UNIVERSITI PUTRA MALAYSIA

PARALLEL EVOLUTION OF MICROSTRUCTURE, PHYSICAL PROPERTIES AND THEIR RELATIONSHIPS IN Co0.5Zn0.5Fe2O4 CERAMICS

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By

MUHAMMAD MISBAH BIN MUHAMAD ZULKIMI

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

November 2013

In appreciation of their love and sacrifices, this thesis is dedicated to Parents
MUHAMAD ZULKIMI AWANG and SUYATI ABD RAZAK.

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

PARALLEL EVOLUTION OF MICROSTRUCTURE, PHYSICAL PROPERTIES AND THEIR RELATIONSHIPS IN $Co_{0.5}Zn_{0.5}Fe_2O_4$ CERAMICS

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MUHAMMAD MISBAH BIN MUHAMAD ZULKIMI

November 2013

Chairman: Associate Professor Mansor Hashim, PhD

Institute: Institute of Advanced Technology

For more than eight past decades, the ferrite research literature has only very superficially dealt with the question of how the evolving microstructure of a ferrite material relates to its accompanying, resultant magnetic properties. The literature has only covered in great detail the answers for the case of ferrite materials obtained from final sintering. Thus, this work is a fresh attempt to critically track the evolution of magnetic properties parallel to the microstructural changes in bulk $Co_{0.5}Zn_{0.5}Fe_2O_4$ samples and to relate the properties to the changes wherever possible.

In this study, high energy ball milling (HEBM) was used to prepare $Co_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles with sintering temperatures from 500 ^oC to 1350 ^oC and with a 50 ^oC increment. Physical characteristics of the as-prepared and sintered samples were studied using X-ray diffraction (XRD), scanning transmission electron microscopy (STEM) and Field Emission Scanning Electron Microscope (FESEM); characterization of magnetic properties was carried out by using An Agilent Model 4291B impedance/material analyser in the frequency range of 1 MHz to 1.0 GHz which was used to measure the permeability and the loss factor. The magnetic properties of the samples were investigated using a MATS-2010SD Static Hysteresisgraph.

The XRD results confirm that Cobalt zinc ferrite cannot be formed directly through milling alone, but heat treatment is necessary. After sintering at 550 ^oC, the cobalt zinc ferrite phase was obtained earliest at this temperature with an average grain size in the nanometric range (0.089µm). The microstructure studies of $Co_{0.5}Zn_{0.5}Fe_2O_4$ showed that the grain size increased, density was increased and porosity was decreased as the sintering temperature increased. The parallel evolution of hysteresis parameters with microstructural properties for sintered samples was studied. The saturation induction, Bs, increased with the enhancement grain size. The highest saturation induction value for sintered samples is 1486.00 G. The coercivity value was increased with increasing grain size and reached a maximum value (24.93 Oe) before dropping with further

increasing grain size. The permeability and loss factor values were observed and can be classified into three different groups with the utilization the similar B-H hysteresis loop results: weak ferromagnetic (first group), moderate magnetic (second group) and strong ferromagnetic (third group) behavior. The resistivity value of the sample under investigation had semiconductor behavior. The Curie temperature varied slightly for the samples due to stoichiometric changes allocated with zinc loss.

Finally, after analysing the results and the observations of the work mentioned above, it is strongly believed that there are three factors found to sensitively influence the samples content of ordered magnetism –their ferrite-phase crystallinity degree, the number of grains above the critical grain size and large enough grains for domain wall accomodation. This research work has shed new light on the microstructure-magnetic properties evolution in ferrites.

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

EVOLUSI SEIRING SIFAT-SIFAT MORFOLOGI, FIZIKAL DAN HUBUNGANNYA DALAM SERAMIK $Co_{0.5}Zn_{0.5}Fe_2O_4$

Oleh

MUHAMMAD MISBAH BIN MUHAMAD ZULKIMI

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Pengerusi: **Profesor Madya Mansor Hashim, PhD**

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Sejak daripada lapan dekad yang lampau, kajian penyelidikan ferit hanya menangani persoalan bagaimana sesuatu mikrostruktur ferit yang sedang mengalami evolusi berkait dengan sifat magnet yang terhasil seiring dengannya dengan tidak begitu mendalam. Maka projek ini adalah cubaan terbaru untuk mencerap secara kritikal evolusi pencirian magnet seiring dengan perubahan mikrostruktur bagi sampel $Co_{0.5}Zn_{0.5}Fe_2O_4$.

Dalam kajian ini, pengisaran bola bertenaga tinggi (HEBM) telah digunakan untuk menyediakan $Co_{0.5}Zn_{0.5}Fe_2O_4$ bersaiz nano dengan suhu pensinteran bermula dari 500 ^oC 1350 ^oC dan kenaikan 50 ^oC. Ciri-ciri fizikal sampel dikaji menggunakan pembiasan sinar-X (XRD), mikroskop pengimbas elektron (STEM) dan mikroskop pengimbas medan elekron (FESEM) dan pencirian sifat magnet dengan menggunakan Agilent Model 4291B Rangkaian / Spectrum analyzer dalam julat frekuensi 1 MHz hingga 1.0 GHz telah digunakan untuk mengukur ketelapan dan faktor kehilangannya. Sifat-sifat magnet sampel telah dikaji dengan menggunakan Hysteresisgraph statik Mats-2010SD.
Keputusan XRD mengesahkan bahawa Cobalt zink ferit tidak boleh dibentuk secara langsung melalui pengisaran sahaja, tetapi rawatan haba diperlukan. Selepas persinteran sampel pada 550 ^oC, fasa kobalt zink ferit diperolehi pada suhu ini dengan saiz purata mikrostruktur dalam lingkungan nano (0.089 μm). Kajian mikrostruktur $Co_{0.5}Zn_{0.5}Fe_2O_4$ menunjukkan bahawa saiz butiran meningkat bersama dengan peningkatan nilai ketumpatan dan liang-liang udara menurun apabila suhu pensinteran meningkat. Evolusi selari parameter histerisis dengan ciri-ciri mikrostruktur untuk sampel tersinter telah dikaji. Induksi tepu, Bs, meningkat dengan peningkatan saiz butiran. Nilai tertinggi induksi tepu untuk sampel tersinter adalah 1486.00 G. Nilai daya paksa (Hc) meningkat dengan peningkatan saiz butiran dan mencapai nilai maksimum 24.93 Oe sebelum ia menurun dengan peningkatan saiz butiran. Ketelapan dan nilai-nilai faktor kehilangan diperhatikan dan boleh dikelaskan kepada tiga kumpulan yang berbeza dengan penggunaan BH keputusan gelung histerisis yang sama iaitu: feromagnet lemah

(kumpulan pertama), magnet sederhana (kumpulan kedua) dan tingkah laku feromagnet yang tinggi (kumpulan ketiga). Nilai kerintangan sampel yang dikaji pula mempunyai sifat semikonduktor. Suhu Curie berubah sedikit untuk sampel yang disebabkan oleh perubahan stoikiometri yang berlaku kerana kehilangan zink.

Akhir sekali, selepas menganalisis keputusan dan pemerhatian kerja yang dilakukan, diyakini bahawa terdapat tiga faktor yang secara sensitif mempengaruhi kandungan kemagnetan bertertib sampel iaitu darjah fasa kehabluran, jumlah butiran melepasi saiz kritikal butiran dan butiran yang cukup besar untuk penempatan dinding domain. Kajian penyelidikan ini telah menyumbangkan pengetahuan baharu tentang terhadap evolusi mikrostruktur-sifat magnet dalam bahan ferit.

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I certify that a Thesis Examination Committee has met on 19 November 2013 to conduct the final examination of Muhammad Misbah bin Muhamad Zulkimi on his thesis entitled “Parallel Evolution of Microstructure, Physical Properties, and Their Relationships in $Co_{0.5}Zn_{0.5}Fe_2O_4$ Ceramics” 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 master science

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This is to confirm that:

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LIST OF ABBREVIATIONS

XRD	X-ray diffraction
STEM	Scanning Transmission Electron Microscopy
HEBM	High Energy Ball Milling
kHz	kilohertz
MHz	megahertz
GHz	gigahertz
wt %	Weight percent
hkl	Miller indices
ICDD	Joint Committee on Power Diffraction Standard
BPR	Ball-to-powder weight ratio
MA	Mechanical alloying
a.u	Arbitrary unit
H	Magnetic field strength
H_C	Coercivity
M	Mass magnetization
B_S	Saturation induction
θ_C	Curie temperature
2θ	2 theta degree
μm	microns
μ’	Real permeability
μ”	Loss factor
Ms	Saturation magnetization

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Microstructure similar to any single-phase polycrystalline ceramic, complex systems of magnetic oxide consisting of crystals pores and grain boundaries are called polycrystalline ferrites. For these materials, their microstructure is strongly affected by few factors such as the calcination temperature, quality raw materials and the sintering temperature regime.

In most cases, the grains, the pore size, the porosity, the density, intra-granular and inter granular distribution of pores and grains are the parameters of microstructure. The quantity, size, shape and distribution of both crystal grains and pores of a ferrite are influenced by different techniques and preparation conditions.

The magnetic properties of these ferrites are determined and strongly influenced by the chemical composition, the crystal structure and the microstructural parameters such as grain size, grain size distribution, porosity, etc. obtained at a particular sintering temperature. Unfortunately, for several decades, studies of the relationship between magnetic properties and morphological properties have been focusing only on the product of the final sintering temperature. These studies abandoned any detailed tracking of the parallel evolutions of morphology and magnetic properties in the 1 nm-to-1μm grain materials, which could have been observed by the early ferrite researchers using wet-chemistry synthesis methods.

The question in the recent work is whether we are able to provide a good explanation of the evolution relationships between magnetic properties and morphological properties of a particular ferrite ($Co_{0.5}Zn_{0.5}Fe_2O_4$) to be prepared by mechanical alloying.

1.2 Ferrite materials

In recent years, there has been considerable interest in the study of properties of nanosized ferrite particles because of the importance in the fundamental understanding of the physics of the properties as well as their proposed applications for many technological purposes (Sharma et al., 2005). For example, these applications include high frequency (HF) choke filters, HF transformer, antenna rods, etc. (Moulson and Herbert, 1990).

Generally, the mixed oxides containing iron oxides as their main component are called ferrite materials. Unlike most materials, they possess both high permeability and moderate permittivity at frequencies from direct current (DC) to the millimeter wavelengths (Harris et al., 2009).

In commercial industry, ferrites materials can be classified in three important types, each one having a specific crystal structure listed below:

a) Spinel ferrites, for example NiZn-, MnZn- and MgMnZn ferrite.
b) Garnet structure microwave ferrites, for example Yitrium iron garnet
c) Magnetoplumbite (Hexagonal) ferrites, for example Barium (Ba) and Sr hexaferrites

In general, types a) and b) can be grouped in one group of “soft” ferrites, while type c) can be grouped into “hard” ferrites. The terms of “soft” and “hard” refer to the characteristics and properties of the ferrites. The “soft” ferrites means that it’s easy to magnetise and easy to dimagnetise the ferrite materials; meanwhile, the “hard” ferrites are difficult to magnetise and also difficult to dimagnetise.
In the present work, the only interest on the soft ferrite and are just focused on the spinel ferrites (type a).

1.3 Problem Statement

Many researchers just focused on the sintering and synthesis to get the better material in the studies of spinel ferrites. However, what would be the composition-microstructure relationship at the various intermediate sintering conditions during the parallel evolutions of the morphology and the materials properties? After more than eight decades of ferrite research, this question has been hardly answered. Hence it is now taken as the problem statement of this research work with specific form an the $Co_{0.5}Zn_{0.5}Fe_2O_4$ ferrite

1.4 Objectives

The ultimate goals of this research to determine the relationship between magnetic properties and microstructure parameter which exists during any states of their parallel evolution and to explain the underlying science for their relationship, it is interesting to study the evolution of the magnetic properties starting with and from nano material powder. However, to achieve the goals above, the necessary work has to be carried out and these research-work steps have the following objectives:

1. To prepare $Co_{0.5}Zn_{0.5}Fe_2O_4$ using mechanically alloyed nanoparticles
2. To study the phase formation of the as-prepared ferrite using X-ray diffraction (XRD)
3. To study the effect of the sintering temperature on the microstructure evolution and magnetic properties

1.5 Thesis outline

This thesis consists of six chapters. First chapter introduces the ferrite material, the problem statement and the objectives of this research. Chapter two is devoted to the literature review on preparation of ferrite, structure of spinel ferrite and application of the theory that will be used for this research. The Methodology of this research is presented in Chapter three. Meanwhile, results and discussion are covered in Chapter four. Finally Chapter five provides the summary of this research and suggestions for future work.

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