UNIVERSITI PUTRA MALAYSIA

STRUCTURAL AND ELECTRICAL PROPERTIES OF ANODIC ALUMINUM OXIDE-TEMPLATE ASSISTED InSb NANOWIRES VIA ELECTROCHEMICAL DEPOSITION

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By

OSAMAH ALI FAYYADH

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Chairman : Suriati binti Paiman, PhD
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The indium antimonide (InSb) material is a remarkable combination of group III-V due to it is narrow band gap and high electron mobility. InSb has been widely employed in various optoelectronic and electronic devices such as in medical application, biosensor, infrared detectors, and emitters. This can be realized by investigating the conductive layer conditions to achieve nanowires with uniform diameter and length as well as structurally controlled nanowires where ratio can be optimized. This thesis aims to study the AAO template conditions on morphology, structural and electrical properties of InSb nanowires via template-assisted electrochemical deposition technique. Various strategies used to produce morphologically controlled nanowires with desired properties includes effects of conductive layer thicknesses of 11 nm, 65 nm, 130 nm and 260 nm. The AAO templates were then covered with insulating tape at the bottom of the conductive gold layer, to study the effects of In and Sb ions direction to the nanowires growth. The morphology images indicate that the AAO template covered with insulating tape have less overgrowth rough film and smoother sidewall of the nanowire. Meanwhile, the long nanowires with length about 6 μm and rough sidewall were observed from uncovered AAO template, which is due to the overgrown rough film that attached to the sidewall of the nanowires. The overall EDX analysis shows that the sample with insulating tape exhibited better morphology and stoichiometric ratio. It was found that the XRD results exhibited the high crystallinity and polycrystalline characteristics in the nanowires. Moreover, the highest conductivity was achieved among the covered and uncovered samples at 12 minutes with insulating tape sample ($7.96 \times 10^{-4}\ \Omega^{-1}cm^{-1}$). In conclusion, the achievement of all these parameters will allow the InSb nanowires to be designed and generated for future nanowires device applications such as biosensor and photovoltaic.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

SIFAT-SIFAT STRUKTUR DAN ELEKTRIK InSb NANOWAYAR MELALUI TERNIK PEMENDAPAN DENGAN BANTUAN TEMPLAT ANODIK ALUMININUM OKSIDA

Oleh

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Bahan indium antimonide (InSb) adalah kombinasi yang luar biasa dari kumpulan III-V oleh kerana leluang jalurnya yang sempit dan mobiliti elektron yang tinggi. InSb telah digunakan dalam pelbagai peranti optoelektronik dan elektronik seperti dalam aplikasi perubatan, biosensor, pengesan inframerah, dan pemancar. Ini dapat direalisasikan dengan menyiasat keadaan lapisan konduktif bagi mencapai diameter dan panjang nanowayar yang seragam, serta boleh dikawal secara berstruktur di mana nisbahnya boleh dioptimumkan. Tesis ini bertujuan untuk mengkaji keadaan AAO templat ke atas ciri-ciri morfologi, struktur, dan elektrik nanowayar InSb menggunakan teknik pemendapan elektrokimia dengan bantuan templat. Pelbagai strategi digunakan untuk menghasilkan nanowayar yang morfologinya terkawal dengan sifat yang dikehendaki meliputi kesan tebal lapisan konduktif berketebalan 11 nm, 65 nm, 130 nm dan 260 nm. Templat AAO kemudian ditutup dengan pita penebat di bahagian bawah lapisan emas konduktif, untuk menyiasat kesan-kesan arah gerakan ion In dan Sb kepada pertumbuhan nanowayar. Imej-imej morfologi menunjukkan bahawa templat AAO yang diliputi dengan pita penebat mempunyai selaput kasar yang lebih sedikit dan dinding sisi nanowayar yang lebih licin. Manakala nanowayar panjang berukuran 6 µm dan dinding sisi kasar dapat diperhatikan dari templat AAO yang tidak diliputi, disebabkan oleh selaput kasar lebih yang melekat di dinding sisi nanowayar tersebut. Keseluruhan analisis EDX menunjukkan bahawa sampel dengan pita penebat mempunyai morfologi dan nisbah stoikiometri yang lebih baik. Dapat dilihat bahawa hasil-hasil XRD menunjukkan kehabluran yang tinggi dan ciri-ciri polihabluran di dalam nanowayar. Tambahan pula, konduktiviti tertinggi dicapai di kalangan sampel-sampel yang diliputi dan tidak diliputi oleh pita penebat pada 12 minit ($7.96 \times 10^{-4}\ \Omega^{-1}cm^{-1}$). Sebagai kesimpulan, pencapaian semua parameter ini akan membolehkan nanowayar InSb direka dan dijana untuk aplikasi peranti nanowayar masa depan seperti biosensor dan fotovoltaik.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

AAO	Anodic Alumina Oxide
CE	Counter electrode
RE	Reference electrode
WE	Working electrode
Au	Gold
Ag/AgCl	Silver Chloride electrode
Al_2O_3	Aluminium Oxide
In	Indium
Sb	Antimony
$C_4H_6O_6$	Tartaric acid
HCl	Hydrochloric acid
$InCl_3$	Indium trichloride
InSb	Indium Antimonide
NaOH	Sodium hydroxide
CVD	Chemical Vapor Deposition
MOCVD	Metal Organic Chemical Vapor Deposition
$SbCl_3$	Antimony trichloride
EDX	Energy dispersive x-ray
FESEM	Field emission scanning electron microscope
XRD	X-ray Diffraction
HPM	Higher Power Microscope
NWs	Nanowires

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Recently, the knowledge of nanoscience and nanotechnology have been providing an important progress and their contribution to research fields has been acknowledged. In material science, nanostructures play a significant role in the applications of nanoscience and other related fields such as information and methods, the source of energy, health and medical treatment and environment. Many nations are very much concerned in this area of research, including the development of front fields nanotechnology, nanoscience, nanoelectronics and device, nano or microfabrication techniques. Moreover, semiconductor nanostructure has become an interesting in the electronic field because of their attractive properties (Xu et al., 2014). Nanowires are cylindrical shaped nanostructures with a diameter less than 100 nm and lengths up to few microns. Nanowires have several fascinating properties that are not found in bulk materials. This due to the electron in nanowires are occupied energy levels and quantum confined laterally that is different from the traditional continuum of energy levels or bands seen in bulk materials. The high density of electronic states, increased surface area, enhanced binding energy and the increased surface scattering for photons and electrons are some of the ways in which nanowires differ from their conforming bulk materials (Dresselhaus, 2003). Nanowires can be grown using various materials such as organic and inorganic, or they can be designated based on their properties as in the case of semiconductor, metallic and oxide (insulator) nanowires. Semiconductor nanowires (NWs) represent a unique system for exploring phenomena at the nanoscale and are also expected to play a critical role in future electronic and optoelectronic devices (Lu and Lieber 2006) (Fang et al., 2014). Their physical properties can be significantly altered within the confines of the nanowire surface (Hochbaum, and Yang 2009). In addition, their large surface to volume ratio allows for distinct structural and chemical behavior as well as greater chemical reactivity. These two-dimensional confinement nanowires with unique properties that stay from those of their corresponding bulk material. III-V compound semiconductor nanowires such as InAs, InP, GaAs, GaP, and InSb, have attracted substantial scientific and technological interests in nanoelectronic devices because of their high electronic transfer characteristic with low leakage currents which make them attractive as building blocks of electronic and photonic nanoscale devices (Kuo et al., 2013). The accumulation layer of electrons usually occurs at the surface of nanoscale materials this causes better electrical conductivity and higher surface sensitivity (Kuo et al., 2013). These properties exhibited by the nanomaterials make group III-V semiconductor materials perfect candidates for nanowire applications.

Indium antimony (InSb) is among the group III-V semiconductors nanowires that have advantages of high electron mobility with speed of about 77000 $cm^2\ V^{-1}\ S^{-1}$ which is associated with smaller mass of the electrons and with ballistic length of up to 0.7 μm at 300 K, these values were found to be higher than those of other types of known semiconductors (Kuo et al., 2013). Due to its unique electronic and optoelectronic properties, InSb has been employed in a wide variety of applications, such as in medical application as biosensor (Singh et al., 2015), infrared detectors (Kuo et al., 2013) and emitters (Gangloff et al., 2004), magnetic field sensors (Takamura et al., 2015), toxic gas sensors for detecting gases of acetone, ammonia, NO_2, ethanol, hydrogen and ozone (Gang 2011), and most recently in low power high-speed electronics devices (Zhang et al., 2005).

In the case of nanowires, InSb is an important semiconductor material to be used in long wavelength optoelectronic devices application. This is due to the high surface to volume ratio and quantum confinement effect, one dimensional (1-D) semiconductive nanostructures exhibit unique optical, electronic, and transport properties, which are widely applied in photodetectors (Y. Yang et al. 2010) electron field emitters (Gangloff et al. 2004) has and dye-sensitized solar cells (Liu and Aydil 2009). Very recently InSb semiconductors nanowires are expected to provide an excellent material platform for the study of Majorana fermions in the solid state system. For example, Majorana fermions are an elusive class of fermions that act as their own antiparticles. The most recent ones are to explore a topological superconductor phase (Deng et al.,2012). InSb nanowires emerged as a very promising candidate as a part of topological properties due to their large spin-orbit interaction and large g factor. Different methods have been used to fabricate InSb nanowires and other III-V nanowires by employing two methods which are bottom-up and top-down techniques. These includes vapor-liquid-solid (Shafa et al., 2016), chemical vapor deposition (Kanti et al., 2010), metal organic vapor phase epitaxy MOVPE (caroff et al., 2008) chemical beam epitaxy (Vogel et al., 2011), while in liquid phase synthesis electrochemical deposition has been successfully applied. However, MOCVD and MBE have a required toxic gasses, high temperatures, high vacuum and even expensive methods. A major drawback of CVD for the growth of InSb NWs which cannot be tuned, depending upon the available direction of that substrate. To overcome all these entanglements, solution methods for synthesis of InSb NWs with controlled stoichiometry and high crystallinity are desirable. Consequently, solution methods have been considered most cost-effective than vapor methods and can be achieved with less expensive apparatus with high yield and throughput (Shafa et al., 2016). Electrochemical deposition technique is one of many techniques have been used to synthesis InSb NWs with assisted of AAO using porous membranes as a template.

This research is concentrated to study morphology, structural and electrical properties of InSb nanowires. In order to get a better morphology and a good chemical composition and high crystal structure, different AAO template conditions were applied before the electrochemical deposition process. Next, the electrical measurement is conducted by using a four-point probe technique. The current vs voltage graph is achieved to conclude the electrical properties of the InSb nanowires. the resistivity and electrical conductivity of the InSb nanowires can be calculated from the current-voltage measurement. Nanowires surface condition, crystal structure, and chemical composition are selected as parameters in this research to explore their effect on the electrical properties of InSb nanowires.

1.2 Problem Statement

Electrochemical deposition method using porous membranes as a template is an effective and low-cost method for fabricating InSb nanowires. Anodic alumina oxide (AAO) templates with 200 nm of pores diameter are widely employed in the nanowire growth methods such as electrodeposition technique due to its ability to withstand higher temperatures, insoluble in organic solvent and cost-effective (Sisman, 2011). This particular technique is also fast and straightforward for fabricating highly ordered and well-aligned nanowires (Khan et al., 2008; Hnida et al., 2013). However, the AAO template were used as a growth substrate and it has a tendency of an overgrown bulk microlayer to be produced on top of it, despite having used the optimized growth parameter (Singh, Algarni, and Philipose, 2017) (Khairudin, 2017). The presence of this bulk microlayer causes for unstable stoichiometry of the nanowires and its rougher surfaces, which will significantly affect the morphology, reduce the performance of its electrical (Fang et al., 2014) and optical properties (Sahu and Nanda 2000). Therefore, the study of the template condition is important as an effort to minimize the bulk microlayer, and hence achieving good stoichiometry as well as the smooth surface of the nanowires. Two parameters of AAO template conditions need to study, which are covered AAO with insulating tape, uncovered AAO and various thickness of the conductive gold layer.

Some literature works have reported on the utilization of insulating tapes in the fabrication of nanowires, which were found to be well-aligned with high aspect ratio and reasonable high pore-filling factor (Irshad et al., 2014). However, detailed analysis of its structural properties due to the presence of the insulating tape was not properly discussed (Irshad et al., 2014). This study aims to investigate the effects of AAO template conditions on the morphology and structural properties of InSb nanowires by comparing the uncovered and covered template with insulating tape.

Furthermore, there is a problem are faced in the synthesis of InSb nanowires where some of the ions did not pass through the pores of the AAO template and got stuck on the surface of the template. During the electrodeposition process , the conductive behavior of gold layer leads the ions to drift down towards the sputtered side of the template instead of the unsputtered side. As a result, the ions cannot drift and enter into the pores this is associated with the gold atoms being blocked at the entrance of the pores (Fu et al., 2008). To overcome this problem, various thickness of the gold conductive layer on AAO templates is maintained to achieve an optimized result. The thickness of the conducting layer depends on the sputtering time of the AAO templates. The thin gold layer is important to allow the pores open and permit the ions to enter through the pores.

1.3 Research Objectives

The Objectives for this research are:

i. To synthesize InSb nanowires using anodic alumina oxide (AAO) template with 200 nm pore diameter by an electrochemical deposition method.
ii. To analyse the morphology and structural properties of InSb nanowires via different AAO template conditions (various thickness of conductive gold layer, covered AAO with insulating tape and uncovered AAO) using Field Emission Scanning Electron Microscope (FESEM), Energy Dispersive X-Ray Analysis (EDX), and X-Ray Diffraction Pattern (XRD).
iii. To investigate the electrical properties of InSb nanowires using a four-point probe technique.

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