SYNTHESIS AND CHARACTERIZATION OF NICKEL ZINC FERRITE NANOPARTICLES PREPARED BY THERMAL TREATMENT METHOD

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By

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

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Chairman: Elias Saion, PhD

Faculty: Science

Cubic structured nickel-zinc ferrite nanoparticles $Ni_{1-x}Zn_xFe_2O_4$ (x = 0, 0.25, 0.5, 0.75 and 1) have been synthesized by thermal-treatment method. The Fourier transform infrared spectroscopy was used to confirm the presence of metal oxide bands at all temperatures and the removal of organic matters at 873 K. The structure and particle size were characterized by X-ray powder diffraction and transmission electron microscopy. The magnetic properties were determined by vibrating sample magnetometer and electron paramagnetic resonance electron paramagnetic resonance at room temperature. By increasing the calcinations temperatures from 723 to 873 K showed an increase of the lattice parameter, the particles size, coercivity, saturation magnetization, peak to peak line width and the value of g- factor. When the concentration of Zinc increased; the lattice parameter increased while the particles size, coercivity, peak to peak line width and the value of g-factor decreased.

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

PENCIRIAN NICKEL ZINC FERIT NANOPARTIKEL DISINTESIS DENGAN KAEDAH THERMAL

Oleh

POH LIN LENG

Julai 2013

Pengerusi: Elias Saion, PhD

Falkuti: Sains

Struktur padu nikel-zink nanopartikel ferit $Ni_{1-x}Zn_xFe_2O_4$ ($x = 0, 0.25, 0.5, 0.75$ and 1) disintesis dengan kaedah terma. Spektro skopi jelmaan Fourier infra merah telah digunakan untuk mengesahkan kehadiran ikatan oksida logam pada semua suhu dan penyingkiran bahan organik pada 873 K. Struktur dan saiz zarah dicirikan oleh pembelauan sinar-X serbuk dan mikroskop electron transmisi. Sifat-sifat magnet telah ditentukan oleh magnetometer pengetar sampel dan paramagnet electron resonans pada suhu bilik. Dengan peningkatan suhu pengkalsinan 723 hingga 873 K menunjukkan peningkatan ketepuan maknit dan g-faktor. Dengan meningkatkan suhu pengkalsinan 723-873 K menunjukkan peningkatan parameter kekisi, saiz zarah, coercivity, pemagnetan tepu, puncak dengan lebar garis puncak dan nilai g-faktor. Apabila kepekatan Zink meningkat; parameter kekisi meningkat manakala saiz zarah, coercivity, puncak dengan lebar garis puncak dan nilai g-faktor berkurangan.

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LIST OF ABBREVIATIONS/ SHORT FORMS/ SYMBOLS

β	full width at half maximum (FWHM)
θ	Bragg angle
ΔH_{pp}	Peak-to-peak line width
a, b, c	lattice parameter
D	crystallite size
EDX	energy-dispersive X-ray spectroscopy
EPR	electron paramagnetic resonance
FTIR	Fourier Transform Infra Red
H	magnetic field strength
H_c	coercivity
h	Plack constant
M	magnetization
M_s	saturation magnetization
M_r	remanence
T_s	Curie temperature
TEM	transmission electron micrograph
TGA	Thermal gravimetric analysis
v	microwave frequency
VSM	vibration sample magnetometer
XRD	x-ray diffraction
χ	susceptibility

CHAPTER 1

INTRODUCTION

1.1 Introduction of nanoparticles

The particles with the size between 1 to 100 nanometers are classified as nanoparticles. Nanoparticles have different properties from the bulk materials due to the large surface atoms of the materials. Besides the size property there are different properties such as electronic quantum confinement in semiconductor particles, superparamagnetism in magnetic materials and the surface plasmon resonance in some metal particles. The last two decades have seen tremendous progress in the synthesis of nanomaterials in search for nanoparticles with improved physical and chemical properties by enhancement of the surface and quantum confinement effects (Bellucci et al., 2008). Nanoparticles can be applied in many fields such as bio-medicals, electronics and optics. As a result; there are many scientific interests on the nanoparticles research.

1.2 Introduction of soft magnetic materials

Soft magnetic materials allow changes in magnetization to occur easily in weak fields. The hard magnetic materials are that they shall retain at high state of magnetization when the field which has produced the state is removed. Soft magnetic materials are required for use at high alternating frequencies usually is non-metallic magnetic materials which are ferromagnetic rather than ferromagnetic. The resistivity varies between 10^{-2} and 10^{7} Ω cm and produce energy losses due to eddy current. The properties required in a soft magnetic material are small coercivity, high saturation magnetization, high permeability, high resistivity and a small hysteresis loss. Hard magnetic must have high remanence . The other important requirement is that its coercivity shall be high. Figure 1 shows the hysteresis loop of the soft magnetic and hard magnetic materials which shows that soft magnetic have smaller hysteresis area compare to hard magnetic materials. The example of soft magnetic material is nickel zinc ferrite and manganese zinc ferrite. Table 1 shows the differences properties between hard magnetic materials and soft magnetic materials.

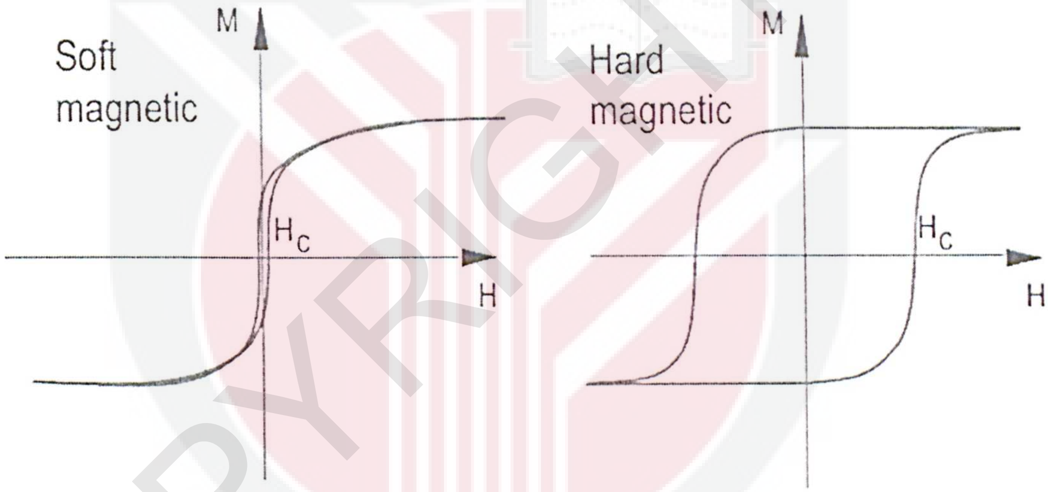

Figure 1: The hysteresis loop of soft magnetic and hard magnetic materials.

Hard magnetic materials	Soft magnetic materials
Coercivity and retentively values are large	Coercivity and retentively values are small
The eddy current loss is high	The eddy current loss is less because of high resistivity
Magnetic energy stored is high	Magnetic energy stored is small
They have large hysteresis loss due to large hysteresis loop area	They have low hysteresis loss due to small hysteresis loop area
Examples: CrO_2, Barium ferrite	Examples: NiZn ferrite, MnZn ferrite

Table 1: The differences between hard magnetic materials and soft magnetic materials.

1.3 Types of the method for preparing nanoparticles

The types of preparation of nanoparticles also can influence the properties of Ni-Zn ferrite. There are several types of method for preparing nanoparticles such as chemical co-precipitate method (Shahane et al., 2009), sol-gel (Zhang et al., 2006), solvothermal (Yan et al., 2010), reverse micelle technique (Thakur et al., 2008, a, b), low temperature solid state reaction (Amiri et al., 2010), radiation and thermal treatment method (Naseti et al.,2011). The radiation method is a simple technique to prepare the nanoparticles with minimum of chemical but the high gamma source would cost a lot. As a result, the chemical co-precipitate method, sol-gel, solvothermal, reverse micelle technique, low temperature solid state reaction and radiation method although successes to produce nanoparticles but some of these methods have disadvantages which are expensive, long reaction times, high reaction temperature and produce by-products. Thermal treatment method used to synthesize the Ni-Zn ferrite nanoparticles with an aqueous solution containing only metal nitrates as precursors, polyvinyl pyrrolidone (PVP) as a capping agent and deionized water as solvent due to the simplicity, is low cost, low reaction temperature and no by- product effluents (Naseri et al.,2011).

1.4 The problem statement

The nickel zinc ferrite nanoparticles prepared by different processes shows different size, structural and magnetic properties. There are many methods used acid or base as controller which will produce a lot of by product and the waste which pollution happen. Besides, some samples prepared with acid or base and different capping agents were requiring higher calcintation temperature to remove the entire unwanted ion. This research is to explore the simple and environmental friendly method in preparation of the nickel zinc ferrite nanoparticles. The concentration of nickel and zinc will influence the particles size, structural and magnetic properties. In this research also will try to find out property differences of the $Ni_{1-x}Zn_xFe_2O_4$ (x = 0, 0.25, 0.5, 0.75 and 1) nanoparticles calcinated at 723 to 873 K.

1.5 The significant of the study

This thermal treatment method is a simple method which can prepare in a big quantity. Besides that, this method is cost-effective and environmentally friendly which do not produces by product effluents and can be prepared pure, crystalline spinel ferrite nanocrystals. The samples can be prepared at low calcinations temperature which from 723 to 873 K.

1.6 Objectives

The main purpose of this project is to systematically study an alternative way to synthesize nickel zinc ferrite with different precursor concentrations by thermal treatment method. The samples obtained from the thermal treatment method in nanometer size and in superparamagnetism. Besides that, the samples prepared in small coercivity, high saturation magnetization, high permeability, high resistivity and a small hysteresis loss which can confirm nickel zinc ferrite is soft magnetic material.

i. To synthesize Nickel Zinc ferrite by thermal treatment method.

ii. To determine the effect of calcination temperature on the size, structural and magnetic properties of spinel ferrite.

iii. To determine the effect nickel and zinc composition on the size, structural and magnetic properties of spinel ferrite.

1.7 Outline of thesis

This thesis is organized into six chapters. Chapter 1 begins with introduction of nanoparticles and soft magnetic materials. The methods for preparing nanoparticles also discussed. The problem statement, significant of study and objectives are presented. Chapter 2 presented literature review on method of preparing NiZn ferrite nanoparticles, characterization of NiZn ferrite nanoparticles and application of NiZn ferrite nanoparticles. Chapter 3 presented introduction of magnetic properties and the types of magnetism. Chapter 4 explains the method of the research. The methodology is given in detail. Characterization technique and the measurements of the samples are discussed. Chapter 5 is on the results of the research followed by discussion. The last chapter is the conclusions for the research and future work

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