



UNIVERSITI PUTRA MALAYSIA

**PREPARATION OF SPINEL AND GARNET FERRITES AND
IDENTIFICATION OF THEIR MAGNETIC-ENERGY LOSSES**

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PREPARATION OF SPINEL AND GARNET FERRITES AND IDENTIFICATION OF THEIR MAGNETIC-ENERGY LOSSES

By

NOORHANA BINTI YAHYA

**Thesis Submitted in Fulfillment of the Requirement for the Degree of Doctor of
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DEDIKASI

Abah dan emak yang dihormati dan disayangi

**YAHYA BIN JAIS
RAHMAH BINTI KULUP HASSAN**

Suami dan anak yang tercinta

**SAZALI BIN YUSOF
MUHAMAD ZIKRI AFWAN BIN SAZALI**

Abang dan Kakak

**BURHANUDDIN BIN YAHYA
NOREHAN BINTI YAHYA**

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**MUHAMMAD ZUL FAZIL BIN ZULKIFLI
ERNA SHAFINAZ BINTI BURHANUDDIN
NUR SAKINAH BINTI BURHANUDDIN
MUHAMMAD AFIQ BIN BURHANUDDIN**

Kejayaan ini adalah milik kita bersama

Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Doctor Philosophy

**PREPARATION OF SPINEL AND GARNET FERRITES AND
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NOORHANA YAHYA

October, 2001

Chairman : Associate Professor Dr. Mansor Bin Hashim

Faculty : Institute of Advanced Technology

The objective of this work was to explain the magnetic-energy loss mechanisms of some magnetic materials. The study was divided into three parts. The first part involved fabrication of NiZn-based and YIG ferrites in toroidal and pellet form, employing ceramic processing technique of the starting oxides. Characterisation of chemical, microstructural, magnetic, electrical, mechanical and thermal properties were carried out. In the second part, sol-gel method was employed to obtain high quality and fine-grained microstructure. The $Y_3Fe_5O_{12}$ and $NiFe_2O_4$ samples were fabricated using this technique. The third part dealt with some preliminary studies on

the magneto-optical Kerr effect, which were carried out on the NiFe_2O_4 and $\text{Y}_5\text{Fe}_5\text{O}_{12}$ samples.

The characterisation of samples in the first part was divided mainly into two parts : the extrinsic-microstructure properties and the intrinsic-composition properties. The results showed that the initial permeability, relative loss factor, impedance, power loss, quality factor, saturation induction, core loss, coercive force, curie temperature and temperature coefficient of the sintered samples depended chiefly on both the microstructure and the composition of the samples. Adopting ZnO, which acted as a modifier, in the NiZn ferrite series (first premise) had greatly influenced the magnetic properties of the samples, as occurrence of Zn loss was a major factor that affected the grain growth kinetics. Adopting an iron-deficit composition (second series) was fruitful when high density and wide operating frequencies were required in the NiZn ferrite composition. Samples with excess Fe_2O_3 (third series) were deleterious in terms of losses due to the formation of Fe^{2+} . There was no significant contribution of the zero magnetostriction affecting the magnetic and electrical properties that was concluded from this premise. CoO was seen to affect the growth anisotropy in the rich NiO content (fourth premise) and thus affected the microstructure of the samples. Interesting, however, was sample with composition $\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$ that gave very homogeneous and moderate grain size ($\approx 10.9 \mu\text{m}$) exhibited large $-K_1$, played a dominant role in the frequency extension. Evidence by the reduced permeability, it was believed that the damping of domain wall was restricted by the anisotropy effects. Simultaneously, the relative loss factor was significantly reduced at higher frequencies. In the fifth premise where both Co^{2+} and Fe^{2+} were adopted in the excess-

iron NiZn based composition, the Co^{2+} content was believed to stabilise the domain wall movement at high frequencies. When a small concentration of cobalt with the formula $\text{Ni}_{0.70}\text{Co}_{0.01915}\text{Zn}_{0.27585}\text{Fe}_{2.005}\text{O}_{4.005}$ was adopted, a vast decrease of power loss was seen to occur. It was speculated that Co^{2+} ions diffused or moved through the vacancies and hence caused them to reside in the vacancies created by the slight iron excess. This reduced the stress and strain created by them and as a result, power loss was reduced significantly.

In the second part of this work, high quality and fine grained single-phase ferrite ($\sim 0.9 \mu\text{m}$) was obtained by using the sol-gel technique. Finally, Kerr rotation ($\sim 1 \text{ deg}$) was observed for both the NiFe_2O_4 and $\text{Y}_3\text{Fe}_5\text{O}_{12}$ samples. Kerr rotation was accompanied by optical energy reflection. This was actually a measure of energy reflected when ferromagnetic order exists. This shed new light in the area of magneto-optics.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

**PENYEDIAAN FERIT SPINEL DAN GARNET DAN PENGENALPASTIAN
KEHILANGAN TENAGA MAGNETNYA**

Oleh

NOORHANA BINTI YAHYA

Oktober 2001

Pengerusi : Profesor Madya Dr. Mansor Bin Hashim

Fakulti : Institut Teknologi Maju

Penyelidikan ini memfokus terhadap mekanisme-mekanisme kehilangan tenaga magnet. Kajian ini melibatkan tiga bahagian utama. Pertama, penyediaan ferit asas-NiZn dan YIG dalam bentuk toroid dan pelet dengan menggunakan teknik pemprosesan lazim yang melibatkan percampuran basah terhadap oksida-oksida logam serta pencirian sampel-sampel merangkumi aspek-aspek kimia, struktur, mikrostruktur, magnet, elektrik, mekanik dan terma. Bahagian kedua pula melibatkan teknik penyediaan secara nobel, iaitu teknik sol-gel, yang bertujuan untuk mendapatkan bahan ferit

berkualiti tinggi serta mikrostruktur yang halus dan homogen. Sampel-sampel NiFe_2O_4 dan $\text{Y}_3\text{Fe}_5\text{O}_{12}$ telah disediakan dengan menggunakan teknik ini. Bahagian ketiga pula melibatkan kajian premier terhadap kesan Kerr yang dilakukan keatas sampel-sampel NiFe_2O_4 dan $\text{Y}_3\text{Fe}_5\text{O}_{12}$. Pencirian sampel-sampel pada bahagian pertama dibahagikan secara amnya kepada dua bahagian iaitu ciri-ciri ekstrinsik-mikrostruktur dan ciri-ciri intrinsik-komposisi. Keputusan eksperimen menunjukkan bahawa ketelapan awal, faktor kehilangan tenaga, impedans, kehilangan kuasa, faktor kualiti, ketumpatan fluks magnet tepu, daya paksa, kehilangan teras, suhu curie dan pemalar suhu, bergantung kepada kedua-dua mikrostruktur dan komposisi sampel-sampel. Dalam premis pertama, penggantian ZnO dalam siri ferit asas NiZn telah mempengaruhi ciri-ciri magnet dan elektrik sampel-sampel kerana berlakunya kehilangan zink yang mempengaruhi kinetik pertumbuhan butiran. Penggunaan defisit-besi dalam premis kedua pula telah menghasilkan sampel berketumpatan tinggi dan julat frekuensi dapat dilebarkan. Pecahan mol sebanyak 0.47 oksida logam dalam ferit NiZn berdefisit- Fe_2O_3 ini dipercayai merupakan nilai yang paling berkesan untuk membentuk sampel defisit berketumpatan tinggi. Sampel-sampel eksis- Fe_2O_3 dalam premis ketiga pula, telah menyebabkan kehilangan tenaga yang tinggi kerana terbentuknya ion-ion Fe^{2+} . Disamping itu, tiada sumbangan yang jelas terhadap magnetostriksi null terhadap ciri-ciri magnet dan elektrik. CoO dilihat dapat mempengaruhi anisotropi pertumbuhan apabila kandungan NiO yang kaya dalam siri ferit NiZn amat mempengaruhi mikrostruktur sampel-sampel ini. Sampel $\text{Ni}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$ membentuk butiran yang bersaiz sederhana dan homogen serta mempamerkan K_1 yang tinggi dan seterusnya

memainkan peranan besar terhadap julat frekuensi. Berdasarkan ketelapan awal yang rendah, adalah dipercayai pelembapan dinding domain adalah hasil daripada kesan anisotropi ini. Justeru itu, faktor kehilangan relatif dapat dikurangkan pada frekuensi yang tinggi. Dalam siri terakhir dimana kedua-dua ion Co^{2+} dan Fe^{2+} digunakan dalam komposisi eksese-besi NiZn , ion-ion Co^{2+} didapati dapat menstabilkan pergerakan dinding domain pada frekuensi yang tinggi. Apabila sedikit kobalt dengan komposisi $\text{Ni}_{0.70}\text{Co}_{0.01915}\text{Zn}_{0.27585}\text{Fe}_{2.005}\text{O}_{4.005}$ digunakan, penurunan kehilangan kuasa berlaku secara mendadak. Ion-ion Co^{2+} dijangka meresap dan seterusnya menduduki kekosongan-kekosongan ini. Ini mengurangkan ketegangan dan hasilnya pengurangan kehilangan kuasa berlaku. Penggunaan teknik sol-gel menghasilkan ferit sefasa yang berkualiti serta saiz butiran yang seni ($\sim 0.9 \mu\text{m}$). Putaran Kerr (~ 1 darjah) dapat dikesan pada kedua-dua sampel NiFe_2O_4 dan $\text{Y}_3\text{Fe}_5\text{O}_{12}$. Putaran Kerr adalah diikuti dengan pantulan tenaga optik yang merupakan satu penemuan baru dalam bidang magneto-optik.

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Noorhana Binti Yahya

This thesis submitted to the Senate of Universiti Putra Malaysia has been accepted as fulfilment of requirement for the degree of Doctor of Philosophy.

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