THE EFFECT OF GROWTH TEMPERATURES AND SUBSTRATE ORIENTATIONS OF INDIUM GALLIUM PHOSPHIDE QUANTUM WIRES

NOR HAMIZAH BINTI NOR HELMI

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To my loved ones:

abah Nor Helmi Zakaria and ibu Johari A. Rahman;

both my family & my in-laws' family;

my other half, Mohd. Farhan Mohd. Rafee;

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ABSTRACT

Indium gallium phosphide (InGaP) nanowires were grown on gallium arsenide (GaAs) substrate by using metal-organic chemical vapour deposition (MOCVD) via vapour-liquid-solid (VLS) technique. The grown InGaP wires were characterized by using scanning electron microscopy (SEM), field emission scanning electron microscopy (FE-SEM), transmission electron microscopy (TEM) and energy dispersive X-ray spectroscopy (EDX). The results showed that the growth of the InGaP wires depend strongly on the growth temperatures and substrate orientations. At low temperature of 380°C, wires grown on GaAs substrates were almost cylinder-like, while with increasing temperatures ranging from 410-500°C, the reverse-tapering phenomenon occurred producing microphone-like InGaP nanowires. With increasing temperature from 380-500°C, Au alloy-seed particle or the head part of the InGaP nanowires were also observed to be thicker in diameter (85-452 nm) than the body part (49.2-224.3 nm), which had been proven by the chemical compositions percentage in EDX analysis. Wires grown on GaAs (100) substrate were less than number of wires on GaAs (111) *B* substrate although wires on both substrates are basically grown on random directions. Twin boundaries crystal defects have been detected on some of the InGaP nanowires structures from TEM analysis. Such defects are not good and should be avoided to prevent further problem when being incorporated into potential devices. The crystal structure for all samples of InGaP wires is zinc blende (ZB), the lattice spacing *(d)* of InGaP wires is 3.342Å, and the lattice parameter *(a)* is 5.788Å.

ABSTRAK

Wayar nano indium galium fosfida (InGaP) telah ditumbuhkan di atas substrat galium arsenida (GaAs) dengan menggunakan pemendapan wap kimia logam organik (MOCVD), melalui teknik wap-cecair-pepejal (VLS). Semua wayar nano InGaP yang tumbuh telah dikaji menggunakan mikroskop imbasan elektron (SEM), mikroskop elektron pengimbas pancaran medan (FE-SEM), mikroskop transmisi elektron (TEM) dan spektroskopi sebaran tenaga sinar-X (EDX). Keputusan menunjukkan bahawa pertumbuhan wayar InGaP adalah sangat bergantung kepada suhu pertumbuhan dan orientasi substrat. Pada suhu yang rendah iaitu 380°C, wayar yang tumbuh di atas substrat GaAs adalah hampir menyerupai silinder, tetapi dengan kenaikan suhu dengan julat 410-500°C, fenomena 'tirus-terbalik' berlaku dalam menghasilkan wayar nano InGaP yang menyerupai mikrofon. Dengan kenaikan suhu dari 410-500°C juga, artikel benih-aloi emas di bahagian kepala wayar InGaP dapat dilihat lebih tebal diameternya (85-452 nm) berbanding bahagian badan wayar tersebut (49.2-224.3 nm), di mana hasil ini dapat ditunjukkan melalui keputusan peratusan komposisi kimia oleh analisis EDX. Bilangan wayar nano yang tumbuh di atas substrat GaAs (100) adalah kurang berbanding bilangan wayar nano di atas substrat GaAs (111) B, walaupun wayar di atas kedua-dua substrat tumbuh pada arah yang rawak dan tidak tersusun. Kecacatan kristal sempadan berkembar pada struktur wayar InGaP dapat dilihat daripada analisis TEM. Kecacatan seperti ini adalah tidak baik dan perlu dielakkan bagi membendung masalah yang berkemungkinan muncul apabila wayar nano tersebut digunakan bagi membina alat yang berpotensi. Struktur kristal bagi semua sampel wayar InGaP adalah zink blend (ZB), jarak kekisi *(d)* bagi wayar InGaP tersebut ialah 3.342Å, manakala parameter kekisi *(a)* pula ialah 5.788Å.

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LIST OF ABBREVIATIONS

InGaP	-	Indium Gallium Phosphide
QWR	-	Quantum wire
QWRs	-	Quantum wires
0D	-	Zero-Dimensional
1D	-	One-Dimensional
2D	-	Two-Dimensional
3D	-	Three-Dimensional
Si	-	Silicon
Ge	-	Germanium
ZnO	-	Zinc Oxide
GaAs	-	Gallium Arsenide
InP	-	Indium Phosphide
ZB	-	Zinc Blende
GaAsP	-	Gallium Arsenide Phosphide
SETs	-	Single-Electron Transistors
SHTs	-	Single-Hole Transistors
LEDs	-	Light Emitting Diodes
VLS	-	Vapour-Liquid-Solid
VSS	-	Vapour-Solid-Solid
Au	-	Aurum (Gold)
AlGaAs	-	Aluminium Gallium Arsenide
MOCVD	-	Metal-Organic Chemical Vapour Deposition
SEM	-	Scanning Electron Microscopy
FE-SEM	-	Field Emission Scanning Electron Microscopy
TEM	-	Transmission Electron Microscopy

EDX	-	Energy Dispersive X-spectroscopy
FCC	-	Face-Centred Cubic
DOS	-	Density of States
CNT	-	Classical Nucleation Theory
CVD	-	Chemical Vapour Deposition
MBE	-	Molecular Beam Epitaxy
PLD	-	Pulsed Laser Deposition
SiI_2	-	Silicon DiIodide
$SiCl_4$	-	Silicon Tetrachloride
Be	-	Beryllium
U	-	Uranium
N_2	-	Nitrogen
H_2	-	Hydrogen
TMIn	-	Trimethylindium
TMGa	-	Trimethylgallium
TMAl	-	Trimethylaluminium
PH_3	-	Phosphine
AsH_3	-	Arsine
RF	-	Radio Frequency
PLL	-	Poly-L-Lysine
ADP	-	Adsorption, Diffusion and Precipitation
eV	-	Electron Volt
ZB	-	Zinc-blende
WZ	-	Wurtzite

LIST OF SYMBOLS

F	-	Dispersions of atoms on structure
A	-	Surface area of a sphere
r	-	Radius of a sphere
V	-	Volume of a sphere
d	-	Diameter of a sphere
n	-	Number of atoms
N	-	Total number of atoms
γ	-	Surface energy
G	-	Gibbs free energy
T	-	Temperature
P	-	Pressure
N_b	-	Number of broken bonds
ε	-	Bond strength
ρ_a	-	Surface atomic density
a	-	Lattice constant/lattice parameter
D_f	-	Number of degrees freedom of electron motions
D_c	-	Number of confinement directions of electrons
kT	-	Thermal energy
δ	-	Gap between the highest occupied and lowest unoccupied state
ΔG_r	-	Change in Gibbs free energy
ΔG_v	-	Gibbs free energy difference per unit volume between solid and liquid phases at same temperature

γ_{SL}	-	Substrate-liquid surface energy
γ_{NS}	-	Nucleus-solid surface energy
L_v	-	Latent heat of fusion per unit volume at equilibrium melting temperature
T_m	-	Melting temperature
ΔT	-	Undercooling temperature
r^*	-	Critical nucleus radius
ΔG^*	-	Critical nucleation barrier
$\Delta\mu_v$	-	Change of volume free energy
$\Delta\mu_s$	-	Change of interfacial or surface energy
ΔG	-	Total free energy
J	-	Rate of nucleation
J_0	-	Available nucleation sites availability
k	-	Boltzmann's constant
θ	-	Contact angle
C_s	-	Solubility limit in the solid phase
C_l	-	Solubility limit in the liquid phase
D	-	Diffusion coefficient in gaseous phase
μ	-	Chemical potential
$\boldsymbol{\Omega}$	-	Volume per atom
σ	-	Surface energy
μ_∞	-	Bulk chemical potential (infinite radius)
L	-	Length of the wire
$\Delta\mu_\infty$	-	Steady-state supersaturation
v_∞	-	Steady-state growth velocity at infinite radius
p	-	Pressure of the system
p_o	-	Equilibrium pressure
b	-	Coefficient independent of supersaturation
Z	-	Atomic number
t	-	Time

d	-	Lattice spacing

CHAPTER I

INTRODUCTION

1.1 Background of Research

Every materials around us has substructure with quantum-scale size, which is basically range from 0.1-100 nm. Nanotechnology is closely related to the science of understanding, controlling and enabling the optimum growth of these quantum-scale structures in terms of length, time and efficiency with as little risk as possible (Bauer *et. al.*, 2006; Ramesh, 2009). The main focus of nanotechnology is basically to save up on materials while building molecules from many assembled atoms and for it to serve its own specific function, to ensure continuous capabilities and development in efficient wafer-scale electronic devices (Chuang *et. al.*, 2005; Bauer *et. al.*, 2006; Ramesh, 2009; Volz, 2009). An isolated quantum-structure has different but as important as the properties in its bulk structure (Chuang *et. al.*, 2005; Roduner, 2005; Dick, 2008; Ramesh, 2009; Volz, 2009). Because of this size reduction from bulk to

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a single quantum-structure, not only the properties are changed, but it also affects the behaviour of the structure and its usage in future electronic devices.

In this size reduction of structure to quantum-scale, it basically cause from two important effects, which is from surface effect and quantization effect. Surfaces and interfaces in solid state behave differently from other bulk materials, so surface effect is closely related to surface-to-volume ratio of a structure (Sharma *et. al.*, 2003; Ponomareva *et. al.*, 2007; Dick 2008; Volz, 2009). In bulk structure or structure bigger than 100nm, since the surface-to-volume ratio is too small, it can easily be neglected since it did not affecting the structure properties. However, in quantum structure, the surfaces give significant effect due to higher surface-to-volume ratio compared to the bulk structure, which somehow causing the change in its properties because of the strain energy in the structure. Quantization effect happen when the diameter of quantum structure is smaller than exciton Bohr diameter and Fermi length. So when the dimension of the structure is small enough (quantum-scale), the electrons will be confined by the limitation of that structure, which resulting in energy quantization inside that confining dimension (Das, 1998; Zanolli *et. al.*, 2007; Dick, 2008; Volz, 2009; Yi, 2012).

The quantum structure can be divided into four groups, two-dimensional (2D) structure: nanofilms and superlattices (stacks of nanofilms), one-dimensional (1D) structure: quantum wires, zero-dimensional object (0D): quantum particles or quantum dots, and carbon nanotubes: graphene sheets rolled into cylinder shape (Volz, 2009). In the recent years, both semiconductor and metal quantum structures have been investigated progressively for the sake of nanotechnology development. However, while there are still limited progresses when it comes to metal quantum structures applications, many accomplishments from nanotechnology were more focused on the applications of semiconductor quantum structures into electronic and

optoelectronic devices. Semiconductors are very suitable to be used in these devices, especially because their conductivity can be modified easily just by altering the use of its electric field (Dick, 2008).

Nanowires or quantum wires (QWRs) is one the quantum materials branch in quantum structure to build semiconductor. With its one-dimensional (1D) properties, nanowire or quantum wire (QWR) has been garnering a lot of attentions since it has been introduced by Wagner *et. al.* (1964) in the hope of that only a single quantum structure can be accessed easily by using external microscopic systems instead using bulk structures (Chuang *et. al.*, 2005). Quantum wires have also been widely studied because of its size reduction effect phenomena and also because of its promising potential to be used in variations of device applications (Novak *et. al.*, 2000; Lu *et. al.*, 2006; Dowdy *et. al.*, 2013). In addition, these semiconductor nanowires or quantum wires (QWRs) also have the ability to interconnect functional quantum-scale components together, with the same wires can actually be used to fabricate those components sequentially. So, in a way, their potential usage in future devices are increase with this ability to incorporate the components that is otherwise too difficult or impossible altogether to realize in two-dimensional (2D) system (Dick, 2008).

Because of these various abilities that has been showed by semiconductor quantum wires structures of their usage, especially in high-efficiency electronics and optical applications (Bauer *et. al.*, 2007; Fakhr *et. al.*, 2010), many studies were started by using elemental and binary compounds materials of semiconductors quantum wires. All these studies of semiconductors are various, which are ranged from silicon (Si), group IV material such as Ge, II-V binary metal oxide compounds like ZnO and binary III-V alloy such as GaAs and InP (Woods *et. al.*, 1994; Chuang *et. al.*, 2005; Dick, 2008; Jabeen *et. al.*, 2008; Fakhr *et. al.*, 2010, Yi, 2012).

However, researchers nowadays are more focusing on overall development of synthesizing and investigating ternary and quarternary III-V compound semiconductor. This is because most of these type semiconductor materials typically exhibit cubic zinc-blende (ZB) structure in their bulk, which are advantages especially in terms of their interesting physical characteristics, mechanical and electronic properties. Thus, these advantages are the basic reason there are many researches done to synthesize good semiconductor quantum wires while maximizing those characteristics and properties for future desired applications (Wang *et. al.*, 2002; Dick, 2008; Chuang *et. al.*, 2005).

Following the prior motivations, many researches have been more focused on phosphorus-based III-V compound semiconductors quantum wires, where from it usage are expanded and varied for many device applications (Ozasa *et. al.*, 1990). Some of these devices which containing phosphorus-based III-V semiconductors quantum wires are like GaAsP for lasers (Ozasa *et. al.*, 1990; Hua *et. al.*, 2009), InAsP for photodetectors (Pettersson *et. al.*, 2006) and InGaP for single-electron transistors (SETs) and single-hole transistors (SHTs) which also suitable for light emitting diodes (LEDs) (Ozasa *et. al.*, 1990; Matsuzaki *et. al.*, 1999; Chuang *et. al.*, 2005; Svensson *et. al.*, 2008; Fakhr *et. al*, 2010).

Among all these phosphorus-based III-V semiconductors, the most appealing phosphorus-based ternary alloy is Indium Gallium Phosphide (InGaP), which has two end binaries, InP alloy and GaP alloy, because of its possibility on accessing wide range of wavelength which ranging from visible wavelength to 925nm at room temperature (Chuang *et. al.*, 2005). According to Dick (2008), GaP is not reactive and the most stable alloy, thus, makes it the easiest to work with. While InP which have properties in between, makes it the most suitable alloy to be used and adapted into many device applications. And recently, due to the photovoltaic capabilities of

III-V semiconductor, InGaP quantum wires also showed that it can be used in a multi-junction design to the improve energy conversion efficiency (Czaban *et. al.*, 2009; Fakhr *et. al.*, 2010). The increase of interests on potentially incorporating InGaP compounds into electronic and optoelectronic device applications also mainly because of two reasons, it has higher chemical stability and also has lower surface recombination (Guimaraes *et. al.*, 1992; de Castro *et. al.*, 1999; Novak *et. al.*, 2000; Kicin *et. al.*, 2001; Kicin *et. al.*, 2004).

In building structures with at least one dimension is less than 100nm, nanofabrication, or ways to fabricate the quantum structure are needed. And generally, there are two basic approaches in fabricating quantum structures, or in specific, bulk semiconductor quantum wires. The two approaches are commonly called as top-down approach, where quantum wire structures are patterned and produced from a bulk structure, and bottom-up approach where the quantum wire is built up one atom or one molecule or one particle at a time (Samuelson, 2003; Cavallini *et. al.*, 2004; Wong *et. al.*, 2005; Dick, 2008; Ramesh, 2009, Luttge, 2011; Qin *et. al.*, 2012). In the top-down approach, a layer of bulk materials semiconductor from the desired composition are added over substrates, and then quantum-scale structures will be patterned out from that material layer by lithography process. However, like what had been reported by Harriott (2001), after some time, lithography process itself will have its own physical limits and when there are needs for this process to continuously developing, the cost of everything also will increase in finding newer technologies that can comply with this demand. In addition, because of the quantum-scale length, the bulk structure is low in uniformity and quality of the structure also will somehow become too difficult to control (Dick, 2008). While for the bottom-up approach, the nanostructure is using self-assembly process to build up itself from finer scales, one atom at a time to form much larger structures. This approach basically requires more control on the materials crystallization aspect, where quantum-scale structures will be produce from chemical reactions of vapour or

liquid sources (Dick, 2008; Ramesh, 2009). Bottom-up approach allows the production of smaller structures than the products of lithography process by top-down approach, so basically most semiconductor quantum wires can be grown through this process.

In past researches, there are five types of method growths that have been successfully used for semiconductor quantum wires. First is the catalyst-assisted vapour-liquid-solid (VLS) method (Wagner *et. al.*, 1964; Givargizov, 1975; Weyher, 1977; Nguyen *et. al.*, 2005; Kim *et. al.*, 2006; Lu *et. al.,* 2006; Chen *et. al.*, 2007; Jung *et. al.*, 2007; Dick, 2008; Jabeen *et. al.*, 2009; Hocevar *et. al.*, 2012; Yi, 2012; Vu *et. al.*, 2013), second is catalyst-free vapour-solid-solid (VSS) method (Umar *et. al.*, 2005; Wang *et. al.*, 2008; Mousavi *et. al.,* 2011, Sotillo *et. al.*, 2013), third is solution-based method (Wong *et. al.*, 2005; Lee *et. al.*, 2010; Qi *et. al.*, 2011), fourth is template-induced method (Hoogenboom *et. al.*, 2004; Urbanus *et. al.*, 2007) and fifth is lithography-based method (Glangchai *et. al.,* 2007; Liu *et. al.*, 2011). According to Wagner *et. al.* (1964), most of semiconductor quantum wires are grown using vapour-liquid-solid (VLS) method and the VLS growth process can be briefly described as when the semiconductor material start to solidify because of the precipitation effect after super-saturation of the eutectic is attained. Semiconductor quantum wires are basically formed between metal catalyst and reactant gases that impinge on the substrate. Many researches has showed that metal catalyst such as gold (Au) particle can be used as seed to the growth substrate in producing good semiconductor quantum wires (Wagner *et. al.*, 1964; Weyher, 1977; Nguyen *et. al.*, 2005; Lu *et. al.*, 2006; Chen *et. al.,* 2007; Jabeen *et. al.,* 2008; Lee *et. al.*, 2010; Hocevar *et. al.*, 2012; Vu *et. al.,* 2013).

1.2 Statement of Problems

Indium Gallium Phosphide nanowires, or also known as InGaP quantum wires (InGaP QWRs) have got so many interests because of its wide-access spectrum range, which is from visible wavelength to 925nm (Chuang *et. al.,* 2005). It has been proposed to have the best potential in substituting AlGaAs quantum wires, which currently had been used widely in many electronic and optoelectronic devices (Castro *et. al.*, 1999; Novak *et. al.*, 2000) which are like lasers, light emitting diodes (LEDs) and transistors (Ozasa *et. al.*, 1990, Matsuzaki *et. al.*, 1999; Chuang *et. al.*, 2005; Svensson *et. al.*, 2008; Fakhr *et. al.*, 2010).

In producing good InGaP QWRs that can be adapted suitably in future potential electronic and optoelectronic devices; like in light emitting diodes (LEDs), laser diodes (LDs), transistors, sensors or photo-detectors, optimum growth conditions should be controlled and be applied in growing such quantum structures, so that these products are homogenous in terms of its size, morphology, crystal structure and chemical compositions. The reason for this study to be conducted is to investigate effects of growth temperature and substrate orientation variations towards the morphology, crystal structure and chemical compositions distributions of InGaP QWRs by using metal-organic chemical vapour deposition (MOCVD), which finally will lead to finding the effect of growth temperatures and substrate orientations in producing good InGaP QWRs.

1.3 Objectives of Research

i. To prepare Indium Gallium Phosphide quantum wires (InGaP QWRs) on GaAs substrates by using Metal-Organic Chemical Vapour Deposition (MOCVD).
ii. To determine growth temperature on the growth of InGaP quantum wires.
iii. To determine effect of substrate orientations on the growth of InGaP quantum wires.

1.4 Significant of Research

The purpose of this study is to investigate further on the effect of growth temperature and substrate orientation variations towards InGaP QWRs in terms of its morphology, crystal structure and distributions of chemical compositions by using MOCVD. Suitable growth temperature that been obtained then can be applied to produce good InGaP QWRs to be grown on a suitable substrate orientation.

1.5 Scope of Research

The whole research is basically focused on the growth of Indium Gallium Phosphide quantum wires (InGaP QWRs) by using metal-organic chemical vapour deposition (MOCVD). MOCVD is used because of its flexible and versatile application to produce mass-scale and wide-range of semiconductor structures. The studies that are conducted were to investigate the effect of growth temperatures and substrate orientations to the morphology, crystalline structure and chemical composition of InGaP QWRs. The growth temperatures that were used are 380°C, 410°C, 440°C, 470°C and 500°C and all of the samples are grown on two different substrate orientations which are on GaAs (100) and GaAs (111) B. The growth parameters are varied and investigated to found the suitable growth mechanism in producing InGaP QWRs. All the growth samples were analyzed using Scanning Electron Microscopy (SEM), Field Emission Scanning Electron Microscopy (FE-SEM), Transmission Electron Microscopy (TEM) and Energy Dispersive X-ray Spectroscopy (EDX) to investigate its morphology, crystalline structure and chemical composition.

1.6 Thesis Outline

Synthesize and structural characterization of Indium Gallium Phosphide (InGaP) quantum wires is reported in this thesis which is discussed in five chapters. In chapter 1, a brief introduction about research study including background of research, statement of problems, objectives of research, significant of research and scope of research are presented. In chapter 2, theoretical backgrounds and information about quantum structure, nucleation, quantum wire, fabrication of quantum structures in terms of bottom-up and top-down approaches, growth method of quantum wires with focus on vapour-liquid-solid (VLS) and characterization techniques are explained. In chapter 3, details on research methodology are presented. In chapter 4, results of the research are discussed in depth. And lastly, conclusions about the research and recommendations are given on chapter 5.

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