UNIVERSITI PUTRA MALAYSIA

SYNTHESIS, MORPHOLOGY, CHARACTERISATION AND EVALUATION OF CARBON NANOTUBES-SUBSTITUTED YIG-PVA COMPOSITES AS ELECTROMAGNETIC WAVE ABSORBING MATERIALS

BEH HOE GUAN

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By

BEH HOE GUAN

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

July 2011

Dedicated to:

My parents, sisters, girlfriend and to all those who has supported me throughout my studies.

Abstract of the thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

SYNTHESIS, MORPHOLOGY, CHARACTERISATION AND EVALUATION OF CARBON NANOTUBES-SUBSTITUTED YIG-PVA COMPOSITES AS ELECTROMAGNETIC WAVE ABSORBING MATERIALS

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Chairman : Associate Professor Irmawati Bt Ramli, PhD

Institute : Institute of Advanced Technology

It is of interest whether carbon nanotubes (CNTs) and Yttrium Iron Garnet (YIG) containing polymer composite would produce any significant electromagnetic wave absorption effects in the composite. Therefore, in this work, carbon nanotubes-substituted YIG-PVA composites (YIG(La)-CNTs-PVA and YIG(Bi)-CNTs-PVA composites) were fabricated. Morphology and its electromagnetic properties as electromagnetic wave materials were studied. Yttrium iron garnet (YIG) substituted with lanthanum and bismuth namely $Y_{3-x}La_xFe_5O_{12}$ and $Y_{3-x}Bi_xFe_5O_{12}$ (where x=0.0, 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0) were synthesized via sol gel technique. The phase formation, surface morphology and magnetic properties of as-prepared YIG nanoparticles were studied using X-Ray diffraction (XRD), transmission electron microscopy (TEM) and vibrating sample magnetometer (VSM), respectively. The synthesized single phases YIG(La) and YIG(Bi) particles have spherical shapes with particles size in the range of 35-80nm. The saturation magnetization of YIG(La) and YIG(Bi) were 17.72emu/g and 18.57emu/g, respectively. Carbon nanotubes (CNTs)

which are used as filler for the composites were synthesized by the decomposition of methane over Co-Mo/MgO catalyst using chemical vapor deposition (CVD) technique. The ratio of Co to Mo supported on MgO and the catalytic reaction time were studied to optimize a CNTs yields. The optimization of CNTs yields was obtained by using the Co:Mo catalyst at ratio 1:1 supported on MgO and the catalytic reaction time of 180min. The microstructure and particles sizes of as-prepared CNTs were determined using TEM. 6M HNO_3 and 6M H_2SO_4 in a volume ratio of 1:3 were used to purify and functionalise the as-prepared CNTs. The purity of CNTs and the functional group introduced on CNTs after purification were determined by using Thermogravimetric Analysis **(**TGA) and identifying the groups using Fourier Transform Infrared Spectroscopy (FTIR), respectively. High purity CNTs of about 98.57% together with COOH and OH functional groups were obtained after purification process. The highest dielectric loss of CNTs-PVA composite was obtained with addition 3wt% of CNTs, while the highest dielectric and magnetic losses of YIG(La)-CNTs-PVA and YIG(Bi)-CNTs-PVA composites were obtained when 3wt% of CNTs were used. The highest tan δ obtained at YIG(La)-CNTs-PVA and YIG(Bi)-CNTs-PVA were 6.75 at frequency 100MHz and 1.12 at frequency 300MHz, respectively. It is suggested that the dielectric loss of the composites understudied are due to the interfacial polarisation of YIG(Bi) or YIG (La), CNTs and PVA within composite and magnetic losses are due to the domain wall movement of YIG(La) or YIG(Bi) in composite.

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

SINTESIS, MORFOLOGI, PENCIRIAN DAN PENILAIAN KOMPOSIT YIG-PVA TERTUKARGANTI TIUB KARBON NANO SEBAGAI BAHAN MENYERAP GELOMBANG ELEKTROMAGNETIK

Oleh

BEH HOE GUAN

Julai 2011

Pengerusi : Profesor Madya Irmawati Bt Ramli , PhD

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Suatu persoalan yang menarik adalah sama ada tiub karbon nano (CNTs) dan Yttrium Iron Garnet (YIG) mengandungi polimer komposit akan menghasilkan kesan menyerapan gelombang elektomagnetik yang ketara. Oleh itu, di dalam penyelidikan ini komposit YIG-PVA (YIG(La)-CNTs-PVA dan YIG(Bi)-CNTs-PVA) tertukarganti CNTs telah difabrikasi. Morfologi dan sifat keelektromagnetannya sebagai penyerap EMI telah dikaji. YIG terganti lantanum dan bismut iaitu $Y_{3-x}La_xFe_5O_{12}$ dan $Y_{3-x}Bi_xFe_5O_{12}$ (dimana x = 0.0, 0.5, 1.0, 1.5, 2.0, 2.5, dan 3.0) telah disintesiskan melalui teknik sol-gel. Kajian terhadap pembentukan fasa, morfologi permukaan, dan sifat kemagnetan bagi zarah nano YIG telah dijalankan masing-masing dengan menggunakan alat pembiasan sinar-X (XRD), pemancaran elektron mikroskopi (TEM) dan magnetometer getaran sampel (VSM). Didapati fasa tunggal YIG(La) dan YIG(Bi) yang terhasil memiliki bentuk sfera dengan saiz partikel dalam lingkungan 35-80nm. Ketepuan pemagnetan bagi YIG(La) dan YIG(Bi) adalah masing-masing 17.72emu/g dan 18.57emu/g. Di dalam kajian ini tiub nano karbon

yang digunakan sebagai pengisi komposit telah disediakan dengan menggunakan teknik pemendapan wap kimia (CVD) melalui penguraian metana dengan kehadiran Co-Mo/MgO sebagai pemangkin. Nisbah Co terhadap Mo tersokong MgO dan masa tindak balas telah dikaji untuk mengoptimumkan penghasilan CNTs. Pengoptimuman penghasilan CNTs telah diperolehi apabila menggunakan mangkin Co:Mo tersokong MgO pada nisbah 1:1 Co terhadap Mo dengan masa tindak balas selama 180min. Ciri-ciri mikrostruktur dan saiz partikel CNTs telah ditentukan dengan menggunakan TEM. Asid HNO_3 6M dan asid sulfurik 6M dalam ratio 1:3 telah digunakan bagi prosess penulenan dan kefungsian CNTs. Ujian terhadap tahap ketulenan dan kumpulan berfungsi yang terbentuk telah dijalankan dengan menggunakan analisis termogravimetri (TGA) dan fourier transformasi inframerah spektroskopi (FTIR). Ketulenan CNTs yang diperolehi adalah disekitar 98.57% dengan kumpulan berfungsi COOH dan OH. Penukargantian CNTs ke dalam PVA menghasilkan komposit CNTs-PVA dengan nilai kehilangan dielektrik tertinggi ditunjukkan apabila berat CNTs adalah 3%. Begitu juga kehilangan dielektrik dan kehilangan kemagnetan tertinggi diperolehi oleh komposit YIG(La)-CNTs-PVA dan YIG(Bi)-CNTs-PVA apabila berat CNTs adalah 3%. Tan δ tertinggi yang diperolehi di YIG(La)-CNTs-PVA dan YIG(Bi)-CNTs-PVA adalah masing-masing 6.75 pada frekuensi 100MHz dan 1.12 pada frekuensi 300MHz. Oleh itu dicadangkan bahawa kehilangan dielektrik CNTs dan PVA dalam komposit adalah disebabkan oleh polarisasi antara muka YIG(Bi) atau YIG(La), dan kehilangan magnetik adalah disebabkan oleh pergerakan dinding domain YIG(La) dan YIG(Bi) dalam komposit.

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I certify that a Thesis Examination Committee has met on 7 July 2011 to conduct the final examination of Beh Hoe Guan on his thesis entitled "Synthesis, morphology, characterization and evaluation of carbon nanotubes-substituted YIG-PVA composites as electromagnetic wave absorbing materials" in accordance with Universiti 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 candidate be awarded the Doctor of Philosophy.

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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 Doctor of Philosophy. The members of the Supervisory Committee were as follow:

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DECLARATION

I hereby declare that the thesis is based on 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.

BEH HOE GUAN

Date: 7 July 2011

TABLE OF CONTENTS