UNIVERSITI PUTRA MALAYSIA

MICROWAVE-ABSORPTION PERFORMANCE OF COMPOSITIONALLY-VARIED FERRITE-CARBON NANOTUBE-POLYMER COMPOSITE AND CVD-SYNTHESIZED CARBON NANOTUBE-POLYMER COMPOSITE

FADZIDAH BT MOHD IDRIS

ITMA 2016 14

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By

FADZIDAH BT MOHD IDRIS

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

November 2016

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DEDICATION

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**. Finally to my lovely husband **MOHD SHAMSUL EZZAD BIN SHAFIE** and my cute son **FIRAZ NURHAKEEM**.

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

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November 2016

Chairman: Associate Professor Mansor Hashim, PhD
Institute : Advanced Technology

Currently the research and development of radar absorbing materials (RAM) have increased where wide range of materials are used for the design, aiming to eliminate or reduce the spurious electromagnetic radiation levels more closely in different applications. The present research attempts to fabricate high absorbing material compositions suitable for microwave absorption from 8 to 18 GHz. Various microwave absorbing composites were fabricated using conventional solid state method and chemical vapor deposition method. There were various different materials being synthesized with different weight percentages, different mixed materials, different catalysts to synthesize carbon nanotube (CNT) and different thicknesses. However, only selected and outstanding results will be explained in details in this thesis. The materials being discussed were divided into four parts; Multiwalled Carbon Nanotubes (MWCNTs) mixed with Nickel-Zinc Ferrite ($Ni_{0.5}Zn_{0.5}Fe_2O_4$), as-synthesized CNTs catalyzed by mill scale, as-synthesized CNTs catalyzed by Nickel-Zinc Ferrite ($Ni_{0.5}Zn_{0.5}Fe_2O_4$) mixed with Cobalt Ferrite ($CoFe_2O_4$) and as-synthesized CNTs catalyzed by Nickel-Zinc Ferrite ($Ni_{0.5}Zn_{0.5}Fe_2O_4$) mixed with Carbonyl Iron. For mixed ferrite with MWCNTs, the starting raw metal oxide powder materials to produce ferrite materials were weighed and milled using the high energy ball milling technique to get nanometer starting particle size while MWCNTs was obtained from commercial source. The ferrite powders were then sintered at different sintering temperature according to different materials being synthesized. For CVD method, the catalyst powder was weighed and heated at 700^0C for 30 minutes. Argon gas and ethanol was used as the carrier gas and carbon source, respectively. The prepared composite samples were then incorporated into epoxy resin as a matrix with different ratio and poured into special manufacture sample holder with thickness kept at 1, 2 and 3 mm. The crystalline phase formation of all samples prepared was further investigated with an X-ray diffractometer (XRD). The particle size was measured using a transmission electron microscope (TEM). The microstructure of the samples was picture using a field emission scanning electron microscope (FESEM) measurement. The elemental analysis was measured using electron dispersive X-ray spectroscopy (EDX). The vibrational phonon modes were determined by Raman spectroscopy. The resistivity was measured using a resistivity measurement setup. The magnetic properties were measured using a Vibrating Sample Magnetometer (VSM). The scattering parameters were measured in the X and Ku-band regions by using a Vector Network Analyzer (VNA) within the frequency range from 8 GHz to 18 GHz. The XRD results for ferrite mixed with MWCNTs showed that the diffraction peaks were slightly shifted and the dominant peaks were given by ferrite materials since the weight percentages amount being added was higher. The average particle size of synthesized ferrite and average diameter of as-synthesized CNTs were in nanometers sized region which enhanced the absorption capability. The resulting aggregated morphology of as-synthesized CNTs were due to Van der Waals forces and this was resolved by incorporated them into an insulated polymer matrix. The carbon structures forms were mostly straight, spiral, hollow tube, netlike and twisted fiber which enhanced the ability of absorption. The EDX measurement for selected samples showed the elements presence in the composite samples. Raman spectrum showed defect (D-band) was higher than graphitize (G-band) which attributed to defects in the tube ends, staging disorders, hollow tube and curved graphene layers. For ferrite mixed with MWCNTs, it was found that as the amount of MWCNTs increased, the coercivity (H_c) of composites increased while the saturation magnetization (M_s) and retentivity (M_r) both decreased. For measurement at higher frequency (X-band and Ku-band), thicker samples resulted in higher microwave absorption. As the thickness increased, reflection loss peak shifted towards lower frequency range due to shifted of matching frequency. For thickness of 3 mm, the reflection loss reached -17 dB at 9.5 GHz for 2 wt% MWCNTs- $Ni_{0.5}Zn_{0.5}Fe_2O_4$ /P with bandwidth 3.5 GHz. As for as-synthesized CNT by mill scale milled at 20 hours for thickness of 3 mm, the value of reflection loss was -25 dB. As for as-synthesized mixed ferrite with thickness of 3mm, the reflection loss of sample with as-synthesized (80% NZF + 20% C)/P gave the most minimum reflection loss value of -29 dB at around 12.5 GHz with bandwidth 4 GHz (10.5 – 14.5 GHz) and -26 dB at 9.5 GHz for as-synthesized (20% NZF + 80% CI)/P. The prepared as-synthesized CNT polymer composites were expected to be very useful in many application especially military applications such as radar cross section reduction and for prevention of electromagnetic interference.

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

PRESTASI PENYERAPAN-MIKROGELOMBANG DARI PELBAGAI-KANDUNGAN FERIT-KARBON NANOTIUB-POLIMER KOMPOSIT DAN CVD-SINTESIS KARBON NANOTIUB-POLIMER KOMPOSIT

Oleh

FADZIDAH BT MOHD IDRIS

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Pada masa kini penyelidikan dan pembangunan bahan-bahan penyerap radar (RAM) telah meningkat dimana pelbagai bahan yang digunakan untuk reka bentuk, bertujuan untuk menghapuskan atau mengurangkan tahap radiasi elektromagnet palsu lebih rapat dalam aplikasi yang berbeza. Kajian masa kini cuba untuk mereka komposisi bahan penyerap yang tinggi sesuai untuk penyerapan mikro gelombang 8 hingga 18 GHz. Pelbagai komposit penyerap mikro gelombang telah direka menggunakan kaedah konvensional keadaan pepejal dan kaedah pemendapan wap kimia. Terdapat pelbagai bahan yang berbeza yang disintesis dengan peratusan berat bahan yang berbeza, bahan-bahan campuran yang berbeza, pemangkin yang berbeza untuk mensintesis CNT dan ketebalan yang berbeza. Walau bagaimanapun, hanya dipilih dan keputusan yang cemerlang akan diterangkan secara terperinci dalam tesis ini. Bahan-bahan yang dibincangkan telah dibahagikan kepada empat bahagian; Multidinding nanotiub karbon (MWCNTs) bercampur dengan nikel zink Ferit ($Ni_{0.5}Zn_{0.5}Fe_2O_4$), Multidinding nanotiub karbon (MWCNTs) dicampur dengan Cobalt Ferit ($CoFe_2O_4$), sedia-disintesis CNTs dimangkinkan oleh sisik besi, sedia-disintesis CNT dimangkinkan oleh Nikel -Zinc Ferit (Ni0.5Zn0.5Fe2O4) dicampur dengan Cobalt Ferit ($CoFe_2O_4$) dan sedia-disintesis sebagai CNT dimangkinkan oleh Nikel-zink Ferit ($Ni_{0.5}Zn_{0.5}Fe_2O_4$) dicampur dengan karbonil ferit (CI). Untuk ferit dicampur dengan MWCNT, bermula bahan mentah logam oksida bagi menghasilkan bahan ferit adalah ditimbang dan digiling menggunakan teknik tenaga gilingan bola yang tinggi untuk mendapatkan bermula saiz zarah nanometer manakala MWCNT digunakan adalah dari sumber komersial. Serbuk ferit kemudian dibakar pada masa pembakaran yang berbeza mengikut perbezaan bahan yang disintesis. Bagi kaedah CVD, serbuk pemangkin telah ditimbang dan dipanaskan pada 700^0C selama 30 minit. Gas Argon dan etanol digunakan sebagai gas pengangkut dan sumber karbon. Sampel komposit disediakan kemudian dimasukkan ke dalam resin epoksi sebagai matriks dengan berbeza nisbah dan dituangkan ke dalam pembuatan istimewa pemegang sampel dengan ketebalan ditetapkan pada 1, 2 dan 3 mm. Pembentukan fasa kristal semua sampel disediakan telah disiasat lanjut dengan sinar-X pembelauan (XRD). Saiz zarah diukur menggunakan mikroskop elektron penghantaran (TEM). Mikrostruktur sampel diambil menggunakan pelepasan bidang imbasan mikroskop elektron (FESEM) ukuran. Analisis unsur diukur menggunakan elektron serakan spektroskopi sinar-X (EDX). Mod getaran fonon ditentukan oleh Raman spektroskopi. Kerintangan diukur menggunakan ukuran kerintangan. Sifat-sifat magnet telah diukur menggunakan Vibrating Sample Magnetometer (VSM). Penyerakan diukur dalam kawasan jalur-X dan Ku dengan menggunakan Network Analyzer Vector (VNA) dalam julat frekuensi daripada 8 hingga 18 GHz. Keputusan XRD bagi ferit bercampur dengan MWCNT menunjukkan bahawa puncak pembelauan telah sedikit beralih dan puncak dominan telah diberikan oleh bahan-bahan ferit setelah peratusan jumlah berat yang ditambah adalah lebih tinggi. Purata saiz zarah ferit disintesis dan diameter purata sedia-disintesis CNT berada dalam kawasan bersaiz nanometer yang telah meningkatkan keupayaan penyerapan. Hasil gumpalan morfologi sedia-disintesis CNT telah disebabkan oleh Van der Waals dan ini diselesaikan dengan menggabungkan mereka ke dalam matriks polimer penebat dengan dicampur menggunakan adunan kelajuan tinggi. Bentuk struktur karbon kebanyakannya lurus, lingkaran, tiub berongga, seperti jaring dan serat berpintal yang meningkatkan keupayaan penyerapan. Pengukuran EDX untuk sampel yang dipilih menunjukkan kehadiran unsur dalam sampel komposit. Raman spectrum menunjukkan kecacatan (jalur-D) adalah lebih tinggi daripada grafitic (jalur-G) yang disebabkan oleh kecacatan pada hujung tiub, gangguan penyusunan, tiub berongga dan lapisan graphene melengkung. Untuk ferit dicampur dengan MWCNT, didapati bahawa jumlah MWCNTs meningkat, coercivity (Hc) komposit meningkat, manakala pemagnetan tepu (Ms) dan baki pemagnetan (Mr) kedua-dua menurun. Untuk mengukur pada frekuensi yang lebih tinggi (jalur-X dan jalur-Ku), sampel tebal menyebabkan penyerapan gelombang mikro yang lebih tinggi. Apabila ketebalan meningkat, perubahan puncak kehilangan pantulan ke arah julat frekuensi yang lebih rendah kerana beralih yang hampir sama frekuensi. Untuk ketebalan 3 mm, kehilangan pantulan mencapai -17 dB pada 9.5 GHz bagi 2 wt% MWCNTs-Ni0.5Zn0.5Fe2O4 /P dengan lebar jalur 3.5 GHz. Bagi sedia-disintesis CNT oleh sisik besi gilingan pada 20 jam untuk ketebalan 3 mm, nilai kehilangan pantulan adalah -25 dB. Bagi sedia-disintesis ferit campuran dengan ketebalan 3mm, kehilangan pantulan sampel dengan sedia-disintesis (80% NZF + 20% C)/P memberikan nilai kehilangan pantulan paling minimum -29 dB pada kira-kira 12.5 GHz dengan lebar jalur 4 GHz (10.5 14.5 GHz) dan -26 dB pada 9.5 GHz untuk sedia-disintesis (20% NZF + 80% CI)/P. Polimer komposit yang disediakan dijangka menjadi sangat berguna dalam aplikasi ketenteraan seperti seksyen pengurangan rentas radar dan pencegahan gangguan elektromagnet.

ACKNOWLEDGEMENTS

It is neither my strength nor my wisdom, but Allah's mercies that made this work a success, thus, I glorify Him. May all praises and salutations of the Lord be upon the Messenger of Allah and upon his Family and Companions, and those who are guided by the light of his 'sunnah' till the Day of Judgment.

I would like to take this opportunity to express my deepest gratitude to my late project supervisor, Assoc. Prof. Dr. Mansor Hashim for his support, great scientific points and tips given with friendly guidance and advice during this study. His encouragement, moral and technical support made this study possible. I would also like to thank to him for giving me the opportunity to work with him and giving me guidance at every step of my work. In fact, he had spent his precious time just to help me during this research. He is such a kindly and knowledgeable supervisor. May Allah reward him with the best of rewards and granted him with Jannah. I would also like to extent my sincere thanks to my co-supervisor, Assoc. Prof. Dr. Zulkifly Abbas, Assoc. Prof. Dr. Jumiah Hassan and also to all my lecturers for their helps throughout this project.

I would like to express my appreciation and thankful to my beloved family who have given me their full moral support although they are not in this field. They always be by my side whenever I am facing problems. In my hard times, they pushed me in the right way. I owe my most sincere thanks to the most important persons in my life, my mother, Hjh Sabariah Bt Mohd Yatim and my lovely sisters, Zanariah and Zuraidah, whose honest support and love gave me energy to complete this work successfully. I am really indebted to them for their warm support and prayers. Al-fatihah to my late father, Allahyarham Mohd Idris Bin Shariat. May his soul rest in peace and be placed amongst the blessed alongside Allah S.W.T. To my cute little niece, Nurnabilah, Muhammad Al-Naim and Muhammad Al-Naufal for cheering me up when I feel down. To my lovely husband, thank you for your love and support. May Allah give us full of happiness in our marriage with our newborn son Firaz Nurhakeem. Thank you very much for joining me "in sacrificing today for a better tomorrow" I LOVE YOU ALL and may Allah reward you with the best of rewards.

To my colleagues in the Magnetics and Nanostructure Evolution Group (MNEG), Dr. Ismayadi, Idza, Rodziah, Ghazaleh, Nora, Hapishah, Mehran, Misbah, Ikhwan, Aizat, Syazwan and Che Sulaiman, I appreciate the discussions, ideas, memorable interactions and time spent throughout this research. Thank you for their support in allowing me to work closely with them, without their support I would not be able to complete this thesis. I am really obliged to them. To my fellow friends during my postgraduate study, Nurzila, Dila, Zarifah, Rahimah, Mutia, Dayang, Fauzana, I say thanks for everything and thanks for the memories together along journey of my studies. Not forgotten to Dr. Shuhazly, Dr. Norlaily and Dr. Rabaah for sharing their ideas throughout my studies.

I also would like to express my thanks to staffs at the Institute of Advanced Technology (ITMA) and Faculty of Science, En. Kadri, En. Azli, Pn. Noor Lina, Pn.

Sarinawani, Pn. Rosnah, Pn. Norhaslinda, Pn. Noriza, Pn. Khamsiah and many others for their great helps and contributions. Finally, my thanks goes to those who have help me directly or indirectly in finishing this research.

I certify that a Thesis Examination Committee has met on 2 November 2016 to conduct the final examination of Fadzidah bt Mohd Idris on her thesis entitled "Microwave-Absorption Performance of Compositionally-Varied Ferrite-Carbon Nanotube-Polymer Composite and CVD-Synthesized Carbon Nanotube-Polymer Composite" 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 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 follows:

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XRD	X-Ray Diffraction
FESEM	Field Emission Scanning Electron Microscopy
EDX	Energy Dispersive X-ray
TEM	Transmission Electron Microscopy
VSM	Vibrating Sample Magnetometry
RAMAN	Raman Spectroscopy
RAM	Radar Absorbing Material
EMI	Electromagnetic Interference
FMR	Ferromagnetic Resonance
UPM	Uniform Precessional Mode
VNA	Vector Network Analyzer
CVD	Chemical Vapor Deposition
MWCNT	Multi-walled Carbon Nanotube
CI	Carbonyl Iron
N	Nickel Zinc ferrite
F	Magnetite
C	Cobalt ferrite
B	Barium Hexaferrite
MS	Mill scale
G	Carbon graphite
CNT	Carbon Nanotube
ICDD	International Centre for Diffraction Data
wt %	Weight percent

MUT	Material under test
BPR	Ball-to-powder weight ratio
MA	Mechanical alloying
HEBM	High-energy ball milling
a. u.	Arbitrary unit
M	Magnetization
H_c	Coercivity field
M_S	Saturation magnetization
M_r	Retentivity
μ’	Real part of permeability/initial permeability
μ”	Imaginary part of permeability/loss factor
ε’	real part of permittivity
ε”	imaginary part of permittivity
2θ	2 theta degree
K	magnetic anisotropy constant
H_a	anisotropy field
RL	Reflection loss (Return loss)
ρ	Resistivity

CHAPTER 1

INTRODUCTION

1.1 Motivating Background of the Study

Recently, rapid development of local electronic devices, microwave communication and the pollution of electromagnetic interference strongly require electromagnetic wave absorbing materials with favorable properties such as low density, low cost and strong absorption in a wide frequency range (Wang et al., 2010). In fact, the rapid development of gigahertz application devices results in serious electromagnetic (EM) interference pollution. The electromagnetic interference pollution can cause disturbances on the equipment and systems for medical, industrial, commercial, and military applications. Microwave radiation is also potentially harmful to biological systems which are continuously exposed to microwave for a considerable period of time. Electromagnetic interference shielding or microwave absorbing materials have been used to attenuate those unwanted electromagnetic energies, which is an important issue to be considered for both civil and military purposes. Therefore, considerable attention has been devoted to the effective electromagnetic wave absorbing materials with lightweight and strong absorption over a broad frequency spectrum (An et al., 2009; Thomassin et al., 2007; Xu et al., 2007; Che et al., 2004).

Those materials with the capability of absorbing electromagnetic signal are widely applied in industrial, commercial and military fields (Tong et al., 2011; Zeng et al., 2010; Zhou et al., 2007). For example, in military applications, particularly in an antiradar system the used materials should have a strong absorbing effect. Thus, EM wave absorbers with wider absorption bandwidths and better absorption properties become more and more important (Qing et al., 2010; Kim et al., 2005). As in current computers, the working frequencies of wireless communication devices and clock frequency of CPU are from hundreds of MHz to several GHz. The broadband absorbers will be preferable to suppress the EM interferences between such signals with different frequencies. Thus, much attention has been paid to radar absorbing materials (RAMs) due to their unique absorbing microwave energy and effectively reducing electromagnetic backscatter. They are specially designed material to suppress the reflected electromagnetic energy incident on the surface of the absorber by dissipating the magnetic and/or electrical fields of the wave into heat. The excellent RAMs should have certain properties as follows: 1) exhibit strong microwave absorption properties over a wide frequency range; 2) need to be thin and lightweight, especially for aircraft; 3) have simple coating-layer structure. Extensive study has been carried out to develop new microwave absorbing materials with a high magnetic and electric loss (Ruan et al., 2000; Babbar et al., 2000).

For the sake of better microwave absorbing capability, a wide range of radar absorbing materials have been fabricated up to now such as powder metals, carbonyl composites, conductive magnetic fibers, hard and soft ferrites and etc. In fact, the fabricated absorbing material should have electric and/or magnetic dipoles in order to interact with the electromagnetic fields in the radiation. Thus, an absorbing material can be classified as magnetic, dielectric or hybrid (a combination of magnetic and dielectric) since pure dielectric or magnetic materials are insufficient for absorbing radiation energy. Other than that, new systems have also been evolved comprising composite powder containing hard and soft magnetic materials. Moreover, with advances in nanotechnology nano microwave absorbers have played an important role in developing better absorbing material. All in all, it would help to develop a proper perspective by having better understanding on the underlying principles of RAM analysis, design and fabrication methods, and the identification of the actual RAM materials. It may also facilitate pioneering the next generation of materials and technology of RAM.

1.2 Overview: Historical Perspective to the Evolution of Radar Absorbing Materials (RAM)

Early research on radar absorbing materials (RAM) was started during 1930’s while the commercial production of RAM started to grow and develop in 1950’s. The first absorber material to be patented was developed in 1936 at Naamlooze Vennootschap Machinerieen, in the Netherlands. In the research, carbon black and TiO_2 was used as a quarterwave resonant material in the 2 GHz region to achieve dissipation and high dielectric constant respectively.

During the World War II, the advents of radars causing absorbers were intensified. According to Schade, 1945 and MacFarlane, 1945, there were two types of materials being used in submarines which were successfully developed in Germany. The first material called Wesch and the second was Jaumann absorber. Wesch material consisted of a semiflexible rubber sheet loaded with carbonyl iron while later material was developed by a multilayer approach. The reflection coefficients were typically lower than -20 dB over a wideband (2 – 15 GHz) for both absorbers for near normal incidence.

According to Emerson, 1973, two types of absorbers (airbone version and rugged shipboard version) were developed during the same time in US which generically called HARP (Halpern-anti-radar-paint). These absorbers offered a reflection reduction of 15 – 20 dB at resonance. The airbone version (MX-410) utilized artificial dielectric materials of high relative permittivity which was actually akin to paint. This version could operate in the X-band with a typical thickness of 0.07 inches while having broad absorption around the resonant frequency (Montgomery et al., 1948). As for rugged shipboard version, it consists of a high concentration of iron particles in a neoprene binder.

A well-known absorber being approached consists of a resistive sheet which was placed at quarter wavelength from the scatterer, was spaced by a low dielectric material. This absorber was known as the Salisbury screen which developed around the same time as HARP (Salisbury, 1952) and was widely used in earlier anechoic chamber designs that operate by the resonant technique. Later, Ruck et al. (1970) introduced another resonant absorber in wide use which was known as the Dallenbach layer. This new approached consisted of a homogeneous lossy layer on a metal plate which the thickness of the lossy layer was selected such that its input impedance matched the intrinsic impedance of the free space.

As discussed above, the absorbers developed in earlier stages were mostly narrowband in nature. As a result, more research was needed towards obtaining broadband RAM. Probably the very first approach to be tried in this context which most practical broadband RAM were constructed was a multilayer structure (Jaumann absorbers discussed above). For example, the broadening of the bandwidth of Salisbury screens was achieved by introducing additional layers of resistive sheets and spacers (Emerson, 1973).

In some applications, bulk absorbers were also used replacing sheets or layers. The conductivity of the bulk material reduces upon infusion of carbon in a matrix of spongy urethane foam. Based on this approach, in the early 1950's, the US Naval Research Laboratory developed a broadband absorber. The conductivity was reduced by dipping or spraying carbon onto a mat of loosely spun animal hair. These bulk absorbers could be readily used in order to control reflections in both indoor (anechoic) and outdoor antenna measurement ranges.

In the 1960's, the possibility of using magnetic materials (e.g., the ferrites) as an absorbing materials was explored intensively. Crispin & Siegel (1968) reported that thin magnetic RAM with low reflection coefficient that was fabricated could operate at lower resonant frequencies that had earlier been feasible with the dielectric materials. However, these materials are much heavier although they were thinner compared to the dielectrics. In addition, they are prone to disintegration at higher frequencies.

It came to light in the 1980's by serendipity that some biotechnology products have ultrawide band absorption characteristics. Therefore, subsequent experiments being carried out and it confirmed that substantial RCS reduction was indeed possible by dissolving these compounds in aircraft structural materials. Following this, the Department of Defense, US, immediately classified the compound. However, there are conjectures that this ultrawide band RAM is a retinyl compound which belongs to the class of Schiff-base salts. This is a powdery black substance which is much lighter than the ferrites and it is also said to have been used in making RAM paints for stealth aircraft.

According to Jaggard & Enghetta (1989), a class of homogeneous materials has also being found to be used as RAM. It is by making use of the chiral property which depend upon optical activity and circular dichroism and the backscatter RCS which is independent of polarization. On the other hand, the chiral RAM have vastly superior RCS characteristics as compared to the dielectric and magnetic RAM discussed above.

Finally, in the 1990's certain polymers when doped showed finite conductivity was discovered. In the context of RAM, it is important to have such conducting polymers since the conductivity provides a loss mechanism by attenuating the EM waves within the polymers. Typical conducting polymers are polyaromatic in nature, such as polyaniline and polypyrrole whereas dopants consist of iodine, bromine, ferric chloride etc. Thus, controlling the dopant concentration can tuned or varied the conductivity of these polymers. This provides an opportunity in designing RAM in order to fulfill the properties/characteristics needed for certain application. Advances in the 1990's, led to 3-dimensional inorganic carbon crystals (the fullerenes) which yet has been reported that C_{60} and other fullerenes when doped results in very effective RAM.

1.3 Selection of Materials

Reflection loss values can be altered in order to achieve maximal absorption of the electromagnetic energy. Nowadays, considerable efforts have been made to design various materials in order to reach the targets. Consequently, in this research various types of materials have been prepared in order to be the candidate as good absorbing materials. The overall main materials to be used as fillers are nickel zinc ferrite, barium hexaferrite, cobalt ferrite, magnetite, carbonyl iron and multiwalled carbon nanotubes. These are all the main starting materials to be used with each of the materials plays their role in obtaining higher absorption. The reasons of choosing those materials are stated in the Table 1.1 below:

Table 1.1: List of materials and reasons for choosing them as an absorbing material.

Materials	Reason
Nickel Zinc Ferrite ($Ni_{0.5}Zn_{0.5}Fe_2O_4$)	$Ni_{0.5}Zn_{0.5}Fe_2O_4$ has moderate saturation magnetization, remarkable chemical stability, and a good mechanical hardness. Their high resistivity is found to be the most versatile technological materials especially suited for high frequency applications. They also have an advantage in applications which can cover from low to microwave frequencies and low to high permeability. It is very useful especially in the microwave frequency range for having a resonant frequency at high frequency.

Barium Hexaferrite ($BaFe_{12}O_{19}$)	$BaFe_{12}O_{19}$ is one of the high performance permanent magnet with fairly large magnetocrystalline anisotropy, high curie temperature, relatively large saturation magnetization and high coercivity. Moreover, it also has high resistance, excellent thermal and chemical stability and corrosion resistivity. Therefore they can be used as microwave absorbers in the GHz frequency range.
Cobalt Ferrite ($CoFe_2O_4$)	$CoFe_2O_4$ is one of the rapid relaxing ions which are good in terms of absorbing and releasing the energy. By having high anisotropy field, it allows absorbing the microwave energy efficiently by precessing the magnetic moment. Moreover, by having large saturation magnetization and high Snoek's limit, it results in high complex permeability values at a wide frequency range. Thus, it makes $CoFe_2O_4$ is highly useful as a thin absorber working at a high-frequency range.
Magnetite (Fe_3O_4)	Fe_3O_4 is a magnetic material with high dielectric permittivity, thus it exhibits both magnetic and dielectric losses. Fe_3O_4 behaves as a total reflector when irradiated by an electromagnetic wave. However, by incorporating into insulating matrix, it may reduce the low dielectric permittivity to ensures low reflection from the sample's front surface.
Carbonyl Iron, $Fe(CO)_5$ (CI)	Carbonyl iron is particles widely used as microwave absorption filler having large saturation magnetization and high Snoek's limit which result in high complex permeability values at a wide frequency range. This factor makes CI particles are useful for microwave absorber at a high-frequency band.
Multiwalled Carbon Nanotubes	Multiwalled carbon nanotubes have properties of lightweight, high specific surface areas, low percolation and high electrical property. It also offers a large flexibility for design and control of microwave absorption behaviors as it being incorporated into polymer. The composites can be tailored through changes in loading fractions, matrix materials, complex permittivity and loss tangent of MWCNTs-polymer composites. The incorporation of CNT may also enhance the thermal stability of the composite as well as possess high real and imaginary permittivity. Thus, by having small additions of MWCNTs, the values of complex permittivity may increase dramatically. Moreover, an optimum conductivity is needed for good microwave absorption since low conductivity may cause partial absorption of the microwave

	whereas highly conducting material may transmits or reflects the wave without any absorption. Thus, the advantages of having free electrons in its skeleton, it can absorb the energy and helps in attenuating it as well. Further the high surface area of MWCNT also may be a reason for its good microwave absorption behavior.

Instead of inorganic samples, this research work have also introduced materials from industrial byproduct (steel mill scale) and agricultural waste (organic) sample which may help in obtaining good microwave absorbers. Mill scale is the flaky surface of hot rolled steel which consist of iron(II) oxide (FeO), iron(III) oxide (Fe_2O_3) and iron(II, III) oxide (Fe_3O_4, magnetite). It was used in order to synthesis of low cost material for lightweight absorber. In addition, iron content is dominant and mill scale can be divided into three types as electric loss, magnetic loss and dielectric loss materials. On the other hand, a coconut husk is an example of organic samples which contains 30wt% of coir fibres and 70wt% coir dust which is also known as coco peat. The coco peat contains lignin, cellulose and hemi-cellulose which are potentially useful as a microwave absorber. The content of carbon in coco peat can also reach to 50%. Carbon plays an important role in microwave absorption since it may easily heated by microwave energy which is suitable for transforming the microwave energy to thermal energy because carbon is impeded as microwave signal pass through. An electric field is produced at the surfaces of the absorber as the microwave pass through the carbon based absorber. Thus, the electrical energy is then transformed into thermal energy and is dissipated.

1.4 Problem Statement

Aiming to eliminate or reduce the spurious electromagnetic radiation levels more closely in different applications, the research and the development of radar absorbing materials (RAM) have increased. However, current technology lacks on the fabrication of microwave absorbing materials with the ability of absorbing electromagnetic radiation in a broad frequency range. The actual barrier to achieve this goal is the lack of detailed scientific understanding of how the molecular units of materials and their combinations reflect, absorb and transmit the incident microwave energy. Therefore, extensive studies, as to be attempted in this research work, on microwave absorption properties of various materials need to be synthesized and investigated to look for the microwave absorbing materials with high absorptive ability and wide band absorption suitable at frequency 8 to 18 GHz.

1.5 Objective

The objective of this research is to fabricate various microwave absorbing composites which are later to be used in microwave absorbing applications. The research will concentrate on observing the behavior of significantly important material properties related with microwave absorption as well as the performance of the material when introduced as a microwave absorber. Thus, this study aims to explore all the information about microwave absorbers in order to scientifically understand the underlying microwave absorption mechanisms through their basic theories.

Work-phase Objectives

1. To synthesize and investigate absorption properties of fillers by varying its materials content and thickness to be incorporated into matrix that would increase the absorption at frequencies 8 to 18 GHz (X to Ku band).
2. To optimize the setup of the reflection loss measurement that would reduce the air gap.
3. To analyze the properties in obtaining the possible materials to be used as an absorber that would increase the performance of reflection loss ≤30 dB.
4. To study various properties especially microwave absorption properties from the research that would increase the microwave absorbing performance.

1.6 Thesis Overview

A general introduction on background of the study, overview historical perspective of the evolution of RAM, problem statements and objectives are discussed in Chapter one. As for Chapter two, the relevant EM parameters and phenomena associated with RAM, which are used in the analysis and design in the later chapters, are also identified and explained. The related literatures on finding the suitable microwave absorbers to solve the electromagnetic interference (EMI) problem are also discussed in this chapter. The attention of the reader is also drawn towards the new approach form this research work. The background theory on magnetism was explained in Chapter three. In addition to that, the important basic theory and background to the process of microwave excitation and the different ways the microwave energy is lost to the system are further explained in detail. Chapter four explained in detail preparation of the samples and reviews the methods in measuring microwave and reflection properties using microwaves characterization equipment. The analysis of the results based on the design of the various types of RAM that were considered and all measurements are discussed in Chapter five. Finally, the research findings are summarized and concluded in Chapter six. In addition to the last chapter, there are some suggestions for further research and the thesis also includes an extensive bibliography of all literature being used.

1.7 Limitations of the study

Although the objectives and scopes in this thesis had been thoroughly investigated and studied, there are few limitations regarding to the research:

1. The measurement was carried out in the X-Band (8-12GHz) and Ku-band (12-18GHz) frequency due to the limitation in experimental equipment constrained.
2. The results of the test involving free space measurement would be best executed in a wideband condition i.e.: 1-18GHz and in anechoic chamber, where most research on the practical absorber was done. However, the horn antenna arrangement was not employed since the absorber plate area needs to be large enough, therefore requiring a rather large amount of expensive ferrite powder. Also it would be difficult to obtain a highly uniform thickness of the ferrite-loaded paint since the plate area required is large (≥ 30 cm × 30 cm).

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