

UNIVERSITI PUTRA MALAYSIA

PARALLEL EVOLUTIONS OF MORPHOLOGY AND MAGNETIC PROPERTIES AND THEIR ATTENDANT RELATIONSHIPS IN POLYCRYSTALLINE YTTRIUM IRON GARNET

RODZIAH BINTI NAZLAN

FS 2012 44

PARALLEL EVOLUTIONS OF MORPHOLOGY AND MAGNETIC PROPERTIES AND THEIR ATTENDANT RELATIONSHIPS IN POLYCRYSTALLINE YTTRIUM IRON GARNET



By

RODZIAH BINTI NAZLAN

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

January 2012

DEDICATION

To my beloved family and friends..

The world is moving faster; We're on a changing course. But you have helped me deal with life; You've been a stable force.

When I have had to follow; New directions, you were there. When the world was hard on me; You always seemed to care.

When nothing held together; Made the slightest bit of sense. You have always helped restore; My inner confidence.

Everyone needs someone; Who's reliable and true. Through the moments I've endured; I'm grateful there was you.

Thanks For Always Being There..

Abstract of thesis presented to Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

PARALLEL EVOLUTIONS OF MORPHOLOGY AND MAGNETIC PROPERTIES AND THEIR ATTENDANT RELATIONSHIPS IN POLYCRYSTALLINE YTTRIUM IRON GARNET

By

RODZIAH BINTI NAZLAN

January 2012

Chairman : Associate Professor Mansor Hashim, PhD

Faculty : Science

The parallel microstructure and magnetic-property evolutions in several polycrystalline Yttrium Iron Garnet (YIG) samples as a result of a sintering scheme were studied in detail, focusing on the attendant occurrence of their relationships: an aspect seemingly neglected, hitherto, in the garnet literature for past seven decades.

Samples with nanometer sized starting powder were synthesized by employing the high-energy ball milling (HEBM) technique and then sintering toroidal compacts of the milled powder. Two batches of samples were produced for single-sample and multi-samples sintering, each covering a range of sintering temperatures from 600°C to 1400°C. The samples were characterized by transmission electron microscopy (TEM), X-ray Diffraction (XRD), scanning electron microscopy (SEM), hysteresisgraph, impedance/material analyzer and picoammeter for their evolution stage in crystalline phases, microstructure, magnetic hysteresis-loop parameters,

magnetic permeability components, Curie temperature and electrical resistivity respectively.

With great experimental care, both the single-sample and multi-samples sintering. batches yielded highly similar variation of magnetic properties versus microstructure of YIG. The results showed an increasing tendency of the saturation magnetization and saturation induction with grain size, which is attributed to crystallinity increase and to reduction of demagnetizing fields in the grains. The variation in coercivity could be related to anisotropy field changes within the samples due to grain size changes. In particular, the starting appearance of room temperature ferromagnetic order suggested by the sigmoid-shaped B-H loops seems to be dependent on a sufficient number of large enough magnetic domain-containing grains having been formed in the microstructure. Viewed simultaneously, the hysteresis loops appear to belong to three groups with different magnetism-type dominance, respectively dependent on phase purity and grain size distributions. A scrutiny of the permeability components, μ' and μ'' , shows that there also tend to similarly belong to the above three sintering temperature related groups. The Curie temperature remained relatively stable, unaffected by the above evolutions, thus confirming its intrinsic character being dependent only on the crystal structure and compositional stoichiometry. The increased electrical resistivity while the microstructure was evolving is believed to strongly indicate improved phase purity and compositional stoichiometry.

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

PENGEVOLUSIAN SELARI HUBUNGAN SEMASA ANTARA MORFOLOGI DAN SIFAT MAGNETIK DALAM POLIHABLUR YTTRIUM IRON GARNET

Oleh

RODZIAH BINTI NAZLAN

Januari 2012

Pengerusi

: Profesor Madya Mansor Hashim, PhD

Fakulti

: Sains

Pengevolusian selari mikrostruktur dan sifat magnet dalam sebilangan sampel polihablur Yttrium Iron Garnet (YIG) sebagai hasil skim pensinteran telah dikaji secara terperinci, dengan memfokuskan kepada kewujudan semasa hubungan mereka: suatu aspek yang sehingga kini diabaikan, di dalam literatur garnet sejak tujuh dekad yang lalu.

Sampel-sampel dengan serbuk permulaan bersaiz nanometer telah disintesis dengan menggunakan teknik pengisaran bola bertenaga tinggi (HEBM) dan seterusnya dengan mensinter kompak toroid serbuk yang telah dikisar. Dua kumpulan sampel telah dihasilkan iaitu pensinteran sampel tunggal dan pensinteran multi-sampel, setiap satunya merangkumi suhu pensinteran daripada 600°C ke 1400°C. Sampel-sampel tersebut telah dicirikan dengan menggunakan mikroskop elektron transmisi (TEM), pembelauan sinar-X (XRD), mikroskop elektron imbasan (SEM), hysteresisgraph, penganalisa impedans/bahan dan picoammeter masing-masing pada

peringkat evolusi mereka dalam fasa-fasa penghabluran, mikrostruktur, parameter magnetic gelung histeresis, komponen-komponen ketelapan magnetik, suhu Curie dan kerintangan elektrik.

Dengan pengawalan eksperimen yang baik, kedua-dua kumpulan sampel tunggal dan multi-sampel telah menghasilkan variasi yang hampir sama dalam sifat magnetik melawan mikrostruktur YIG. Hasil kajian menunjukkan kecenderungan peningkatan dalam kemagnetan tepu dan induksi tepu dengan saiz butiran, yang disebabkan oleh penghabuluran bertambah dan pengurangan medan nyahmagnet di dalam butiran. Kepelbagaian dalam daya paksa pula boleh dikaitkan dengan perubahan medan anisotropi di dalam sampel yang disebabkan oleh perubahan saiz butiran. Secara khususnya, permulaan kewujudan sifat ferromagnetik pada suhu bilik telah dicadangkan melalui gelung B-H berbentuk sigmoid yang kelihatan bergantung kepada bilangan mencukupi domain magnetik-mengandungi butiran yang cukup besar yang terbentuk di dalam mikrostruktur. Dilihat pada masa yang sama, gelung histeresis kelihatan tergolong kepada tiga kumpulan dengan dominasi jenis kemagnetan yang berbeza, masing-masing bergantung kepada ketulenan fasa dan taburan saiz butiran. Penelitian terhadap komponen-komponen ketelapan, μ ' dan μ '' menunjukkan bahawa terdapat juga kecenderungan untuk bersama tergolong dalam tiga kumpulan suhu pensinteran seperti di atas. Suhu Curie relatifnya kekal stabil, tidak terjejas oleh pengevolusian seperti di atas, sekaligus mengesahkan bahawa ciriciri intrinsik hanya bergantung kepada struktur hablur dan stoikiometri komposisi sahaja. Peningkatan kerintangan elektrik semasa mikrostruktur mengevolusi dipercayai menunjukkan ketulenan fasa dan stoikiometri komposisi menjadi lebih baik.

ACKNOWLEDGEMENT

My sincere gratitude goes to the Creator of the heavens and earths and what's in between; the Almighty Allah, glorified be He, then to His prophet, Nabi Muhammad S.A.W.

This dissertation would not have been possible without the supervision of my supervisor, Assoc. Prof. Dr. Mansor Hashim who was always readily available for discussion and guidance. I wish to express my deepest appreciation for his continued encouragement, guidance and support during this research and the preparation of this thesis. I would also like to extend my appreciation to my supervision committee member, Dr. Khamirul Amin Matori who was ever willing to give his help throughout this project.

Besides that, I gratefully acknowledge staff of Material Synthesis and Characterization Laboratory (MSCL), Institute of Advanced Technology and Material Physics Laboratory, Faculty of Science for allowing me to utilize their lab facilities. I would also like to thank Universiti Putra Malaysia for financial support by Fundamental Research Grant Scheme (FRGS; grant no. 5523649), Research University Grant Scheme (RUGS; grant no. 91553) and Graduate Research Fellowship Scheme.

Next, a lot of thanks to all my colleagues in the Magnetic and Nanostructure Evolution Group (MNEG), especially Idza Riati Ibrahim, Encik Ismayadi Ismail, Wan Norailiana Wan Abdul Rahman, Norhapishah Abdullah, Ghazaleh Bahmanrokh, Fadzidah Mohd Idris, Shamsul Ezzad Shafee and Masni Manap for their concerns, moral support and encouragement.

Finally, I am forever indebted to family: parents, brothers and sister for their sacrifice, patience, love, understanding, support and for their prayers during these years that I have spent in pursuit of my master's degree. Without their support, patience and encouragement this thesis would never have started much less finished. I thank them for their never ending unconditional love, for their support, and for everything.

I certify that an Examination Committee has met on 31 January 2012 to conduct the final examination of Rodziah binti Nazlan on her Master of Science thesis entitled "An Exposition of Attendant Relationship Between Parallel Evolving Morphology and Magnetic Properties in Polycrystalline Yttrium Iron Garnet (YIG)" 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 rewarded the Master of Science degree.

Member of the Thesis Examination Committee were as follows:

Jumiah binti Hassan, PhD

Associate Professor Faculty of Science Universiti Putra Malaysia (Chairman)

Lim Kean Pah, PhD

Faculty of Science Universiti Putra Malaysia (Internal Examiner)

Wan Mahmood bin Yunos, PhD

Professor Faculty of Science Universiti Putra Malaysia (Internal Examiner)

Abdul Kariem bin Aroff, PhD

Professor Faculty of Science Universiti Malaya (External Examiner)

> **SEOW HENG FONG, PhD** Professor and Deputy Dean School of Graduate Studies Universiti Putra Malaysia

Date:

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

Mansor bin Hashim, PhD Associate Professor Faculty of Science Universiti Putra Malaysia (Chairman)

Khamirul Amin bin Matori, PhD Lecturer Faculty of Science Universiti Putra Malaysia (Member)

BUJANG BIN KIM HUAT, PhD Professor and Dean School of Graduate Studies

School of Graduate Studies Universiti Putra Malaysia

Date:

DECLARATION

I declare that the thesis is my original work except for quotations and citations which have been duly acknowledge. I also declare that it has not been previously and is not concurrently, submitted for any other degree at Universiti Putra Malaysia or at any other institution.

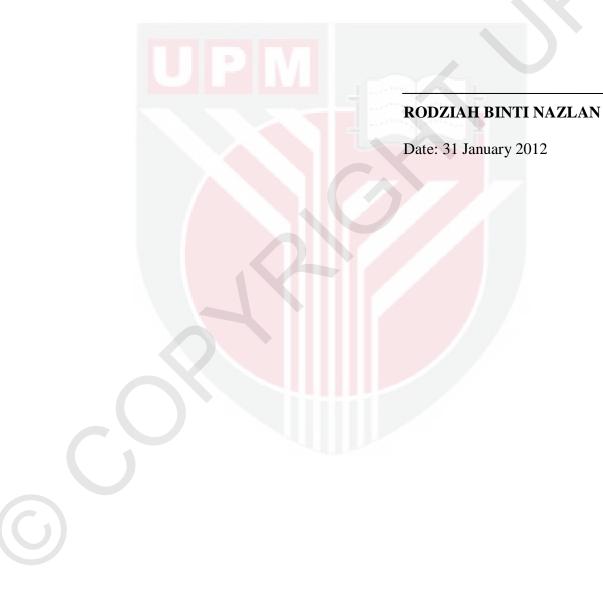


TABLE OF CONTENTS

Page **DEDICATION** ii ABSTRACT iii ABSTRAK v ACKNOWLEDGEMENT vii APPROVAL ix DECLARATION xi LIST OF TABLES xiii LIST OF FIGURES xv LIST OF ABBREVIATIONS xix

CHAPTER

1

2

3

INTRODUCTION	
1.1 General Introduction	1
1.2 Ferrites	2
1.3 Basis of Work and Statement of Objective	5
LITERATURE REVIEW	
2.1 Processing Technique of YIG	7
2.1.1 Oxide Mixture Route	8
2.1.2 Sol-Gel Route	9
2.1.3 Co-precipitation Route	10
2.1.4 High Energy Ball Milling	11
2.2 Chemical Aspects of Ferrites	12
2.3 Some Microstructural Aspects of Ferrites	16
	10

2.4 Some Electrical Properties of Ferrites	19
--	----

THEORY

3.1 The Origin of Magnetism	21
3.2 Types of Magnetism	22
3.3 The Ferrimagnetic Garnet Structure	26
3.4 Basic Magnetic Properties of Garnet	
3.4.1 Intrinsic properties	29
3.4.2 Extrinsic properties	35
3.5 Microstructure of Garnet	41
3.6 Sintering	42
3.7 Dependency of Coercivity on Particle Size	48

4 **METHODOLOGY** 4.0 General Introduction

4.1 Sample Preparation4.1.1Chemical formula, weighing and mixing of constituent powders

50

52

xi

4.1.2 High-energy ball milling (HEBM)	52
4.1.3 Moulding	54
4.1.4 Sintering	55
4.2 Samples' Measurement and Characterization	
4.2.1 Starting particle size confirmation	56
4.2.2 Multiphase determination	56
4.2.3 Microstructure analysis	58
4.2.4 Density measurement	60
4.2.5 Complex permeability measurement	61
4.2.6 B-H hysteresis parameters	62
4.2.7 Curie temperature	63
4.2.8 Electrical resistivity	64
4.3 Errors of Measurement	67

5

6

RESULTS AND DISCUSSION

5.0 General Introduction

5.1 N	Microstructural-related Analysis	
	5.1.1 Transmission Electron Microscope	
	(TEM)	69
	5.1.2 X-ray Diffraction (XRD)	71
	5.1.3 Effect of amorphous grain boundary	
	volumes in materials with nanometer	
	and micrometer grain size	76
	5.1.4 Morphological properties analysis	78
5.2 N	Magnetic Properties Measurement	
	5.2.1 Development of B-H Loop	96
	5.2.2 Hysteresis parameter	101
	5.2.3 Net magnetic moment	108
	5.2.4 Curie temperature	110
	5.2.5 Complex permeability	113
5.3 E	Electrical Properties Measurement	
	5.3.1 Electrical resistivity	119

68

CONCLUSION AND SUGGESTIONS

6.1 Conclusion	122
6.2 Suggestion	124

REFERENCES 126 **BIODATA OF THE STUDENT** 132 LIST OF PUBLICATIONS 133