UNIVERSITI PUTRA MALAYSIA

SYNTHESIS AND CHARACTERIZATION OF LOW DIMENSIONAL AND HETEROSTRUCTURE MAGNETIC QUANTUM AND NANOSTRUCTURE MATERIALS PREPARED USING ELECTRODEPOSITION METHOD

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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Chairman: **Associate Professor Mansor Hashim, PhD**
Faculty: **Institute of Advanced Technology**

Low dimensional structures such as nanowires, thin films and hetreostructures have attracted considerable attraction in advanced electronic devices since they exhibit diverse and interesting physical phenomena compared with bulk structures. One of the most rapidly growing fields in electronics is spintronics which combines both the spin and charge of the electrons. Compared with conventional semiconductors, dilute magnetic semiconductors (DMSs) offer better performance for new generation devices. For this purpose, group II-VI semiconductors of ZnSe and ZnS and dilute magnetic semiconductors of (Zn,Mn)Se and (Zn,Mn)S were chosen as the materials in this research. The incorporation of magnetic manganese ions in semiconductor ZnSe and ZnS structures was the source of spin-polarized electrons.

Highly technological low dimensional systems are fabricated by expensive and sophisticated methods such as molecular beam epitaxy. The problem is that the synthesis method for fabrication of such structures is very expensive. Our main attempt is to solve this problem by applying inexpensive and easy synthesis method like electrodeposition for preparation of low dimensional and heterostructures. The properties of thin films, heterostructures and nanowires were studied by applying various characterization methods.

The XRD results showed formation of highly crystallined ZnSe and ZnS thin films and the crystallinity increased at lower deposition temperatures. All deposited thin films showed mixed cubic a hexagonal structures. XRD results of Mn doped ZnSe and ZnS thin films indicated well doped samples that perfectly matched with $Zn_{1-x}Mn_xSe$ and $Zn_{1-x}Mn_xS$ (x = 0.01, 0.05 and 0.1).. The calculated strain and dislocation density from XRD data showed a gradual increase with increasing manganese concentration. FESEM images showed that films cover perfectly the surface of ITO substrate. In addition no defects or cracks were observed on the surface of the thin films. The optical measuremets indicated that with increasing grain size and film thickness the absorption edge shifted to higher wavelengths and a red shift is observed in bandgap. The grain size and film thickness were increased

with increasing deposition temperature and deposition time. Mn doped thin films revealed a red shift with increasing manganese concentration. Very high bandgap for both semiconductor and dilute magnetic semiconductors were obtained and the bandgap blue shifted compared with bulk ZnSe and ZnS. Photoluminescence spectra for semiconductor and DMS thin films were obtained and the results showed a characteristic strong green emission peak centred at 440 nm (2.82 eV) for ZnSe. Mn-doped ZnSe systems showed a dominant yellow/orange emission present at 599 nm which results from the $^4T_1$-$^6A_1$ transition of the Mn^{2+} impurity excited by energy transfer from the host ZnSe lattice. The blue emission at 376 nm (3.28 eV) is obtained for ZnS thin films that attributed to surface states. The non-ohmic I-V characteristic was observed for ZnSe and ZnSe-Mn thin films. With increasing Mn concentration in ZnSe thin films the resistivity dereased. In addition the resistivity decreased with increasing the film thickness. Moreover, ZnS and ZnS thin films doped with Mn revealed ohmic behaviour. In ZnS thin films the resistivity increased as the film thickness increased. More doping leads to higher conductivity in ZnS-Mn thin films.

Single quantum well ZnSe/(Zn,Mn)Se and ZnS/(Zn,Mn)S heterostructures prepared by electrodeposition method. The thickness of quantum wells increased with increasing the deposition time. The variation in bandgap between the layers of heterostructures is invesitaged. The bandgap differences between barrier and quantum well materials were in the range of 100-300 meV. The quantum mechanical tunnelling and transport properties were investigated by photoluminescence, 2-point probe and electron spin resonance data. The amount of tunnelling and spin transport strongly depended on the quantum well thickness as well as the concentration of Mn in the barrier.

ZnSe, ZnS and Mn doped nanowires embedded in polycarbonate membranes revealed an increase in optical and electrical properties with decreasing the pore diameter. The higher bandgap was obtained in nanowires with smaller diameters. In addition the bandgap enhanced with increasing Mn concentration in nanowires. High resistivity and low conductivity was observed for nanowires with larger diameters.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SINTESIS DAN PENCIRIAN KUATUM MAGNET BERDIMENSI RENDAH DAN STRUKTUR HETERO KUANTUM MAGNET DAN BAHAN STRUKTUR NANO YANG DISEDIAKAN DENGAN MENGGUNAKAN TEKNIK PENGELEKTROENAPAN

Oleh

GHAZALEH BAHMAN ROKH

November 2016

Pengerusi: Profesor Madya Mansor Hashim, PhD
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Struktur dimensi rendah seperti nanowayar, filem nipis dan struktur hetero telah menarik tarikan yang besar dalam peralatan elektronik canggih kerana mereka mempamerkan fenomena fizikal yang pelbagai dan menarik berbanding dengan struktur pukal. Salah satu bidang yang paling pesat berkembang dalam elektronik adalah spintronik yang menggabungkan kedua-dua spin dan caj elektron. Berbanding dengan semikonduktor konvensional, semikonduktor magnet cair menawarkan prestasi yang lebih baik untuk peranti generasi baru. Untuk tujuan ini, semikonduktor Kumpulan II-VI yang terdiri dari ZnSe, ZnS semikonduktor magnet cair (Zn, Mn)Se dan (Zn, Mn)S telah dipilih sebagai bahan dalam kajian ini. Penggabungan ion mangan magnet semikonduktor dalam ZnSe dan ZnS adalah sumber sistem electron spin-terkutup. Sistem dimensi rendah berteknologi tinggi adalah difabrikasi dengan kaedah mahal dan canggih seperti epetaksi alur molekul. Usaha utama kami adalah untuk menyelesaikan masalah ini dengan menggunakan kaedah sintesis murah dan mudah seperti pengelektroenapan untuk penyediaan struktur dimensi rendah dan struktur hetero. Sifat-sifat filem nipis, struktur hetero dan wayar nano telah dikaji dengan menggunakan pelbagai kaedah pencirian.

Keputusan XRD menunjukkan pembentukan kristal ZnSe yang sangat jita dan filem nipis ZnS yang penghablurannya meningkat pada suhu pemendapan yang lebih rendah. Semua filem nipis yang didepositkan menunjukkan struktur heksagon bercampur. Keputusan XRD untuk Mn ZnSe yang didopkan Mn dan filem nipis ZnS menunjukkan pendopan sampel yang sempurna dipadankan dengan $Zn_{1-x}Mn_xSe$ dan $Zn_{1-x}Mn_xS$ (x = 0.01, 0.05 dan 0.1). Terikan (ε) yang dikira dan ketumpatan kehelan (δ) dikiradari data XRD menunjukkan peningkatan beransur-ansur dengan meningkatnya kepekatan mangan. Imej FESEM menunjukkan bahawa filem menutup dengan sempurna permukaan substrat ITO. Disamping itu tiada kecacatan atau keretakan diperhatikan pada permukaan filem nipis. Pengukuran optik menunjukkan bahawa dengan meningkatnya saiz butiran dan ketebalan filem pinggir penyerapan taranjak kearah panjang gelombang yang lebih tinggi dan anjakan merah

dicerap di dalam jurang jalur. Saiz butiran dan ketebalan filem telah meningkat dengan peningkatan suhu pemendapan dan tempoh pemendapan. Mn didopkan filem nipis mendedahkan anjakan merah dengan meningkatkan kepekatan mangan. Nilai jurang yang sangat tinggi untuk kedua-dua semikonduktor dan DMSs diperolehi dan memberikan nilai jurang anjakan biru berbanding ZnSe pukal dan ZnS. Spektrum fotoluminisen untuk filem semikonduktor dan DMS nipis diperolehi dan keputusan menunjukkan pencirian puncak pelepasan hijau yang kuat berpusat pada 440 nm (2.82 eV) untuk ZnSe. Sistem Mn-didopkan ZnSe menunjukkan dominan kuning / oren pelepasan hadir pada 599 nm yang terhasil daripada peralihan $^4T_1$-$^6A_1$ bendasing Mn^{2+} yang teruja dengan pemindahan tenaga dari kekisi tuan ZnSe. Pelepasan biru pada 376 nm (3.28 eV) diperolehi untuk ZnS filem nipis disebabkan oleh keadaan permukaan juga. Ciri I-V bukan ohm diperhatikan untuk ZnSe dan ZnSe-Mn filem nipis. Dengan meningkatkan kepekatan Mn dalam filem nipis ZnSe kerintangan menurun. Di samping itu kerintangan menurun dengan peningkatan tebal filem. Selain itu, ZnS dan ZnS filem nipis didopkan dengan Mn mendedahkan tingkah laku ohm. Dalam filem nipis ZnS kerintangan meningkat apabila ketebalan filem meningkat. Lebih pengedopan membawa kepada kekonduksian tinggi dalam ZnS-Mn nipis films.

Telaga tunggal kuantum ZnSe/ (Zn,Mn)Se dan struktur hetero ZnS/(Zn,Mn) disediakan dengan kaedah pengenapan. Ketebalan telaga kuantum meningkat dengan peningkatan masa pemendapan. Perubahan dalam memberikan nilai jurang antara lapisan struktur hetero disiasat. Perbezaan nilai jurang antara halangan dan telaga kuantum adalah dalam lingkungan 100-300 meV. Terowong kuantum mekanik ini dan sifat-sifat pengangkutan mekanik kuantum telah disiasat oleh data fotoluminisen, prob 2-poin dan elektron spin resonans. Jumlah terowong dan pengangkutan spin amat bergantung kepada ketebalan telaga kuantum dn juga kepekatan Mn dalam halangan. Wayar nano didopkan ZnSe, ZnS dan Mn tertanam dalam membran polikarbonat mendedahkan peningkatan dalam sifat-sifat optik dan elektrik dengan mengurangkan diameter liang itu. Nilai jurang yang lebih tinggi telah diperolehi dalam wayar nano dengan diameter yang lebih kecil. Selain itu, nilai jurang dipertingkatkan dengan peningkatan kepekatan Mn dalam wayar nano. Kerintangan yang tinggi dan kekonduksian rendah diperhatikan untuk wayar nano dengan diameter yang lebih besar.

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I attribute the level of my Doctor of Philosophy degree to all your supports. To you all I dedicate this thesis.

I certify that a Thesis Examination Committee has met on 7 November 2016 to conduct the final examination of Ghazaleh Bahman Rokh on her thesis entitled "Synthesis and Characterization of Low Dimensional and Heterostructure Magnetic Quantum and Nanostructure Materials Prepared using Electrodeposition Method" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Doctor of Philosophy.

Members of the Thesis Examination Committee were as follows:

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Date: 26 January 2017

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

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Name of Chairman of Supervisory Committee: Mansor Hashim, PhD

Signature: _____
Name of Member of Supervisory Committee: Khamirul Amin Matori, PhD

Signature: _____
Name of Member of Supervisory Committee: Lim Kean Pah, PhD

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Å	Angstroms
AAM	Anodic alumina membran
AFM	Antiferromagnet
BSE	Backscattered electrons
1D	One-dimensional
2D	Two-dimensional
CB	Conduction band
σ	Conductivity
D	Diameter
DMS	Dilute magnetic semiconductors
DL	Double layer
δ	Dislocation density
emu/g	Electromagnetic unit per gram
ESR	Electron spin resonance
ED	Electrodeposition
EDX	Energy dispersive x-ray spectroscopy
FWHM	Full width at half maximum
FM	Ferromagnet
g	Gram
GMR	Giant magneto resistance
H	Magnetic field strength
H_C	Coercivity
H_E	Exchange bias filed
nm	Nanometer
NPs	nanoparticles
M_r	Remanent magnetization
M_S	Saturation magnetization
MBE	Molecular beam epitaxy
MR	magnetoresistance
Oe	Orsted
PCTE	Polycarbonate
PL	Photoluminescence
ρ	Resistivity
QD	Quantum dot
QW	Quantum well, quantum wire
R	Resistance
UHV	Ultra-high vacuum
SE	Secondary electrons
V	Volume
χ	Magnetic susceptibility
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Low dimensional structures refer to systems in which at least one of the three dimensions is intermediate between the characteristic of atoms/molecules generally in the range of 1 to 100 nm. In low dimension electron systems, one or more spatial dimensions are small enough to restrict the quantum mechanical wave-function of electrons. The electrons contained inside, exhibit some diverse and interesting physical phenomena in all of the condensed matter physics. Two-dimensional structures, nanowires, nanotubes and quantum dots are examples of low dimensional systems. In these systems the surface states become important and even dominant due to high surface area to volume ratio. Additionally the dimensional restriction on the system gives raise to quantum size effects that significantly change their behaviour and energy spectrum of electrons. Consequently the properties of such systems are identically different from those of bulk counterparts and they show extraordinary electronic, optical, magnetic and chemical properties, which may result in their use in wide range of nanotechnology.

In recent years an extraordinary volume of research has been carried out on low dimensional semiconductors, metals, ceramics, and composites containing nanostructure materials for application in health/disease, sensing systems, electronics and communication, information processing and storage using multiple functional nanomaterials. Quantum structures have superior charge transport and optical properties thus much intensive research has been done to use them in making diode lasers, transistors and biological sensors. Improved technology for growing semiconductor thin film of different structural and electronic properties with layer thickness approaching atomic dimensions have provided new opportunities for basic scientific studies and device applications.

Advances in multilayer thin film growth techniques such as molecular beam epitaxy (Bimberg, *et al.*, 1999) and metal organic chemical vapour deposition (MOCVD)(Warren, *et al.*, 2015) have made it possible to produce semiconductor heterojunctions between IV-IV, III-V, and II-VI compounds with tailored electronic and optical properties that do not occur in nature. The epitaxial layers using the advanced methods are so thin that quantum mechanical effects in the operation of heterostructure devices have been realized. It is also possible to control the alloy composition and doping in ternary and quaternary IV-IV, III-V, and II-VI semiconductor compounds over atomic distances.

1.2 Introduction to spintronics

For many years magnetism has been the basis for information process and storage. The field of spintronics was initiated with the discovery of the giant magneto-

resistive (GMR) effect in 1988 (Maekawa and Shinjo, 2002). In GMRs the resistance of the sandwiched thin film ferromagnetic and nonmagnetic multi-layers are strongly depend on magnetic field. Nowadays GMR effect is applied almost in every modern computer hard drive. Further advances in this area come from new type of magnetic devices which manipulate both spin and charge of the electron. These devices are called spintronics which lead to more exotic information devices and capable of wide variety of functionality.

The combination of magnetism (spin) and semiconductivity (charge) in one device to store and process the information is tomorrow's information technology. The technology utilizing the degrees of freedom in both charge and spin, called spintronics or spin electronics, is one of the hot topics today and has raised much research interest by implementing novel functionalities in the community of electronics and magnetics. The charge, mass and spin of electrons form the foundation of present information technology.

Most magneto-electronic devices expanded are based on magnetic semiconductor systems, where the magnetic ions like Mn, Cr, Co and Fe are introduced into non-magnetic semiconductors. In these magnetic semiconductors, a part of the lattice is made up of substitutional magnetic atoms. They are called diluted magnetic semiconductors (DMS). To employ spins in semiconductor, it is necessary to be able to generate, inject, transport, manipulate, and detect spins. In recent years there has been a wide research towards introducing ferromagnetic property such as spontaneous magnetization at room temperature in semiconductors to make new class of spintronic devices like spin valves, transistors, spin light emitting diodes, magnetic sensors, non-volatile memory, logic devices and ultra-fast switches. The potential advantages of spintronic devices will be higher speed, greater efficiency, and better stability, as well as low energy required to flip a spin.

1.3 Choice of materials

The major difficulty for practical application of spintronic devices is that efficient spin polarized carrier injection is required. Spin injection efficiency is often very low in conventional ferromagnetic metals due to resistivity differences and to the formation of Schottky barrier. A novel class of materials, dilute magnetic semiconductors (DMS), could be a best solution to this problem. The group II-VI semiconductors such as (Zn,Mn)Se or (Zn,Mn)S are examples of DMS. In DMS alloys the stoichiometric fraction of the constituent atoms is replaced by magnetic transition metals atoms to utilize the electrons spin in addition to its charge. These alloys are semiconductors and are able to process well defined magnetic properties such as paramagnetism, ferromagnetism and antiferromagnetism in compare with conventional semiconductors. DMS have been used to form magnetic and non-magnetic interfaces formed in heterostructures. It is been observed that incorporation of magnetic materials in these structures is a source of spin-polarized electrons. In addition the technological importance of group II-VI semiconductors make them a best choice for advanced research studies specially in the spintronic applications. The glass or ceramic substrates can be applied as substrate based on their special applications. Mostly used substrate is indium tin oxide. The most important factor to

facilitate nucleation and to avoid tension in growth of layers is the thermal expansion coefficient of substrate and grown layer should be similar.

1.4 Importance of the study

During the past decades, tremendous progress has made in basic units of the microelectronic industry. Devices continue to diminish in size in order to achieve higher speeds with less consumption of electricity. Several long-term alternative concepts are being investigated in order to attain the above matters by exploiting multifunctional properties of materials.

The theoretical understanding of semiconductor devices have developed rapidly since the first transistor was invented in 1949 (Kröger, 1978). Improved new technologies for growing semiconductor thin films of different structural and electronic properties have provided new opportunities for basic scientific studies and device applications. Current transistors operate much faster than conventional ones and have led many thousand-fold increases in speed, which is very crucial to the electronic and optical communication and computer industries. An increase in the speed of transistors has resulted from a reduction of charge transit time between terminals. Shorter charge transit time indicates minimum time delay of input signal to the output. Charge transit time has been shortened very efficiently by choosing semiconductor materials and device structures in which signals propagate faster by employing semiconductors with high electron mobility and velocity (Kang and Lee, 2013).

Over the past decade the experimental and theoretical study of quantum confinement of carriers in spatially modulated semiconductor structures has been an area of intense activity. Relatively new magnetic semiconductor quantum wells and super-lattices have extended this field to include novel spin dependent phenomena. The purpose in spintronic devices is to use wide bandgap semiconductors such as ZnS and ZnSe which are suitable candidates to produce carrier-induced magnetism. A good knowledge about the mechanism of the magnetic interactions coupled with spins of the band carriers and localized spins of magnetic ions, could obtain which may result in different magnetic phases such as ferromagnetism, paramagnetism and antiferromagnetism (Xu and Thompson, 2006).

In this research 1D and 2D quantum structures are prepared using low cost electrodeposition method instead of expensive and sophisticated methods such as molecular beam epitaxy. The first part discussed the basic physics involved II-VI MS quantum structures. Clear and stablished underlying science and the relationship between structure and properties of these quantum structures are discussed. In addition more practical device from II-VI MS heterostructures is developed and showed spin injection and transport properties. The group II-VI MS alloy quantum structures are of interest that transition metal Mn^{2+} incorporated on the group II site of zinc blend lattice. Figure 1.1 shows the relation between lattice parameter and the energy band gap of some ternary alloys of II-VI magnetic semiconductors.

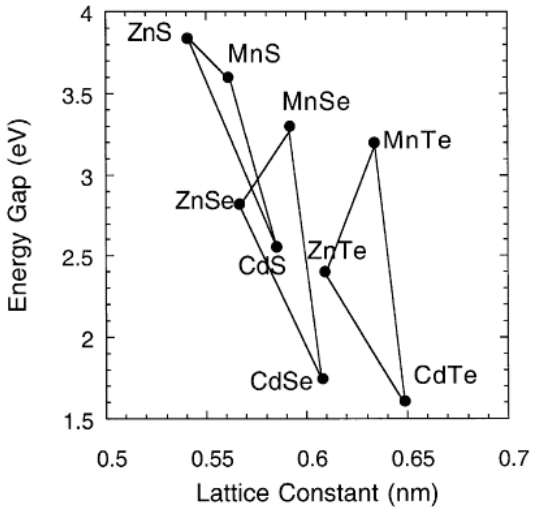

Figure 1.1: Summary of the energy gap at 4.2 K and lattice parameter of some II-VI MS alloys. The lines showing ternary alloys (Awschalom and Samarth, 1999).

There are different examples of 'spin engineered' MS heterostructures which are fabricated using sophisticated methods such as MBE. Here we only concentrate on the simplest MS heterostructures consist of binary non-magnetic quantum well flanked by MS barriers of ZnSe/(Zn,Mn)Se and ZnS/(Zn,Mn)S prepared by electrodeposition method.

In the second part we focused on semiconductor and magnetic semiconductor nanowires with different diameter prepared by electrodeposition method into polycarbonate membrane templates. The template assisted synthesis has recently proved to be a well-designed chemical approach for synthesis of nanoscale materials and an alternative to MBE method. There are a wide range of nanoprorous materials. The most studies were performed using track-etched polymer membranes and anodic porous alumina membranes. Both membranes enable to make fibrils of uniform diameter in the range of nanometer. We will discuss the preparation method of NWs using polycarbonate (PCTE) membrane and presented morphological characterization dependent optical and electrical properties.

1.5 Problem statements

Nowadays, the most successful technologies are led by the data storage industries and the integrated circuits. Most of the current semiconductor devices use the charge of electrons and holes in order to perform their specific functionalities and ignore the spin of electrons. However the integrated circuits have advantages such as high speed and good reliability, but their memory elements are volatile which means that when the power is switched off the stored information is lost. Moreover, power dissipation and heat generation are becoming critical issues. On the other hand, the spin of electron is the key parameter in magnetic data storage technology and if we can harness the spin of the carriers within a material, the devices will show highly increased speed and storage density. Devices based on spin polarized can be used as

non-volatile memory because of the natural remanence characteristic of ferromagnetic materials without the need of the electrical power.

Fabrication of highly desirable heterostructures with homogeneous compounds, exactly controlled stoichiometry and atomically abrupt interfaces has been performed using advanced methods like molecular beam epitaxy. Unfortunately such methods are very expensive and many attempts have been performed to solve this problem. In this work our aim is to fabricate these heterostructures using simple and inexpensive method like electrodeposition. The valuable features of the electrodeposition method are its convenience in producing large area devices inexpensively and its low temperature growth which yields sharper junctions and control film thickness, morphology and composition by adjusting electrical parameters in an isothermal process.

The ability to achieve zincblende (Zn,Mn)Se having appreciable Mn content is crucial to quantum well studies, because a small variation in bandgap with Mn concentration make it necessary to grow barrier layers with a high Mn fraction to achieve sufficient band offset for carrier confinement.

1.6 Objectives of the study

The principal objective of this thesis is to synthesize and characterize low dimensional materials by low cost electrodeposition method, understand the basic science, particularly physics of the nanostructures and explore their technological applications. Thus the proposed research project has the following clear work-phase objectives.

1. To synthesize and optimize the deposition condition of ZnSe and ZnS thin films and characterize the structural dependent properties of 2D quantum structures.

2. To investigate the effect of manganese doping concentration on morphology, optical, electrical and magnetic properties.

3. To fabricate single quantum well heterostructures of ZnSe/(Zn,Mn)Se and ZnS/(Zn,Mn)S using electrodeposition method.

4. To demonstrate spin polarized injection using magnetic element as spin aligner and observe spin polarized transport effect.

5. To obtain ZnSe, ZnS, ZnSe-Mn and ZnS-Mn nanowires using template assisted method by electrodeposition and investigate the effect of pore diameter on their properties.

1.7 Outline of the thesis

Given the importance of detailed understanding of the structure, morphology and optical, electrical and magnetic properties of nanostructure materials for the application described above, this study will focus on the general introduction about the research background, problem statement and objectives of the study. Chapter 2 concerns with the review on background, synthesis and some characterization of low dimensional structures. Chapter 3 focused on theoretical background which includes brief introduction to spintronics, basic physics of thin film growth and heterostructures and fundamental of electrodeposition method. Chapter 4 highlights the methodology of the study; including materials, sample preparation and characterization methods applied. Chapter 5 deals with the results and discussion on characterization of single layer, three-layer thin films and nanowires. Chapter 6 summarizes the results and gives some suggestions for future work.

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