SYNTHESIS OF LOW-LOSS YTTRIUM IRON GARNET ($Y_3Fe_5O_{12}$) VIA THE MICROEMULSION TECHNIQUE AND ITS SYNTHESIS VARIATION EFFECTS ON ITS PROPERTIES

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By

MASNI BINTI MANAP

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

July 2013

This thesis is dedicated to my late father
Allahyarham Haji MANAP BIN ABDULLAH,
my beloved mother Hajah KULSHAM BINTI PONIMIN,
my brothers and sisters, MAHIDAN, MARIAH, MAHADIR,
MARIYANTI, MARIZA, and MUHAMAD,
in appreciation of their love and supports.

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

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Chairman : Associate Professor Mansor Hashim, PhD

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Yttrium iron garnet, $Y_3Fe_5O_{12}$ (YIG), is a material widely used in electronic devices for the microwave region from 300 MHz to 100 GHz range. Even though the technology involving ferrites is advancing, there is still a lack of understanding and systematic examinations on how losses in ferrites occur and how to control them especially at UHF or higher frequencies. In this work, YIG powders were prepared via the microemulsion technique and different approaches of bulk preparation were employed to attempt production of extremely low loss YIG. The loss of conventional-uniaxial sample, monodisperse-uniaxial sample and monodisperse-CIP sample was studied for samples sintered at relatively low temperature and up to fairly high temperature. YIG was produced by the microemulsion technique as the growth of the precursor was controlled by a water-in-oil emulsion. The aqueous solution consists of ($Y(NO_3)_3$•6 H_2O) and ($Fe(NO_3)$ •9 H_2O). Cetyltrimethyammonium Bromide (CTAB) was used as the surfactant and n-octane as the oil phase. Ammonium hydroxide solution which acts as a reducing agent was added to the aqueous solution to form precipitates. Then, the precipitates were separated by centrifugation, washed with ethanol and then dried at 80 °C for 12 h. The dried precipitate was calcined at 600 °C for 2 h and ground into powder form. Three torroidal samples were prepared which is conventional-uniaxial sample, monodispersion-uniaxial sample, and monodisperse-CIP sample were sintered at different temperatures. The particle size was confirmed by Transmission Electron Microscopy (TEM), the thermal analysis was performed using a Thermal Thermogravimetric Analyzer (TGA), the phase was characterized using X-ray diffraction (XRD) and morphology was observed by Field Emission scanning electron microscopy (FeSEM). The permeability and rf energy loss of the samples was studied using an impedance material analyzer. The TEM results show that the particles are in the nanometer range with an average of 24 nm. The crystallization temperature of the sample can be deduced to be at 1145°C as observed from the TGA curve. The XRD results show that the full phase of YIG is formed at 1200°C. FeSEM micrographs and grain size distributions for the samples with different preparation techniques show the evolution of microstructure as the grain size increases with the increase of the sintering temperature. The micrographs clearly illustrate the evolution of the particle constituent to the formation of necks which lead to grains development over the sintering temperature range. The conventionally prepared and monodisperse sample show the correlation between magnetic loss, phase purity and grain size as it decrease with the decrease of grain size and phase purity. The monodisperse-CIP sample shows a different trend where it has a big grain size but low loss. We speculate that the monodisperse-CIP samples have significant numbers of pores that can act as pinning centers to the domain wall movements thus decreasing the magnetic loss of the sample. All the samples exhibit lower loss with tan δ lower than 10^{-1} comparable to previous research results.

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

PENYEDIAAN YTTRIUM IRON GARNET ($Y_3Fe_5O_{12}$) YANG MEMPUNYAI KEHILANGAN TENAGA YANG SANGAT SEDIKIT MELALUI TEKNIK MICROEMULSION DAN KESAN VARIASI PENYEDIAAN TERHADAP CIRI-CIRINYA

Oleh

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Yttrium Iron Garnet, $Y_3Fe_5O_{12}$ (YIG), merupakan bahan yang digunakan secara meluas dalam alat-alat elektronik bagi rantau gelombang mikro daripada 300 MHz hingga 100GHz. Walaupun teknologi ferit semakin maju, masih terdapat kekurangan pemahaman dan pemeriksaan yang sistematik tentang bagaimana kehilangan tenaga dalam ferit berlaku dan bagaimana untuk mengawalnya terutama pada frekuensi yang sangat tinggi atau frekuensi yang lebih tinggi. Dalam kajian ini, serbuk YIG telah disediakan melalui teknik mikroemulsi dan pendekatan yang berbeza bagi teknik penyediaan pukal telah dilakukan dalam percubaan untuk menghasilkan kehilangan YIG amat rendah. Kehilangan tenaga bagi sampel proses penyediaan biasa-unipaksi, sampel monodisperse-unipaksi dan sample monodisperse-CIP dikaji pada suhu pensinteran yang agak rendah dan sehingga ke suhu pensinteran yang tinggi. YIG telah dihasilkan oleh teknik mikroemulsi dengan pertumbuhan prekursor telah dikawal oleh emulsi air dalam minyak. Larutan akueus terdiri $Y(NO_3)_3$ dan $Fe(NO_3)$, Cetyltrimethyammonium bromida (CTAB) telah digunakan sebagai surfaktan dan n-oktana sebagai fasa minyak.

Larutan Ammonium hidroksida yang bertindak sebagai agen penurunab ditambah kepada larutan akueus bagi membentuk mendakan. Kemudian, mendakan telah dipisahkan oleh sentrifugasi, dibasuh dengan etanol dan kemudian dikeringkan pada 80°C selama 12 jam. Mendakan kering telah dikalsin pada 600°C selama 2 jam dan dihancurkan kepada bentuk serbuk. Tiga sampel berbentuk torroid iaitu sampel proses penyediaan biasa-unipaksi, sampel monodisperse-unipaksi dan sample monodisperse-CIP telah disinter pada suhu yang berbeza. Saiz partikel telah disahkan oleh mikroskop transmisi elektron (TEM), analisis terma telah dilakukan menggunakan analisia terma termogravimetri (TGA), fasa dicirikan menggunakan pembelauan sinar-X (XRD) dan morfologi telah diperhatikan oleh mikroskop imbasan elektron pelepasan medan (FeSEM). Kebolehtelapan dan kehilangan tenaga rf sampel telah dikaji menggunakan penganalisis bahan impedans. Keputusan TEM menunjukkan bahawa partikel dalam julat nanometer dengan purata sebanyak 24 nm. Suhu pembentukan hablur boleh disimpulkan pada 1145°C seperti yang dilihat dari lengkung TGA. Keputusan XRD menunjukkan bahawa fasa penuh YIG telah terbentuk pada 1200°C. Mikrograf FeSEM dan taburan saiz butiran untuk sampel bagi semua teknik penyediaan menunjukkan evolusi mikrostruktur apabila saiz butiran meningkat dengan peningkatan suhu pensinteran. Mikrograf jelas menggambarkan evolusi butiran kepada pembentukan leher yang membawa kepada pembangunan butiran seiring dengan suhu pensinteran. Sampel penyediaan biasa dan monodisperse menunjukkan korelasi antara kehilangan tenaga, ketulenan fasa dan saiz butiran kerana ia berkurangan dengan penurunan saiz butiran dan ketulenan fasa. Sampel CIP menunjukkan tren yang berbeza di mana ia mempunyai saiz butiran yang besar tetapi mempunyai kehilangan tenaga yang rendah. Kami berspekulasi bahawa sampel CIP mempunyai bilangan liang yang signifikan yang boleh menyematkan pusat untuk pergerakan dinding domain sekali gus mengurangkan kehilangan tenaga sampel. Semua sampel mempamerkan kehilangan tenaga yang lebih rendah yang mempunyai tan δ lebih rendah daripada 10^{-1} jika dibandingkan dengan penyelidikan sebelumnya.

ACKNOWLEDGEMENTS

In the name of Allah, the Most Gracious and the Most Merciful

Alhamdulillah, all praises to Allah for the strengths and His blessing in completing this thesis. Special appreciation goes to my supervisor, Associate Professor Dr. Mansor Hashim, for his supervision and constant support. His invaluable help of constructive comments and suggestions throughout the experimental works and thesis have contributed to the success of this research. Not forgotten, my appreciation to my co-supervisor, Dr. Zulkifly Abbas for his support and knowledge regarding this topic.

I would like to express my appreciation to the Magnetics and Nanostructured Polycrystals Evolution Group (MNPEG): Dr. Ismayadi Ismail, Dr. Jumiah Hassan, Dr. Norlaily Mohd Saiden, Dr. Md. Shuhazly Mamat, Dr. Rabaah Syahidah Azis, Idza Riati Ibrahim, Rodziah Nazlan, Fadzidah Mohd Idris, Mohd Shamsul Ezzad Shafie, Ghazaeh Bahmanrokh, Wan Norailiana Wan Ab. Rahman, Nor Hapishah Abdullah, Mutia Suhaibah Abdullah, Muhammad Misbah Muhamad Zulkimi, Mohd Khairul Ikhwan Mohd Zawawi, Aizat Noor Ismail, Muhammad Syazwan Mustaffa, Che Sulaiman Ahmad, Mehran Masudi, Abdollah Hajalilou, Hatef Sabba, and Zahra Behjat for their help on setting off trains of thought and spark ideas or understandings. My acknowledgement also goes to all the technicians and office staffs of Faculty of Science and Institute of Advanced Technology (ITMA), En. Md. Ali Rani, En. Kadri Masaud, Pn. Sarinawani Abd. Ghani, Pn. Norhaslinda Noruddin, Pn. Kamsiah Alias, Pn. Noriza Maslan and others for their helps and co-operations.

Sincere thanks to all my friends especially Nor Eliza Mat Jusoh, Mastura Mohamad, Nor Shafina Sahinin, Aisyah Ruqayyah Mohd Sabbri, Syuhada Abu Bakar, Sumaiyah Megat Nabil Mokhsin, Nor Lyana Adnan, Wan Nurul Ain Wan Shaaidi, Dayang Nur Fazliana Abdul Halim, Siti Nur Ain Rusly, Nurzilla Mohamed, Nur Fadilah, Nur Amalina Hafiza, Zarifah Alassan, Arlina Ali, Emma Ramli, Nur Fauzana Adnin, Norni Hidayawati Mat Daud and others for their kindness and moral support during my study. Thanks for the friendship and memories.

Last but not least, my deepest gratitude goes to my late father, Manap Bin Abdullah, my beloved mother Kulsham Binti Ponimin, to my brothers and sisters, and to my nephews and nieces for their endless love, prayers and encouragement. To those who indirectly contributed in this research, your kindness means a lot to me. Thank you very much.

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LIST OF ABBREVIATIONS

YIG	Yttrium Iron Garnet
f	frequency
kHz	kilohertz
MHz	megahertz
GHz	gigahertz
μ'	real permeability
μ''	magnetic loss
μ^*	complex permeability
ρ	density
$YFeO_3$	Yttrium Orthoferrite
Fe_2O_3	Iron Oxide
XRD	X-ray diffraction
SEM	Scanning Electron Microscopy
TEM	Transmission electron microscopy
nm	nanometer
MUT	Material under test
JCPDS	Joint Committee on Power Diffraction Standard
a.u	Arbitrary unit
2θ	2 theta degree

CHAPTER 1

INTRODUCTION

1.1 Background of the study

In the earlier part of electrical technology development, iron and its alloys were used as magnetic materials to supply the need of the electrical industry for a long time. However, with the introduction of higher frequencies, the standard techniques of using lamination or iron powder cores, reducing eddy current losses, were no longer cost effective or efficient. This realization stimulated a renewed interest in "magnetic insulators" since first reported by Hilpert in Germany (1909). The high electrical resistivity of oxides could be combined with desired magnetic characteristics and a magnetic material would result that was well suited for high frequency operation.

Various laboratories all over the world have done research to develop such a material, such as by V. Kato, T. Takei, and N. Kawai in the 1930's in Japan and by Snoek of the Philips' Research Laboratories in the period 1935-45 in the Netherlands (McLyman and McLyman, 2004). By 1945 Snoek had laid down the basic fundamentals of the physics and technology of practical ferrite materials. In 1948, the Neel theory (1948) of ferromagnetic provided the theoretical understanding of this type of magnetic material. These ferrites are ceramic, homogeneous materials composed of various oxides with iron oxide as their main constituent.

Generally, ferrites are classified into three classes based on three different crystal types which are:

i) Soft Ferrite with spinel cubic structure, for example; Nickel Zinc Ferrite and Manganese Zinc Ferrite.

ii) Soft Ferrite with garnet structure, for example yttrium-based garnets that are used in microwave devices.

iii) Hard Ferrite with magnetoplumbite (hexagonal) structure. The hexagonal ferrites develop high coercivity and are an important member of the permanent magnet family.

Among most common representatives of soft ferrite with the garnet structure is Y_3 Fe_5 O_{12} (YIG) which has been widely investigated as it is an interesting ferrimagnetic material. This material has high resistivity and low magnetic loss at high frequency which has made it among the best UHF magnetic materials. It is widely used in the microwave frequency range and optical-communication devices and other applications (Vaqueiro et. al, 1997).

1.2 Ferrite for Microwave Application

Microwave technology is moving up to higher frequencies and higher bandwidths, into the mm wave range, up to 100 GHz. Nonconducting materials are essential to ensure total penetration of electromagnetic fields. Ferrite materials are unique because they are one of a few classes of insulating magnetic oxides that possess moderate value of magnetization, high permeability, moderate to high permittivity, and low losses at frequencies from dc to sub-millimetre wavelengths. These properties add to them a great value in high frequency devices that require strong coupling to electromagnetic signals while experiencing low losses (Harris et al., 2009). Ferrite elements are widely used in microwave devices such as isolators, circulators, and phase shifters. For applications requiring nonreciprocal operation, as in circulators and isolators, there is no alternative to magnetic devices. Due to their very high specific resistance, remarkable flexibility in tailoring the magnetic properties, ease of preparation, and, last but not the least, price and performance considerations, ferrites are the first choice materials for microwave applications. However, the frequency range of operation, the power handling capacity and the temperature sensitivity of ferrite devices should be improved.

Nanostructured materials have a number of desirable electromagnetic and mechanical properties. Electromagnetic absorption properties can be controlled by changing the particle size distribution in nano-materials and application-specific, tailored materials can be produced. Eddy -current and magnetic losses are minimized in nano-materials and very sharp resonances can be set up leading to high-Q filter characteristics. This can be directly exploited in antenna technology. Few key aspects that make nano-materials very attractive candidates for antenna technology development are:

i. Physical properties different from bulk and often superior
ii. Superior mechanical properties
iii. Better control of microstructure, porosity
iv. Selective enhancement of desirable parameter

1.3 Problem Statement

To appreciate the value of ferrites in microwave applications, it is important to understand the basic physical phenomena that are involved in successful device operation. Even though the technology in ferrites is advancing, there is still a lack of understanding and systematic investigation on how losses in ferrites occur and how to control them especially at UHF or higher frequencies.

Some magnetic properties of a ferrite depend critically on the structure and microstructure of the material. The dependence towards microstructure of the materials leads to the development of techniques to produce garnets with a strict control of the composition, homogeneity, size and particle shape (Vaqueiro et. al, 1997). This work would attempt to obtain extremely low loss nanometer-sized YIG particles by the water-in-oil microemulsion technique.

1.4 Objectives

The goal of this research is to synthesize a high quality yttrium iron garnet with extremely low electromagnetic loss via the microemulsion technique. In order to accomplish this work, an experimental investigation was carefully conducted. The objectives are as follows:

1) To prepare uniform and monodisperse Yttrium Iron Garnet nanoparticles from microemulsion technique.

2) To study the effect of phase purity, crystal structure and microstructure on EM energy losses.
3) To synthesize a garnet that can transmit and receive microwave energy with very little EM energy loss, employing synthesis variation effects.

1.5 Thesis Outline

The earliest chapter of this thesis gives an introduction on ferrites, ferrites for microwave application and some research questions. Chapter two presents aspects on the related literature on low loss ferrites, synthesis methods, and some microstructural of ferrites. Chapter three reports the basic theories of magnetism, the ferromagnetic structure of garnet, and microemulsion synthesis. The preoccupations in chapter four are methodologies employed for the preparations and the characteristics measurement of the prepared YIG samples. The discussion of the results obtained forms chapter five. Chapter six summarizes and concludes the research findings, in addition to some suggested recommendations. The list of publications by the author is attached at the end of the thesis, preceded by the author's biography, appendices and references/bibliographies.

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