UNIVERSITI PUTRA MALAYSIA

ISOCHRONAL RECOVERY OF ELECTRO-MAGNETIC ENERGY LOSS AND ELECTRICAL RESISTIVITY IN YTTRIUM-IRON GARNET ($Y_3Fe_5O_{12}$)

NOR HAPISHAH BINTI ABDULLAH

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By

NOR HAPISHAH BINTI ABDULLAH

Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Master of Science.

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

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Chairman : Associate Professor Mansor b Hashim, PhD

Faculty: Science.

Ferrites, including garnets are magnetic ceramics which have been used in the microwave and non-microwave frequency region for more than eight decades. Radio antenna, and high frequency transformers are just a few examples of devices in which ferrites are applied. The high electrical resistivity and excellent magnetic properties make ferrites a preferred choice in electronics and telecommunications in higher frequency region (upper MHz and GHz).

One long standing research interest which covers both fundamental concerns and practical importance is a clear understanding of the origin or causes of electromagnetic (EM) energy loss in ferrites. This knowledge would greatly contribute towards producing very low-loss antenna and circuit-component ferrite materials. Hence yttrium-iron garnet (YIG) was chosen for the present research on account of its being the best low-loss applications. Since microstructural effects on EM energy loss are well

understood, we propose instead to establish whether or not crystal atomic defects have any influence on such loss. For this intention, we have borrowed from metallurgists, a simple but powerful probing technique involving defects creation and their subsequent elimination within equal time-length durations: this is a technique for establishing isochronal recovery behaviour of any atomic defects-dependent properties.

We chose to investigate experimentally the effect on a sample's properties which are important to microwave scientists and engineers, after undergoing quenching and annealing treatments. In this study, YIG was prepared via mechanical alloying involving a 24 hours milling time of a mixture of yttrium oxide (Y_2O_3) and iron oxide (Fe_2O_3). The samples were then sintered at different temperatures between 900°C to 1350°C and optimized for microstructure. The samples were then heated for 2 hours at 1000°C and quenched in cooking oil. Then, annealing of the samples at 1000°C for 2 hours was carried out.

The microstructure, magnetic and electrical properties before and after quenching also after annealing were studied in order to understand the physical, magnetic and electrical properties of the resulting materials. The X-Ray diffraction results confirmed the formation of the crystalline phase after sintering at 900°C. The microstructure studies of $Y_3Fe_5O_{12}$ showed that the grain size increased and the pore size was estimated to be decreased as sintering temperature increased. The permeability and loss factor showed an isochronal recovery behaviour in which a parameter's value was decreased after quenching and increased back even higher after annealing. This suggests that magnetic permeability and energy loss in YIG could significantly show an atomic defect-dependent behaviour. The resistivities of samples also could be observed to have an isochronal recovery behaviour in which

the value dropped after quenching and increased almost gradually back even higher after an isochronal annealing series starting from 500°C to 1000°C. The values of resistivity were dropped after quenching due to defects created which are known as atomic vacancies and interstitials that might act as an electron donor which increased the value of the conductivity hence giving lower resistivity. Isochronal annealing on the other hand could be understood as a filling back of the vacancies slowly from low to high annealing temperature, yielding after the recovery on the values which is equal or higher than that of the as prepared sample. The corresponding results on the values of the permeability and the loss factor, strongly suggest that the decreases and increases in those values were associated with the creation and elimination, respectively, of atomic scale defects.

The findings on the isochronal recovery behaviour of the permeability, energy loss and resistivity for these samples hopefully will help produced a better quality YIG with very low energy loss and very high resistivity to be used in electronic devices operating in the frequency range of 1 MHz to 1 GHz, as covered in this study.

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

PEMULIHAN ISOKRONAL BAGI KEHILANGAN TENAGA DAN KERINTANGAN ELEKTRIK PADA FREKUENSI ELEKTROMAGNET DALAM GARNET YTTRIUM FERUM ($Y_3Fe_5O_{12}$)

Oleh

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Ferit, termasuk garnet seramik magnet yang telah diaplikasikan pada julat frekuensi gelombang mikro telah dikenalpasti selama lebih daripada lapan dekad. Antenna radio dan transformer frekuensi tinggi adalah hanya beberapa contoh alat-alat yang digunakan. Dengan nilai rintangan elektrik yang tinggi dan sifat magnet yang baik menjadikan ferit ini sejenis pilihan yang baik sebagai bahan dalam aplikasi elektronik dan telekomunikasi pada julat frekuensi tinggi (MHz ke atas hingga GHz).

Suatu minat penyelidikan yang telah lama mantap dalam aspek asas dan kepentingan praktikal ialah pemahaman yang jelas mengenai asalan dan sebab-sebab terjadinya kehilangan tenaga elektromagnet (EM) dalam bahan ferit. Pemahaman yang jelas mengenai sebab-sebab terjadinya kehilangan tenaga elektromagnet akan menyumbang hebat ke arah menghasilkan antenna dan komponen litar dengan kehilangan tenaga yang amat rendah. Maka garnet yttrium-ferum (YIG) telah dipilih untuk penyelidikan ini kerana ia adalah bahan magnet terbaik untuk kegunaan

sebagai antenna dan kegunaan kehilangan-rendah yang lain. Oleh kerana pengaruh mikrostruktur ke atas kehilangan tenaga telah difahami dengan baik, kami sebaliknya bercadang menentu kukuh sama ada kecacatan atom hablur mempengaruhi kehilangan tenaga sedemikian. Untuk tujuan ini, kami meminjam daripada ahli metalurgi suatu teknik pengesan yang mudah tetapi berkuasa, melibatkan pembentukan kecacatan atom dan penghapusannya dalam jangkamasa yang sama panjang: ini adalah teknik untuk mengesahkan wujudnya tabiat pemulihan isokronal bagi sebarang sifat bahan yang boleh dipengaruhi oleh kecacatan atom.

Oleh itu, dengan menggunakan garnet yttrium ferum sebagai bahan ujian, kami memilih untuk menyiasat tentang kesan ke atas sampel selepas menjalani proses pelindapan dan penyepuhlindapan ke atas beberapa sifat-sifat yang penting untuk ahli-ahli sains dan jurutera gelombang mikro. Dalam kajian ini, YIG telah disediakan melalui teknik pengaloian mekanik selama 24 jam untuk penyempurnaan campuran yttrium oksida (Y_2O_3) dan besi oksida (Fe_2O_3). Sampel kemudiannya disinter pada suhu antara 900°C hingga 1350°C dan dioptimumkan untuk mikrostruktur yang berbeza. Sampel dipanaskan selama 2 jam pada 1000°C dan dilindapkan ke dalam minyak masak. Kemudian, penyepuhlindapan sampel pada 1000°C selama 2 jam telah dijalankan.

Mikrostruktur, sifat-sifat magnet dan elektrik sebelum dan selepas pelindapan dan juga selepas penyepuhlindapan dikaji untuk memahami sifat-sifat fizikal, magnetik dan elektrik bahan-bahan yang terhasil. Keputusan pembelauan sinar-X mengesahkan pembentukan fasa kristal selepas pensinteran pada 900°C. Kajian mikrostruktur $Y_3Fe_5O_{12}$ telah menunjukkan bahawa saiz butiran meningkat dan saiz

liang dianggarkan menurun dengan peningkatan suhu pensinteran. Ketelapan dan faktor kehilangan menunjukkan sifat pemulihan isokronal di mana nilainya telah menurun selepas proses pelindapan dan meningkat kembali lebih tinggi selepas penyepuhlindapan. Ini menunjukkan bahawa ketelapan magnet dan kehilangan tenaga dalam YIG boleh mempunyai pengaruh daripada kecacatan pada peringkat atom. Kerintangan sampel juga menunjukkan sifat pemulihan isokronal apabila nilai yang menurun setelah proses pelindapan dan meningkat hampir secara beransur-ansur kembali lebih tinggi selepas penyepuhlindapan isokronal bermula dari 500°C hingga 1000°C. Nilai-nilai yang telah menurun selepas proses pelindapan disebabkan oleh kecacatan yang dinamakan kekosongan atom dan anjakan atom yang mungkin bertindak sebagai penderma elektron lalu meningkatkan nilai kekonduksian dan memberi nilai kerintangan yang lebih rendah. Penyepuhlindapan isokronal pada sisi lain dapat difahami sebagai mengisi kekosongan atom secara perlahan-lahan daripada penyepuhlindapan yang rendah ke suhu tinggi, lalu memberi pemulihan kepada nilai yang lebih baik daripada nilai asalnya. Keputusan yang sama pada nilai ketelapan dan faktor kehilangan, sangat menyarankan bahawa kenaikan nilai yang dikaitkan dengan kebergantungan pada kecacatan pada skala atom.

Penemuan mengenai tabiat pemulihan dalam ketelapan, kehilangan tenaga dan kerintangan bagi sampel ini diharapkan akan menghasilkan kualiti YIG yang lebih baik dengan kehilangan tenaga yang rendah dan tinggi nilai kerintangan yang akan digunakan dalam alat-alat elektronik yang beroperasi pada julat frekuensi di antara 1 MHz ke 1 GHz seperti yang diliputi dalam kajian ini.

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I certify that a Thesis Examination Committee has met on 13 01 2012 to conduct the final examination of **NOR HAPISHAH BT ABDULLAH** on her thesis entitled " "**ISOCHRONAL RECOVERY OF ELECTRO-MAGNETIC ENERGY LOSS AND ELECTRICAL RESISTIVITY IN YTTRIUM-IRON GARNET ($Y_3Fe_5O_{12}$)"** accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] xx xxx 2011. The committee recommends that the student be awarded the Master of Science.

Members of the Thesis Examination Committee were as follows:

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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 Supervisory Committee were as follows.

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DECLARATION

I declare that this thesis is my original work except for quotations and citation 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 other instituitions.

NOR HAPISHAH ABDULLAH
Date: 13 January 2012

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