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Materials' Properties of Lightweight Spiral Hybrid CNT/Epoxy Composites Enhanced Reflection Loss

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ABSTRACT

Recently, various electronic devices have been developed to meet the requirements of higher frequency technology applications. This widely used application without realizing has created more electromagnetic interference pollution that is harmful to human health and other equipment. Therefore, more research interest focuses on fabricating the electromagnetic (EM) wave absorbing materials that can absorb the EM wave interference. In this regard, this research highlights the use of Iron Oxide and Cobalt Oxide as catalyst to synthesize hybrid CNT by using Thermal Vapor Deposition Tube (TVDT) method. The spiral hybrid CNT/epoxy composites were prepared at thickness of 1mm, 2mm and 3mm. The phase formation, microstructural, particle size and structural analysis of the hybrid CNT were analyzed by using X-ray diffractometer (XRD), Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM) and RAMAN spectrometer respectively. The microwave characterization of the hybrid CNT/epoxy composite samples was analyzed by using Vector Network Analyzer (VNA) at GHz frequency range. The phase analysis confirmed the existence of Carbon and iron carbide in the sample. The microstructural of CNT formation are mostly in spiral and straight like structure. On the other hand, the structural analysis shows the sample are more towards defective structure with higher and broader D-band peak. This could enhance the EM wave absorption performance. The minimum reflection loss (RL) peak was ~-23dB (t=3mm) obtained for all hybrid CNT composite samples. The differences of minimum reflection loss peak at different weight percentages are most likely shown by the shift of frequency range. Thus, this lightweight spiral hybrid CNT/epoxy composites results in better EM wave performance at different thin thickness used for different applications.

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1. Introduction

Rapid growth of the use of electronic devices that work at microwave frequency range have increased in the electromagnetic (EM) interference and non-ionizing radiation that are harmful to human health and other electronic equipment. In this regard, it attracts more public attention and researcher interest in conducting research focusing on the EM wave absorbing materials that could absorb and reduce the unwanted EM wave signal. Developing an effective EM wave absorption material requires strong absorption ability and wide bandwidth as reported by Lu *et al.,* [1]. Moreover, the absorber materials should have lower material density, thinner absorption coating and have stability at high temperatures with material compatibility as also being reported by several authors [2-4]. The way EM wave absorbing material works is that it absorbs EM wave energy, gradually transforms it into heat or other types of energy, and then dissipates that energy through attenuation and loss as reported by Lu *et al.,* [1]. The increase in the transmission route and transfer from one medium to another medium contributes to higher efficiency of the EM wave absorber.

Binary combinations such as Nickel (Ni), Iron (Fe) and Cobalt (Co) are often utilized and act as active catalysts that exhibit greater activity than the individual elements. Carbon nanotubes (CNTs) growth from an active catalyst are well-known due to their unique chemical, physical and mechanical properties that makes it suitable for certain modifications for lightweight, wide and strong absorption bandwidth and act as a potential EM wave absorber. The dielectric and resistance loss of EM waves in CNTs is converted into thermal energy dissipation because of surface polarization and dielectric relaxation in the conductive network generated by CNTs. This implies the features of absorption type of EM wave absorbing materials of CNTs. In comparison to other carbon-based EM wave absorbing materials, CNTs have greater EM wave absorption capability because of their flexibility, lighter, thinner and larger specific surface area as reported by Konrath *et al.,* [5]. Other research conducted by Hashim *et al.,* [6] also reported on the highest mechanical strength enhancement was given by the MWCNT-Al5Si where the MWCNT was homogeneously distributed into the matrix and MWCNT helps in reducing the crack propagation.

Therefore, this research highlights the modification of CNTs, with special emphasis on their EM wave absorbing ability. In this study, Magnetite (Fe_3O_4) and Cobalt Ferrite ($CoFe_2O_4$) were mixed and used as catalyst to synthesize spiral hybrid multiwalled carbon nanotubes (MWCNTs) by using thermal vapor deposition tube (TVDT). The prepared sample was then further incorporated into an epoxy resin as matrix. The materials and microwave characteristics were studied to analyze the synthesized MWCNTs from mixed ferrite as catalyst.

2. Methodology

2.1 Synthesized of Spiral Hybrid MWCNTs by Using TVDT

Nanometer particle size of Fe_3O_4 and $CoFe_2O_4$ were prepared by crushing the powder using high energy ball milling (HEBM) as shown in Figure 1(a). The nanometer powder was further sintered at 900^0C and 1100^0C respectively to obtain the full phase. The sintered powder was then used as catalyst to grow CNTs. Different weight percentages of Fe_3O_4 and $CoFe_2O_4$ were mixed and used as catalyst to grow CNTs. The weight percentages of Fe_3O_4 (F) mixed with $CoFe_2O_4$ (C) varies from 20%, 50% and 80%. The sintered powder was mixed and labelled as follows S1 (20wt% F + 80wt%C), S2 (50wt% F + 50wt%C) and S3 (80wt% F + 20wt%C). On the other hand, as for 100% F and 100% C, it was denoted as SF and SC respectively. In the preparation of MWCNTs, 0.1 g catalyst was first placed in the ceramic boat and was placed in the middle of the furnace by using the thermal vapour

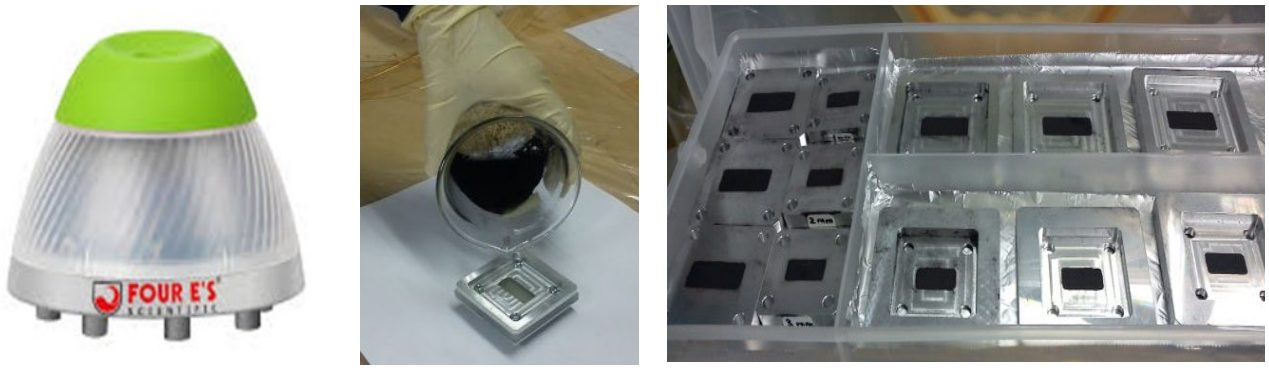
deposition tube (TVDT) as shown in Figure 1(b). The reaction temperature was set to 700^0C for 30 minutes since the hydrocarbon was decomposed at 700 – 900^0C. Ethanol was used as the carbon source, while Argon gas was used as carrier gas. Diameters of the synthesized MWCNTs were determined by the size of catalyst particles used at the beginning. After the reaction process, the synthesized MWCNTs were collected and weighed as shown in Figure 1(c).

(a) (b) (c)

Fig. 1. (a) High energy ball milling (b) furnace for TVDT setup (c) synthesized MWCNTs

2.2 Incorporation of Filler into Polymer Matrix

Synthesized MWCNTs used as filler in this process were collected after TVDT process and was dispersed in the epoxy (E) resin as matrix since epoxy resin has almost no absorption. The weight percentage of filler incorporated into matrix was fixed to 8wt%. The mixing process was conducted by using a mini vortex mixer at 3000 rpm as shown in Figure 2(a). Hardener was then added into the mixture as a curing agent and was continuously mixed. The ratio of epoxy resin to hardener was fixed to 10:1 for 10 to 15 minutes. The prepared composite samples were poured into sample holder (Figure 2(b)) at two different sizes, for measurement at different frequency (X-band and Ku-band). The thickness was fixed at 1mm, 2mm and 3 mm and were cured at room temperature as shown in Figure 2(c). The composite samples were labelled as SF/E, SC/E, S1/E, S2/E and S3/E.



(a) (b) (c)

Fig. 2. (a) High energy mixer (b) pouring polymer composite into sample holder (c) cured polymer composite

2.3 Measurement and Analysis

The materials' properties and characterization were conducted to analyze the properties of the synthesized spiral hybrid CNT/epoxy composite. The crystalline phase formation of the prepared composite sample was confirmed by X-ray Diffraction (XRD) spectrum in the 2θ range of 20^0 to 80^0. The particle size and microstructural analysis were measured by using Transmission Electron

Microscope (TEM) and Field Emission Scanning Electron Microscope (FeSEM) respectively. The vibrational phonon modes were determined by using Raman Spectroscopy. The microwave characterization of the composite samples was measured with metal back by using Vector Network Analyzer (VNA) in the frequency range of 8 – 12 GHz (X-band) and 12 – 18 GHz (Ku-band) as shown in Figure 3.

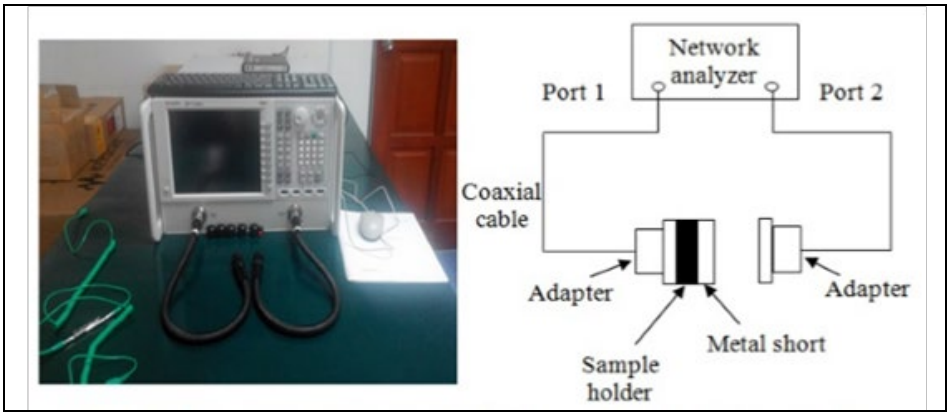


Fig. 3. Microwave characterization setup using Vector Network Analyzer

3. Results and Discussion

3.1 Phase Analysis

Figure 4 shows the X-ray diffraction pattern of different composite samples by incorporating synthesized spiral hybrid MWCNTs into epoxy resin as matrix. Based on the diffraction pattern, two main phases obtained are carbon (G) and Iron Carbide (Fe_3C). The peaks were compared with the standard reference code data for carbon graphite (98-001-7175) and iron carbide (98-009-1656). A well-defined diffraction peak appeared at (hkl) peak of (002) at an angle of $2\theta = 26.577^0$ correspond to Carbon peak generated by the reflections from basal hexagonal carbon atomic networks and parallel nanotube stacking layers as also reported by Somiya [7]. It also presents as the X-ray beam strikes the single wall of CNTs. CNT strains have different sizes and shapes with curved graphene layers. Thus, it makes most of the carbon peak could not be observed due to the suppression of few reflection peaks that leads to peak shifting and broadening as reported by Tessonnier *et al.,* [8]. However, the remaining iron carbide (Fe_3C) peaks occurred because of the presence of iron (Fe) and carbide (G) during the TVDT process. The formation of iron carbide on the active catalyst phase replaces all the iron-based catalyst during the synthesized regardless of the type of CNT that was emanating from the particle of catalyst, or the amount of carbon deposited. The morphological orientation of CNT determined the CNT diffraction peak intensities.

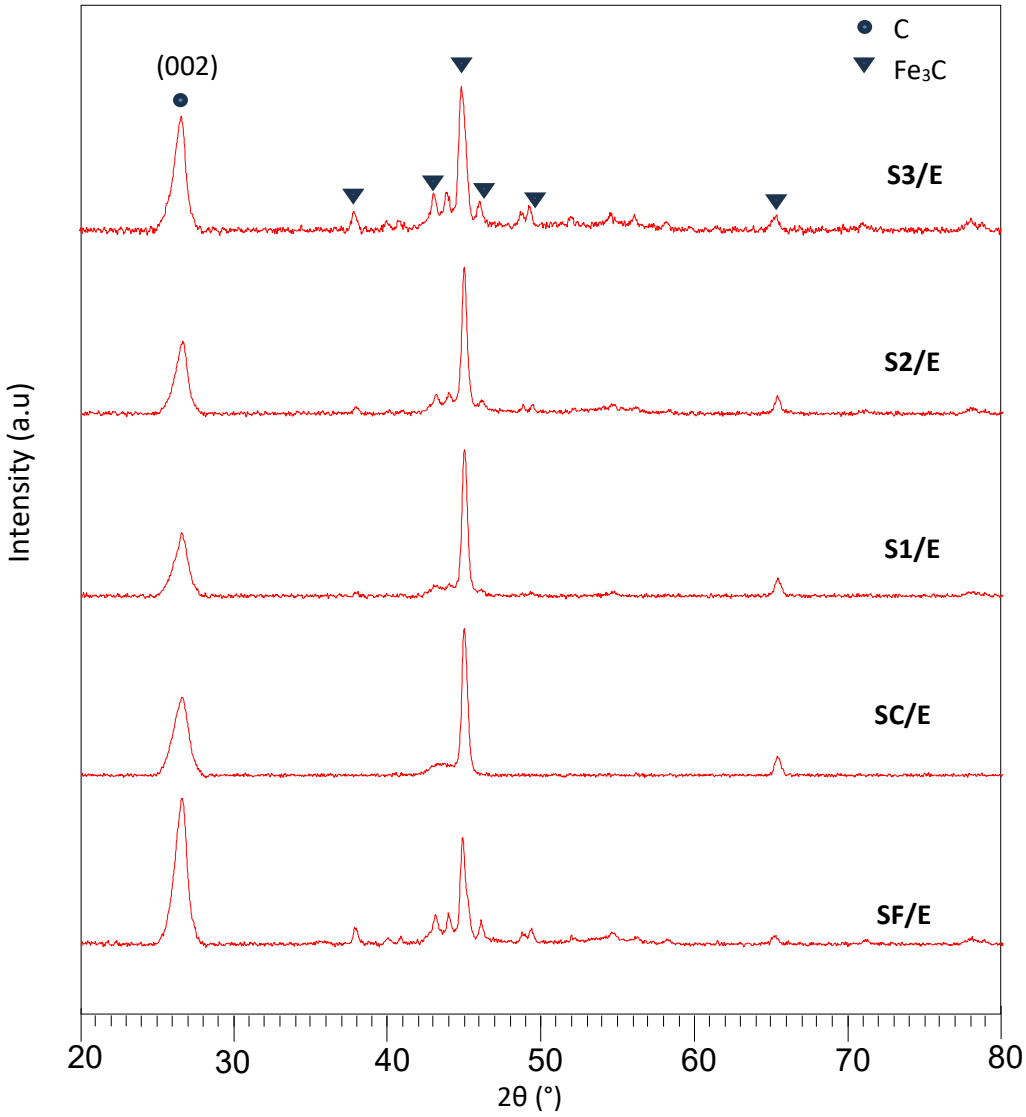


Fig. 4. X-ray diffraction pattern of composite samples SF/E, SC/E, S1/E, S2/E and S3/E

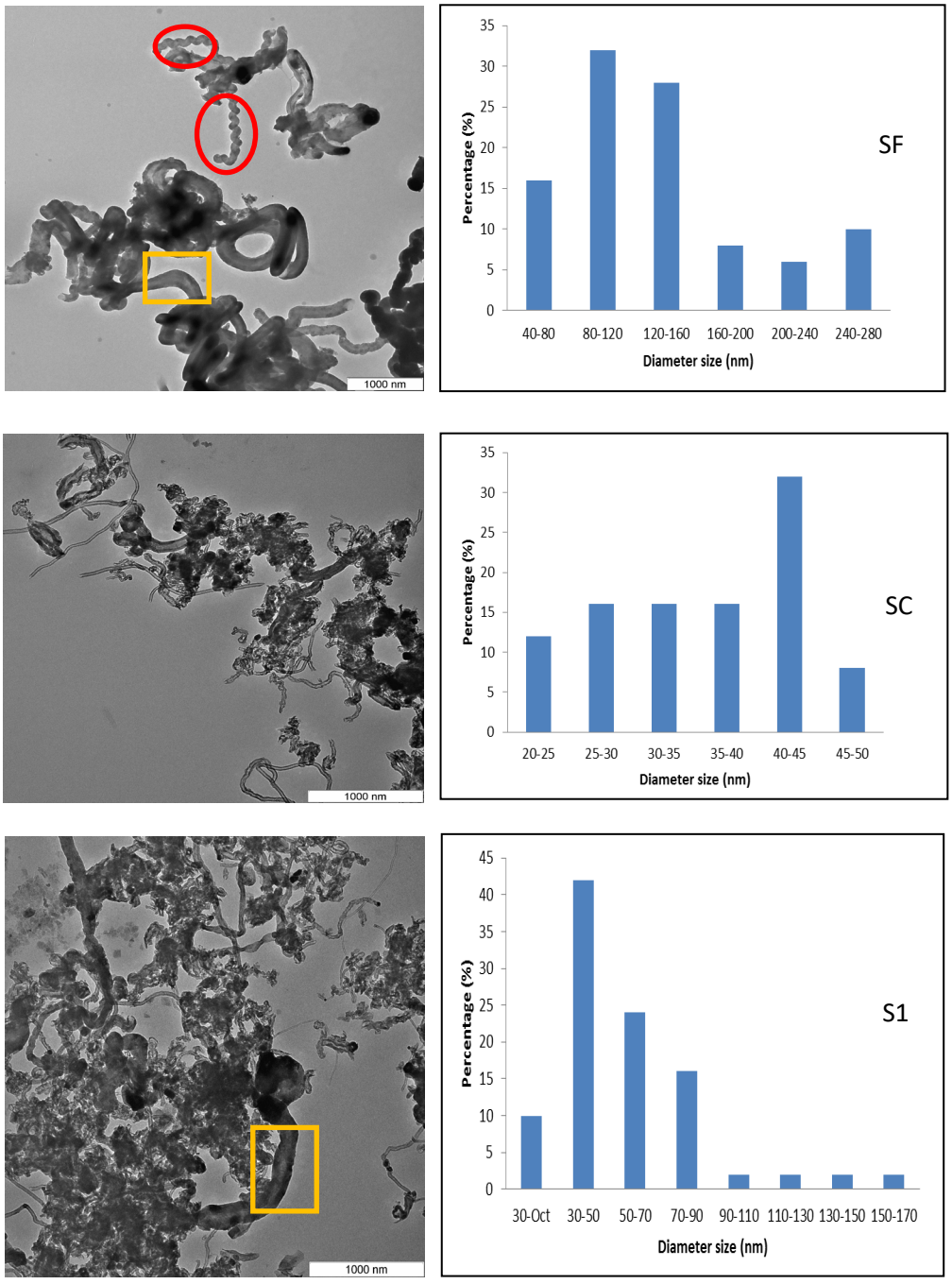
3.2 Diameter Size Analysis

TEM micrograph and diameter size distribution of synthesized spiral hybrid MWCNTs of SF, SC, S1, S2 and S3 is shown in Figure 5. The micrograph shows that the structure is mix of straightlike, curved and twisted together. There also exist spiral and hollow tube CNTs that are shown in circle and rectangle shape respectively. The black spots present refers to the agglomerated growth happened because of the repulsive interactions are not high enough to block their access due to Van der Waals attraction. Those structure characteristics appeared in all synthesized samples. Moreover, micrograph shows that some dispersed iron particles are trapped inside the CNTs walls. On the other hand, the black spot present at the walls surface refers to the combination of particles of Fe and FeC as also reported by Brachetti-Sibaja *et al.,* [9].

Catalyst particles size influenced the diameter formation of the synthesized MWCNTs. Smaller catalyst particle size results in smaller diameter of synthesized MWCNTs. There are two types of CNTs growth mechanisms that take place in the formation of MWCNTs. The related growth mechanisms are tip growth mode and base growth mode. These two mechanisms are depending on the adhesion between the catalyst particle and the substrate. Tip growth mode produced when the

catalyst particle size is large (>> 5nm) that results in synthesizing MWCNTs (>10 walls). On the other hand, base growth mode take place when the catalyst particle size is small (< 5nm) producing single or few wall CNTs that is typically less than seven walls. Thus, it shows that the synthesized CNTs in this research is MWCNTs that are suitable for application as EM wave absorption in microwave frequency range due to its multiple reflections in the synthesized MWCNTs.

The average diameter size of SF and SC are 133.6 nm and 36.2 nm respectively. On the other hand, the average diameter size of S1, S2 and S3 are 56.7 nm, 64 nm and 75.4 nm respectively. The coils forming the helical structure indicate the presence of defects that correspond to D band that can be observed in Raman spectra.



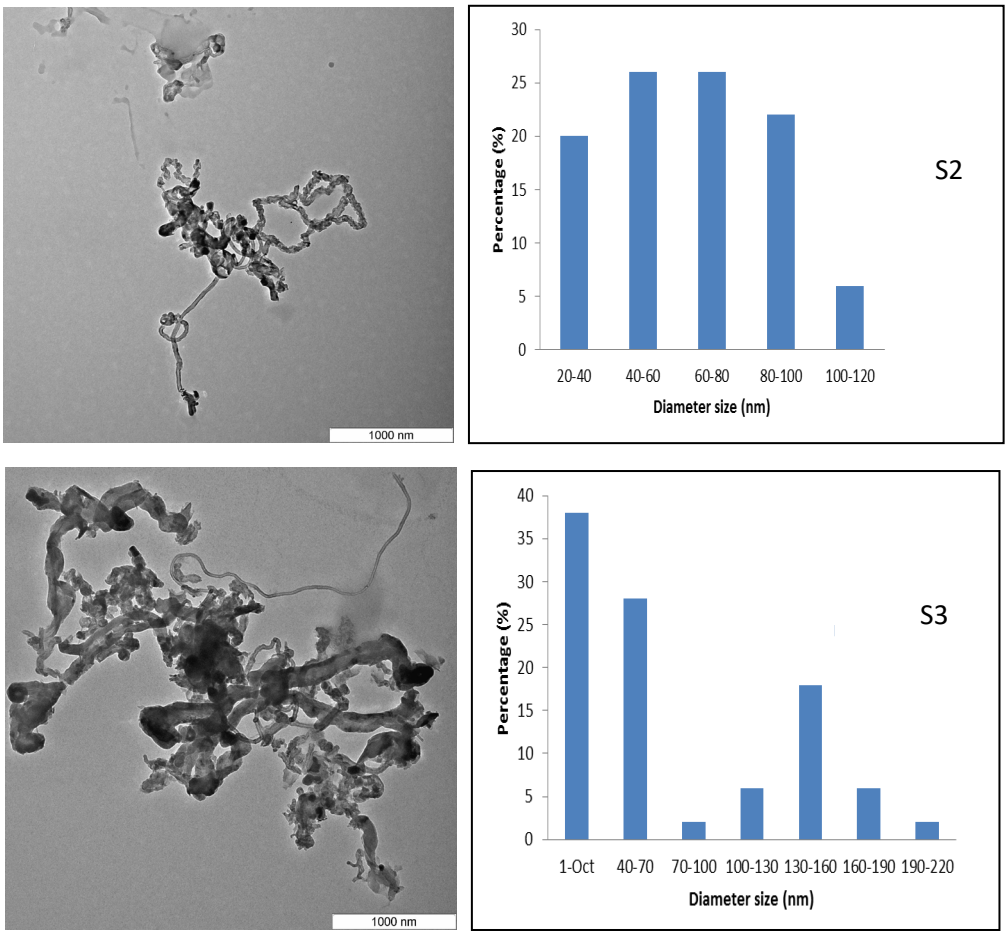


Fig. 5. TEM micrograph and diameter size distribution of synthesized spiral hybrid MWCNTs SF, SC, S1, S2 and S3

3.3 Microstructural Analysis

FeSEM micrograph of synthesized spiral hybrid MWCNTs of SF, SC, S1, S2 and S3 is shown in Figure 6. The micrograph shows the images of MWCNTs without being loaded into polymer matrix are in the form of bundles and entanglements. The higher attraction to each other that consist of 50 to a few hundred of individual CNTs that forms together is due to strong Van der Waals forces. The average diameter size of synthesized MWCNTs of SF, SC, S1, S2 and S3 are 105.9 nm, 49.8 nm, 21.2 nm, 22.4 nm and 94.4 nm respectively. The microstructural image shows that the synthesized CNTs are mix of fiber that is straight, curved and twisted together. However, most of the samples shows spiral structure that results in having more total internal reflection and multiple reflection in the sample that could enhance the microwave transmission route, hence results in higher EM wave absorption properties.

Figure 7 shows the FeSEM micrograph of synthesized spiral hybrid MWCNTs/epoxy matrix of SF/E, SC/E, S1/E, and S3/E. The aggregation of bundles CNT due to Van der Waals forces can be overcome by well dispersion of MWCNTs into an insulated polymer matrix. The incorporation of filler into matrix was fixed at 8wt% since incorporation of more than that could result in not homogenously dispersed in the polymer matrix. Moreover, loading amount of 10wt% would result in not much improvement compared to 8wt% CNT loading as also conducted by Wang and Zhao [10]. In addition, by using high speed mixer, it may improve the dispersion of MWCNTs in the epoxy matrix. Study conducted by Razak *et al.,* [11], the best combination of kenaf loading fiber is 5wt% with stirring time 10 mins, showing the best polymer composite tensile strength performance. This

also support that the weight percentage of fiber to be loaded into epoxy resin as matrix should not be more than 10wt%. Moreover, Thanh, *et al.,* [12] reported that a good adhesion interaction between all components of filler and matrix must be uniformly distributed within the epoxy resin matrix. The agglomerations that form larger particles will reduce the dispersion ability within the epoxy matrix that results in reducing mechanical properties of the polymer composites.

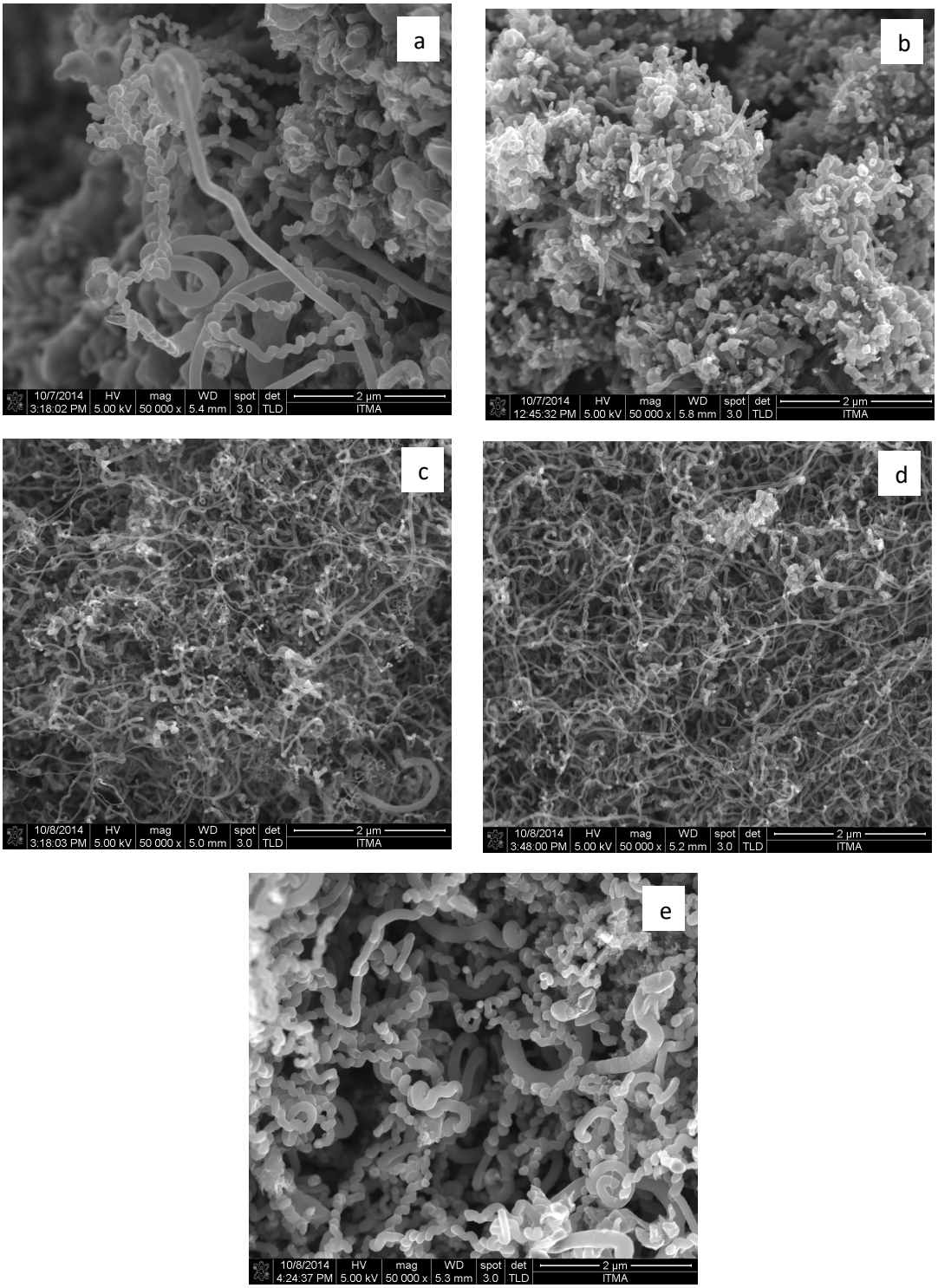


Fig. 6. FeSEM micrograph of synthesized spiral hybrid MWCNTs (a) SF (b) SC (c) S1 (d) S2 and (e) S3

The images show that the composite samples forming a nearly interconnected network, with small distances between CNT ends (Figure 7). By having larger amount of CNTS loaded into the polymer matrix, precaution needs to be taken to well-disperse the CNTs since aggregated CNTs in a large volume could increase the electrical conductivity of the EM wave signal than those uniformly dispersed CNTs as reported by Aguilar and Aviles [13]. By having higher conductivity, it leads to having higher reflection due to moving of free electrons. The dispersed MWCNTs and isolate MWCNTs bundles in the epoxy composites may increase the EM wave absorption energy. The interaction between the interior electrons and exterior microwave radiation may increase the attenuation of the EM wave radiation as reported by Qin and Brosseau [14]. Thus, it results in increase of the EM wave transmission route. However, higher electrical conductivity is essential to be used as an EMI shielding materials due to the electric and varying magnetic fields generate currents in the material as reported by Shueb *et al.,* [15].

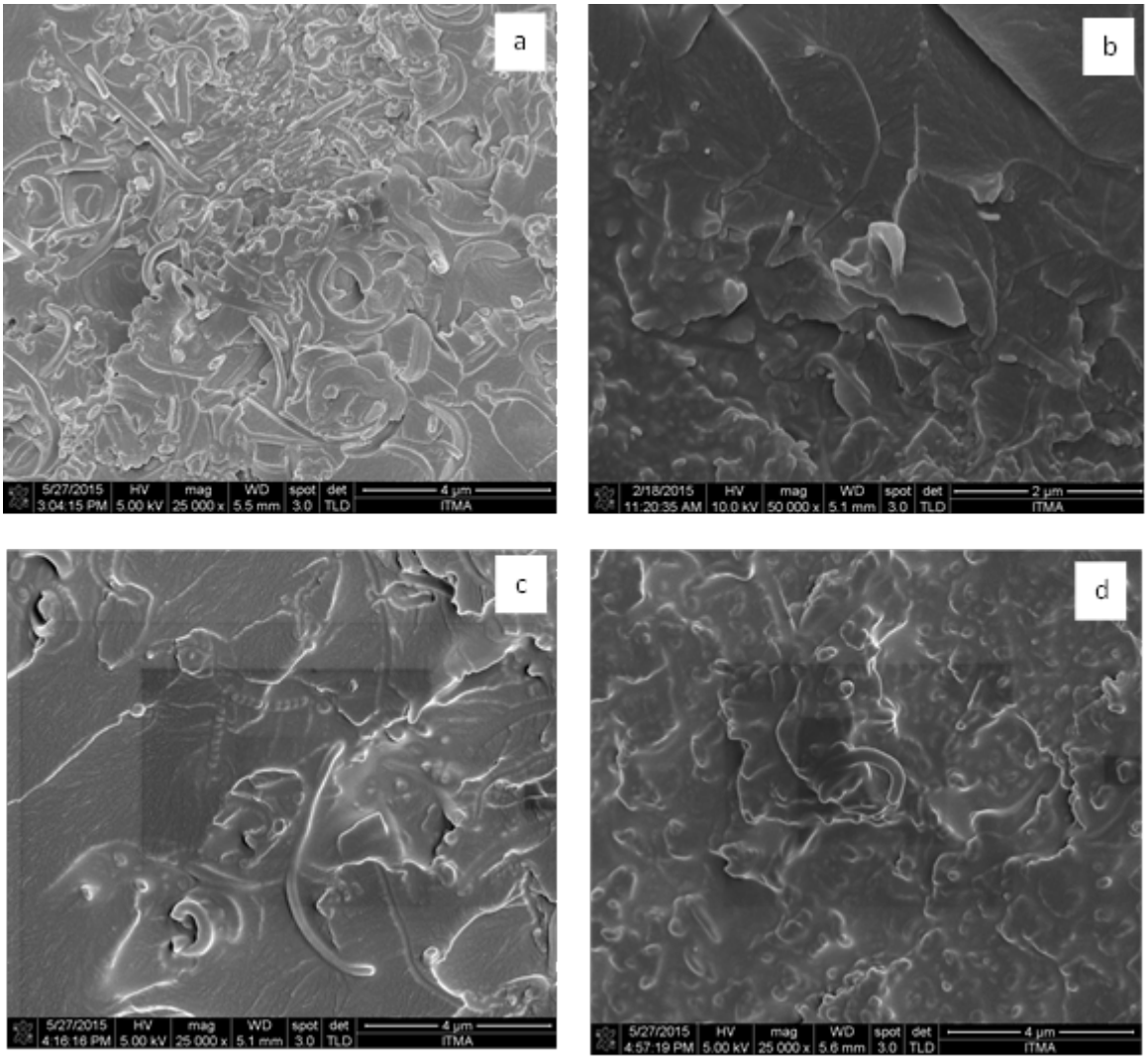


Fig. 7. FeSEM micrograph of synthesized spiral hybrid MWCNTs/epoxy resin (a) SF/E (b) SC/E (c) S1/E and (d) S3/E

3.4 Structural Analysis

Raman scattering of the composite was measured to further validate the presence of CNT and is also a well-accepted characterization method to evaluate the degree of structural order of

carbonaceous materials by using the ratio of the intensity of the D-band (I_D) to that of G-band (I_G). Figure 8 shows that the D-peak is around 1346 cm^{-1}, which corresponds to the defects and disorder, and the G-peak at 1579 cm^{-1}, associated with the signal form crystalline graphite. Larger peaks pf D-band and broad peaks of G-band indicate lower graphitization of carbon nanomaterials. According to Hiura *et al.,* [16], detected G-band corresponds to movement in the opposite direction of two neighbouring carbon atoms in a graphene sheet, while Cheng *et al.,* [17] reported that D mode attributed to defects in the tube ends, staging disorders and curved graphene layers. The ratio of the intensities of these two peaks (I_D/I_G) for SF, SC, S1, S2 and S3 are 0.98, 1.15, 1.07, 1.1 and 1.06 respectively. Higher degree of graphitization results in higher conductivity. This suggest that many defects occur contributed from hollow and spiral structure results in defective structure or lower degree of graphitization in the synthesized CNTs. The ratio of I_D/I_G also used to determine the quality of CNTs as also reported by Cheng *et al.,* [17]. Moreover, broad half width is also the evidence of the presence of disorder and/or distortion in the synthesized CNTs as reported by Zhang *et al.,* [18]. Interestingly for all samples, the ratio of I_D/I_G for the D and G band are constant at around 1.1. Thus, it indicates that the samples probably contain long-range disordered graphitic carbon that is consistent with weak and broad diffraction peak at 26.577^0 in the XRD patterns.

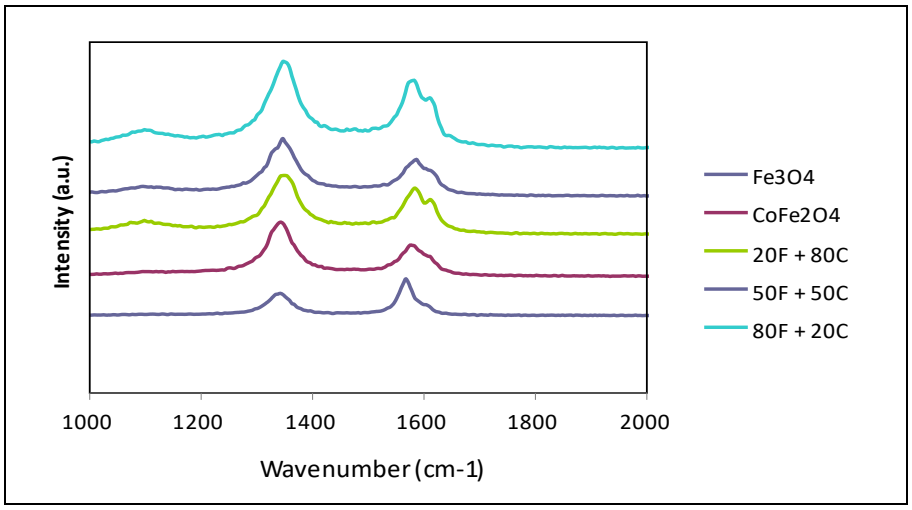


Fig. 8. Raman spectra of synthesized spiral hybrid MWCNTs SF, SC, S1, S2 and S3

3.5 Microwave Characterization

Figure 9 shows the reflection loss of synthesized spiral hybrid MWCNTs/epoxy resin of S1/E, S2/E and S3/E at different thicknesses and measured by using Vector Network Analyzer (VNA) at frequency range of 8 to 18 GHz. As for thickness fixed at 1 mm, the reflection loss peak was not observed since it resonated at much higher frequency range. On the other hand, for sample at thickness of 2 mm, the reflection loss peak start to appear at much lower frequency range as the mixture amount of Fe_3O_4 (F) as catalyst is increase from 20wt% to 80 wt%. As the thickness increase from 1 mm to 3 mm, the reflection loss peaks formation is shifting and resonates towards lower frequency range. This can be understood according to the geometrical effects of matching condition in which the thickness of the layer is quarter wavelength to the thickness of the material. In general, the reflection loss peak that can be observed in the figure also showing reflection loss value of less than -10 dB absorption. This shows that the sample is suitable to be used as an EM wave absorber by referring to the absorption peak refers to application at certain frequency. Reflection loss (RL) value of less than -10 dB indicates that the sample can absorbed 90% of the EM

wave signal. As for S1/E and S2/E, it can be observed that the RL peaks of thickness 3 mm are -23 dB (12 GHz) and -21 dB (14 GHz) respectively. However, for sample S3/E, two thicknesses of 2 mm and 3 mm can be observed with the RL values of -19 dB (16 GHz) and -21 dB (9.5 GHz) respectively.

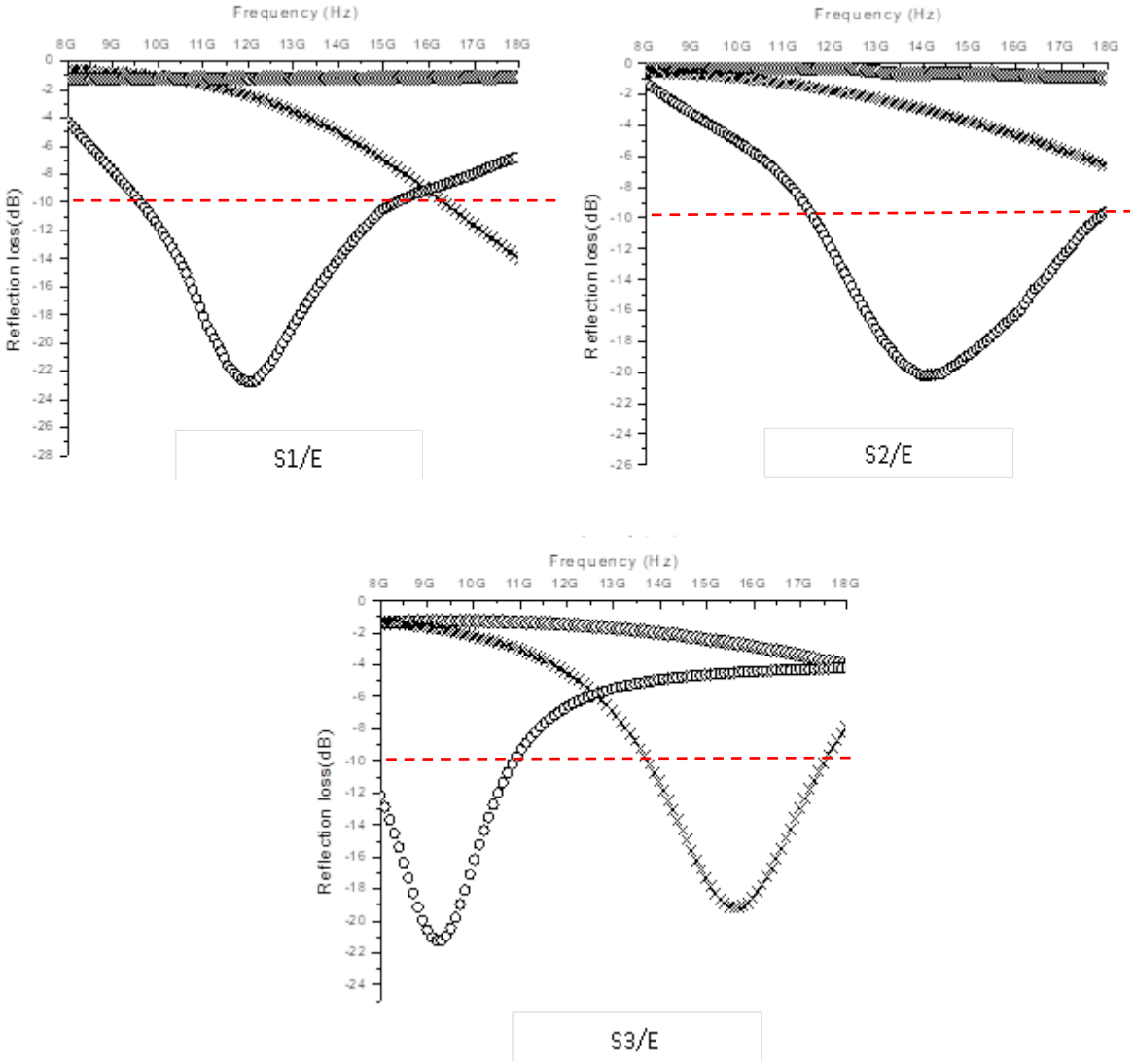


Fig. 9. Reflection loss of synthesized spiral hybrid MWCNTs/epoxy resin (a) S1/E (b) S2/E and (c) S3/E

As marked by dotted line in the figure, it indicates that the frequency bandwidth having RL characteristics of more than 90% of the EM wave signal being absorbed. The bandwidth for S3/E at thickness of 2 mm is 4 GHz (13.5 – 17.5 GHz). On the other hand, the bandwidth values for thickness 3mm for S1/E, S2/E and S3/E are 6 GHz (9.5 – 15.5 GHz), 6 GHz (11.5 – 17.5 GHz) and > 4GHz (<8 – 11 GHz) respectively. The reflection loss of the synthesized MWCNTs loaded into epoxy matrix plays the role by absorbing the EM wave and attenuating the radiation via the interaction between interior electrons and exterior microwave radiation. Defects form in the polymer composite samples act as polarization centers that contribute to stronger microwave absorption and Qin and Brosseau [14] and Michielssen *et al.,* [19] reported that it was attributed mainly to the dielectric relaxation in the sample.

The multiwalled CNTs can effectively guide and dissipate the incident EM wave when it is homogeneously dispersed in the matrix. The epoxy composites play a critical role in the absorption

of EM waves by absorbing the EM wave energy and mitigating the radiation. When the MWCNTs is well dispersed in the matrix, the whole composite sample present lower conductivity that causes the incident EM wave to effectively enter the absorbing material rather than being reflected on the surface. These events occur via the interaction between interior electrons and exterior EM wave radiation and inner electrons of the epoxy composites. The hollow tube makes the absorbing material has large internal cavity that makes it difficult to the EM wave entering the material to escape and be consumed by multiple reflection effects. Other than that, spiral MWCNTs also can enhance the transmission route as well and results in multiple reflection in the spiral coil. Moreover, the multiwalled structure that makes up the outer wall can effectively scatter most of the EM waves, disperse the concentrated EM energy and convert it into heat energy during the EM wave propagation. Yu *et al.,* [20] and Zhang *et al.,* [21] reported that the EM wave that are trapped in the fiber, can be reflected multiple times and converted it into heat because of the impedance mismatch between the fiber and the wall.

4. Conclusions

In summary, mixing nanometer particle size of two different magnetic materials (Fe_3O_4 and $CoFe_2O_4$) that act as catalyst have successfully synthesized spiral hybrid carbon nanotubes by using thermal vapour deposition tube (TVDT). Nanometer particle size of catalyst provides larger specific surface area that is enough for the reaction sites during synthesis. In this research work, a series of different weight percentages of Fe_3O_4 and $CoFe_2O_4$ mixture used as catalysts was introduced into the EM wave absorption field for the first time and have achieved outstanding results. By having mutiwalled carbon nanotubes, it enhanced the transmission route of EM wave that propagates through sample. Moreover, combination of spiral, straightlike, hollow tube, curved and twisted fiber gives advantages to the sample by having multiple reflection in the polymer composite samples. Broad X-ray diffraction peak indicate lower graphitization of the sample that was related with higher D-band peak from Raman spectrum. The ratio of I_D/I_G also indicate higher defects produce in the polymer composite sample. This defect can enhance the EM wave absorption in the sample, since higher graphitization results in higher conductivity in the sample. The effective absorption bandwidth obtains for all sample with only 8wt% of spiral hybrid MWCNT being well dispersed into epoxy resin can reach to 6 GHz at thickness of 3 mm. The RL peak with the minimum value was obtained by S1/E with -23dB at 12 GHz. As the thickness increase, the RL minimum peak shift towards lower frequency range and was not observed in the graph. However, for thickness 3 mm for S1/E, S2/E and S3/E showing the values of RL is less than -10 dB that indicates 90% of the EM wave was absorbed in the sample. Thus, it shows that the mixture of two different magnetic materials that use as catalyst to synthesize MWCNT results in enhance EM wave absorption performance at higher GHz frequency range. In addition, the synthesized spiral hybrid CNTs/epoxy resin have the advantages of lightweight properties that provides good performance and efficient EM wave absorber.

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