

Article

Enhancing Microwave Absorbing Properties of Nickel-Zinc-Ferrite with Multi-walled Carbon Nanotubes (MWCNT) Loading at Higher Gigahertz Frequency

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Abstract— The rapid growth of electronic systems and devices operating within the gigahertz (GHz) frequency range has increased electromagnetic interference. In order to eliminate or reduce the spurious electromagnetic radiation levels more closely in different applications, there is strong research interest in electromagnetic absorber technology. Moreover, there is still a lack of ability to absorb electromagnetic radiation in a broad frequency range using thin thickness. Thus, this study examined the effect of incorporating magnetic and dielectric materials into the polymer matrix for the processing of radar absorbing materials. The experiment evaluated the sample preparation with different weight percentages of multi-walled carbon nanotubes (MWCNT) mixed with Ni_{0.5}Zn_{0.5}Fe₂O₄ (Nickel-Zinc-Ferrite) loaded into epoxy (P) as a matrix. The prepared samples were analysed by examining the reflectivity measurements in the 8 – 18 GHz frequency range and conducting a morphological study using scanning electron microscopy analyses. The correlation of the results showed that different amounts of MWCNT influenced the performance of the microwave absorber. As the amount of MWCNTs increased, the reflection loss (RL) peak shifted towards a lower frequency range and the trend was similar for all thicknesses. The highest RL was achieved when the content of MWCNTs was 2 wt% with a thickness of 2 mm with an RL of – 14 dB at 16 GHz. The 2.5 GHz bandwidth corresponded to the RL below -10 dB (90% absorption) in the range of 14.5 – 17 GHz. This study showed that the proposed experimental route provided flexible absorbers with suitable absorption values by mixing only 2 wt% of MWCNTs.

Keywords— magnetic; dielectric; composite; reflection loss.

I. INTRODUCTION

Microwave absorbing materials have continued to attract significant interest in recent decades, for both commercial and military purposes [1] in the reduction of spurious radar cross-sections (RCS) as well as in the telecommunications field, particularly in the gigahertz (GHz) frequency. Hence, microwave absorbers have been explicitly designed to remove the reflected electromagnetic energy that exists on the absorber's surface. This can be achieved by dissipating the magnetic and/or electrical fields of the wave in the form of heat since electromagnetic waves have both components. The

loss of absorber characteristics may help to dissipate the signals by attenuating signals entering the absorber. Compounds of relatively heavy metals or ceramics are typical magnetic absorbers that play a role in regulating both complex permeability and permittivity. These materials are often used at high mixing ratios, which result in high aerial weight and the addition of more magnetic materials resulted in magnetic fillers having low complex permeability within the GHz frequency range.

Conductive fillers such as carbon black (CB), multi-walled carbon nanotubes (MWNT), carbon nanofibre (CNF), short

carbon fibre (SCF), silver powder, etc. have been used as dielectric absorbers [2-5]. These conductive fillers, with relatively low density and low mixing ratios, were used to control the complex permittivity alone. Carbon nanotubes (CNT) have been found to have an overwhelming response in CNT-based product development studies. Intensive investigations have therefore been carried out to test them as a potential microwave absorber [6].

$\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ (Nickel-Zinc-Ferrite) has moderate saturation magnetisation, remarkable chemical stability, and good mechanical hardness, and is the most versatile technological material specially suited for high-frequency applications due to its high resistivity. It also has an advantage in applications that covers low to microwave frequencies and low to high permeability. Furthermore, it is very useful, especially in the microwave frequency range, for having a resonant frequency at a high-frequency. However, Nickel-Zinc-Ferrite ($\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$) alone cannot satisfy the demand of being the “ideal” absorbing material, having defects including a narrow absorbing band and mass density. Therefore, it is important to match the magnetic and dielectric loss components simultaneously to increase reflection loss (RL). In order to meet the requirements of thin thickness, lower weight, wider absorption bandwidths, and strong absorbing characteristics, Nickel-Zinc-Ferrite was gradually substituted by new absorbing materials such as ferrite-CNT composites, which are widely regarded as efficient wave absorbing materials. In this study, the NZF-MWCNTs/P (epoxy) composites were synthesised using a solution casting method and the aim was to investigate the effect of different contents of MWCNTs on the magnetic properties, electromagnetic parameters, and wave absorbing properties of the composites.

II. METHODOLOGY

A. Synthesis of ferrite sample

Nickel-Zinc-Ferrite with a composition of Ni content ($x = 0.5$) was prepared using a conventional method. The starting raw materials of Nickel (II) Oxide (NiO) 99%, Zinc Oxide (ZnO) 99.9%, and Iron (III) Oxide (Fe_2O_3) 99.5% from Alfa Aesar were used without further purification. They were weighed according to intended proportions and mixed for homogeneity using ball milling. A SPEX8000D mill was used to mix the powder in a hardened steel vial and further milled together with 10 grinding balls with 12 mm diameter to produce nanosized particles. For the preparation of ferrite nanoparticles, the milling time and ball to powder weight ratio (BPR) were set at 10 hours and 10:1, respectively. The powders were then sintered at 900 °C for 10 hours using a heating rate of 4 °C/min and the obtained Nickel-Zinc-Ferrite phase was confirmed by X-ray Diffraction (XRD) analysis. Heating at a moderately low temperature of – 900 °C was required to form the full phase. Samples sintered at 900 °C contained a mixture of single-domain and multi-domain grains. A mixture of various grain sizes was important to achieve significant microwave absorption in a wide frequency range.

B. Mixing filler incorporated into a polymer matrix

The total weight percentages of the synthesised ferrite sample mixed with commercial MWCNTs (< 10 nm diameter, 5 – 15 μm length, 95 % purity) were fixed at 60 wt%, while the amount of polymer matrix was fixed at 40 wt%, as these were the optimised weight percentages. The composite samples were synthesised by incorporating different amounts of MWCNTs (2 wt%, 4 wt%, and 6 wt%) mixed with ferrite and dispersed into epoxy as a matrix. The mixtures were then distributed and uniformly mixed using a high-speed mixer at 3000 rpm in 20 minutes. Subsequently, a hardener was added as a curing agent to the mixtures and regularly mixed. The addition ratio for the epoxy resin and hardener was 10:1. Prepared samples were then poured into a sample holder of different thicknesses (for X-band and Ku-band) of 1 mm, 2 mm, and 3 mm, and the polymer composite samples were cured overnight at room temperature.

C. Characterisations of sample

The phase analysis was observed from the XRD spectra in the 2θ range 20° to 80° using an X-Pert PANalytical diffractometer (PW3050/60) with a step size of 0.03° operating at 40 kV and 30 mA. The starting diameter sizes of carbon nanotubes were measured using the LEO 912AB Energy Filter Transmission Electron Microscope (TEM), while cross-section surface morphology was obtained from Field Emission Scanning Electron Microscope (FeSEM) micrographs taken using the FEI NOVA NanoSEM230 machine. The diameter and particle size were obtained by estimating a mean diameter of at least 200 different diameters and particles by using the intercept method. The elemental study was performed using the Energy Dispersive X-ray (EDX) and magnetic properties were determined using a vibrating sample magnetometer (VSM) with a maximum field of 12000 G at room temperature. The Vector Network Analyser was used to analyse the electromagnetic parameters and absorbing properties of the composites in the 8 – 18 GHz frequency range. The calculation was carried out using only one port and metal short, as in Fig. 1.

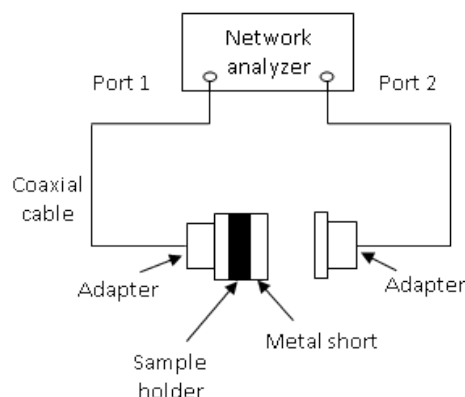


Fig. 1 Instrumental setup for measuring RL using a metal short.

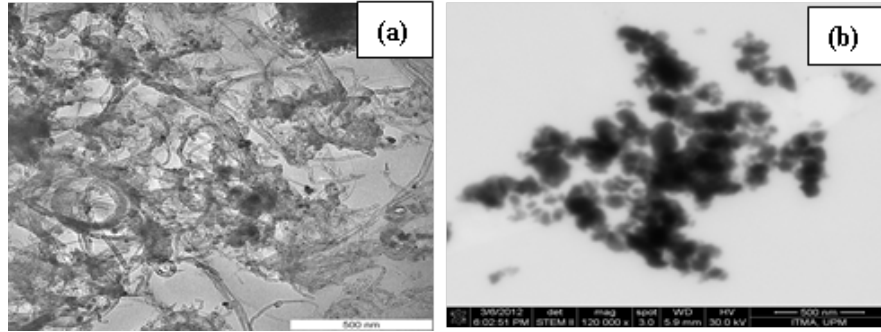


Fig. 2 TEM micrograph of a) commercial MWCNT and b) nanosized Nickel-Zinc-Ferrite powder milling 10 hours.

III. RESULTS AND DISCUSSION

Fig. 2 (a) and (b) show the TEM micrographs of commercial multi-walled carbon nanotubes and synthesised Nickel-Zinc-Ferrite nanosized powder after milling for 10 hours, respectively. The TEM micrograph of MWCNTs was randomly entangled and cross-linked with nanotubes with an average diameter size of 13.5 nm. On the other hand, Nickel-Zinc-Ferrite was found to have an agglomeration of particles due to the milling process. The distribution of particle size was in the range of 40 – 160 nm with an average of 89 nm.

It was previously suggested that the smallest size of the single-domain particles for Nickel-Zinc-Ferrite was 0.1 μm [7]. However, a more recent study reported that 0.3 μm was the critical threshold instead, and samples smaller than 0.3 μm exhibited single-domain behaviour [8]. Small particles have superparamagnetism and single-domain states, and even when larger grains are formed, very small grains that exhibit superparamagnetism or single-domain behaviour cannot be prevented [9]. Magnetic nanoparticles, which are small enough, exhibit single-domain behaviour that can absorb more energy and result in high-frequency applications.

Generally, two different magnetising mechanisms, which are spin rotation and domain wall movement, are due to the permeability of polycrystalline ferrite ceramics. This relationship can be described as:

$$\mu_i = 1 + X_w + X_{\text{spin}}, \quad (1)$$

where X_w is the domain wall susceptibility and X_{spin} is the intrinsic rotational susceptibility. Meanwhile, the X_w and X_{spin} may be written as follows:

$$X_w = 3\pi M_s^2 D / 4\gamma \quad (2)$$

and

$$X_{\text{spin}} = 2\pi M_s^2 / K, \quad (3)$$

where M_s is the saturation magnetisation, K is the total anisotropy, D is the average grain diameter, and γ is the domain wall energy. It is known from the formulae provided that the spin rotation and domain wall motion are affected by grain size. Therefore, there should be no domain walls within

the grains for sufficiently small grains where the permeability was only due to the rotational processes (equation 3).

The XRD patterns of synthesised Nickel-Zinc-Ferrite, pure multi-walled carbon nanotubes and pure epoxy resin are shown in Fig. 3. The diffraction peak at 26.2° assigned to the (002) plane of the MWCNT structure could be differentiated as a result of the basal hexagonal carbon atomic network reflections and parallel nanotube stacking layers [10]. A weak peak was also observed in MWCNTs at $2\theta = 42.9^\circ, 44.3^\circ, 52.0^\circ, 56.0^\circ, 61.8^\circ$ and 74.9° corresponding to (100), (101), (200), (004), (110), (112) and (006) respectively. The remaining peaks at $2\theta = 34.5^\circ, 38.1^\circ, 39.6^\circ$ and 69.6° demonstrated the presence of the catalysts (Co and Mo) in CNT, which were labelled as (*) [11]. The XRD spectrum of the pure epoxy sample displayed a hump that was assigned to the amorphous structure of the epoxy while the XRD spectrum of the Nickel-Zinc-Ferrite showed diffraction peaks at $2\theta = 30.3^\circ, 35.7^\circ, 37.4^\circ, 43.4^\circ, 53.7^\circ, 57.2^\circ, 62.8^\circ, 70.9^\circ$ and 74.9° corresponding to (220), (311), (222), (400), (422), (511), (440), (026) and (226) planes respectively, confirming the proper formation of the ferrite crystallographic phases with reference code 98-006-8765.

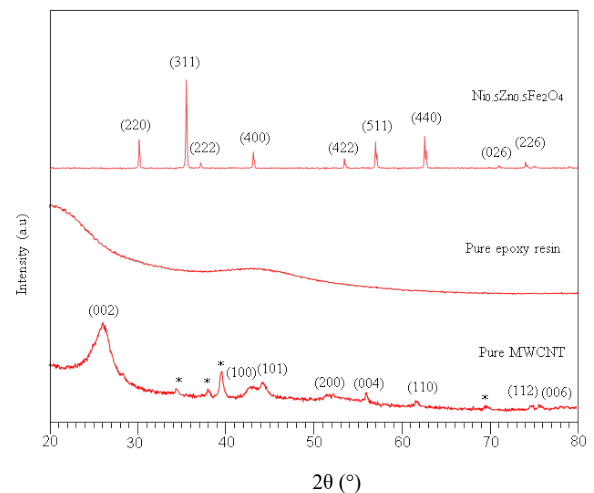


Fig. 3 XRD patterns of pure epoxy resin, pure multi-walled carbon nanotubes, and Nickel-Zinc-Ferrite.

In order to study the phase evolution of the MWCNTs-Nickel-Zinc-Ferrite composites with different MWCNT loadings, further XRD analysis was performed, as shown in Fig. 4. It can be observed that all peaks were shifted to the right relative to the synthesised Nickel-Zinc-Ferrite powders mixed with varying weight percentages of MWCNTs and then incorporating into epoxy by using a high-speed mixer. The shift in XRD peaks reflected the presence of defects in the crystal structure, which contribute to the higher RL in the sample. Additional peaks were absent in the sample although mixed with MWCNTs. This was due to the limitation of XRD in the detection of lower concentrations of MWCNTs that were mixed in the composite sample. On the other hand, increasing the amount of MWCNTs did not cause many changes in the intensity of the peaks because the amount being mixed was too low.

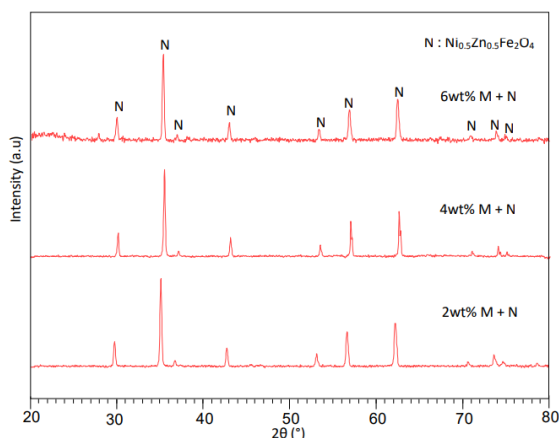


Fig. 4 XRD pattern of different weight percentages of MWCNT(M) mixed with Nickel-Zinc-Ferrite incorporated into the matrix.

Fig. 5(a) shows the FeSEM image taken for morphological studies of the fractured cross-section area. Typical FeSEM composite images containing 2 wt%, 4 wt%, and 6 wt% of MWCNTs mixed with Nickel-Zinc-Ferrite and incorporated into the epoxy matrix are shown in Fig. 5 (b), (c), and (d). All the composite samples shown were after being freeze-fractured in liquid nitrogen and gold coated. It has been established that MWCNTs, with their weak van der Waals forces between them, will spontaneously attract each other to form MWCNTs bundles. Thus, incorporation into the insulating polymer matrix is one way to reduce or weaken forces between them. The FeSEM image clearly showed that the MWCNTs-Nickel-Zinc-Ferrite was dispersed uniformly in the epoxy matrix. The arrows and rectangular shapes in Fig. 5 (b), (c), and (d) were indicating the epoxy resin and MWCNTs, respectively, while the circles and oval shapes were indicating the Nickel-Zinc-Ferrite particles and MWCNTs immersed in the epoxy resin.

The amounts to load were fixed at 2 wt%, 4 wt% and 6 wt% as MWCNTs bundles formed tend to agglomerate with irregular sizes and shapes in the epoxy matrix above 8 wt%. Furthermore, microwave absorption of the composite sample loaded with 10 wt% MWCNTs did not display significant improvements compared to the composite sample with 8 wt% MWCNTs [12]. This indicated that microwave absorption was optimised up to the loading of 8 wt%. The FeSEM images showed that it was possible to obtain complex microstructures of the composites with multiple MWCNTs adjacent to Nickel-Zinc-Ferrite particles. 3-dimensional network structures were generated through the interfacial interaction of MWCNTs/Nickel-Zinc-Ferrite/epoxy resin. These 3-dimensional network structures created multiple internal reflections in the sample that helped absorb the electromagnetic wave.

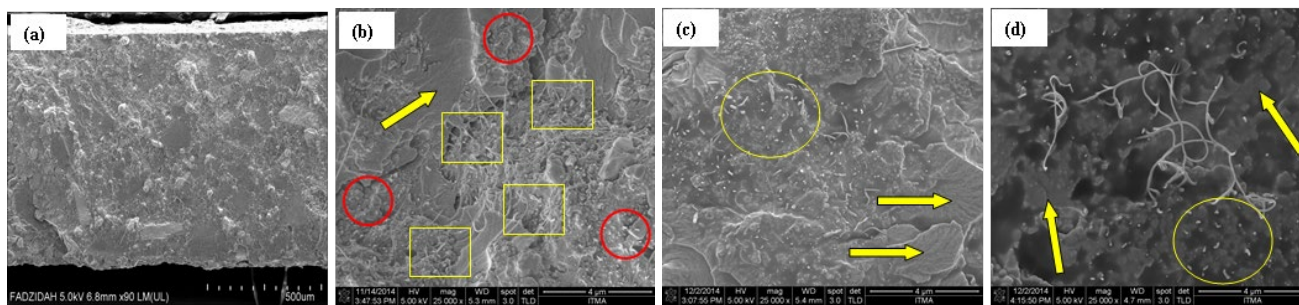


Fig. 5 FeSEM image of a) cross-section b), c) and d) of 2 wt%, 4 wt%, and 6 wt% MWCNTs mixed with Nickel-Zinc-Ferrite and being incorporated into epoxy matrix.

In order to determine the presence of elements in the composite sample, EDX measurement was carried out on one of the composite samples as shown in Fig. 6. The EDX pattern revealed that the composite sample of MWCNTs-Nickel-Zinc-Ferrite consisted of carbon (C), which indicated the presence of MWCNTs, and Nickel (Ni), Zinc (Zn), Ferrite (Fe) and Oxygen (O) from Nickel-Zinc-Ferrite. The element Au originated from the gold coating on the sample surface, which indicated that impurity was absent in the sample and the loss of reflection was due to the 3-dimensional structure network.

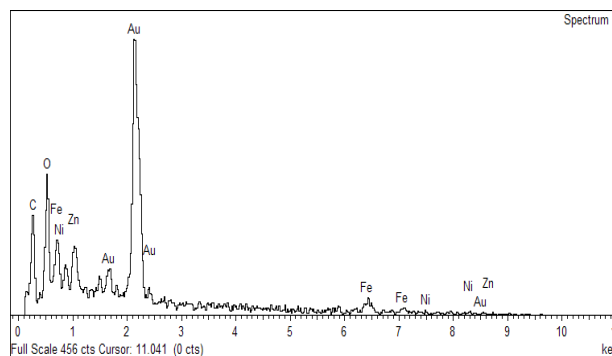


Fig. 6 EDX pattern of the 2 wt% MWCNTs-Nickel-Zinc-Ferrite-epoxy resin.

Fig. 7 displays the VSM plot of pure Nickel-Zinc-Ferrite, 2 wt%, 4 wt%, and 6 wt% MWCNTs mixed with Nickel-Zinc-Ferrite incorporated into the epoxy matrix. The magnetisation curves of the composite sample indicated the presence of mixed states of ferrimagnetism (sigmoid shape of hysteresis with the existence of coercivity values) and superparamagnetism (small grains below the critical diameter and crystalline phase formation) and despite the presence of non-magnetic MWCNTs, the composite showed significant retention of the Nickel-Zinc-Ferrite magnetic behaviour.

The main magnetic properties of different composite samples are described in Table 1. It was obvious that the composite coercivity (H_c) increased with the amount of MWCNTs, while both saturation magnetisation (M_s) and retentivity (M_r) decreased with the increase in the amount of MWCNTs as previously reported by [13]. The reductions in M_s and M_r were most likely due to the presence of higher amounts of MWCNTs and structural distortion in the ferrites' surface that reduced the magnetic moment. This could be due to the small size and surface defects of ferrite crystallites and the strain between the ferrite nanoparticles and MWCNTs surfaces [14].

The maximum M_s was 44.496 emu/g for (2wt% M + N)/P, which was lower than that of the pure Nickel-Zinc-Ferrite/P, and this lower M_s value was due to the presence of non-magnetic MWCNTs. It has been reported that 0.009 emu/g was the saturation magnetisation value for pure epoxy [15], while the M_s value for pure $Ni_{10.5}Zn_{0.5}Fe_2O_4$ sintered at 900°C was 68.200 emu/g [8], which was higher than Nickel-Zinc-Ferrite/epoxy as the polymer compound was absent. Essentially, the H_c of a grain size below the threshold of 0.3 μm was dominated by shape and magnetocrystalline anisotropy, with shape anisotropy being the major factor affecting the H_c of smaller grain size. Since the grain was too small to accommodate the multi-domain formation, it could only contain single domains. Therefore, the absorption of the magnetisation was only due to the response by spin. In addition, the ferrite nanoparticles' surface spins were disordered and the exchange coupling between surface and core gave rise to a variety of spin distributions within a single-domain particle. These surface spins resulted in a high hysteresis loss, which could increase the absorption of the microwave.

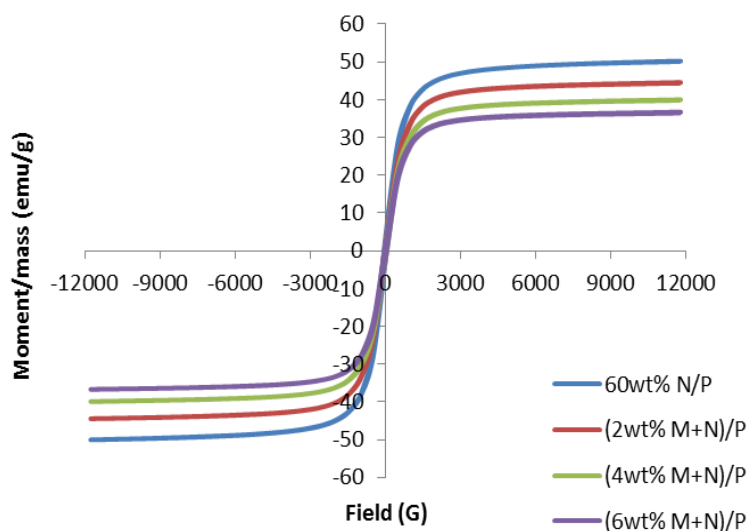


Fig. 7 M-H hysteresis shape of 60 wt% Nickel-Zinc-Ferrite, 2 wt%, 4 wt%, and 6 wt% MWCNTs mixed with Nickel-Zinc-Ferrite incorporated into the epoxy matrix.

TABLE I
MAGNETIC PROPERTIES OF 2 wt%, 4 wt% and 6 wt% MWCNTS MIXED WITH NICKEL-ZINC-FERRITE INCORPORATED INTO THE EPOXY MATRIX

List of samples	Coercivity (H_c) (G)	Magnetisation (M_s) (emu/g)	Retentivity (M_r)
60wt% N/P	52.810	50.133	3.219
(2wt% M + N)/P	39.194	44.496	1.961
(4wt% M + N)/P	40.786	39.937	1.861
(6wt% M + N)/P	41.618	36.697	1.782

A Vector Network Analyser was used to measure the RL of 2 wt%, 4 wt%, and 6 wt% MWCNTs mixed with Nickel-Zinc-Ferrite and loaded into the epoxy resin fixed with a thickness of 1 mm, 2 mm, and 3 mm. The prepared composite samples were backed by a metal plate and directly measured using a one port measurement to obtain the S_{11} parameter or RL. The microwave frequency and RL relationship were measured within the 8 – 18 GHz frequency range (X and Ku-band).

Fig. 8 shows the frequency dependence of Nickel-Zinc-Ferrite/P, 2 wt%, 4 wt%, and 6 wt% MWCNTs-Nickel-Zinc-Ferrite/P RLs. As reported by [16], a pure epoxy resin matrix has almost no absorption at the frequency range of 2 – 18 GHz. Therefore, the RL was primarily due to the effects of the filler itself. However, the epoxy resin as a matrix helped in providing multiple reflections in the composite sample. The RL peak of 60 wt% Nickel-Zinc-Ferrite/P for 3mm thickness occurred at 14 GHz with a RL of -8 dB, while RL peaks were not observed for the other two thicknesses due to higher frequency resonance. The relationship between the matching thickness (d_m) and matching frequency (f_m) is described in

the equation $d_m = \frac{c}{2\pi f_m \mu''}$ where c is the velocity of light.

It was apparent that the corresponding frequency decreased with an increase in absorber thickness.

MWCNTs had an obvious effect on the composites' microwave absorbing properties, especially in terms of peak position. The positions of microwave absorptions peaks for each thickness shifted to lower frequencies as the amount of MWCNTs increased. For the thickness of 1 mm, no peak was observed for 2 wt% MWCNTs-Nickel-Zinc-Ferrite/P, whereas for 4 wt% and 6 wt% MWCNTs-Nickel-Zinc-Ferrite/P, the RL reached ~ -10.5 dB (17.3 GHz) and ~ -9.5 dB (16.2 GHz) respectively. The RL for the thickness of 2 mm was highest at 16 GHz with -14 dB for 2 wt% MWCNTs-Nickel-Zinc-Ferrite/P, followed by -11 dB at 11.5 GHz for 4 wt% MWCNTs-Nickel-Zinc-Ferrite/P, and -6 dB at 10.5 GHz for 6 wt% MWCNTs-Nickel-Zinc-Ferrite/P. On the other hand, the composite with a thickness of 3 mm displayed a RL of -11 dB at 9.5 GHz for 2 wt% MWCNTs-Nickel-Zinc-Ferrite/P and resonated at much lower frequencies for both 4 wt% and 6 wt% MWCNTs-Nickel-Zinc-Ferrite/P. Therefore, it was evident that within the 8 – 18 GHz frequency range, the thickness of 2 mm with 2 wt% MWCNTs had the highest RL among all samples. The RL of the MWCNTs-Nickel-Zinc-Ferrite/P was below -10 dB (90% absorption) in the range of 14.5 – 17 GHz, corresponding to a bandwidth of 2.5 GHz.

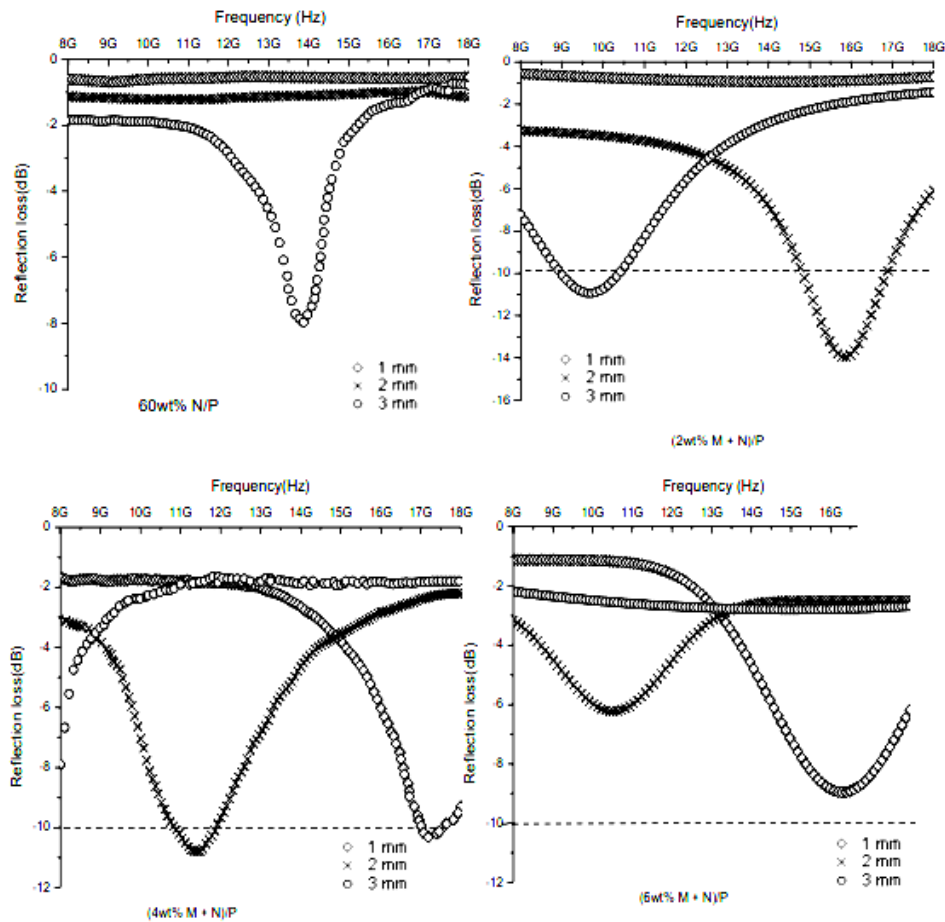


Fig. 8 RL of 60 wt% Nickel-Zinc-Ferrite, 2wt%, 4wt%, and 6wt% MWCNTs mixed with Nickel-Zinc-Ferrite incorporated into the epoxy matrix

IV. CONCLUSIONS

Different weight percentages of MWCNTs mixed with Nickel-Zinc-Ferrite were synthesised, uniformly mixed, and dispersed in the epoxy matrix as shown by the microstructure. It was demonstrated that the addition of only 2 wt% of MWCNT helped enhance the absorbing ability of the microwave. The electromagnetic absorption of pure MWCNTs contributed to dielectric loss, although for pure Nickel-Zinc-Ferrite particles, the effects of magnetic losses including eddy current and other magnetic losses, were more dominant than dielectric loss. The RL peak shifted towards a lower frequency range as the amount of MWCNTs increased, which suggested that the working frequency could be tuned by increasing MWCNTs and changing the corresponding thickness according to specific frequency ranges for the requirements of certain applications. This study has demonstrated that these samples have potential applications as electromagnetic absorbers.

ACKNOWLEDGEMENT

The authors express deepest gratitude to their late project supervisor Assoc. Prof. Dr. Mansor Hashim and acknowledge the Ministry of Higher Education Malaysia for financial support through the Long-Term Research Grant Scheme (LRGS/B-U/2013/UPNM/Defence & Security-P2) and Fundamental Research Grant Scheme (FRGS/1/2020/STG05/USIM/02/3). The authors also acknowledge Universiti Sains Islam Malaysia for financial support under USIM-RACER Grant (PPPI/USIM-RACER_0120/KGI/051000/11920).

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest regarding the publication of this paper.

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Received 1st November 2020; Revised 1st January 2021;
Accepted 15th March 2021; Published 1st April 2021

Academic Editor: Azira Khalil
USIM Press
Malaysian Journal of Science, Health & Technology
Vol. 7, No. 1 (2021), 7 pages
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