MICROSTRUCTURAL AND MAGNETIC PROPERTIES OF YTTRIUM IRON GARNET DERIVED FROM STEEL WASTE PRODUCT

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By

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Chairman: **Raba'ah Syahidah Azis, PhD**
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In this research work, the morphology and magnetic properties-evolutions in Yttrium Iron Garnet (YIG) were studied in details, focusing on parallel evolving relationship with their dependences on sintering temperature. The iron oxide that has been used to synthesize YIG was obtained from the steel waste product, mill scale. There is less study and no reports published regarding the evolution of microstructure and magnetic properties YIG from low sintering temperature to high sintering temperature by mill scale waste product. The raw mill scale went through the milling and purification process by magnetic and non-magnetic separation and curie temperature separation technique to produce high purity iron oxide powder as main raw material in preparing YIG and fabricate YIG by high energy ball milling process. The obtain iron oxide was characterized by X-ray Diffractometer (XRD), X-ray fluorescence (XRF), X-ray photoelectron spectroscopy (XPS), Fourier transform infrared spectroscopy (FTIR) and Raman spectroscopy for mineralogical composition and chemical analysis. The results of XRD indicated that the synthesized iron oxide identified as a α-Fe_2O_3 (hematite). Moreover, XRF, XPS, FTIR and Raman data was found to correspond with pure hematite.

Among the characterization procedures, the highlight properties were phase identification by XRD, microstructure by FESEM, magnetic permeability by impedance material analyzer and saturation magnetization by VSM. The XRD pattern of YIG showed an improvement of crystallinity with increasing sintering temperature. Full YIG phase was seen for samples sintered at 1100^{o}C and upwards. The temperature obtain of YIG fabricate is comparable with high energy milling method by commercial iron oxide raw material. FESEM micrographs showed larger grains as the sintering temperature increased, and the amounts of porosity were decreased, as the some grains grew at the expense of others. The initial

permeability, μ_i showed the highest value of 51.70^o at 1400^{o}C, and decrement values of initial permeability at 700^{o}C to 800^{o}C because of the presence of weak ferromagnetic phases. The results of VSM showed an increasing tendency of saturation magnetization, M_s with increased grain sizes, and the decrement value for samples sintering at 700^{o}C to 800^{o}C. These results can be associated with the formation of weak ferromagnetic behavior of α-Fe_2O_3 and $YFeO_3$ phases. A particular pattern of the magnetic properties with sintering temperature is a manifestation of the phase purity level and microstructural factors. Thus, three groups of ferromagnetic behavior of YIG can be classified based on those factors.

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

MIKROSTRUKTUR DAN SIFAT-SIFAT MAGNET DALAM YTTRIUM IRON GARNET YANG DIPEROLEHI DARIPADA BAHAN BUANGAN BESI

Oleh

NURAINE MARIANA BINTI MOHD SHAHRANI

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Dalam kerja-kerja penyelidikan ini, morfologi dan sifat-sifat magnet-evolusi dalam Yttrium Iron Garnet (YIG) telah dikaji secara terperinci, memberi tumpuan kepada hubungan perkembangan selari mereka dengan suhu pensinteran. Oksida besi yang telah digunakan untuk mensintesis YIG diperoleh daripada bahan buangan keluli, di kilang. Terdapat kurang kajian dan tiada laporan bercetak mengenai evolusi mikrostruktur dan sifat-sifat magnet YIG dari suhu pensinteran rendah kepada suhu pembakaran tinggi oleh bahan buangan besi dari kilang. Sisik besi mentah telah melalui proses pengilangan dan pemurnian oleh pemisahan magnet dan bukan magnet dan teknik pemisahan suhu curie untuk menghasilkan serbuk oksida besi berketulenan tinggi sebagai bahan mentah utama dalam penyediaan YIG dan pembuatan YIG oleh proses pengilangan bola tenaga tinggi. Penghasilan oksida besi telah dicirikan oleh pembelauan sinar-X (XRD), pendarfluor sinar-X (XRF), spektroskopi fotoelektron sinar-X (XPS), spektroskopi inframerah transform Fourier (FTIR) dan spektroskopi Raman untuk komposisi mineralogi dan analisis kimia. Keputusan XRD menunjukkan bahawa oksida besi yang disintesis dikenal pasti sebagai α-Fe_2O_3 (bijih besi). Selain itu, data XRF, XPS, FTIR dan Raman telah didapati sepadan dengan bijih besi tulen.

Di antara prosedur pencirian, sifat-sifat yang diketengahkan adalah pengenalan fasa oleh XRD, mikrostruktur oleh FESEM, kebolehtelapan magnet oleh penganalisa bahan galangan dan pemagnetan tepu oleh VSM. Pola XRD daripada YIG menunjukkan peningkatan penghabluran dengan peningkatan suhu pensinteran. Fasa YIG penuh dapat dilihat pada sampel sinter pada 1100^{o}C dan ke atas. Suhu ini adalah setanding dengan kaedah pengilangan bola tenaga tinggi daripada bahan mentah oksida besi yang komersial. Mikrograf FESEM

menunjukkan bijirin lebih besar kerana suhu pembakaran meningkat, dan jumlah keliangan telah menurun disebabkan oleh beberapa bijirin berkembang sesama sendiri. Kebolehtelapan awal, μ_i menunjukkan nilai tertinggi 51.70^o pada suhu 1400^{o}C, dan kebolehtelapan awal susut pada 700^{o}C dan 800^{o}C kerana kehadiran fasa lemah feromagnet. Keputusan VSM menunjukkan pemagnetan tepu, M_s yang semakin meningkat dengan saiz bijirin meningkat, dan nilai susut pada sampel pensinteran 700^{o}C dan 800^{o}C. Kesusutan pada suhu ini boleh dikaitkan dengan pembentukan tingkah laku fasa lemah feromagnet α-Fe_2O_3 dan $YFeO_3$. Corak tertentu sifat-sifat magnet dengan pensinteran suhu adalah manifestasi daripada kesucian tahap fasa dan faktor-faktor mikrostruktur. Maka, tiga kumpulan tingkah laku feromagnet daripada YIG boleh dikelaskan berdasarkan faktor-faktor tersebut.

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

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS AND ABBREVIATIONS

Ni	Nickel
Mn	Manganese
Zn	Zinc
Ba	Barium
Sr	Strontium
Fe_2O_3	Iron (III) oxide; hematite
Y_2O_3	Yttrium oxide
$Y_3Fe_5O_{12}$; YIG	Yttrium iron garnet
Y	Yttrium
FeO	Iron (II) oxide
Fe_3O_4	Iron (II,III) oxide
Fe	Iron
CTS	Curie temperature separation
MNM	Magnetic and non-magnetic separation
XRF	X-ray fluorescence
XRD	X-ray diffraction
Cu	Copper
Cr	Chromium
$YFeO_3$; YIP	Orthoferrite
FMR	Ferromagnetic resonance
ΔH	Linewidth
Fe^{3+}	Iron (III) ion; ferric ion
Fe^{2+}	Iron (II) ion; ferrous ion
H_c	Coercivity
M_r	Remanence
M	Magnetization
T_c	Curie temperature
M_s	Saturation magnetization
H	Applied field
P	Resistivity
HEBM	High energy ball milling
FTIR	Fourier transform Infrared spectroscopy
XPS	X-ray photoelectron spectroscopy
μ'	permeability
μ_i	Initial permeability
μ''	Permeability loss factor
μ_B	Bohr magneton
m	Magnetic moment
Ca	Calcium
V	Vanadium
Sn	Tin
K	Anisotropy
DC	Direct current
AC	Alternating current
VSM	Vibrating sample magnetometer

FESEM	Field emission scanning electron microscope
a-sublattice	Octahedral sublattice
d-sublattice	Tetrahedral sublattice
c-sublattice	Dodecahedral sublattice
D	Grain size
R	Resistance
P	Porosity
d_b	Bulk density

CHAPTER 1

INTRODUCTION

1.1 Background of study

Oxide ceramics which exhibit ferrimagnetic behavior play an important role in the electronic industry. They are commonly known as ferrites. Ferrites are the mixed metal oxides containing iron oxide as their main component. There are three important classes of commercial ferrites, and each of one having a specific crystal structure. The first one is soft ferrites with cubic spinel structure, such as NiZn- and MnZn ferrites. The second one is the soft ferrites with garnet structure, such as the microwave ferrites, for example yttrium iron garnet, and the third one is hard ferrites with the hexagonal structure such as Ba and Sr hexaferrites. The industrial importance of ferrites becomes obvious when one examines the diversity of their applications. Through the years, the demand of ferrites has steadily increased together with the specific applications of ferrites, and have changed remarkably and kept pace with the development in electronic technologies.

Ceramic technology has historically developed from an empirical method of fabrication to one which is based more and more on the application of scientific knowledge. It has become quite evident that the parameters determining the suitability of materials for particular applications are chemical composition, crystallographic structure and the so-called microstructure, which comprises such important aspects as size of constituent crystallites (grains), their size distributions, their boundaries (grain boundaries), their orientation (texture), and the voids (pores) between them (Sun, 2007). To control and manipulate these parameters, there must be a range of methods available for preparing ceramic powders. There are three basic methods, which are mechanical, chemical, and vapor phase methods.

Mechanical methods use coarse-grained materials which is often necessary to eliminate aggregates and to reduce particle size. They are subjected to a series of processes called as comminution. The steps involved crushing, grinding, and milling. Mechanical methods of powder production are widely used in the industry due to ability for high mass production of the product. Chemical methods such as sol-gel processing offer several advantages over mechanical methods. This method allows exceptional control over particle morphology and purity at lower temperatures. Chemical processes are widely used in the production of advanced ceramic materials such as fibers, coatings, etc. (Cheng et al., 2007). The other method is vapor-phase processes. Vapor-phase processes offer many advantages, such as the ability to produce particles of non-oxides and nanoparticles, as they can give high purity powders, discrete and non-aggregated particles, and nanoparticles with narrow size distributions (Guo et al., 2005).

In this chapter, the general information on ferrite magnetic garnets is introduced. An introduction on raw steel waste (well-known as mill scale) and its potential in producing iron oxide powder will be highlighted. In the thesis, we will discuss the previous work on the synthesis compound within the Fe_2O_3-Y_2O_3 system by mechanical alloying method and the statistical analysis data by this method. Theories that are related with the results are also discussed. The materials used and the procedure of the research will be compile followed with the results and discussion. Based on the results, we will summarize and conclude the research findings, in addition to some suggested recommendations for future work.

1.2 Magnetic ceramics

In recent years, ferrimagnetic garnet have attracted much attention as microwave device materials, circulators, isolators, phase shifters, etc. due to its excellent electromagnetic properties, including low dielectric loss, narrow ferromagnetic resonance linewidth in microwave region and tailorable saturation magnetization (Yu et al., 2011). Thus, research has been committed to the investigations of YIG and doped-YIG by controlling the preparation conditions to tailor their microstructure in addition to the design of chemical composition.

1.3 Introduction of Garnets

Generally, the chemical formula for garnet system is written as $3M_2O_3.5Fe_2O_3$ or $M_3Fe_5O_{12}$. M represents yttrium (Y) or one of the rare earth ions. The rare earth ions are large, so that they occupy the largest cation sites in the garnet structure, which are dodecahedral sites. Note that in the magnetic garnet, the metal ions are all trivalent. There are 24 dodecahedral, 16 octahedral, and 24 tetrahedral sites in a unit cell containing 8 formula units. The prototype for ferrimagnetic iron garnet is yttrium iron garnet (YIG). Garnets provide superior performance in microwave devices because they have narrow resonance line width, one of the most important parameters from application point of view besides wide range of magnetization and very low dielectric loss (Soleimani et al., 2012).

1.4 Mill scale as a potential of raw materials

The processes of manufacturing steel product from industry produce two times more waste materials than the final product obtained. These results in the formation of large amounts of waste materials in the form of slags, scale and dusts (Martin et al., 2012). Currently, most of the waste is utilized in the same steelworks and passed to external customers or it is reused as a feeds for other technological processes. In the case of Malaysia’s steel Industry, Malaysia’s consumption on steel is expected to trend up along with on-going construction and mass transit projects. The demand of steel is expected to rise up by 12.6% year-of-year (Export-

Import Bank of Malaysia, 2015). In 2013, 11.69% million tons of steels are produced and increased to 12.5% million tons in 2014 ("World Steel Association," 2014). This trend is expected to increase up in year 2020. The steel industry in Malaysia can be categorically subdivided into two main segments, namely long products and flat products. Long products include billets, bars, wire rods, sections, nails, wire mesh, nuts, bolts, etc. which are predominantly used in the construction industry. Flat products are the products consumed mostly by the manufacturing, construction and oil & gas sectors like hot-rolled plates and sheets, cold-rolled coils, tubes, pipes, boiler and pressure vessels, etc. Irrespective of the products, during processing of steel to yield long or flat product, mill scale is generated and considered as a waste (Bagatini et al., 2011).

Mill scale is flaky that can be found on metals that have been rolled. Mill scale forms an exterior surface of sheets and plates as they are being manufactured through rolling steel billets and hot iron into rolling mills. Mill scale which represents 2% of steel produced contains iron in elemental form and three types of iron oxides: wustite (FeO), hematite (α-Fe_2O_3) and magnetite (Fe_3O_4) (Legodi and de Waal, 2007). The chemical composition of mill scale depends on the type of steel produced and the process used. The iron content in the mill scale is normally around 72% Fe with small amounts of non-ferrous metals. Mill scale is contaminated with remains of lubricants and other oils and greases from the equipment associated with rolling operations (Martin et al., 2012). The oil content usually ranges between 0.1 % and 2.0%, and can reach up to 10.0%. In steelworks plants, almost 85% of the mill scale generated is recycled within the steelmaking industry and small amounts are used for ferroalloys, in cement plants and in the petrochemicals industry. The balance part of the mill scale with particle size less than 0.5 mm (finer mill scale or mill scale sludge), which is more heavily contaminated with oils, ends up in landfills. Mill scale sludge cannot be recycled via sintering since its fine particles contains a high oil level which is 5.0-20.0% and is normally treated as a landfill waste. Mill scale with oil content more than 3.0% is recycled after extracting the oil in a pre-treatment stage. Mill scale with oil content less than 1.0% with the particle size between 0.5-5.0 mm can be returned without any pre-treatment (Legodi and de Waal, 2007). Thus, the allowable limit for oil content in the mill scale is less than 1% for all usage.

Although most of the mill scales are consumed after recycling, and only small amounts are used in ferroalloys, cement and petrochemicals industry, there is a potential for production of steel increase up to 1.0 million tons with the increase in construction activities, current economic needs in emerging markets and growing concern over the use of steel in heavy industries. All the above gives an initiative to find suitable means for effective utilization of mill scale and a low cost by-product in the steelmaking industry. In the present research, we have employed the mill scale waste product from the Malaysian steel factories. The steel are collected from Perwaja Steel Terengganu. The immense amount of steel waste give a challenge for us to extract the iron oxide contained in the mill scale by a purification technique and producing iron oxide for magnetic materials.

1.5 Aim of study

In several past decades, studies on YIG by pure materials are reported in ferrite literature. Instead, fabrication of YIG ferrite by using recycled steel waste product (mill scale) is still investigated. There are no reports from literature on this work. Steel waste products are collected from steel industry in Malaysia. It has high content of iron (Fe) and gives us a challenge to purify and recycled the powder to produce YIG ferrites. This project will highlight the low cost ferrite fabrication from the waste material.

Ferrite magnetic materials are known very rely to their microstructure. Nowadays, the fundamental on scientific enquiry of the microstructure-property evolution has been neglected. Therefore, in this work, the evolutions of the microstructure-magnetic properties relationship at various sintering temperature on the morphology and material properties will be investigated.

1.6 Objectives

The interest of this research is to track down the parallel evolution of microstructural and magnetic properties from lower sintering temperature (500^{o}C) to higher temperature (1400^{o}C) of YIG. Hence, the study embarks on the following objectives:

I. To purify recycled mill scale waste product by magnetic and non-magnetic separation (MNM) and Curie temperature separation technique (CTS) collected from steel industries in Malaysia to produce high purity Fe_2O_3 as main raw materials in fabrication YIG ferrite by using mechanical alloying technique.
II. To elucidate the parallel evolution of the magnetic properties with microstructural changes and their relationship in nano-micro yttrium iron garnet ferrites at various sintering temperatures.

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