UNIVERSITI PUTRA MALAYSIA

FABRICATION AND CHARACTERISATION OF SELECTED MICROWAVE ABSORBING FERRITE-POLYMER COMPOSITES

FADZIDAH BT MOHD IDRIS

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By

FADZIDAH BT MOHD IDRIS

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

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In appreciation of their love and sacrifices, this thesis is dedicated to my family especially my beloved mother HJH SABARIAH BT MOHD YATIM and my sisters ZANARIAH BT MOHD IDRIS and ZURAIDAH BT MOHD IDRIS who have been giving me full moral support throughout the years. Not forgotten to my late father Allahyarham MOHD IDRIS BIN HJ SHARIAT.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Master of Science

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Chairman: Associate Professor Mansor Hashim, PhD

Institute: Institute of Advanced Technology

Although absorbing materials are a useful part of modern-day defence systems, very little published knowledge exists on the fabrication of such materials especially microwave absorbing materials. The present research attempts to fabricate absorbing material compositions suitable for microwave absorption from 8 to 18 GHz. Various compositions of composite ferrites were prepared using mechanical alloying and sintering. The starting metal oxide raw materials were weighed according to the targeted proportion and milled for 10 hours using a SPEX8000D mill to get nanosized particles. The resulting mixture was poured into a PVA solution as a binder and stirred while drying it using an ultraviolet lamp until the powder contained ~1 wt% PVA. It was then pressed into pellet/toroid-shaped samples and sintered at temperature 900 °C for 10 hours. Then, a composite of ferrite powder with polymer paint as matrix was prepared. The composite paint produced was applied on the surface of a metal sheet of specified surface dimensions. Physical characteristics of the as-prepared filler samples were studied using X-ray diffraction (XRD), scanning transmission electron microscopy

(STEM) and Field emission Scanning Electron Microscope (FESEM). The toroidal sample was further studied using an Agilent 4291B Impedance Analyzer with the frequency range from 1 MHz to 1 GHz in order to investigate the material's complex permeability components μ' and μ". The absorption of the composite paint-coated metal sheet was characterized using an Agilent 85071E Network Analyzer in the frequency range from 8 GHz to 18 GHz. The XRD results show that at 900ºC the full phase of nickel zinc ferrite was formed. The average particle size for all the compositions is in nanometers(sub-micron sized). The resulting morphology was a homogeneous microstructure with small grain size and a uniform grain size distribution obtained via the mechanical alloying technique. From the complex permeability component μ' and μ" results, a significantly important result was established: that it was possible to extend the em energy absorption frequency range by reducing the grain size from micrometer to nanometer, using samples of the same chemical composition. For measurement at higher frequency (X-band and Ku-band), physical sample thickness influences the reflection loss and absorption of the ferrite-in-polymer-matrix composites with a metal back. Thicker samples result in higher microwave absorption. A sample with a thickness of 3.22 mm yields higher absorption compared to a sample with a thickness of 2.35 mm backed by an aluminium plate. Consequently, materials with different compositions give different levels of microwave absorption with the aluminium plate giving ~ 0 dB reflection losses. For other compositions, trends can be observed as the Ni^{2+} content is increased. As the Ni^{2+} content increases, the minimum reflection loss or maximum absorption is decreased. Therefore, $Ni_{0.5}Zn_{0.5}Fe_2O_4$ gives the highest absorption compared to that of other compositions. In addition, by comparing all the compositions involving combined nickel zinc ferrite (a mixture of nickel zinc ferrites with different

NiZn ratios-NZF combine), $Ni_{0.5}Zn_{0.5}Fe_2O_4$ alone gives the most minimum reflection loss which corresponds to the highest absorption by the sample. The minimum reflection loss given by $Ni_{0.5}Zn_{0.5}Fe_2O_4$ at frequency 9.0 GHz and 12 GHz reaches – 6.72 dB and – 11.2 dB respectively. Furthermore, the percentage amount of sintered $Ni_{0.5}Zn_{0.5}Fe_2O_4$ being added into the paint are 5 wt% and 20 wt% with different thicknesses. A higher ferrite content in the matrix resulting in higher absorption. The composite paints are expected to be very useful in military applications such as radar cross section reduction and prevention of electromagnetic interference.

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

PEMBUATAN DAN PENCIRIAN BAGI PENYERAP MIKROGELOMBANG FERIT-POLIMER KOMPOSIT TERPILIH

Oleh

FADZIDAH BT MOHD IDRIS

Disember 2012

Pengerusi: **Profesor Madya Mansor Hashim, PhD**

Institut: **Institut Teknologi Maju**

Walaupun bahan penyerap adalah sebahagian daripada kegunaan sistem pertahanan moden, sangat sedikit pengetahuan yang diterbitkan wujud pada pembuatan bahan-bahan tersebut terutama bahan penyerap gelombang mikro. Penyelidikan ini adalah percubaan untuk menghasilkan komposisi bahan penyerap yang sesuai untuk penyerapan pada 8 hingga 18 GHz. Pelbagai komposisi komposit ferit telah disediakan dengan menggunakan pengaloian mekanikal dan pensinteran. Bahan permulaan oksida logam besi di timbang mengikut nisbah yang ditetapkan dan dikisar selama 10 jam menggunakan pengisar SPEX8000D untuk mendapatkan zarah saiz nano. Campuran yang terhasil telah dicurahkan ke dalam larutan PVA sebagai pengikat dan dikacau sambil dikeringkan menggunakan lampu ultraviolet sehingga serbuk mengandungi ~1% berat PVA. Sampel kemudiannya ditekan kepada bentuk pellet/toroid dan dipanaskan pada suhu 900 °C selama 10 jam. Kemudian, serbuk komposit ferit dengan cat polimer sebagai matrik telah disediakan. Cat yang dihasilkan telah digunakan pada permukaan kepingan logam mengikut dimensi yang ditetapkan. Ciri-ciri fizikal sampel dikaji

menggunakan pembiasan sinar-X(XRD), mikroskop pengimbas elektron (STEM) dan mikroskop pengimbas medan elektron (FESEM). Sampel toroid terus diukur menggunakan Impedance Analyzer Agilent model 4291B dengan julat frekuensi dari 1 MHz hingga 1 GHz untuk mengkaji komponen ketelapan kompleks μ' dan μ". Penyerapan kepingan logam bersalut cat komposit dicirikan menggunakan Agilent 85071E Network Analyzer dalam julat frekuensi 8 GHz hingga 18 GHz. Keputusan XRD menunjukkan bahawa pada suhu 900 °C fasa penuh ferit nikel zink telah terbentuk. Purata saiz zarah bagi semua komposisi adalah bersaiz nanometer (bersaiz sub-mikron). Morfologi yang terhasil adalah mikrostruktur homogen dengan saiz butiran kecil dan taburan saiz butiran seragam melalui teknik pengaloian mekanikal. Dari keputusan komponen ketelapan kompleks μ' dan μ", hasil yang ketara telah terbentuk: bahawa adalah mungkin untuk melebarkan julat frekuensi penyerapan tenaga em dengan mengurangkan saiz butiran dari mikrometer kepada nanometer, dengan menggunakan komposisi sampel kimia yang sama. Bagi pengukuran pada frekuensi yang lebih tinggi (Jalur-X dan Jalur-Ku), pengaruh fizikal ketebalan sampel mempengaruhi kehilangan pantulan dan penyerapan ferit dalam matrik polimer komposit yang disokong plat logam. Sampel yang lebih tebal menghasilkan keputusan penyerapan yang lebih tinggi. Keputusan sampel dengan ketebalan 3.22 mm menghasilkan penyerapan yang lebih tinggi berbanding dengan sampel dengan ketebalan 2.35 mm yang disokong kepingan aluminium. Seterusnya, bahan dengan komposisi berbeza menghasilkan keputusan penyerapan gelombang mikro yang berbeza. Keputusan kepingan aluminium menghasilkan ~ 0 dB kehilangan pantulan. Bagi komposisi lain, corak boleh diperhatikan apabila kandungan Ni^{2+} meningkat. Apabila kandungan Ni^{2+} meningkat, kehilangan pantulan minima atau penyerapan maksima menurun. Oleh itu, $Ni_{0.5}$

$Zn_{0.5}Fe_2O_4$ memberi penyerapan tertinggi berbanding dengan komposisi lain. Di samping itu, dengan membandingkan semua komposisi dengan gabungan nikel zink ferit (campuran berbagai bahan ferit-nikel-zink dengan nisbah NiZn yang berbeza-NZF gabungan), $Ni_{0.5}Zn_{0.5}Fe_2O_4$ sahaja memberikan kehilangan pantulan yang paling minimum sepadan dengan penyerapan tertinggi oleh sampel. Kehilangan pantulan minimum diberikan oleh $Ni_{0.5}$ $Zn_{0.5}Fe_2O_4$ pada frekuensi 9 GHz dan 12 GHz masing-masing boleh mencapai -6.72 dB dan 11.2 dB. Tambahan pula, jumlah peratusan $Ni_{0.5}$ $Zn_{0.5}Fe_2O_4$ bersinter yang ditambah ke dalam cat adalah 5 wt% dan 20 wt% dengan ketebalan yang berbeza. Kandungan ferit yang tinggi dalam matrik menghasilkan penyerapan yang tinggi. Cat komposit dijangka menjadi sangat berguna dalam aplikasi ketenteraan seperti pengurangan keratan rentas radar dan pencegahan pencemaran elektormagnet.

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I certify that a Thesis Examination Committee has met on 6th December 2012 to conduct the final examination of Fadzidah Bt Mohd Idris on her thesis entitled "**Fabrication and Characterisation Of Soelected Microwave Absorbing Ferrite-Polymer Composites**" in accordance with Universities and University Colleges Act 1971 and the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Degree of Master of Science.

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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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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.

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FADZIDAH BT MOHD IDRIS

Date:

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