UNIVERSITI PUTRA MALAYSIA

EFFECTS OF SINTERING TEMPERATURE ON MICROSTRUCTURAL, ELECTRICAL AND MICROWAVE PROPERTIES OF GADOLINIUM IRON GARNET

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ITMA 2018 13

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By

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

June 2018

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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June 2018

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The effect of various sintering temperatures towards microstructural of GdIG has not been carried out in the previous research, which can provide the information on the fundamental knowledge of magnetic behaviour from nanometer to micrometer grain size regime. Therefore, the relationship between microstructural evolution from nano to micron grain size and their physical, magnetic, electrical and microwave properties of the gadolinium iron garnet ($Gd_3Fe_5O_{12}$, GdIG) has been investigated systematically. Raw materials were milled using mechanical alloying method for 3 hours, shaped into toroid and pellet and sintered from 600 ^oC to 1400 ^oC with a 100 ^oC increment.

The average particle size measured after milling using Scanning Transmission Electron Microscope (STEM) was ~36.9 nm. GdIG phase which was identified using X-ray diffraction (XRD) began to form a single phase with cubic garnet structure at 1000 ^oC. At this temperature, magnetization studied using vibrating scanning magnetometer (VSM) had started to show an improvement with highest magnetization value of 3.86 emu/g. The Curie temperature (T_c) obtained which was measured using LCR (Inductance, Capacitance and Resistance) meter for GdIG was ~300 ^oC. As the sintering temperature increased, the crystallinity improved with the highest intensity of XRD peak for sample of 1000 ^oC. The microstructure evolution observed using Field Emission Scanning Electron Microscope (FESEM) had influenced the changes in magnetic, microwave and electrical properties. For magnetic measurement, the maximum value obtained through impedance analyser for permeability was 1.33 and the highest induction value of 108.9 G was obtained using *B-H* static hysteresis graph. The density which was measured using densimeter, observed to be increased at higher sintering temperature with the maximum value of 6.01 g/cm^3 for sample sintered at 1400 ^oC. The resistivity value that was measured using picoammeter showed a maximum value for sample sintered at 1200 ^oC of 4.18×10^{10} Ω cm. The linewidth from microwave measurement that was carried out using Vector Network Analyzer

(VNA) in the frequency range of 4 to 8 GHz (C-band), showed the maximum value of 25.10 Oe which validates the suitability for low loss microwave material.

Therefore, the microstructure evolution of starting powder from nanosize grain would allow one to observe the critical change in transformation of phase and also the change in grain size that influenced the change in the properties studied. Samples with good crystallinity and large grain size would result in better properties.

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

KESAN SUHU PENSINTERAN KE ATAS SIFAT MIKROSTRUKTUR, ELEKTRIK DAN GELOMBANG MIKRO GADOLINIUM IRON GARNET

Oleh

FARAH NABILAH BINTI SHAFIEE

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Kesan pelbagai suhu pensinteran terhadap mikrostruktur GdIG belum dijalankan dalam penyelidikan terdahulu, yang dapat memberi maklumat mengenai pengetahuan asas tentang sifat magnet daripada saiz butiran nanometer kepada mikrometer rejim. Oleh itu, hubungan antara evolusi mikrostruktur daripada saiz butiran nano kepada mikron dengan sifat fizikal, magnet, elektrik dan gelombang mikro garnet besi gadolinium ($Gd_3Fe_5O_{12}$, GdIG) telah disiasat secara sistematik. Bahan mentah digilingkan menggunakan kaedah pengaloian mekanikal selama 3 jam, dibentuk menjadi toroid dan pelet dan disinter dari 600 ^oC hingga 1400 ^oC dengan kenaikan 100 ^oC.

Purata saiz zarah selepas penggilingan yang diukur dengan menggunakan Pengimbasan Penghantaran Elektron Mikroskop (STEM) adalah 36.9 nm. Fasa GdIG yang dikenal pasti menggunakan pembelauan sinaran X (XRD) mula membentuk fasa tunggal dengan struktur garnet kiub pada 1000 ^oC. Pada suhu ini, pemagnetan yang dikaji menggunakan magnetometer pengimbasan bergetar (VSM) telah mula menunjukkan peningkatan dengan nilai pemagnetan tertinggi 3.86 emu/g. Suhu Curie (Tc) yang diukur menggunakan meter LCR (Aruhan, Kapasiti, dan Rintangan) untuk GdIG ialah ~300 ^oC. Apabila suhu pensinteran meningkat, penghabluran bertambah baik dengan keamatan XRD tertinggi untuk sampel 1000 ^oC. Evolusi mikrostruktur yang diperhatikan menggunakan Mikroskop Elektronik Pengimbasan Pelepasan Medan (FESEM) telah mempengaruhi perubahan sifat-sifat magnet gelombang mikro dan elektrik. Untuk pengukuran magnet, nilai maksimum yang diperoleh melalui penganalisis impedans untuk kebolehtelapan adalah 1.33 dan nilai aruhan tertinggi 108.9 G diperoleh dengan menggunakan graf hysteresis statik *B-H*. Ketumpatan yang diukur menggunakan densimeter, diperhatikan meningkat pada suhu pensinteran yang lebih tinggi dengan nilai tetinggi sebanyak 6.01 g/cm^3 untuk sampel yang disinter pada 1400 ^oC. Nilai kerintangan yang diukur dengan menggunakan pikoammeter menunjukkan nilai maksimum untuk sampel yang disinter pada 1200 ^oC iaitu 4.18×10^{10} Ω cm. Pelebaran talian daripada pengukuran gelombang mikro yang dijalankan

menggunakan Penganalisa Rangkaian Vecktor (VNA) dalam julat frekuensi 4 hingga 8 GHz (jalur C), menunjukkan nilai maksimum iaitu 25.10 Oe, yang mengesahkan kesesuaian untuk bahan gelombang mikro dengan kehilangan yang rendah.

Oleh itu, evolusi mikrostruktur yang bermula daripada butiran nano saiz akan membolehkan perubahan kritikal dalam transformasi fasa dan juga saiz butiran yang mempengaruhi perubahan dalam sifat yang dikaji di mana sampel dengan penghabluran yang baik dan saiz butiran yang besar akan menghasilkan sifat yang lebih baik.

ACKNOWLEDGEMENTS

First and foremost, I would like to say my gratitude to the Almighty Allah s.w.t. Alhamdulillah, with His continuous blessings and guidance, I finally managed to finish off this tough yet interesting journey.

To the late (Allahyarham) Prof. Madya Dr. Mansor Hashim who was always the best educator that we could ever have. I would like to acknowledge this appreciation to him since it was a great honour to be able to be part of his magnetic team that I could learn a lot of new things albeit it was merely for a short time. May Allah bless him and place him in the best place.

I would also like to extent this appreciation to Dr. Raba'ah Syahidah Azis who was willing to accept and guide my friends and me throughout this research, especially with motivational advices. Next, to my co supervisor Dr. Ismayadi Ismail who has always encouraged us and shaped our minds to think critically. He always taught us on the theoretical part of magnetism which I find very fascinating once the rudimentary basis is well understood. Not to forget to my other co supervisor Dr. Zulkifly Abbas for his guidance and encouragement.

I could not have done this project without the support from my beloved parents, En. Shafie bin Harith and Puan Nor Baya binti Taha. I could manage to encounter the hardship and challenges from the research and keep moving forward with their wise advices and prayers.

I would like to thank my colleagues, Dr. Rodziah Nazlan that was willing to guide and teach me throughout the project, Dr Nor Hapishah Abdullah, Dr Idza Riati Ibrahim, Dr Fadzidah Mohd Idris, Dr Mohd Shamsul Ezzad Shafie and Dr Muhammad Syazwan Mustaffa who were always lending me their hands in guiding me and willing to share their knowledge, Dr. Wan Norailiana Wan Ab Rahman,, Dr Samikannu Kanagesan, Ms. Nur Fadillah Baharudin, Ms. Azdiya Suhada Abdul Rahim, Ms. Nur Izzaiti Ibrahim, Mrs. Siti Nor Ain Rusly, Dr. Low Zhi Huang, and Mr. Muhammad Misbah Zulkimi that helped me with the lab work and their ideas.

Last but not least, thanks to staffs of Institute of Advanced Technology and Faculty of Science of UPM and Faculty of Science of UKM, Mr. Mohd Kadri Masaud, Dr. Mohd Shamsul Ezzad Shafie, Mr Mohd Ali Rani, Mrs Sarinawani Abd Ghani, Mrs Rosnah Nawang, Mrs Roslina Abdul Rashid, Mrs Norhaslinda Noruddin, Mrs Kamsiah Alias and Mrs Hani for their great contribution to accomplish this research.

May Allah ease and bless all of us with His blessings.

I certify that a Thesis Examination Committee has met on 6 June 2018 to conduct the final examination of Farah Nabilah binti Shafiee on her thesis entitled "Effects of Sintering Temperature on Microstructural, Electrical and Microwave Properties of Gadolinium Iron Garnet" 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 of Science.

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LISTS OF ABBREVIATIONS AND SYMBOLS

$Gd_3Fe_5O_{12}$	Gadolinium iron garnet
GdIG	Gadolinium iron garnet
$GdFeO_3$	Gadolinium orthoferrite
wt.	Weight
T_c	Curie temperature
T_{comp}	Compensation temperature
B	Magnetic induction
M	Magnetization
H	Magnetic field
H_c	Coercivity
ρ_{DC}	Electrical resistivity
STEM	Scanning Transmission Electron Microscopy
XRD	X-ray Diffraction
FWHM	Full wave half maximum
FESEM	Field Emission Electron Microscopy
PVA	Polyvinyl alcohol
VSM	Vibrating Sample Magnetometer
ESR	Electron Spin Resonance
EDX	Energy Dispersive X-ray Spectroscopy
ρ	Density
ρ_{xrd}	X-ray density
ρ_{exp}	Experimental density
Q	Activation energy
REIG	Rare-earth iron garnet

VNA	Vector Network Analyzer

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Recently, there has been a growing interest in the development of new magnetic materials in the field of nanotechnology and their properties. This increases the efficiency of current technology and promotes progress in emerging fields. Magnetic materials possess remarkable properties and widely used in many applications such as transformers, inductors, circulators, mobile phones, electric motors and hybrid vehicles (Jiles, 1998; Winkler, 1981). Magnetic insulators or well known as ferrites had surfaced as new materials to replace the needs of minimizing the loss caused by generation of eddy current for their property of high resistive material (Spaldin, 2010).

The advent of higher frequency devices with low eddy current loss have caused the utilization of ferromagnets such as iron and alloy to be no longer effective as the materials that employ the standard techniques using lamination or iron powder core.

There are three types of ferrites; spinel, garnet and hexagonal where garnet has become the preference to be used in microwave devices. Garnet is a low loss material by virtue of no divalent ions in the crystal structure, leading to less possibility of divalent iron ion occurring (Fuller, 1987). The stability to sustain the low loss property at that range is also contributed by the valency of ions apart from the composition and the crystal structure. Spinel ferrites on the other hand consist of 2+ ion metals, having a high tendency to form the ferrous ion, Fe^{2+}, which would reduce the resistivity. The narrowest linewidth exhibited yet is by yttrium iron garnet (YIG) (Ozgur, 2009). This property is important for telecommunication applications. However, other materials of the same type such as gadolinium iron garnet should not be neglected as it has its own properties that should be investigated further.

Microstructure of polycrystalline ferrites consists of grains, grain boundaries, pores and phases. The microstructure results in various properties of the same material. The starting particle size in nanometer size with an average particle or grain size of less than 100 nm has shown different behavior compared to its bulk counterparts (Lassri, 2011). The phase formation of nanoparticle ferrites has successfully been formed at lower temperature than the micron size particles due to large surface, leading to high reactivity of the particles (Chen and He, 2001; Paesano et al; 2005, Zanatta et al., 2005; Tsagaroyannis et al., 1992). The assumption that pores and secondary phase formation were undesirable was eliminated by the fact that there are turning points to the two components as they could control the properties according to the desired applications. The microstructure influence can be more delved into by investigating the evolution of the microstructure where one material with a starting particle size is sintered at different temperatures with a constant increment. This would give rise to the evolution of the size of particle or grain which consequently would affect the properties of the material.

Therefore, the underlying science of the effect of microstructure towards the magnetic properties, electrical and microwave properties was further investigated. The evolution from nanometer to micrometer grain size accompanied by the evolution of the properties has been elucidated. This parallel occurrence is a phenomenon that should be scrutinized as it can be a great medium in providing massive information in describing the mechanism of what is occurring within the material that gives rise to such properties for each sintered sample. In this research, gadolinium iron garnet, $Gd_3Fe_5O_{12}$ (GdIG) was chosen as the material to be studied. Raw materials of gadolinium (III) oxide, Gd_2O_3, and iron oxide, α-Fe_2O_3, were mixed and milled using a high-energy ball milling machine in order to produce nanosize powder and subsequently sintered starting from a low temperature of 600 ^oC to 1400 ^oC. The physical, structural, electrical, magnetic and microwave properties were studied through several characterizations that will be explained further in Chapter 4.

1.2 Selection of materials

GdIG has been a promising candidate that commonly used in microwave devices besides YIG. YIG has the narrowest ferromagnetic resonance (FMR) linewidth yet. Narrow linewidth indicates a very low magnetic loss of a material. This is very important for antenna applications which would require a material that would transmit most of the signal from the source. GdIG is superior to YIG in terms of temperature stability. This has been proven from previous research to why yttrium ion, Y^{3+} in YIG is substituted with gadolinium ion, Gd^{3+} based on the composition of $Y_{3-x}Gd_xFe_5O_{12}$. The Curie temperature of ~273 ^oC and temperature stability of magnetization are increased where the saturation magnetization can be retained from 300 K to ~525 K with increasing concentration of Gd^{3+} (Ramesh et al., 2017). These properties would allow the characterization to be carried out at room temperature and above. Substituting Gd^{3+} ion into a material, for instance YIG would help in maintaining the resulting saturation magnetization of YIG in broader temperature range, ranging from room temperature to Curie temperature (Kuanr, 1997 & Lataifeh, 1996). GdIG has remarkable properties at low temperature due to the strong contribution from ferromagnetic Gd^{3+} ion to Fe^{3+} ions in *a* and *d* sites (Nguyet, 2009). However, temperature effect changes the behavior of ferromagnetic to paramagnetic Gd^{3+} which causes the phenomenon of zero magnetization at ~ 16 ^oC to exist. This temperature is called compensation temperature. This difference from the behavior shown by diamagnetic Y^{3+} has become an interest to how it affects the magnetic, electrical and microwave properties at all. Therefore, GdIG was chosen as the material in this evolution study.

1.3 Problem statement

Fundamental knowledge has been a while neglected since researchers at present time are extensively producing solely the final product of doped rare earth iron garnet due to the industrial demand. GdIG material in previous research was studied in a range of 800 to 1000 ^{0} C only (Opuchovic et al., 2017). The important information such as the crtical sintering temperature of structural transformation could be missed. In addition, the effect of various sintering temperatures towards microstructural of GdIG in wide range of temperature can contribute to rudimentary basis. The evolution of microstructure from nanometer range to micrometer which results from various sintering temperature (600 to 1400 ^{0} C) with a constant increment of 100 ^{0} C and the influence of microstructure evolution towards the magnetic, electrical and microwave properties had not also been investigated.

The problems above are the main drive to this research as those questions have not been studied widely. Despite focusing on the final product, this study would help comprehend the underlying physics changes that lie in the material through its structure and microstructure from nanometer regime to micrometer regime of grain size and the effect from the changes towards the magnetic, electrical and microwave properties for each sample of GdIG, from low, intermediate and high sintering temperature to be critically tracked. Therefore, the information obtained could be utilized to identify the sample with optimum properties that suit the requirements in any applications.

1.4 Objectives of the study

The primary goal of this research is to investigate the relationship between the evolution of microstructure and the magnetic and electrical of GdIG sintered from low to high sintering temperature. This study embarks on the following objectives:

i) To determine the effect of sintering temperatures on the microstructure evolution of GdIG.
ii) To investigate the parallel relationship of microstructure evolution on the physical, structural, electrical, magnetic and microwave properties of GdIG.

1.5 Limitation of study

This study is focused on the changes of behavior occurred that were studied through the electrical, magnetic and microwave properties along the structural, phase changes and microstructural evolution from nanometer to micrometer grain size of samples of GdIG at varying sintering temperature with a constant increment of 100 ^{o}C. All measurements were conducted at room temperature.

1.6 Outline of the thesis

This thesis begins with Chapter 1 that is consists of research background, selection of materials, problem statement and the objectives of this study. Chapter 2 provides the information on recent progress on GdIG and other rare-earth iron garnet, synthesis methods used to prepare ferrite samples, the effect of microstructure towards ferrite properties, and summary of previous research. From there, new approach of this study is briefed in the last part of this chapter. Chapter 3 presents the crystal structure of GdIG and magnetic theory related to this study. The mechanism of high-energy ball milling process and sintering are also included in this chapter. The method, instruments used and characterization that had been carried out are explained in Chapter 4. All the detailed discussion on the analyzed data from the measurement is elucidated in Chapter 5. Lastly, the conclusions and achievements from the study are disclosed in the Chapter 6 which is the final chapter of this thesis.

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