UNIVERSITI PUTRA MALAYSIA

ELECTROMAGNETIC CHARACTERIZATION OF Sm-YIG AND Sm-YIG-PVDF COMPOSITES PREPARED USING MODIFIED CONVENTIONAL MIXING OXIDE TECHNIQUE

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By

RAMADAN MASOUD AL-HABASHI

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

September 2009
Appendixes
DEDICATION

This thesis is dedicated to my son, daughters, wife and parents; to whom their true love and support motivated my success.
Samarium substituted-yttrium iron garnet (Sm-YIG) nanoparticles were fabricated via a modified conventional mixing oxides (MCMO) method according to the $Y_{3-x}Sm_xFe_5O_{12}$ system ($0 \leq x \leq 3$). In this research, utilization of an organic compound (ethanol) and metal oxides in conjunction with mixing the reactants directly without adding water are the key techniques of this method. Using ethanol solution instead of water could produce nanoparticles with better homogeneity and smoother surface structure. Single-phase garnet structure of Sm-YIG nanoparticles was produced at $1350 \, ^{0}\text{C}$ sintering temperature with an average particle size ranged from 25 to 39 nm. XRD results of Sm-YIG samples at $x = 2$ and 2.5, presented some unknown peaks which speculated to, the time or/and sintering temperature is/are not enough to form the garnet structure phase of the samples. The true density values of 5.245 and 6.221 g.cm$^{-3}$ were calculated for pure yttrium iron garnet (YIG, $x=0$) and samarium iron garnet (SmIG, $x=3$) samples, respectively which reached around 99% of the theoretical density of the samples.
Real permittivities of the Sm-YIG samples presented almost flat values ranged from 7 to 10 with loss factors around 0.1 to 0.3, for YIG (x=0) and SmIG (x=3) respectively, within 10 MHz to 1 GHz frequency range. The real permeability value 19.5 is presented by pure YIG at 13.4 MHz and declined rapidly to be around 2 at 1 GHz, and decreased with increasing x. The higher permeability resulted in lower permittivity and vice versa for all the Sm-YIG samples.

This work was also carried out to prepare the 10 wt% Sm-YIG in Poly-vinylidene-fluride (PVDF) composite samples and study their electromagnetic properties. Sm-YIG samples prepared via MCMO method, PVDF powder and Ethyl-methyl-ketone (MEK) were used to prepare such composites. High permittivities of composite samples observed at lower frequency range indicated to the heterogeneous conduction in the multiphase structure of the composites. The real permeabilities presented almost flat values through all the range of the frequency from 10 MHz to 1 GHz, with value of 1.06 at x=0 and 1.13 at x=3, for 10 wt% Sm-YIG loading in the composites. MCMO technique appears to be another alternative to the conventional (manufactured) technique, due to the decreasing of the particle size with better homogeneity, high purity, reduction of the cost, and high yield in a nano-scale product compared to other preparations techniques.

The numerical optimization method performed using MATLAB program is to estimate the effective complex permittivity and/or permeability of each component of the 10 wt% Sm-YIG-PVDF composite samples. It is found that, the optimum impedance values are very close to the measured ones for each composite. The optimized values of the complex permittivities and permeabilities for both
components [Sm-YIG and PVDF] are within the estimated ranges. The optimization process eliminated the difference between the measured impedance and the calculated one from Maxwell-Garnett (MG) formula via a specific objective function.

Results of a developed formula based on MG formula with a comparison of various theoretical models including the MG, Looyenga, Bruggeman and Sen-Scala-Cohen, have been carried out and discussed with comparisons to the measurements for the 10 wt% Sm-YIG-PVDF composite samples. This was to calculate the complex permittivity and permeability of such composite materials. The lowest mean error percentage values were detected from the developed MG formula for each composite, which was different from composite to composite depend on the mole fraction x. The developed MG model appears to add a new contribution to the theoretical models to calculate the effective permittivity and permeability of mixture ferrite-polymer materials, due to its accuracy as compared with others.
Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENCIRIAN ELEKTROMAGNETIK KOMPOSIT Sm-YIG DAN Sm-YIG-PVDF MENGGUNAKAN TEKNIK DIMODIFIKASI KONVENSIONAL CAMPURAN OKSIDA

Oleh

RAMADAN MASOUD AL-HABASHI

September 2009

Pengerusi: Zulkifly Bin Abbas, PhD
Institut: Teknologi Maju

Zarah nano Samarium-YIG telah difabrikasikan melalui kaedah pengubahsuaian konvensional pencampuran oksida (MCMO) berdasarkan kepada sistem $Y_{3-x}Sm_xFe_5O_{12}$ ($0 \leq x \leq 3$). Dalam kajian ini, penggunaan sebatian organik (etanol) dan oksida logam bersama dengan campuran reaktan secara terus tanpa penambahan air adalah kunci teknik untuk kaedah ini. Menggunakan larutan etanol menggantikan air boleh menghasilkan zarah nano dengan keseragaman yang lebih baik dan struktur permukaan yang lebih halus. Zarah nano Sm-YIG berstruktur garnet fasa tunggal telah dihasilkan pada suhu pemanasan 1350 °C dengan purata saiz zarah berjulat antara 25 ke 39 nm. Keputusan XRD bagi sampel Sm-YIG pada $x=2$ dan 2.5 menunjukkan beberapa puncak yang tak diketahui yang menggambarkan masa atau/suha pemanasan adalah tidak mencukupi untuk membentuk struktur fasa garnet pada sampel. Nilai ketumpatan sebenar adalah 5.245 dan 6.221 g.cm$^{-3}$ telah dikira untuk yttrium iron garnet (YIG, $x=0$) tulen dan samarium iron garnet (SmIG, $x=3$) masing-masing dimana mencapai 99% ketumpatan sampel secara teori.
Ketelusan sebenar bagi sampel Sm-YIG menunjukkan adalah kebanyakan nilai rata berjulat daripada 7 ke 10 dengan faktor kehilangan sekitar 0.1 ke 0.3 untuk YIG (x=0) dan SmIG (x=3) masing-masing dalam julat frekuensi 10 MHz ke 1 GHz. Nilai sebenar ketelapan 19.5 didapati dari YIG tulen pada 13.4 MHz dan berkurang dengan cepat sekitar 2 pada 1GHz dan berkurang dengan peningkatan jumlah x. Semakin tinggi nilai ketelapan menyebabkan semakin rendah nilai ketelusan dan sebaliknya untuk semua sampel Sm-YIG.

Kerja ini juga telah dilaksanakan untuk menyediakan 10 wt% Sm-YIG dalam sampel komposit Poly-vinylidene-fluride (PVDF) dan mengkaji sifat-sifat elektromagnetiknya. Sm-YIG sampel disediakan melalui kaedah MCMO, Serbuk PVDF dan Ethyl-methyl-ketone (MEK) telah digunakan untuk menyediakan komposit seperti ini. Ketelusan tinggi bagi sampel komposit telah diperhatikan pada julat frekuensi rendah menunjukkan kepada konduksi heterogen di dalam struktur multifasa komposit. Ketelapan sebenar menunjukkan kebanyakan nilai rata sepanjang kesemua julat untuk frequency dari 10 MHz hingga 1 GHz, dengan nilai 1.06 pada x=0 dan 1.13 pada x=3, bagi 10% Sm-YIG di dalam komposit. Ini menunjukkan percampuran baik campuran homogen komposit ferrite-polimer. Teknik MCMO juga adalah sebagai alternative lain bagi teknik konvensional (penghasilan), kerana dapatmenghasilkan pengurangan saiz zarah dengan homogen yang baik, ketulenan yang tinggi, pengurangan kos, dan hasil yang tinggi dalam produk skala-nano berbanding dengan teknik penghasilan yang lain.

Teknik pengoptimuman berangka telah dilakukan menggunakan program MATLAB. Ini adalah untuk menganggarkan ketelusan dan/atau ketelapan kompleks berkesan.
bagi setiap komponen sampel komposit 10 wt% Sm-YIG di dalam PVDF. Didapati bahawa, nilai impedan optimum adalah sangat hampir dengan nilai yang diukur bagi setiap komposit. Nilai optimum bagi ketelusan dan ketelapan kompleks pada kedua-dua komponen [Sm-YIG dan PVDF] adalah didalam julat anggaran. Proses pengoptimuman telah menghapuskan perbezaan di antara impedan yang telah diukur dan yang dikira daripada formula Maxwell-Garnett (MG) dengan membezakan antara pelbagai model teori termasuk MG, Looyenga, Bruggeman dan Sen-Scala-Cohen, telah dijalankan dan dibincangkan dengan membezakan nilai pengukuran pada 10% Sm-YIG di dalam komposit sampel PVDF. Ini adalah untuk mengira ketelusan dan ketelapan kompleks bagi bahan komposit itu. Bagi setiap komposit, nilai purata ralat yang terendah telah didapati daripada formula MG yang telah dibangunkan, yang mana ini adalah berbeza daripada kebergantungan komposit kepada komposit pada pecahan mol x. Formula model MG yang telah dibangunakan dapat memberikan sumbangan baru kepada model teori untuk mengira ketelusan dan ketelapan efektif bagi bahan campuran polimer ferrite, berdasarkan kepada ketepatannya berbanding dengan yang lain.
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I certify that a Thesis Examination Committee has met on 17 September 2009 to conduct the final examination of Ramadan Masoud Al-Habashi on his thesis entitled “Electromagnetic Characterization of Sm-YIG and Sm-YIG-PVDF Composites Prepared Using Modified Conventional Mixing Oxide Technique” 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 Doctor of Philosophy.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been
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Date: 16 November 2009
DECLARATION

I hereby declare that the thesis is my original work except for quotations and citations which have been duly acknowledged. 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.

RAMADAN MASOUD AL-HABASHI

Date:
**TABLE OF CONTENTS**

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>DEDICATION</td>
<td>ii</td>
</tr>
<tr>
<td>ABSTRACT</td>
<td>iii</td>
</tr>
<tr>
<td>ABSTRAK</td>
<td>vi</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
<td>ix</td>
</tr>
<tr>
<td>APPROVAL</td>
<td>xi</td>
</tr>
<tr>
<td>DECLARATION</td>
<td>xiii</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>xvi</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>xix</td>
</tr>
<tr>
<td>LIST OF ABBREVIATIONS</td>
<td>xxiv</td>
</tr>
</tbody>
</table>

**CHAPTER**

1 INTRODUCTION

1.1 General 1
1.2 Nanomaterials and Nanostructures 2
1.3 Polymer Composite of Magnetic Particles 3
1.4 Preparation Techniques of Ferrite Material 4
1.5 Permittivity and Permeability Concepts 5
1.6 Soft Ferrites 6
1.7 Problem Statement 7
1.8 Aims/Objectives 8
1.9 Significant of This Study 9
1.11 Thesis Layout 9

2 LITERATURE REVIEW

2.1 Introduction 11
2.2 Preparation and Characterization of YIG Samples 11
2.3 Ferrite-Polymer composite 27
2.4 Optimization and Numerical models 30

3 THEORY

3.1 Introduction 35
3.2 Classification of Magnetic Materials 35
3.3 Garnet Structure 38
3.4 Bragg’s Law and Lattice Parameters 40
3.5 Sintering and Grain Growth 43
3.5.1 Solid-state sintering 44
3.5.2 Sintering stages 45
3.5.3 Grain growth 47
3.5.4 Factors affecting solid-state sintering 47
3.6 Permittivity and Permeability Study 49
3.6.1 Parallel-plate capacitor measurement method 49
3.6.2 Induction measurement method 52
3.6.3 Dielectric properties of materials 55

xiv
3.6.4 Magnetic Properties of materials

4 METHODOLOGY

4.1 Introduction

4.2 MCMO Technique for Sm-YIG Samples Preparation

4.3 Sm-YIG-PVDF Composite Samples Preparation

4.4 Materials Characterizations

4.4.1 Shrinkage

4.4.2 XRD for structure and phase analysis

4.4.3 FESEM, EDX and TEM for microstructure observations

4.4.4 RF-Impedance/material analyzer

4.5 Numerical Optimization and Theoretical Models Study

4.5.1 Optimization method

4.5.2 Developed Maxwell-Garnett formula

5 RESULTS AND DISCUSSIONS

5.1 Introduction

5.2 Structure Characterization of Sm-YIG Samples

5.2.1 XRD profiles

5.2.2 Lattice constant

5.2.3 Density

5.2.4 Crystallite size

5.3 Shrinkage of Sm-YIG Samples

5.4 Microstructure Observations of Sm-YIG Samples

5.4.1 FESEM and EDX results

5.4.2 TEM micrographs

5.5 RF-Impedance/Material Analyzer Results

5.5.1 PTFE permittivity results

5.5.2 Permittivity and permeability of PVDF sample

5.5.3 Permittivity and permeability of Sm-YIG samples

5.5.4 Permittivity and permeability of Sm-YIG-PVDF

5.6 Optimization Results of Sm-YIG-PVDF Samples

5.6.1 Estimated permittivity results

5.6.2 Estimated permeability results

5.7 Developed MG Formula and Different Models Results

6 SUMMARY, CONTRIBUTIONS AND SUGGESTIONS

6.1 Summary of the Study

6.2 Main Contributions

6.3 Suggestions for Future Study

REFERENCES
### List of Tables

<table>
<thead>
<tr>
<th>Table</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.1</td>
<td>Amounts of the raw materials used to prepare Sm-YIG samples.</td>
</tr>
<tr>
<td>4.2</td>
<td>Amount of the raw materials used to prepare 10 wt% Sm-YIG in PVDF composite samples.</td>
</tr>
<tr>
<td>5.1</td>
<td>Intensity and 2Theta for Sm-YIG samples calcined at 1350 °C.</td>
</tr>
<tr>
<td>5.2</td>
<td>Lattice constant 'a' for Sm-YIG samples calcined at 1350 °C.</td>
</tr>
<tr>
<td>5.3</td>
<td>True density of Sm-YIG samples sintered at 1350 °C.</td>
</tr>
<tr>
<td>5.4</td>
<td>Crystallite size of Sm-YIG samples sintered at 1350 °C.</td>
</tr>
<tr>
<td>5.5</td>
<td>Percentage shrinkages values of Sm-YIG samples.</td>
</tr>
<tr>
<td>5.6</td>
<td>Quantitative analysis of Y3 sample sintered at 1350 °C in air.</td>
</tr>
<tr>
<td>5.7</td>
<td>Quantitative analysis of Y2.5 sample sintered at 1350 °C in air.</td>
</tr>
<tr>
<td>5.8</td>
<td>Quantitative analysis of Y2 sample sintered at 1350 °C in air.</td>
</tr>
<tr>
<td>5.9</td>
<td>Quantitative analysis of Y1.5 sample sintered at 1350 °C in air.</td>
</tr>
<tr>
<td>5.10</td>
<td>Quantitative analysis of Y1 sample sintered at 1350 °C in air.</td>
</tr>
<tr>
<td>5.11</td>
<td>Quantitative analysis of Y0.5 sample sintered at 1350 °C in air.</td>
</tr>
<tr>
<td>5.12</td>
<td>Quantitative analysis of Y0 sample sintered at 1350 °C in air.</td>
</tr>
<tr>
<td>5.13</td>
<td>Estimated and optimized values of relative permittivity and, objective function of 10wt% Y3 in PVDF composite.</td>
</tr>
<tr>
<td>5.14</td>
<td>Estimated and optimized values of relative permittivity and, objective function of 10wt% Y2.5 in PVDF composite.</td>
</tr>
<tr>
<td>5.15</td>
<td>Estimated and optimized values of relative permittivity and, objective function of 10wt% Y2 in PVDF composite.</td>
</tr>
<tr>
<td>5.16</td>
<td>Estimated and optimized values of relative permittivity and, objective function of 10wt% Y1.5 in PVDF composite.</td>
</tr>
<tr>
<td>5.17</td>
<td>Estimated and optimized values of relative permittivity and, objective function of 10wt% Y1 in PVDF composite.</td>
</tr>
</tbody>
</table>
5.18 Estimated and optimized values of relative permittivity and, objective function of 10wt% Y0.5 in PVDF composite.

5.19 Estimated and optimized values of relative permittivity and, objective function of 10wt% Y0 in PVDF composite.

5.20 Estimated and optimized values of relative permeability and, objective function of 10wt% Y3 in PVDF composite.

5.21 Estimated and optimized values of relative permeability and, objective function of 10wt% Y2.5 in PVDF composite.

5.22 Estimated and optimized values of relative permeability and, objective function of 10wt% Y2 in PVDF composite.

5.23 Estimated and optimized values of relative permeability and, objective function of 10wt% Y1.5 in PVDF composite.

5.24 Estimated and optimized values of relative permeability and, objective function of 10wt% Y1 in PVDF composite.

5.25 Estimated and optimized values of relative permeability and, objective function of 10wt% Y0.5 in PVDF composite.

5.26 Estimated and optimized values of relative permeability and, objective function of 10wt% Y0 in PVDF composite.

5.27 Impedance error percentage calculated via various permittivity models and, mean relative permittivity of 10wt% Y3 in PVDF composite.

5.28 Impedance error percentage calculated via various permittivity models and, mean relative permittivity of 10wt% Y2.5 in PVDF composite.

5.29 Impedance error percentage calculated via various permittivity models and, mean relative permittivity of 10wt% Y2 in PVDF composite.

5.30 Impedance error percentage calculated via various permittivity models and, mean relative permittivity of 10wt% Y1.5 in PVDF composite.

5.31 Impedance error percentage calculated via various permittivity models and, mean relative permittivity of 10wt% Y1 in PVDF composite.

5.32 Impedance error percentage calculated via various permittivity models and, mean relative permittivity of 10wt% Y0.5 in PVDF composite.

5.33 Impedance error percentage calculated via various permittivity models and, mean relative permittivity of 10wt% Y0 in PVDF composite.
5.34 Impedance error percentage calculated via various permeability models and, mean relative permeability of 10wt% Y3 in PVDF composite. 138
5.35 Impedance error percentage calculated via various permeability models and, mean relative permeability of 10wt% Y2.5 in PVDF composite. 139
5.36 Impedance error percentage calculated via various permeability models and, mean relative permeability of 10wt% Y2 in PVDF composite. 139
5.37 Impedance error percentage calculated via various permeability models and, mean relative permeability of 10wt% Y1.5 in PVDF composite. 140
5.38 Impedance error percentage calculated via various permeability models and, mean relative permeability of 10wt% Y1 in PVDF composite. 141
5.39 Impedance error percentage calculated via various permeability models and, mean relative permeability of 10wt% Y0.5 in PVDF composite. 141
5.40 Impedance error percentage calculated via various permeability models and, mean relative permeability of 10wt% Y0 in PVDF composite. 142
### LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.1</td>
<td>(a) Random orientation of atomic magnetic moments in an unmagnetized substance. (b) When an external field B is applied, the atomic magnetic moments tend to align with the field.</td>
<td>36</td>
</tr>
<tr>
<td>3.2</td>
<td>Different magnetic behaviours.</td>
<td>37</td>
</tr>
<tr>
<td>3.3</td>
<td>Structural units and positions of cations in 'a', 'c' and 'd' sites of YIG structure (Barsoum, 1997).</td>
<td>38</td>
</tr>
<tr>
<td>3.4</td>
<td>(a) Two dimensional lattice showing the set of planes with the Miller indices (1, 1). (b) Three dimensional unit cell showing the plane (1 1 1).</td>
<td>40</td>
</tr>
<tr>
<td>3.5</td>
<td>(a) Scattering of waves by crystal planes. (b) and (c) both have the same lattice type, but quite different unit cells and crystal structures (Barsoum, 1997).</td>
<td>41</td>
</tr>
<tr>
<td>3.6</td>
<td>(a) liquid-phase sintering; (b) solid-state sintering (Barsoum, 1997).</td>
<td>44</td>
</tr>
<tr>
<td>3.7</td>
<td>Schematic of two possible paths by which a collection of particles can lower its energy, (a) Densification followed by grain growth. (b) Coarsening where the large grains grow at the expense of the smaller ones (Barsoum, 1997).</td>
<td>45</td>
</tr>
<tr>
<td>3.8</td>
<td>(a) Initial stage of sintering model represented by spheres in tangential contact, (b) Near end of initial stage; spheres have begun to coalesce, (c) Intermediate stage; grains adopted shape of dodecahedra, enclosing pore channels at grain edges, (d) Final stage; pores are tetrahedral inclusions at corners where four dodecahedra meet (Barsoum, 1997).</td>
<td>46</td>
</tr>
<tr>
<td>3.9</td>
<td>Schematic of parallel plate electrodes structure (Agilent, 1999).</td>
<td>49</td>
</tr>
<tr>
<td>3.10</td>
<td>Material has some loss for permittivity measurement.</td>
<td>50</td>
</tr>
<tr>
<td>3.11</td>
<td>Edge effect (Agilent, 1999).</td>
<td>51</td>
</tr>
<tr>
<td>3.12</td>
<td>Basic relationship of magnetic flux density, magnetic flux, and current (Agilent, 1999).</td>
<td>53</td>
</tr>
<tr>
<td>3.13</td>
<td>Schematic structure of 16454A fixture (Agilent, 1999).</td>
<td>54</td>
</tr>
<tr>
<td>3.14</td>
<td>Material has loss for permeability measurement (Agilent, 1999).</td>
<td>55</td>
</tr>
<tr>
<td>3.15</td>
<td>Frequency dependence of permittivity for a hypothetical dielectric material.</td>
<td>56</td>
</tr>
</tbody>
</table>
3.16 Frequency dependence of permeability for hypothetical magnetic material.  
4.1 Preparation via MCMO technique and characterization of Sm-YIG samples and 10 wt% Sm-YIG in PVDF composite samples flow chart.  
4.2 Heating and cooling rate during the pre-sintering process.  
4.3 Dimensional measurement of toroidal sample.  
4.4 Structure of the (a) 16453A fixture & (b) 16454A fixture (Agilent, 1999).  
4.5 Applicable MUT size of the (a) 16453A & (b) 16454A (Agilent, 1999).  
4.6 The structure chart of the optimization MATLAB program.  
5.1 XRD profile of Y3 sample calcined at different temperatures.  
5.2 XRD profile of Y2.5 sample calcined at different temperatures.  
5.3 XRD profile of Y2 sample calcined at different temperatures.  
5.4 XRD profile of Y1.5 sample calcined at different temperatures.  
5.5 XRD profile of Y1 sample calcined at different temperatures.  
5.6 XRD profile of Y0.5 sample calcined at different temperatures.  
5.7 XRD profile of Y0 sample calcined at different temperatures.  
5.8 XRD profile of Sm-YIG samples calcined at 1350 °C.  
5.9 Lattice constant vs mole fraction for Sm-YIG samples sintered at 1350 °C.  
5.10 True density vs mole fraction for Sm-YIG samples sintered at 1350 °C.  
5.11 Crystallite size vs mole fraction for Sm-YIG samples sintered at 1350 °C.  
5.12 Percentage shrinkage vs mole fraction for Sm-YIG samples sintered at 1350 °C.  
5.13 FESEM micrograph of Y3 sample sintered at 1350 °C in air.  
5.14 EDX spectrum of Y3 sample sintered at 1350 °C in air.  
5.15 FESEM micrograph of Y2.5 sample sintered at 1350 °C in air.  
5.16 EDX spectrum of Y2.5 sample sintered at 1350 °C in air.  
5.17 FESEM micrograph of Y2 sample sintered at 1350 °C in air.  
5.18 EDX spectrum of Y2 sample sintered at 1350 °C in air.  

xx
5.19 FESEM micrograph of Y1.5 sample sintered at 1350 °C in air. 95
5.20 EDX spectrum of Y1.5 sample sintered at 1350 °C in air. 95
5.21 FESEM micrograph of Y1 sample sintered at 1350 °C in air. 96
5.22 EDX spectrum of Y1 sample sintered at 1350 °C in air. 96
5.23 FESEM micrograph of Y0.5 sample sintered at 1350 °C in air. 97
5.24 EDX spectrum of Y0.5 sample sintered at 1350 °C in air. 97
5.25 FESEM micrograph of Y0 sample sintered at 1350 °C in air. 98
5.26 EDX spectrum of Y0 sample sintered at 1350 °C in air. 98
5.27 TEM images of nanosized pure YIG (Y3) powders sintered at 1350 °C in air. 100
5.28 TEM images of nanosized pure SmIG (Y0) powders sintered at 1350 °C in air. 100
5.29 Measured impedance of PTFE standard sample. 101
5.30 Relative permittivity of PTFE standard sample. 102
5.31 Measured impedance of PVDF sample for permittivity calculation. 103
5.32 Measured relative permittivity of PVDF sample. 103
5.33 Measured impedance of PVDF sample for permeability calculation. 104
5.34 Measured relative permeability of PVDF sample. 105
5.35 Measured impedance of Sm-YIG samples for permittivity calculation. 107
5.36 Relative real permittivity vs frequency of Sm-YIG samples. 107
5.37 Loss factor permittivity vs frequency of Sm-YIG samples. 108
5.38 Measured impedance of Sm-YIG samples for permeability calculation. 111
5.39 Relative real permeability vs frequency of Sm-YIG samples. 111
5.40 Loss factor permeability vs frequency of Sm-YIG samples. 112
5.41 Measured impedance of 10wt% Sm-YIG in PVDF composites for permittivity calculation. 114
5.42 Real permittivity vs frequency 10wt% Sm-YIG in PVDF composite. 114
5.43 Loss factor vs frequency of 10wt% Sm-YIG in PVDF composite. 114
5.44 Measured impedance of 10wt% Sm-YIG in PVDF composites for...
permeability calculation.

5.45  Real permeability vs frequency of 10wt% Sm-YIG in PVDF composite.

5.46  Loss factor vs frequency of 10wt% Sm-YIG in PVDF composite.

5.47  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y3 in PVDF composite.

5.48  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y2.5 in PVDF composite.

5.49  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y2 in PVDF composite.

5.50  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y1.5 in PVDF composite.

5.51  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y1 in PVDF composite.

5.52  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y0.5 in PVDF composite.

5.53  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y0 in PVDF composite.

5.54  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y3 in PVDF composite.

5.55  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y2.5 in PVDF composite.

5.56  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y2 in PVDF composite.

5.57  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y1.5 in PVDF composite.

5.58  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y1 in PVDF composite.

5.59  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y0.5 in PVDF composite.

5.60  Optimized (optimum values) MG, calculated MG and measured impedance vs frequency of 10wt% Y0 in PVDF composite.

5.61  Various impedance results calculated via different permittivity models vs frequency of 10wt% Y3 in PVDF composite.