deposition
PVD - Physical Vapor Deposition
p-ZnO - Pure ZnO
QCEs - Quantum Confinement Effects
QDs - Quantum Dots
RF - Radio Frequency
RF - Radio Frequency
RT - Room Temperature
SEM - Scanning Electron Microscopy
SEM - Scanning Electron Microscopy
TEM - Transmission Electron Microscopy
UV - Ultraviolet
UV - Ultraviolet
VB - Valance Band
Vis - Visible
V_{zn} - Zinc Vacancies
XPS - X-ray photoelectron spectroscopy
XRD - X-ray Diffraction
Zn_i - Zinc Interstitials
ZnO - Zinc Oxide
Zn_o - Zinc Antisites
ZNPs - Zinc Oxide Nanoparticles
ZNSs - Zinc Oxide Nanostructures
ZAD - Zinc acetate dehydrate
MEA - Monoethanolamine
IPA - Isopropanol

LIST OF SYMBOLS

I - Current

V - Voltage

α - thermal diffusivity

λ - Wavelength

μ - electron mobility

ρ - Resistivity

D - grain size

V - volume

δ - dislocation density

Θ - diffraction angle

β - Full Width Half Maximum

ϵ - Strain

Tc - texture coefficient

σ - Stress

L - bond length

μH - Hall mobility

μ - Mobility

R - Resistance

ρ - Density

n_e - electron concentration

hkl - Miller indices

d_{hkl} - inter planer distance

a,c - lattice parameters

E_g - energy gap

X_c - the fraction of crystalline phase

E_L - laser energy

S - sensitivity

M - molecular weight

Ov - Oxygen Vacancies

Oi - Oxygen Interstitials

O_{Zn} - Oxygen Antisites

n-type - Donor type

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

At present, the sophisticated tools needed for functional materials designs, characterisations, synthesis, and applications encompass the manipulation of diverse materials in the range of 1 to 100 nm (nanoscale). Distinctive facets including indirect, direct and conceptual aspects are exploited for the evolution of nanoscience and nanotechnology (Shastri et al., 2010). The indirect aspect of nanotechnology refers to the advancement in the miniaturisation of existing technologies which open up new avenues of applications. In addition to indirect, direct signifies the appliance of new nanoscale objects to develop a novel process, material, mechanism and performance for completely exotic purposes. Lastly, the conceptual aspect of nanotechnology encompasses all nanomaterials and processing technologies at the molecular or atomic level particularly involving the living organism and bio-system. For instance, information and communication technology (ICT) benefitted tremendously from emerging nanotechnology concepts where novel semiconductor and optoelectronic materials were synthesised to achieve new nanodevices (Michael et al., 2008).

Nanotechnology has penetrated every field of science and technology including the environmental sciences (filtration), the energy sectors (for energy cut, tackle increased energy consumption, efficient production of energy, cleaning up nuclear accidents and storing and recycling of wastes), heavy industries (aerospace engineering and chemicals), consumer products, bio-medicine, food technology, forensics, and defence. In the past few decades, rapid progress in nanomaterials synthesis and characterisations has opened up several new avenues of nanotechnology. Many advanced functional materials with unusual properties have been developed (Poole et al., 2003).

In the area of wide bandgap semiconductor materials, ZnO thin films have extensively been used in many applications including piezoelectric transducers, windows in solar panels, gas sensing, devices for the detection of surface acoustic waves, optical waveguides, acousto-optic devices, transparent conductive electrodes and varistors (Look, 2001). ZnO is a unique semiconductor because of its broad bandgap (~3.37 eV) and high exciton binding energy of 60 meV (Triboulet et al., 2003). Furthermore, it is a potential candidate for blue and ultraviolet (UV) light-emitting diodes and laser diodes, backlight diodes for liquid crystal display (LCD), mobile phones and automotive lighting. Nowadays, it is intensively used in optoelectronic devices because of its strong excitonic absorption and recombination at room temperature (Yamamoto et al., 2001).

The existence of excess conduction electrons in ZnO nanostructure leads to an enhanced electrical conductivity. This is useful in transparent conductive electrodes as a substitute to indium tin oxide, commonly referred to as ITO. The primary sources of these excess electrons are defects (Kohan et al., 2000), which can be intrinsic or extrinsic impurity defects. Extrinsic defects are recognized as dopants when deliberately introduced to a system to enhance various properties (Maller, 2016). Three types of defects are present in the ZnO crystal: vacancies, interstitial atoms and antisites of oxygen and zinc (Flemban, 2017).

Zinc oxide is a natural wurtzite structure which is an n-type semiconductor due to intrinsic defects such as O vacancies (V_O) and Zn interstitials (Zn_i). Al acts as a donor on a lattice site [Al_{Zn}] and an acceptor on an interstitial site [Al_{Zni}]. Al atoms are substituted into the structure due to the fact that the Al^{3+} radius (0.054nm) and Zn^{2+} radius (0.074nm) are similar. Thus, Al^{3+} ions easily substitute for Zn^{2+}. Zinc has two valence electrons and aluminium has three valence electrons.

It is confirmed that the concentration of oxygen vacancies and Zn interstitials are the most abundant native defects and can be changed with the Zn partial pressure. The results of an electron concentration displayed that with a high partial pressure of Zn, electrons dominate the content of the hole (Kohan et al. 2000). Thus, intrinsic ZnO donors do not significantly participate in the electrical conduction in ZnO. Elements in-group III (Al, Ga and In) with valence cell configuration of ns_2 np_1 possess lower ionisation energy compared with Zn (ns_2). Thus, such elements can substitute Zn^{2+} ions in the lattice site of ZnO crystal. Donation of excess electrons increases the carrier concentrations to a value greater than 10^{20} cm^{-3}, resulting in an enhancement in electrical conductivity (Yamamoto et al., 2001).

To synthesise ZnO nanoparticles, many researchers have used physical methods such as radio frequency (RF) sputtering (Uikey et al., 2016) and molecular beam epitaxy (MBE) (Maller, 2016). However, structural inhomogeneity and cost remain disadvantageous for large-scale synthesis by physical means (Diaz De Leon et al., 2017). On the other hand, many researchers have synthesised ZnO nanoparticles using chemical methods such as chemical bath deposition (Flemban, 2017), hydrothermal synthesis (Wei et al., 2013) and the sol-gel method (Maache et al., 2017). Amongst these techniques, sol-gel is the preferred for the preparation of thin film owing to its low cost, outstanding control on the precursor solution stoichiometry, simple modifications of compositions, homogeneity, low temperature and non-requirement of a vacuum (Vajargah et al., 2013). Moreover, sol-gel deposited ZnO thin film samples reveal a high performance and a smaller number of defects which are beneficial for many applications. Several authors have reported the impact of sol-gel on controlling ZnO nanoparticle's shape and size (Diaz De Leon et al., 2017). Compared to other chemical methods in terms of reaction mechanisms such as hydrolysis and condensation, the sol-gel method offers highly uniform dispersion of dopants in the ZnO crystal lattice. Some researchers have used the sol-gel method with polymerising agents such as starch, nitric acid and dextrose to control the nanoparticles' shape and size (Diaz De Leon et al., 2017). Moreover, wide bandgap materials are chemically and thermally more stable, which is an advantage for devices operating in harsh environments (Liu et al., 2010). Among these materials is ZnO which has been studied extensively in recent years for its unique properties and potential application in electronic and optoelectronic devices (Liu et al., 2010). It has strong radiation hardness, high chemical stability, low cost and a large bandgap of 3.37 eV at room temperature (Liu et al., 2010).

Hydrogen is a non-toxic, non-poisonous, colourless, odourless and tasteless gas that cannot be detected by human senses. Hydrogen has a very low density (0.0899 kg/m^3) with a high diffusion coefficient in the air (0.61 cm^2/s). The H_2 content in the air at sea level is 0.5 ppm and has no threshold limit value (Schwandt & Fary 2000). Hydrogen can be ignited easily with a very small amount of energy, as low as 0.02 mJ (Hübert et al. 2011) and the explosive range is wide, from 4% to 75% (Al-Hardan et al. 2010). The monitoring of hydrogen concentration is essential to nuclear reactor safety (Hübert et al. 2011). In coal mines, hydrogen can be produced in the ppm^2 range by methane or coal-dust explosions or by the spontaneous heating and low-temperature oxidation of coal (Brungs et al. 1992).

Hydrogen is an energy carrier and can contribute to overcoming problems of dwindling fossil fuel reserves, energy supply security and climate change (Hübert et al. 2011). In 2006, 10,638 thousand tons of H_2 was produced in the United States. Most of this H_2 was used in petrochemical plants, fertiliser plants (ammonia production) and the methanol industry (Korotcenkov et al. 2009). A minuscule fraction of H_2 (50 tons in 2006) was used as an alternative fuel for electric power generation and transportation. This small fraction of H_2 used in power generation and transportation is growing rapidly, resulting in a greater presence and distribution of H_2 in society. In this emerging hydrogen economy, the detection of hydrogen leaks and the measurement of hydrogen concentration are necessary during production, storage, transportation and use in both stationary and mobile applications (Hübert et al. 2011). Sensors will, therefore, be used for safety monitoring of hydrogen production plants, pipelines, storage tanks, refuelling stations and automotive vehicles (Hübert et al. 2011).

Alternative hydrogen detection methods employ instruments such as gas chromatographs, mass spectrometers or specific ionisation gas pressure sensors. Traditionally, these instruments are large, expensive, high maintenance and slow in terms of their sampling and reaction times (Hübert et al. 2011). Therefore, it has become a priority in research to investigate various techniques to develop a H_2 sensor with a high sensitivity and a fast response time. Hydrogen sensors have several advantages over the conventional hydrogen detection methods, including their lower cost, smaller size, and faster response. These advantages make them more suitable for portable and in situ hydrogen detection in a range of applications (Hübert et al. 2011).

Commercially available sensors are primarily based on semiconductor metal oxides, electrochemical H_2 oxidation, catalytic response, thermal conductivity response or optical response technology. Although these sensors are sensitive and reliable, electrochemical based sensors particularly suffer from a decrease in sensitivity with time due to deterioration of the electrode catalyst. Catalyst based sensors are not specific to H_2 and often need high temperatures or high power for their operation. The most successful commercial sensors based on metal oxides are small in size, highly responsive, cheap and operate using a relatively simple electric circuit. However, these sensors require a significant pre-heating of the sensing element up to 300-700 °C to achieve the desired sensitivity and accuracy (Lupan et al. 2010). Recently, efforts in H_2 sensor development using metal oxides have focused on improving the sensitivity and decreasing the operating temperature. In-room temperature sensing ZnO has shown improved response and short recovery time due to its wide band-gap and surface adsorbed oxygen species. Furthermore, it is a cost-effective, easy, fast reproducible synthesis in nanostructured form.

Research has found that the resistivity of AGZO thin films (2.14×10^{-3} Ω-cm) is lower than AZO thin films (6.40×10^{-3} Ω-cm) (Lee et al. 2009). Furthermore, (Han et al. 2010) reported that depending on the Ga doping level, the grain size increases to reduce grain boundary scattering. In their study, the resistivity of AZO films decreased from 3.5×10^{-3} to 8.1×10^{-4} Ω.cm by Ga doping at 2.1 at.%. By Ga doping, the Hall mobility was improved, and the carrier concentration was increased. Ga acted as an intrinsic donor. Enhancing the conductivity of AZO nanostructures by Ga doping using sol-gel and spin coating which can synthesize tow and three-dimensional nanostructure, the efficiency of devices can be enhanced. Thus, the synthesis of co-doped ZnO nanostructures using sol-gel and spin coating is reported.

1.2 Problem Statement

Nanostructured ZnO can be synthesised using a number of methods including vapour solid (VS), vapour-liquid-solid (VLS), radio frequency (RF) and sol-gel. VS and VLS require high temperatures for the growth of nanostructures. RF requires expensive instruments and specialised laboratory setup. On the contrary, the sol-gel method is cost-effective and can synthesise ZnO with controlled nanostructure morphology and dopants. However, the sol-gel methods for the synthesis of a ZnO nanostructure on Si substrate are not well characterised.

One dimensional (1D) and two-dimensional (2D) ZnO nanostructures have a high surface to volume ratios, which make them valuable for a variety of applications (Ali et al., 2010). Although there are different methods used to fabricate 2D ZnO nanostructures, the sol-gel spin coating method is the easiest and is able to fabricate 2D ZnO nanostructures at low temperatures (Cheng, et al., 2009).

It has been found that the structural, electrical and optical properties of ZNS-based thin films can remarkably be improved by co-doping with Aluminium and Gallium (Al/Ga) but a few studies have explored this possibility thus far. Moreover, the capacity of combined sol-gel and spin-coating (a chemical approach) for the preparation of ZNSs-film co-doped with Al/Ga has not been reported in the literature, but they were used many (physical approaches) . It is necessary to prepare high-density ZnO NSs-film co-doped with Al/Ga using a novel combined technique of laser annealing with different energies (Vajargah et al., 2013). This method needs to be implemented under different growth conditions to determine the influence of changing substrates type, annealing temperature and time, laser annealing energy and Al/Ga concentration ratio on the structures, morphologies, electrical as well as optical properties of ZnO NSs-film samples co-doped with Al/Ga. Growth optimization is a prerequisite to achieving optimum ZnO NSs-film co-doped with Al/Ga which is advantageous for high-performance gas sensing applications (Ivanova et al., 2015). The limitations associated with the existing synthesis techniques of Al/Ga co-doped ZNSs-film must be overcome by developing a simple, economical and eco-friendly sol-gel method. For diverse applications, high-density ZnO nanorods are required.

Moreover, in some cases, the sensor fabrication step requires the addition of various dopants which are very complicated and time consuming. The mono-doped (single element doping) system including Al:ZnO and Ga:ZnO thin films have some issues related to their stability, mechanical strength, efficiency morphology and hall mobility that are disadvantageous for applications. By co-doping ZnO with more than one of the group III metallic elements these issues can be resolved (Bu, 2016). In addition to this, the expensive nature of Ga limits the mass production of Ga doped ZnO nanofilm. The impact of substrate type (Si) and changing laser annealing energy on the surface morphology of ZnO thin film grown by sol-gel route remains unresolved.

Researchers have used Al as a substrate to form the porous structure of ZnO nanoplates. Using Al as a substrate has some potential problems: (i) Al is quickly oxidised by atmospheric oxygen, (ii) Al is a good conductor and therefore a thick layer of Al under the ZnO nanostructure might be problematic in its application in electrical devices. The substrate used is p-type (Si) and changing laser annealing energy on the surface morphology of ZnO thin film grown by the sol-gel route remains unresolved.

To measure the gas sensing potency of optimum Al/Ga co-doped ZNSs-film sample grown by the sol-gel method, a sensor must be indigenously designed, and the sensing mechanism must be understood. Sample characterisations are necessary to determine the optical, morphological and electrical correlation effects useful for the development devices. A comprehensive understanding of the mechanisms behind the modifications of the overall characteristics of ZnO NSs-film co-doped with Al/Ga is significant. Experimental results must be compared and validated with other similar findings. Thus, high-density AGZO nanofilm with diverse morphology was synthesised and subsequently characterised to evaluate the structural, optical, electrical and gas sensing properties of the NSs-film. However, despite extensive research on ZnO nanostructure synthesis and characterisation, well-controlled ZnO nanostructures-based films with desirable electrical and optical properties are far from being achieved.

To summarise the issues that arise in this process, the sensor fabrication step requires the addition of various dopants which is very complicated and time-consuming. Reproducible and controlled growth of ZnO nanostructures with desirable attributes using an easy method is necessary for many applications. Although several methods have been used to synthesise ZnO nanostructures, they suffer from many challenges. Vapour solid (VS) and vapour-liquid-solid (VLS) require a high temperature for the nanostructures' growth, while radio frequency (RF) sputtering requires costly instruments and a particular laboratory setup. The sol-gel method is cost-effective and can generate controlled nanostructures of ZnO with morphology and dopants. However, the uses of sol-gel method for synthesising are not well described. One dimensional (1D) and two dimensional (2D) ZnO nanostructures have high surface to volume ratios, which make them valuable for many applications (Ali et al., 2010). Although there are different methods to fabricate 2D ZnO nanostructures, the sol-gel spin coating method is the easiest and is able to fabricate 2D ZnO nanostructures at low temperatures. Based on the above-mentioned issues, this research proposes alternative methods in order to minimize cost and time.

1.3 Research Objectives

Based on the previously mentioned research and the research gaps the following objectives are set:

i) To synthesise pure, ZnO NSs-thin films doped with Al content from (0 to 5 at.%), and co-doped with Al/Ga with varying Ga level (0-5) at.% by using a combined sol-gel and spin coating technique on Si-substrate by thermal annealing.

ii) To determine the processing parameters (annealing temperature, time annealing, Ga dopant concentration and laser energy) dependent structure, morphology, optical and electrical properties of synthesised AZO samples through comprehensive characterisations on Si-substrate.

iii) To measure the hydrogen gas sensitivity of grown AGZO nanofilm (containing vertically aligned nanorods morphology) by manufacturing a gas sensor for the two types of annealing (thermal and laser).

1.4 Scope of the Study

This research involves the synthesis and thorough characterisations of ZnO NSs-thin films doped with Al and co-doped with Al/Ga. Samples will be analysed to determine their structural, morphological, optical and electrical features. The design of a gas sensor using the optimum AGZO film sample is the application-oriented target. The preparation of a series of NSs-thin films of pure zinc-oxide (ZO), Al-doped zinc-oxide (AlZO), and ZnO co-doped with Al/Ga (AGZO) with diverse morphologies on the p-type Si (100) substrates will be carried out using combined sol-gel and spin-coating techniques. Raw materials of zinc-acetate-dehydrate, ZnAc [$Zn(CH_3COO)_2.2H_2O$], aluminium-nitrate-nonahydrate [$Al(NO_3)_3{\cdot}9H_2O$], gallium nitrate nonahydrate [$Ga(NO_3)_3{\cdot}9H_2O$], 2-propanol [C_3H_8O] and ethanolamine (EA) will be used for growing such ZnO thin films. Zn precursor solution will be prepared by the dissolution of [$Zn(CH_3COO)_2.2H_2O$] in 2-propanol (0.1 M) and EA. Spin-coating at 3000 rpm speed and drying at 300 °C for 20 min on Si wafer will be completed. Furthermore, the execution of thermal and laser annealing treatment on the prepared thin films for the improvement of properties will be carried out. Samples of thermal annealing will performed at different temperature (425, 450, 475 and 500) ºC and for varying time durations (between 0 to 3 h) and for the laser energy annealing to examine their impact of overall properties. Al doping concentration will fixed at 1 at.% and Ga contents will varied (0 to 5 at.%) to optimize the composition of AGZO nanofilms.

Room temperature structural and morphological characterisations of the as-grown thin films samples will be taken using atomic force microscopy (AFM), scanning electron microscopy (SEM), field emission scanning electron microscopy (FESEM), X-ray diffraction (XRD) and energy-dispersive X-ray (EDX) spectroscopy. The optical properties of these samples will be examined using photoluminescence (PL), Raman and UV-Vis spectroscopy. The 4-point probe method will be used to measure the electrical resistivity and (I-V) characteristics of samples. Finally, a hydrogen gas sensor will be designed based on the optimum AGZO nanofilm sample to demonstrate its potential for gas sensing. The sensing performance of the synthetic

AGZO NSs film will be evaluated at 100 ºC and 150 ºC under varying hydrogen gas concentrations of 250, 500, 1250 and 1750 ppm.

1.5 Significance of the Study

Understanding the growth mechanism and improvement of various properties of AGZO nanofilm is fundamentally important. Despite this, a systematic approach for depositing such film with high quality and desired morphology using the sol-gel assisted method has not yet been developed. Thus, this study is significant in terms of developing a systematic method for the synthesis of good quality AGZO films required for diverse applications especially for gas sensors and transparent electrodes. This study is expected to resolve some of the issues related to traditional growth methods. Furthermore, the achieved new findings will contribute towards the development of ZnO-NSs thin films co-doped with Al/Ga which are useful for efficient gas sensors. So far, the effects of substrate, annealing laser shooting, growth time and temperature on the structure, morphology, optical and electrical properties of AGZO films have not been systematically determined. Growth of high quality samples requires careful optimization of the processing parameters.

Controlled morphology and structure with high crystallinity required for the optoelectronic industries are believed to be achievable by sol-gel assisted spin-coating and laser annealing methods. The performance evaluation of the deposited AGZO nanofilms in terms of efficiency, cost, large scale reproducibility and environmental friendliness will be an interesting aspect to investigate. Using the proposed characterisation tools, it is possible to measure the defects, crystallinity, NSs morphology, optical band gap, optical absorbance, elemental traces, NPs shape, size and distribution, surface roughness, crystal density, Hall mobility, resistivity, carrier concentrations and gas sensing attributes. Furthermore, the proposed method can also be extended to grow another kind of semiconductor NSs in a well-controlled and reproducible manner.

1.6 Thesis Outline

This thesis is composed of five chapters with the following highlights:

Chapter 1 provides a brief research background to introduce the problem and to show why this research topic is relevant and worth undertaking. A brief review is provided to show the existing research gap and the issues to be resolved. The problem statement, research objectives, scope of the research and the significance are described.

Chapter 2 presents a detailed literature review to justify the problem statement and the need for further research to fill the current gaps in the literature. A gentle introduction will be presented to the development of ZnO NSs, various methods of synthesis with their notable merits and demerits, characterisation tools, un-doped and doped ZnO films, and applications of these NSs. Detailed theories of ZnO nanostructure growth mechanisms, the principle of the techniques used to grow ZnO films and the mechanisms for gas sensing are discussed.

Chapter 3 describes the research methodology in terms of raw materials, compositions, sample preparation techniques, optimization procedure, characterisation tools and their models, methods of data collection and subsequent analysis.

Chapter 4 discusses the experimental results and analysis, interpreting them using various mechanisms, and comparing the present results with earlier findings in the literature. This chapter demonstrates how the results obtained can fulfil the proposed research objectives. The role of different annealing temperatures, time and laser energy shoots on the surface morphological, structural, electrical and optical behaviours of ZnO-NSs thin films co-doped with Al/Ga synthesised by sol-gel and laser annealing are underlined. Furthermore, the gas sensing attributes of the optimum sample are discussed.

Chapter 5 concludes the thesis by proposing recommendations for future research in this field.

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APPENDIX A

LIST OF PUBLICATIONS

Journal with Impact Factor

1. **Hayder J. Al-Asedy**, Noriah Bidin, Khaldoon N. Abbas, Mohammed A. Al-Azawia. Structure, morphology and photoluminescence attributes of Al/Ga co-doped ZnO nanofilms: Role of annealing time.Materials Research Bulletin 97 (2018) 71–80. https://doi.org/10.1016/j.materresbull.2017.08.050**.(Q2,IF: 2.873)**
2. **Hayder J. Al-Asedy**, Ali A. Ati, Noriah Bidin, Siew-Ling Lee. Gallium contents-dependent improved behavior of sol–gel-grown Al:Ga co-doped ZnO nanostructures. Appl. Phys. A (2017) 123:665. https://doi.org/10.1007/s00339-017-1271-0 **.(Q2.IF: 1.604)**
3. **Hayder J. Al-Asedy** ,Al-Khafaji, S.A., Bakhtiar, H. and Bidin, N., 2018. Properties of Al-and Ga-doped thin zinc oxide films treated with UV laser radiation. Applied Physics A, 124(3), p.223. https://doi.org/10.1007/s00339-018-1619-0.**(Q2,IF:1.604)**
4. **Hayder J. Al-Asedy**, H.J., Bidin, N., Al-khafaji, S.A. and Bakhtiar, H., 2018. Sol-gel grown aluminum/gallium co-doped ZnO nanostructures: Hydrogen gas sensing attributes. Materials Science in Semiconductor Processing, 77, pp.50-57. https://doi.org/10.1016/j.mssp.2018.01.011**.(Q2,IF:2.593)**
5. Khaldoon N. Abbas, Noriah Bidin, Raad S. Sabry, **Hayder J. Al-Asedy**, Mohammed A. Al-Azawi and Shumaila Islam b. Structures and Emission Features of High-Density ZnO Micro/Nanostructure Grown by an Easy Hydrothermal Method. Materials Chemistry and Physics, 2016. 182: 298-307. https://doi.org/10.1016/j.matchemphys.2016.07.035.**(Q2,IF:2.210)**

Indexed Scopus Journal

1. **Hayder J. Al-Asedy**, Noriah Bidin, Khaldoon N. Abbas and Mohammed A. Al-Azawi.ZnO QDs Deposited on Si by Sol-Gel Method: Role of Annealing Temperature on Structural and Optical Properties.Modern Applied Science, 2016. 10(4): 12. DOI:10.5539/mas.v10n4p12.(**(Indexed by SCOPUS)**

Indexed Conference Proceedings

1. Khaldoon N. Abbas, Noriah Bidin, Mohammed A. Al-Azawia and **Hayder J. Al-Asedy.** Nanostructural and Optical Properties of Hierarchical ZnO Grown via Hydrothermal Method.Jurnal Teknologi, 2016. 78(3): 1-5. **(Indexed by SCOPUS)**
2. Khaldoon N. Abbas, Noriah Bidinb, Mohammed A. Al-Azawia and **Hayder J. Al-Asedy**. Facile Hydrothermal Synthesis of Flower-like ZnO Nanorods without Catalysts. *Jurnal Teknologi*, 2015. 74(8): 15-18. **(Indexed by SCOPUS)**
3. Mohammed A. Al-Azawi, Noriah Bidin, M Abdullah, Abdulrahman K. Ali, Khaleel I. Hassoon, Khaldoon N. Abbas and **Hayder J Al-Asedy**. Surface Plasmon Resonance Effects of Gold Colloids on Optical Properties of N719 Dye in Ethanol. Optoelectron. Adv. Mater, 2015. 17(3-4): 264-269. **(Indexed by SCOPUS)**
4. Mohammed A Al-Azawi, Noriah Bidin, M Bououdina, Khaldoon N. Abbas, **Hayder J Al-Asedy**, Omar H Ahmed and Asad A Thahe. The Effect of the Ambient Liquid Medium on the Ablation Efficiency, Size and Stability of Silver Nanoparticles Prepared by Pulse Laser Ablation in Liquid Technique.Jurnal Teknologi, 2016. 78(3): 7-11. **(Indexed by SCOPUS)**