UNIVERSITI PUTRA MALAYSIA

ELUCIDATION OF THE PARALLEL EVOLUTIONS OF MICROSTRUCTURE AND MAGNETIC PROPERTIES AND THEIR RELATIONSHIP IN NICKEL-ZINC FERRITE

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

ELUCIDATION OF THE PARALLEL EVOLUTIONS OF MICROSTRUCTURE AND MAGNETIC PROPERTIES AND THEIR RELATIONSHIP IN NICKEL-ZINC FERRITE

By

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In this research work, parallel evolving morphology and magnetic properties in nickel zinc ferrite with composition $Ni_{0.3}Zn_{0.7}Fe_2O_4$ were studied. For several past decades, studies of the relationship between morphological properties and magnetic properties of ferrites have been focusing only on the product of the final sintering temperature, largely neglecting the parallel evolutions of morphological and magnetic properties and their relationships at various lower sintering temperatures. Hence, here, in this thesis we report some research findings on the parallel evolutions of such morphological properties and magnetic properties; we attempt to elucidate their relationships. Nickel zinc ferrite was prepared via High-energy ball milling in a hardened steel vial for 2 hours using a SPEX8000D mill. The toroidal samples went through two different sintering routes which were multi-sample sintering and single-sample sintering. In the multi-sample sintering, the samples were sintered from 600^{o}C to 1400^{o}C using 100^{o}C increments with any one sample being subjected to

only one sintering temperature. Nevertheless, for the single-sample sintering, the same sample was subjected to repeat sintering from 600^{o}C to 1400^{o}C with 100^{o}C increments. The completion of the solid state reaction was confirmed by X-ray diffraction (XRD) using a Philips X-ray diffractometer. The evolution of microstructural properties was studied using an FEI Nova NanoSEM 50 series. The magnetic studies were carried out by using a Linkjoin Technology MATS-2010SD B-H Hysteresisgraph and an Agilent Model HP4291B Impedance/Material Analyzer. The XRD patterns showed an improvement of crystallinity with increasing sintering temperature. Ni-Zn ferrite peaks were the only observed peaks for samples sintered at 800^{o}C upwards for both sintering routes. SEM micrographs showed larger grain size as the sintering temperature increased, consequently increasing the multi-domain grains. The density values for the samples in both sintering routes proportionally increased with sintering temperature up to 1300^{o}C but dropped at 1400^{o}C showing an increased presence of pores. The real permeability, μ', and loss factor, μ", increased generally with increasing sintering temperature but dropped at the 1400^{o}C sintering temperature; the drop is attributed to increased porosity in grains. An integrated analysis of phase, microstructural, hysteresis and permeability data would point to the existence of three distinct shape-differentiated and sintering-temperature dependent groups of B-H hysteresis loops which belong to samples with weak, moderate and strong magnetism. One interesting result can be deduced from the phase, grain-size distribution and B-H curve data: the first appearance of strong ferromagnetic behaviour is indicated by the first occurrence of a strikingly erect sigmoid-shape B-H curve. That well-defined shape was observable only when sufficient single-phase purity and crystallinity and a sufficiently high volume fraction of grains with diameters >0.25 μm were attained. The B-H hysteresis shape evolution

was thus strongly influenced by the parallel evolution of the microstructure which developed from being predominantly single-domain to predominantly multi-domain. A scrutiny of the permeability parameters, μ' and μ", shows that these also seem to belong to the same three sintering-dependent groups mentioned above, consistent with the parameters' known dependence on the magnetization and the microstructure. The measured Curie temperature remained unaffected by the above evolutions since the exchange strength, on which the Curie temperature depends, is dependent only on the crystal structure and compositional stoichiometry. However the measured resistivity was found to decrease with increasing sintering temperature due to the combined effect of increased grain size and Fe^{2+} ions due to increasing zinc loss. Both single-sample and multi-sample sintering would led to highly similar results and conclusions.

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

PENJELASAN EVOLUSI SELARI SIFAT-SIFAT MIKROSTUKTUR, MAGNET DAN HUBUNGAN KEDUA-DUANYA DALAM FERIT NIKEL ZINK

Oleh

IDZA RIATI BINTI IBRAHIM

Disember 2011

Pengerusi: Prof. Madya Mansor Hashim, PhD

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Dalam kerja penyelidikan ini, kajian ke atas evolusi sifat-sifat mikrostruktur dan magnet dalam ferit nikel zink berkomposisi $Ni_{0.3}Zn_{0.7}Fe_2O_4$ telah dijalankan secara selari. Untuk beberapa dekad yang lalu, kajian ke atas hubungan sifat-sifat mikrostruktur dan sifat-sifat magnet hanya tertumpu kepada produk pada suhu pensinteran akhir dengan mengabaikan evolusi sifat-sifat mikrostruktur dan magnet yang selari pada pelbagai suhu pensinteran yang rendah. Oleh itu, kami melaporkan beberapa penemuan penyelidikan mengenai sifat-sifat magnet yang mempunyai sifat-sifat mikrostruktur tersebut. Ferit nikel zink telah disediakan dengan menggunakan pengisar bebola berkuasa tinggi selama 2 jam menggunakan pengisar SPEX8000D di dalam bekas keluli dan bebola terkuat. Sampel-sampel toroid melalui dua proses pensinteran iaitu pensinteran pelbagai sampel dan pensinteran sampel tunggal. Dalam pensinteran pelbagai sampel, sampel disinter dari 600^oC hingga 1400^oC dengan kenaikan 100^oC dengan menggunakan sampel berlainan bagi setiap suhu.

Tetapi, dalam pensinteran sampel tunggal pula, hanya satu sampel digunakan dan dilakukan pensinteran berulang ke atas sampel tersebut dari suhu 600^{o}C hingga 1400^{o}C dengan kenaikan 100^{o}C. Penyempurnaan tindakbalas keadaan pepejal diperiksa dengan menggunakan pembelauan sinar-x (XRD) dengan menggunakan Philips X-ray diffractometer. Evolusi sifat-sifat mikrostruktur dikaji dengan menggunakan FEI Nova NanoSEM 50 series. Kajian sifat-sifat magnet telah dijalankan dengan menggunakan B-H Hysteresisgraph Linkjoin Technology model MATS-2010SD dan Penganalisis Impedan/Bahan model Agilent HP4291B. Corak-corak pembelauan sinar-x menunjukkan peningkatan kehabluran dengan meningkatnya suhu pensinteran. Keseluruhan fasa ferit nikel zink mula ditemui pada suhu pensiteran 800^{o}C untuk kedua-dua proses pensinteran. Mikrograf-mikrograf mikroskop pengimbas elektron menunjukkan peningkatan saiz butiran dengan peningkatan suhu pensinteran, dan dengan itu telah meningkatkan juga butiran yang mempunyai domain berbilang. Nilai-nilai ketumpatan untuk sampel bagi kedua-dua proses pensinteran meningkat sehingga suhu 1300^{o}C, tetapi menurun pada suhu 1400^{o}C akibat peningkatan liang-liang. Ketelapan dan faktor kehilangan meningkat dengan peningkatan suhu pensinteran tetapi menurun pada suhu 1400^{o}C disebabkan oleh peningkatan liang-liang di dalam butiran. Suatu analisis fasa, mikrostruktur, histerisis dan ketelapan bersepadu akan menunjukkan kepada kewujudan tiga kumpulan gelung histeresis B-H yang berasaskan bentuk, dengan pergantungan kepada suhu pensinteran, yang mewakili sampel yang mempunyai kemagnetan lemah, sederhana dan kuat. Suatu penemuan yang menarik boleh disimpulkan dari fasa, taburan saiz butiran dan data lengkung B-H: kemunculan pertama sifat feromagnetik kuat ditunjukkan dengan kemunculan pertama lengkung B-H bentuk sigmoid yang ketara tegak. Bentuk yang jelas itu hanya mampu dilihat apabila

ketulenan fasa tunggal dan kehabluran yang mencukupi dan juga apabila terdapat pecahan saiz butiran melebihi 0.25 μm yang cukup tinggi dicapai. Maka evolusi bentuk histeresis B-H ternyata kuat dipengaruhi oleh evolusi selari mikrostruktur yang berkembang daripada sifat domain-tunggal menonjol kepada sifat domain-berbilang menonjol. Suatu penelitian parameter ketelapan, μ' dan μ", menunjukkan parameter-parameter ini juga tergolong kepada tiga kumpulan yang bergantung kepada suhu pensinteran seperti di atas, selaras dengan pergantungan biasa parameter-parameter ini terhadap pemagnetan dan mikrostruktur. Suhu Curie didapati tidak terkesan oleh evolusi di atas kerana kekuatan tukarganti yang menentukan suhu Curie hanya bergantung kepada struktur hablur dan komposisi stoikiometri. Bagaimanapun, kerintangan didapati menurun disebabkan oleh kesan gabungan pertambahan saiz butir dan bilangan ion Fe^{2+} daripada peningkatan kehilangan zink.

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I certify that a Thesis Examination Committee has met on 20 December 2011 to conduct the final examination of Idza Riati binti Ibrahim on her thesis entitled "Elucidation of the Parallel Evolutions of Microstructure and Magnetic Properties and Their Relationship in Nickel-Zinc Ferrite" 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 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 declare that this 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 other institutions.

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IDZA RIATI BINTI IBRAHIM

Date: 20 December 2011

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