UNIVERSITI PUTRA MALAYSIA

MECHANOCHEMICAL SYNTHESIS OF NANOSTRUCTURED NICKEL AND NICKEL-ZINC FERRITES AND INVESTIGATION OF THEIR STRUCTURAL AND MAGNETIC PROPERTIES

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By

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Chairman: **Associate Prof. Mansor Hashim, PhD**
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For several past decades, studies of the relationship between morphological and magnetic properties of ferrites have been focusing only on the final sintering temperature, largely neglecting the parallel evolutions of morphological and magnetic properties and their relationship at various sintering temperatures. Hence, here, a new method of high energy ball milling was employed to attempt synthesizing two technologically applicable soft magnetic materials, namely Ni-ferrite and $Ni_{1-x}Zn_xFe_2O_4$ ferrite, for Zn contents x = 0.36 and 0.64, and to elucidate the relationship between morphological, magnetic and electrical properties at different sintering temperatures. Subsequently, common oxides of 0.4CaO + $0.8SiO_2$ were added to the $Ni_{0.36}Zn_{0.64}Fe_2O_4$ ferrite to observe the resulting property changes.

In the first work section, $NiFe_2O_4$ nanoparticles were synthesized by a mechanochemical reaction of NiO and Fe_2O_3 powders in a high energy planetary ball mill. The X-ray diffraction (XRD) results indicate that the NiO/Fe_2O_3 particles reacted in a solid-state reaction mode, producing nickel ferrite particles ranging from 5 to 18 nm in size after 18 h of milling. The effects of milling time, rotation speed, and ball to powder weight ratio were investigated and the contribution of each parameter was evaluated by using the Taguchi robust design method. It was found that rotational speed had the most effect on the crystallite size. By sintering 30 h-activated compacted samples from 900 to 1300 ºC, with 100 ºC increments, a dependence of magnetic and electrical properties on sintering temperature was found, thus improving magnetic properties i.e. saturation magnetization and reduction of electrical resistivity with increase in the sintering temperature.

In the second work section, high-energy ball milling with a subsequent heat treatment method was carried out to synthesize nanocrystalline $Ni_{1-x}Zn_xFe_2O_4$ ferrite with x = 0.36 and 0.64 from a powder mixture of pure metal Zn, Fe_2O_3 and NiO. The effect of milling atmospheres (argon, air and oxygen), milling time and sintering temperature was investigated. The XRD results indicated that a single phase of Ni-Zn ferrite was not produced after 30 h milling in the all three atmospheres of air, argon and oxygen for

both compositions except for the milled samples in argon for Zn content at x = 0.64. However, single phase Ni-Zn ferrite was later produced after sintering the other samples of 30 h-milled powders at 500 ºC for 2 h. The 30 h-milled powders in different atmospheres were pressed into pellet/toroid form and subjected to sintering in air in the same conditions from 400 to 900 ºC for 2 h. Increasing sintering temperature improved the magnetic properties but degraded the DC electrical resistivity. In terms of milling atmosphere, however, the milled-sintered samples in argon presented the lowest crystallite size, as compared to the two other atmospheres, they exhibited the highest Ms. By increasing the Zn content the lattice parameter and density of the samples increased while the saturation magnetization, crystallite size, porosity and resistivity decreased. Furthermore, by increasing milling time from 6 h to 18 h a synthesis temperature of $Ni_{0.36}Zn_{0.64}Fe_2O_4$ remarkably was reduced from 500 to 300 ºC, respectively. The Ni-Zn ferrite formation mechanism was detected to be in three stages: oxidation of zinc, diffusion of ZnO in Fe_2O_3 thus the forming $ZnFe_2O_4$, and diffusion of NiO in $ZnFe_2O_4$ thus forming Ni-Zn ferrite. Furthermore, Fourier transform infrared spectroscopy (FT-IR) results suggested the presence two absorption bands for octahedral and tetrahedral sites in the range of 350-700 cm^{-1}.

Finally, the common oxides (X = 0.4CaO + $0.8SiO_2$) were added in different moles (X = 0, 0.02, 0.06, 0.12, 0.24 and 0.48) to Fe_2O_3, Zn, and NiO. The mixed powders were mechanically alloyed for 12 h and then were sintered at 1200 ºC for different times. It was found that there was a dependence of spinel ferrite properties i.e. microstructure, electrical and magnetic properties with both X contents and sintering time. For example, magnetic parameters such as saturation magnetization (Ms) and induction magnetization (Bs) degraded while resistivity improved by increasing the X content.

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SINTESIS MEKANOKIMIA FERIT NIKEL AND FERIT NIKLE-ZINK BERSTRUKTUR NANO DAN PENYELIDIKAN SIFAT-SIFAT STRUKTUR DAN MAGNETIK BAHAN-BAHAN TERSEBUT

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Untuk beberapa dekad yang lalu, kajian mengenai hubungan diantara sifat morfologi dan sifat magnetik ferit hanya tertumpu kepada suhu pensiteran akhir, dengan mengabaikan evolusi selari sifat morfologi dan sifat magnetik serta hubungan kedua-duanya pada suhu pensiteran yang pelbagai. Oleh itu, suatu kaedah baru pengisaran bebola berkuasa tinggi telah digunakan untuk mensintesis dua bahan yang boleh diaplikasikan dalam teknologi bahan magnet lembut, iaitu ferit-Ni dan ferit-$Ni_{1-x}Zn_xFe_2O_4$ bagi kandungan Zn, x = 0.36 dan 0.64, dan untuk menjelaskan hubungan diantara sifat-sifat morfologi, magnetik dan elektrik pada suhu pensinteran yang berbeza. Seterusnya, oksida-oksida biasa iaitu $0.4CaO + 0.8SiO_2$ telah ditambah ke ferit $Ni_{0.36}Zn_{0.64}Fe_2O_4$ untuk melihat hasil kepada perubahan sifat bahan.

Dalam bahagian kerja pertama, zarah nano $NiFe_2O_4$ telah disintesis menggunakan tindak balas mekanokimia dari serbuk NiO dan Fe_2O_3 dalam pengisar bebola planetari berkuasa tinggi. Keputusan pembelauan sinar-X (XRD) menunjukkan zarah-zarah NiO/Fe_2O_3 bertindak balas dalam mod tindak balas keadaan pepejal, menghasilkan zarah ferit nikel bersaiz dari 5 hingga 18 nm selepas 18 jam tempoh pengisaran. Kesan masa pengisaran, kelajuan putaran dan nisbah berat bebola kepada serbuk telah diselidik dan sumbangan setiap parameter telah dinilai menggunakan pendekatan reka bentuk mantap Taguchi. Didapati kelajuan putaran sangat memberi kesan ke atas saiz kristalit. Dengan mensinter dari suhu 900 hingga 1300 ºC, dengan kenaikan 100 ºC, bagi sampel-sampel yang diaktifkan 30 jam dan telah dimampatkan, didapati satu kebergantungan sifat-sifat magnetik dan elektrik terhadap suhu pensinteran; meningkatkan sifat-sifat magnetik iaitu pemagnetan tepu dan pengurangan kerintangan elektrik dengan peningkatan suhu pensinteran.

Dalam bahagian kerja kedua, pengisaran bebola berkuasa tinggi dengan kaedah rawatan haba seterusnya telah dijalankan untuk mensintesis hablur nano ferit-$Ni_{1-x}Zn_xFe_2O_4$ dengan x = 0.36 dan 0.64 dari campuran serbuk logam asli Zn, Fe_2O_3 dan NiO. Kesan medium persekitaran semasa pengisaran (argon, udara, dan oksigen), masa pengisaran, suhu pensinteran telah disiasat. Keputusan XRD menunjukkan fasa tunggal ferit-Ni-Zn tidak terhasil selepas 30 jam dikisar dalam tiga medium persekitaran iaitu udara, argon dan oksigen untuk kedua-dua komposisi kecuali untuk pengisaran dalam medium argon

bagi sampel yang mengandungi Zn pada x = 0.64. Namun begitu, fasa tunggal ferit-Ni-Zn bagi sampel-sampel yang lain yang telah dikisar selama 30 jam terhasil selepas pensinteran pada suhu 500 ºC selama 2 jam. Peningkatan suhu pensinteran menambah baik sifat magnetik tetapi merendahkan kerintangan elektrik arus terus. Dari segi persekitaran pengisaran, sampel-sampel yang dikisar dan disinter dalam argon, walaubagaimanapun menghasilkan saiz kristalit terkecil dibandingkan dengan dua persekitaran lain, yang mempamerkan Ms tertinggi. Dengan meningkatkan kandungan Zn, parameter kekisi dan ketumpatan sampel bertambah manakala pemagnetan tepu , saiz hablur, keliangan dan kerintangan berkurang. Tambahan pula, dengan meningkatkan masa pengisaran dari 6 jam hingga 18 jam suhu sintesis $Ni_{0.36}Zn_{0.64}Fe_2O_4$ telah amat berkurang dari 500 kepada 300 ºC, masing-masing. Mekanisme pembentukan $NiZnFe_2O_4$ telah dikenalpasti berada dalam bentuk tiga fasa: pengoksidaan zink, peresapan ZnO ke dalam Fe_2O_3 dengan demikian membentuk $ZnFe_2O_4$, dan peresapan NiO ke dalam $ZnFe_2O_4$ dengan demikian membentuk ferit Ni-Zn. Seterusnya, keputusan FT-IR mencadangkan dua jalur penyerapan bagi tapak-tapak oktahedral dan tetrahedral dalam julat 350 – 700 cm^{-1}.

Akhirnya, oksida biasa (X = 0.4CaO + $0.8SiO_2$) telah ditambah dalam mol yang berbeza (X = 0, 0.02, 0.06, 0.012, 0.24 dan 0.48) ke dalam Fe_2O_3, Zn, dan NiO. Campuran serbuk-serbuk itu telah dialoi secara mekanikal selama 12 jam dan telah disinter pada suhu 1200 ºC pada masa berbeza. Kajian mendapati, terdapat suatu kebergantungan sifat ferit spinel seperti struktur mikro, sifat-sifat elektrik dan magnetik terhadap kandungan X dan juga masa pensinteran. Sebagai contoh, parameter-parameter magnet seperti pemagnetan tepu (Ms) dan aruhan pemagnetan (Bs) telah berkurang, manakala kerintangan telah bertambah dengan penambahan kandungan X.

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LIST OF ABBREVIATIONS AND SYMBOLS

XRD	X-ray diffraction
SEM	Scanning electron microscopy
FeSEM	Field emission scanning electron microscopy
EDX	Energy-dispersive X-ray
TEM	Transmission electron microscopy
AFM	Atomic force microscopy
BET	Brunauer–Emmett–Teller
TGA	Thermogravimetric analysis
DSC	Differential scanning calorimetry
DTA	Differential thermal analysis
FT-IR	Fourier transform infrared spectroscopy
VSM	Vibrating sample magnetometer
JCPDS	Joint Committee on Power Diffraction Standard
FWHM	Full width at half maximum
ICDD	International Centre for Diffraction Data
PVA	Polyvinyl alcohol
A	Cross sectional area
ε	Lattice strain
HEBM	High-energy ball milling
BPR	Ball-to-powder weight ratio
Fig.	Figure
eq.	Equation
a.u	Arbitrary unit
2θ	2 theta degree
wt%	Weight percent
hkl	Miller indices
MPa	Megapascal
W_a	Weight of the sample in air
W_w	Weight of the sample in water
ρ_{xrd}	X-ray diffraction density

ρ_x	Experimental density
ρ	Resistivity
M	Molecular weight
N_a	Avogadro's constant
P	Porosity
λ	Wavelength
λ	Magnetostriction
E_k	Magneto anisotropy energy
K	Magnetocrystalline anisotropy
μ_s	Spin magnetic moment
Hc	Coercivity
H	Magnetic field strength
B	Magnetic induction/Flux magnet
B_s	Saturation induction/ saturation flux magnet
Ms	Saturation magnetization
μ'	Real part of permeability/initial permeability
μ''	Imaginary part of permeability/ loss factor
MA	Mechanical alloying
d	Average crystallite size
n_B	Magnetic moments
μ_b	Bohr magneton
Q_b	bound charges
Q_f	free charges
ε_r'	Dielectric constant
γA	Total interfacial energy of a powder compact
γ	Specific surface (interface) energy
A	Total surface (interface) area of the compact
$\Delta\gamma$	The change in interfacial energy
CR	Charge ratio
μ	Diffusion potential
C	Capacitance
Co	Capacitance without dielectric
Q	Magnitude of charge stored
V	Voltage

E	Permittivity
ε_o	permittivity of free space
Mr	Remanent magnetization
V	Volume
T_N	Neel temperature
T_C	Curie temperature
X	Magnetic susceptibility

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Research in nanoparticle/nanocrystalline materials can be framed within three significant goals: synthesis, understanding of new advanced materials and correlated phenomena between them. From the microscopic counterparts, nanomaterials are mostly considered as materials in which the size of the particles is below 100 nm in at least one or more dimensions. At this length scale, a huge fraction of the atoms exists at/or close to the surface of particles which gives the unique characteristics to the materials. Nanomaterials have a large surface to volume ratio, thus, often exhibiting amazing properties that differ from the bulk ones. Recently, synthesis of nanocrystalline spinel ferrites has attracted the interest of many researchers due to their unique and desirable electrical and magnetic behaviors as well as due to the presence of unexpected behavior in the nanoscale regimes with respect to their chemical and physical properties. Ferrites are considered those ceramic materials in which the main constituent of them is metal oxides such as iron oxide. According to the magnetization and their application, ferrite materials fall into two main groups: if a material is easily magnetized and demagnetized, it is considered as a soft magnetic material. If a magnetization and demagnetization of a material is difficult, it is well-known as hard or permanent magnetic material. The soft magnetic materials are mostly utilized in applications such as a microwave communication system, and as core materials for transformers and inductors. In contrast, the hard magnetic materials are broadly used in loudspeakers, motors, and other electrical-mechanical energy conversion devices. Generally speaking, based on the crystal structure type, ferrites are divided in three main groups: (1) the spinel type, considering spinel ferrites, (2) the garnet type, considering garnet ferrites and (3) the magnetoplumbite type, considering hexagonal ferrites. The first and second type of ferrites are often considered a subdivision of soft magnetic ferrites, while, the third type belongs to or under the hard magnetic ferrites.

Relatively, a low externally applied field is required to magnetize the soft magnetic ferrites in a way that a low magnetism is retained in these materials by removing the applied field. The general chemical composition formula of soft ferrites is considered as AB_2O_4, where A includes one or more divalent metallic ions such as Ni, Zn, Mg, Cd, and so on, while B includes the trivalent iron ions, and O stands for oxygen. On the other hand, a hard magnetic material becomes magnetized by a high applied magnetic field. The properties of these materials are characterized with high remnant magnetism. These ferrites are often prepared from iron oxides and strontium or barium oxides.

Soft magnetic materials such as Ni-Zn ferrite and Ni-ferrite are important ceramic-magnetic materials in our daily lives due to their extensive use in electrical devices and telecommunication industries. Due to their unique characteristics such as low eddy current loss, good thermal and chemical stability, high resistivity, low coercivity and high Curie temperature (Ravindranathan and Patil 1987; Bid and Pradhan, 2003), they are also found in a wide variety of applications. Such applications include being used in microwave devices, rod antennas, power transfer systems, and read/write heads for high speed digital tapes.

Furthermore, in respect to their preparation, these soft ferrites are being produced from inexpensive raw materials. They are also more stable ceramic compounds as compared to other soft ferrites. The electrical and magnetic properties of $NiFe_2O_4$ and $Ni_{1-x}Zn_xFe_2O_4$ spinel ferrites are sensitive to the microstructure characteristics and the compositional variability, which are influenced by the applied technique to prepare them (Verma et al., 2005). Moreover, due to the evaporation of zinc at higher temperatures, it gives rise to the formation of non-stoichiometric composition which can alter the Ni-Zn ferrite properties. In this study, firstly, the attempt was to produce $NiFe_2O_4$ ferrite then a Ni_{1-x}-$Zn_xFe_2O_4$ ferrite, involving two different compositions in which the Zn content was chosen to be X = 0.36 and 0.64.

1.2 The Microstructural-magnetic relationship of Soft Ferrites

Nanometer size particles of spinel ferrites, which were produced by the high-energy ball-milled method, offer fascinating chemical, physical and magnetic properties which remarkably differ from their bulk counterparts. They have been carried out through several studies on the comparison of nano-sized and bulk spinel ferrites properties (Kodama et al., 1996, Chinnasamy et al., 2002, Oliver et al., 2000). Šepelák et al., (1997) recognized that the mechanical activation route can increase the chemical reactivity of nano-sized powders in the aim of the production of spinel ferrites. In another study, preparing nanocrystalline spinel ferrites by the high energy ball milling method, they found a disordered spin configuration, nonequilibrium cation distribution as well as unstable nano-sized particles in the final product. The sintering process leads to the recrystallization of the milled samples and results in their transition from excited metastable state to the low-energy crystalline state. Furtheremore, the desirable properties of nano-sized ball-milled spinel ferrites are lost during the sintering process (Šepelák et al., 1998). Due to this, it is required to scrutinize the relaxation mechanism of mechanically alloyed induced metastable states as well as the thermal stability of a spinel ferrite nanostructure. To grasp the response of nanosized crystalline spinel ferrites to variations in temperature is essential not only for basic science (the improvement of microscopic and atomistic theory of the mechanically-alloyed process), but also due to the industrial and technological high-temperature utilizations in information storage, ferrofluids and catalysis. To broaden the knowledge on the correlations microstrurral-magnetic behavior evolution, this project undertakes on the response of fine nano-size initial powders made up of mechanically alloyed Ni-ferrite and Ni-Zn ferrite to vary in the sintering conditions. Although several investigations have recently been carried out on the nano-sized-milled soft magnetic materials (Jiang et al., 1999, Oliver et al., 2000, Šepelák et al., 2000, Chinnasamy et al., 2002), no measurement of the thermally induced magnetic and structural evolutions in these metastable materials have been stated.

1.3 Problem Statement

Since the electrical and magnetic properties of soft ferrites are strongly dependant on the microstructure and the preparation route, thus, the parallel evolution in the changes in microstructure with controlling process factors must be considered. Furthermore, according to literature studies, the higher sintering temperatures destroy some advantages of the nanostructured materials. Therefore, these questions would seem to be apparent: how to find the best way involving method and raw materials to synthesize nanocrystalline soft magnetic ferrites like nickel ferrite and nickel-zinc ferrite. How to optimize the variable parameters of the used method to save time and energy? What would be the magnetic-microstructure as well as composition-microstructure correlations for milled samples in different atmospheres, at intermediate sintering process, during the parallel evolutions of the microstructure- magnetic and electrical properties?

1.4 Objectives

The main aim of this study is to examine the parallel evolution of microstructural, electrical and magnetic behaviors of the milled samples in different atmospheres as well as at different sintering temperatures. The achieved results from this research work can be used to develop the new general theoretical models on the parallel evolution of the microstructure and various properties of advanced materials in future studies.
Here, in this research work, the work-step objectives are involved in three sections as follows;

Section I
1) To prepare nanocrystalline $NiFe_2O_4$ via mechanically alloyed nanoparticles and to optimize the variable parameters of ball milling process using the Taguchi Robust design.
2) To study the effect of a sintering temperature on the microstructure characteristics, electrical and magnetic properties of Ni-ferrrite.

Section II
1) To prepare nanocrystalline $Ni_{1-x}Zn_xFe_2O_4$ ferrite with two different compositions (x = 0.36, 0.64) via mechanically alloyed (high-energy ball milling) and study the effects of the milling time and milling atmosphere including argon, air and oxygen on the products.
2) To examine the evolution of ferrite properties with microstructure modification as a consequence of sintering temperature from 400 to 900 ºC.

Section III
1) Adding common additive oxides of $0.4CaO + 0.8SiO_2$ to the $Ni_{0.36}Zn_{0.64}Fe_2O_4$ and investigation the effects of additives percentage, sintering time and temperature on the microstructure as well as electrical and magnetic properties.

1.5 Thesis Outline

This chapter briefly describes the general introduction of ferrite, correlation between microstructure and magnetic behaviors of a ferrite, and the problem statement as well as the research objectives. In chapter two, it reports the views of previous literatures about the performed synthesis techniques, high-energy ball milling or mechanical alloying with its effective variable parameters and the effects of microstructure changes on some magnetic behaviors. Chapter three focuses on the basic theories that have been stated about magnetic ferrites and sintered materials. In fact, this chapter describes the basic of magnetization, spinel ferrites structure, sintering process parameters and mechanism of mechanical alloying. Chapter four presents the methodology of sample preparation and the equipments that were used to characterize the occurred changes in the products; such as phase changes, structural variations, as well as electrical and magnetic behaviors under various conditions. Chapter five is about the obtained results from the current research work and discussion. The summary of the concluded results was presented in Chapter Six which was followed by future research suggested recommendations. Finally, the references, appendix and list of publications were attached, accordingly.

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Publications in ISI Journals during the Ph.D program

N.	Title of paper	Corresponding Author
1	Structure and magnetic properties of $Ni_{0.64}Zn_{0.36}Fe_2O_4$ nanoparticles synthesized by high-energy milling and subsequent heat treatment" *Journal* of Materials *Science*: *Materials in Electronics.* DOI 10.1007/s10854-014-2597-4. (IF: 1.966, Q2) **Abdollah Hajalilou**, Mansor Hashim, Halimah Mohamed Kamari	**A.Hajalilou**
2	A comparative study of in-situ mechanochemically synthesized $Mn_{0.5}Zn_{0.5}Fe_2O_4$ ferrite nanoparticles in the $MnO/ZnO/Fe_2O_3$ and $MnO_2/Zn/Fe_2O_3$ systems, Ceramics International. Ceramics International, Ceramics International41 (2015)8070–8079. (IF: 2.086, Q1) **Abdollah Hajalilou**, Mansor Hashim, Mohamad Taghi Masoudi	**A.Hajalilou**
3	Synthesis and structural characterization of nano-sized nickel ferrite obtained by mechanochemical combustion". Ceramics International l40 (2014)5881–5887. (IF: 2.086, Q1) **Abdollah Hajalilou**, Mansor Hashim, Reza Ebrahimi-Kahrizsangi, Halimah Mohamed Kamari, Nahid Sarami	**A.Hajalilou**
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