



UNIVERSITI PUTRA MALAYSIA

***SYNTHESIS AND CHARACTERIZATION
OF NICKEL ZINC FERRITE THIN FILMS DEPOSITED USING SPIN
COATING TECHNIQUE***

YUSNITA BINTI YUSUF

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COATING TECHNIQUE**

By

YUSNITA BINTI YUSUF

**Thesis Submitted to the School of Graduate Studies, Univetsiti Putra
Malaysia, in Fullfillment of the Requirement for the Degree of Master of
Science**

December 2016

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DEDICATION

To my lovely:

Parents

&

Siblings

Thank you for supports and encourages until I completed this project.



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

SYNTHESIS AND CHARACTERIZATION OF NICKEL ZINC FERRITE THIN FILM DEPOSITED USING SPIN COATING TECHNIQUE

By

YUSNITA BINTI YUSUF

December 2016

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Faculty : Institute of Advanced Technology

The trend for downsizing electronic devices and the potential applications of soft ferrite materials have leads to the fabrication of ferrite thin films. However, the main problem concerning the existing research is the fabrication of ferrite films, to make a simple technology at low temperature. Thus, in this work, we report some research findings on relationship of low annealing temperature towards morphological, magnetic and optical properties. Nickel zinc ferrite thin film was prepared via sol-gel and spin-coating technique. The films were coated with indium tin oxide (ITO) glass substrates and spin-coated. The samples were annealed from 400 °C to 700 °C using 100 °C increments with any one sample being subjected to only one annealing temperature. The structural and microstructural of $\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$ thin films were carried out by using X-ray diffraction (XRD) and field emission scanning electron microscope (FESEM). The *M-H* hysteresis loop of thin film was observed by using vibrating sample magnetometer (VSM). The formation of films and the vibration of molecules were also confirmed by using Fourier Transform Infrared (FTIR) spectroscopy. The XRD patterns showed an improvement of crystallinity with increasing annealing temperature. Plane (311) most intense plane formed nickel zinc ferrite phases. The spinel structure was also confirmed by FTIR. FESEM image showed films have dense and homogenous grains with average grain size are 18.61 nm (400 °C), 26.25 nm (500 °C), 28.12 nm (600 °C) and 41.32 nm (700 °C). The measured resistivity was found to increase with increasing temperature, however decreased after annealed at 700 °C due to the combined effect of increased grain size and Fe^{2+} ions due to increasing zinc loss. The synthesized of $\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$ ferrite thin films showed narrow with a low saturation magnetization (M_s). The value of M_s are 1.28, 2.39, 2.65 and 3.42 emu/g respectively. These attributed to the presence of small nanoparticles containing a spin-glass-like surface layer and ferrimagnetically

lined-up core spins. The coercivity is decreases as increase the average grain size, 16.18, 16.54, 12.28, and 8.30 Oe respectively. The highest of energy bandgap obtained for direct and indirect bandgap 3.57 and 3.30 eV.



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

SINTESIS DAN PENCIRIAN TERHADAP SAPUT NIPIS NIKEL ZINK FERIT MENGUNAKAN TEKNIK SALUTAN PUTAR

Oleh

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Permintaan untuk teknologi termaju dalam elektronik bersaiz kecil telah menarik minat terhadap ciri struktur, magnetik dan elektrik ferit. Walau bagaimanapun, masalah utama berkaitan dengan kajian yang sedia ada adalah fabrikasi filem ferit menggunakan teknologi yang mudah dengan suhu yang rendah. Suhu pemendapan yang tinggi menghadkan pilihan untuk bahan substrak serta menyekat aplikasi bahan yang berbeza dari saput nipis ferit. Jadi dalam kajian ini, kami melaporkan beberapa penemuan penyelidikan mengenai hubungan suhu rendah terhadap sifat-sifat mikrostruktur, magnetik, dan optik. Ferit nikel zink telah disediakan dengan kaedah sol-gel dan teknik spin-salutan. Filem disalut atas substrak kaca indium tin oksida (ITO). Sampel telah penyepuhlandapan dari 400 °C hingga 700 °C dengan kenaikan 100 °C dengan menggunakan sampel berlainan bagi setiap suhu. Sifat struktur dan mikrostruktur sapus nipis diperiksa dengan menggunakan pembelauan sinar-X (XRD) dan mikroskop elektron imbasan pengeluaran medan (FESEM). Kajian magnet dijalankan dengan magnetometer sampel bergetar (VSM). Pembentukan saput nipis juga dipastikan dengan menggunakan spektroskopi transform Fourier gelombang merah (FTIR). Corak pembelauan sinar-X menunjukkan peningkatan penghabluran dengan meningkatnya penyepuhlandapan. Struktur spinel juga dipastikan dengan FTIR. Mikrograf FESEM menunjukkan saiz butiran meningkat dengan suhu penyepuhlandapan dengan purata butiran 18.61 nm (400 °C), 26.25 nm (500 °C), 28.12 nm (600 °C) dan 41.32 nm (700 °C). Kerintangan didapati meningkat disebabkan oleh kesan gabungan pertambahan dan bilangan ion Fe^{3+} daripada peningkatan kehilangan zink. Pemagnetan tepu saput nipis $\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$ yang diperolehi dalam suhu bilik dari gelung histerisis adalah meningkat dengan suhu penyepuhlandapan. Nilai pemagnetan tepu 1.28, 2.39, 2.65 dan 3.42 emu/g. Ini dikaitkan dengan kehadiran nanopartikel kecil yang mengandungi lapisan

permukaan sampingan kaca dan ferrimagnetik yang berbaris. daya paksa adalah penurunan sebagai meningkatkan saiz butiran purata. Nilai daya paksa menurun dengan peningkatan saiz butir, 16.18, 16.54, 12.28, dan 8.30 Oe. Nilai jurang tenaga yang tertinggi diperoleh secara langsung dan tidak langsung memberikan nilai jurang 3.57 dan 3.30 eV.

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I certify that a Thesis Examination Committee has met on 23 December 2016 to conduct the final examination of Yusnita binti Yusuf on her thesis entitled “Synthesis and Characterization of Nickel Zinc Ferrite Thin Film Deposited Using Spin Coating Technique” 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 degree of Master of Science.

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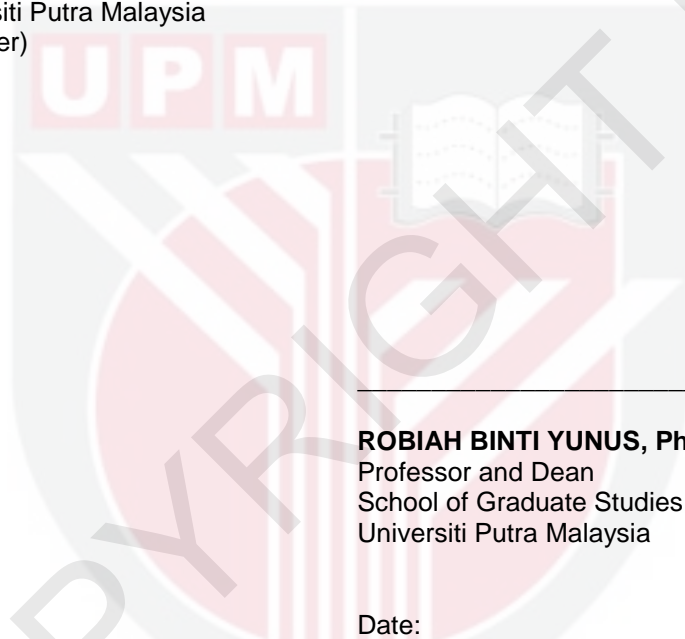
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LIST OF SYMBOLS AND ABBREVIATIONS

XRD	X- ray diffraction
FESEM	Field Emission Scanning Electron Microscopy
VSM	Vibrating Sample Measurement
FTIR	Fourier Transform Infrared Spectroscopy
UV-Vis	Ultraviolet Visible NIR Spectroscopy
M_s	Magnetic saturation
H_c	Coercivity
2θ	Theta degree
ρ	Resistivity
I -V	Current-voltage
Wt %	Weight percent
a.u	Arbitrary unit
σ	Conductivity
H	Magnetic field strength
E_g	Optical bandgap energy
E_p	Photon energy
nB	Magnetic moment per formula unit
M_{oct}	Magnetic moment octahedral
M_{tet}	Magnetic moment tetrahedral

D	Crystallite size
β	Angular line width at half maximum intensity
θ_b	Bragg angle
A	Absorption
I_0	Incident light intensity
I	Transmitted light intensity
h ν	Photon energy
t	Thickness
N _a	Avogadro number
Fe ₂ O ₃	Iron oxide
M	Molar mass



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CHAPTER 1

INTRODUCTION

1.1 Background of study

Ferrites ceramic that have both electrical and magnetic properties been considered as highly important in electronic technological materials. Among the various types of ferrites, spinel ferrites are widely used in electrical and magnetic applications. Ferrites constitute a class of materials that has been perceived to have imperative potential in applications region from millimetre wave integrated circuitry to attractive recording. In contrast to technology of bulk ferrite for example, microwave magnetic devices and transformer cores, these potential applications in ferrite films perform to the guarantee of critical bulk ferrite properties. Ferrites are one of the magnetic ceramic materials which all are ferromagnetic oxides somehow have different in crystals structure. Ferrites have known as a spinel structure (Suzuki *et al.*, 2001), and recognized as iron oxides group that incorporates to garnets, spinels, orthoferrites and hexaferrites. Ferrites molecular formula is $M^{2+}OFe_2^{3+}O_3$ where M represent the divalent metal. For the example Cu, Fe, Mn, Co, Zn, Ni, Mg and so on (Huq *et al.*, 2013).

Soft ferromagnetic materials find extensive applications as a result of their ability to enhance the flux produced by an electrical current. Consequently, the uses of soft materials are closely connected with electrical applications such as electrical power generation and transmission, radio signals receiver, microwaves, inductors, relays and electromagnets (Chavan *et al.*, 2010). The new applications such as radio, television, video tape recorders, carrier telephone, computer circuitry and microwave devices (Sugimoto *et al.*, 1999). Hence, the requirements of ferrites have also changed. The properties such as a strong in magnetic coupling, a high resistivity, and low loss at high frequencies. Their important potential have led to extensive intention in finding of fabrication and characterization in thin film of ferrite materials to particular applications (Suzuki *et al.*, 2001).

In 2000's, the electronic devices miniaturizing, nanostructured thick and thin films have been fabricating in term spinel ferrites. Nanocrystalline ferrite thin film with spinel cubic structure have been a subjects of huge investigation because of their potential applications in high density magneto-optic recording devices, ferrofluids, colour imaging and magnetic refrigeration (Gupta, Verma, *et al.*, 2007). In view of storage applications, some ferrite thin films have been studied as potential perpendicular recording and magneto-optical media material. Barium ferrite and cobalt ferrite films are particularly promising

because of their large coercivity, as well as their high corrosion resistance, good mechanical stability, and low noise (Suzuki *et al.*, 2001). More recently, high-permeability insulating ferrites have been identified as candidate materials for flux guides and sensors in thin film recording heads. While microwave devices using bulk ferrites can be traced back to the 1950s, the development of ferrite thin films will provide several advantages for microwave and millimeter wave devices. For example, the propagation characteristics of magnetostatic wave devices can be significantly improved by films that can provide better uniformity of effective internal field compared with bulk crystals. Recent studies of ferrite thin films have focused on understanding the microstructure and its implications on the electronic and magnetic properties. Studies range from spinel and hexaferrite thin films for perpendicular recording media, integrated inductors, and micromagnetic devices to garnet structure ferrite films for magneto-optical applications. Despite variations in crystallinity and crystal structure, the fundamental issues of structure-property relationships in ferrite thin films remain the same in these studies. Ferrite thin films under study range from nanocrystalline or polycrystalline to epitaxial thin films. Given a particular growth process, it has often been difficult to reproduce results from one laboratory to the next; however, the development and refinement of deposition techniques have resulted in the convergence of results. In polycrystalline and epitaxial ferrite thin films, electroplating, magnetron sputtering, pulsed laser deposition, evaporation, and molecular beam epitaxy have been used extensively.

1.2 Problem statement

Ni-Zn ferrite is commercially important materials because of their excellent electrical and soft magnetic properties. Technique involving preparation in ferrite film can simply prepared at low temperature. But, nickel-zinc substituted nickel ferrite thin films using sol-gel and spin-coating technique have not been reported in detail for its structural, magnetic, electrical and magnetic properties. Considering the significant of nickel ferrite thin film for wide applications, it worth to study the relationship between microstructure, magnetic properties, electrical and optical properties of thin film ferrite with nominal $\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$. Most of the previous research focussed on various annealing temperature, different substrate used, and dependent of thicknesses with increased the zinc contents. While, this research proposes are to establish and understand the relationship of the microstructure, magnetic, and optical properties in $\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$ ferrite thin film.

The hypothesis of each objective are listed as following:

1. The grain size and thickness of the film would increase with annealing temperature. That would result M_s increases while H_c decreases.

2. As the annealing temperature increase, the direct and indirect energy bandgap would decrease. That would increase the resistivity.

1.3 Objectives

The main objective of this project is to synthesis and characterization of NiZn ferrite thin film prepared by spin coating technique. In order to optimize the sol-gel spin coating NiZn thin film, the following objectives have been performed, which are:

1. To study the morphology and magnetic proeprties of $\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$ ferrite thin film.
2. To study the effect of annealing temperature of $\text{Ni}_{0.3}\text{Zn}_{0.7}\text{Fe}_2\text{O}_4$ ferrite thin film on optical and electrical properties.

1.4 Thesis structure

The thesis consists five chapters which include Introduction, Literature Review, Theory, Results Discussion and Conclusion. An introduction to NiZn ferrite thin films and its applications is given in Chapter 1. The research background of study, problem statement and the objectives of this project also discuss in this chapter. The theoretical explain in Chapter 2. Literature review on synthesis method, characterization of Ni-Zn ferrite film on structural, magnetic, electrical and optical is included in Chapter 3. The research experimental works used in this research is presented in Chapter 4. This chapter explain the starting materials used to prepare the film, film deposition and samples characterization. In Chapter 5, discussion and results of data of the experiment are listed, analysed and interpreted in the forms of graphs, tables, diagrams as well as in pictures.

In Chapter 6, conclusion and recommendations for future research are present.

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