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Preparation and Evaluation of Carbon Black-MWCNT Nano-composites for Microwave Absorption

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Abstract: ***Nano-composites in toroidal shaped sample have been prepared and electromagnetic (EM) and microwave absorbing properties at different thicknesses have been studied using simulation code for metal backed single layer absorber. The vector network analyser (Model PNA E8364B, Software module 85071E) attached with coaxial measurement set up has been utilised to investigate the EM and microwave absorbing properties of the samples in the frequency range of 2–18 GHz. The fabrication of the sample has been carried out using carbon black powder and multiwall carbon nano-tubes (MWCNT) in different ratios as filler in two component polyurethane (PU) matrix. The complex permittivity of the composite has been found to be frequency dependent. The findings reveals that the absorption performances of the composite sample having 250 mg carbon black and 50 mg MWCNT in one ml polyurethane showed minimum absorption peak of -26.2 dB ($R_{L,\ min}$) at a matching frequency (f_m) of 11.6 GHz and more than -10 dB loss in the frequency range of 10.3 GHz to 13.5 GHz for the sample thickness of 2.0 mm. The morphology and thermal behavior of the nano-composite samples have also been investigated through scanning electron microscope (SEM) and thermo gravimetric analysis (TGA) techniques.***

Keywords: MWCNT, Carbon black, Nano composite, EM properties, Reflection loss

1. Introduction

High level of integration for miniaturization of electronic circuits has generated enormous electromagnetic interference (EMI) problems at microwave frequencies, which has drastically increased the demand of effective and efficient microwave absorbers in civil as well as defense sectors because of their noble property to eliminate or mitigate electromagnetic wave pollution. These materials are also strategically utilized to reduce the radar signatures for stealth applications.

Electromagnetic radiation, particularly at microwave frequencies are likely to interfere with electronics sub modules having poor EMI shielding and thus leading substantial damage to their intended functionality. The EMI shielding of both electronic modules and radiation sources is hence needed and is inevitably required for effective address of such interference problems. With the advent of ultra-fast electronic and telecommunication systems, demands for reliable microwave absorber to improve the EMI shielding has been ramped up. Further, Stealth technology for defence is another area, where these microwave absorbers are employed for effective counter measures against radar surveillance [1]. Application of such microwave absorbing coatings on the exterior surfaces of military aircraft and aerial vehicles helps to conceal from radar with phenomenal wider range of operation [2].

The first electromagnetic wave absorber was deployed, to improve the front to back ratio of a 4 GHz antenna, in around 1930s [3]. Since then various methods, techniques and material compositions have been reported towards development for microwave absorbers. Microwave absorbing materials can be manufactured by a variety of magnetic and dielectric materials composition in powdered form, loaded in various kinds of polymeric binders [4–13]. The most of the polymeric matrices are transparent to microwaves, and absorption in these materials occurs mainly due to interactive loss processes of dielectric and magnetic dipoles of the particulates suspended in these matrices. As fillers, Graphene and MWCNTs have been reported potential candidates for Microwave absorption [14].

Being resonant type, sample thickness plays a crucial role in the absorbing properties of such composites. In case of resin type, excessive loading of particulates of pigments quiet often leads to heavy cracks in the cured samples, as the resin molecules are unable to bind the pigments-particulates together, while low loading of pigments in the same resin reduces absorption properties. Thus optimised controlling of the composition, fill factor, and thickness improves the performance of these absorbers significantly.

Hence, in principle, the following are the key parameters of the dielectric composite governing the microwave absorption properties;
1. Complex permittivity ($j\varepsilon''$)
2. Fill factor of particulate

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3. Thickness of the composite

Although we have prepared many nano-composite samples with different composition of fillers, in this work, we have reported the performances of the best nano-composite sample with the composition of 250 mg carbon black and 50 mg MWCNT in one ml polyurethane (PU). The nano-composite has shown minimum absorption peak of -26.2 dB ($R_{L,\ min,}$) at a matching frequency (f_m) of 11.6 GHz for 2.0 mm thickness.

2. Experimental

2.1 Composite Preparation

The nano-composite preparation has been carried out by using MWCNT (sigma Aldrich, 95% pure) and conducting carbon black powder (CBP) (Senka Carbon, India). Both the components have been thoroughly mixed in acetone medium in mortar and pestle in two pack polyurethane matrix consisting of polyol-8 (Ciba-Geigy, Switzerland) and hexamethylene di-isocynate (E-Merck, Germany) mixed in the ratio of 50:50. Materials used and their sources are given in table 1. The mixture was homogenized in mortar and pestle and then poured in the mould followed by curing it under heat and pressure in a hydraulic press (Heating rate-20^0C/min, $Temp._{max}$-120^0C, Pressure-100kg/m^2, 2 hrs heating).

Materials	***Sources***
Two pack polyurethane (PU) matrix consists of Polyol-8 and Hexa-methylene di- isocynate mixed in ratios 50:50 in Acetone medium	-
Polyol-8	Ciba-Geigy, Switzerland
Hexa-methylene di- isocynate	E-Merck, Germany
MWCNTs	Sigma Aldrich, 95% pure
Carbon black powder (CBP)	Senka Carbon, India

TABLE -1

50 mg MWCNTs were mixed with 250 mg CBP in one ml PU and prepared the specimen in toroidal shape with an outer diameter of 7.0 mm, an inner diameter of 3.0 mm so as to fit in a co-axial waveguide sample holder.

2.2 Microwave Measurements

Microwave absorbing properties have been measured using coaxial line method. Electromagnetic parameters (complex permittivity and Complex permeability) of CBP/PU composite filled with MWCNTs were determined using AGILENT vector network analyser Model PNA E8364B (figure 1) in the frequency range of 2–18 GHz.

Further the reflectivities (R) with different thicknesses have been calculated by using the following equations

$$R_L(dB) = 20 \log_{10} \left| \frac{Z_{in} - 1}{Z_{in} + 1} \right| \quad (1)$$

$$Z_{in} = \left(\frac{\mu_r}{\varepsilon_r} \right)^{\frac{1}{2}} \tanh \left[j \left(\frac{2\pi f d}{c} \right) (\quad r \quad r)^{\frac{1}{2}} \right] \quad (2)$$

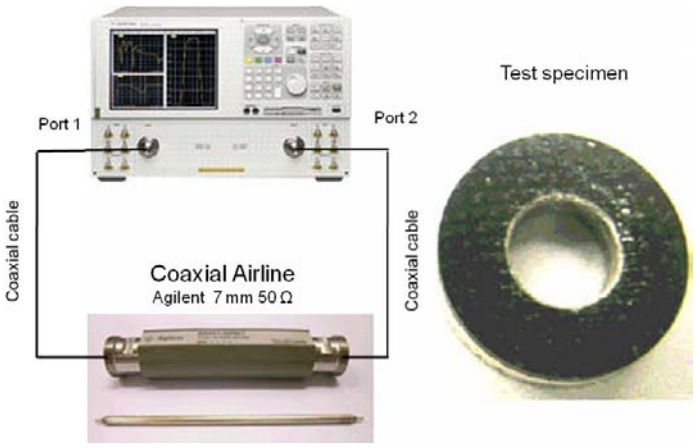


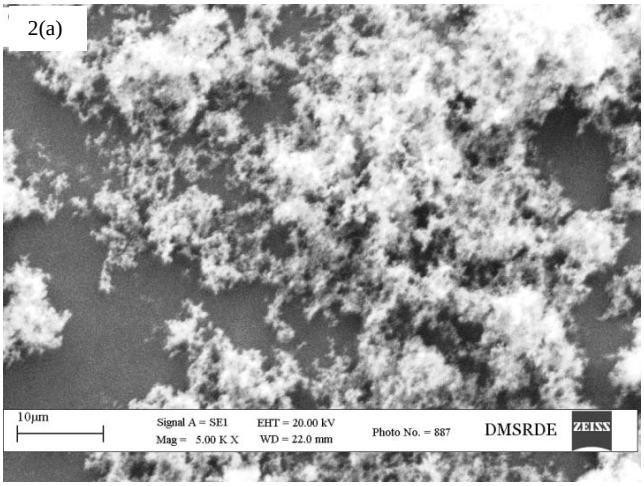
Figure 1: System setup and prepared specimen for microwave measurements

where Z_{in} is the normalized input impedance at the interface of the material and free space. $\varepsilon_r = \varepsilon' - j\varepsilon''$ and $\mu_r = \mu' - j\mu''$ are respectively the complex permittivity and complex permeability of the material. The real part of the permittivity/ permeability is a measure of the extent to which the material will be polarized or magnetized by the application of electric or magnetic field respectively, whereas the imaginary part is a measure of the energy loss incurred in re-arranging the alignment of the electric or magnetic dipoles according to applied ac fields, d is the thickness of the absorber, c is the velocity of light and f is the frequency of microwave in free space.

3. Results and discussion

3.1. Morphological Properties

Morphological properties of Powder sample of conducting carbon black, MWCNT and prepared nano-composite have been analyzed by scanning electron microscopy (SEM) (Carl Zeiss EVO-50).



SEM images in figure 2(a) shows highly agglomerated globular particles of carbon black powder where as in figure 2(b) depicts entangled tubes of MWCNTs.

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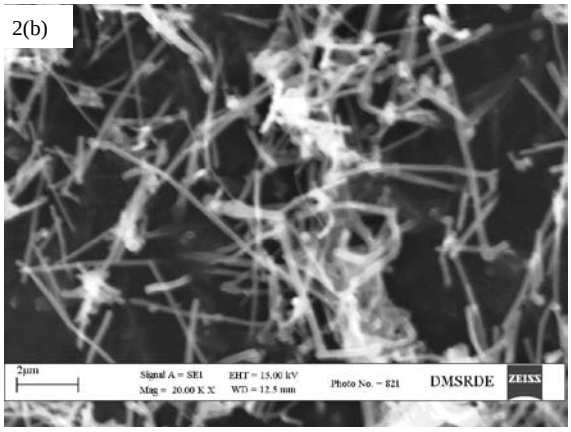

Figure 2: SEM micrographs of (a) Carbon Black Powder (CBP), (b) MWCNTs Powder

Figure 3 shows the SEM image of prepared nano-composite showing localised MWCNT dispersed in CBP/PU matrix.

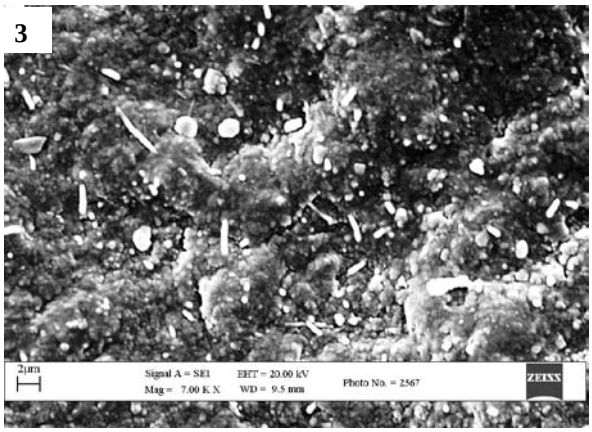

Figure 3: SEM micrographs of nanocomposite of MWCNT dispersed in CBP /PU Matrix

3.2 Thermal Properties

Thermo gravimetric analysis (TGA) has been carried out to study the thermal stability of the prepared nano-composite sample.

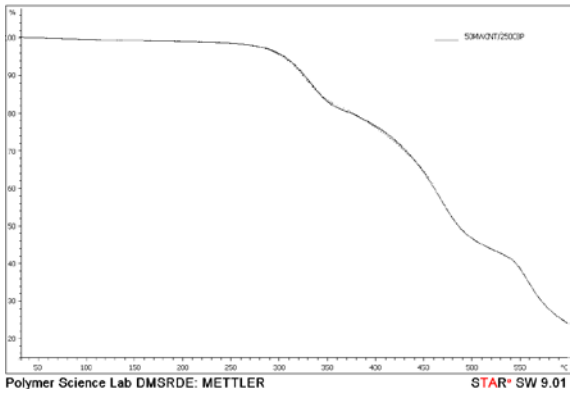

Figure 4: TGA of MWCNTs dispersed in CBP/PU

Figure 4 shows the TGA plot of prepared nano-composite which exhibits weight loss in several steps. But the prepared nano-composite is found to have a thermal stability at least up to 290 ^{0}C.

3.3. Permittivity Spectra

Figure 5 (a) and (b) shows the variations of the dielectric ε_r (ε' and ε'') and magnetic parameters μ_r ($\mu' \approx 1$ and $\mu'' \approx 0$) respectively of nano-composite material in the frequency range of 2–18 GHz. Conducting MWCNT and Carbon black powder mixed in PU (insulating) matrix leads to variation of the dielectric and magnetic parameters with frequency producing large accumulation of charges due to the dynamic nature of the incident electromagnetic wave.

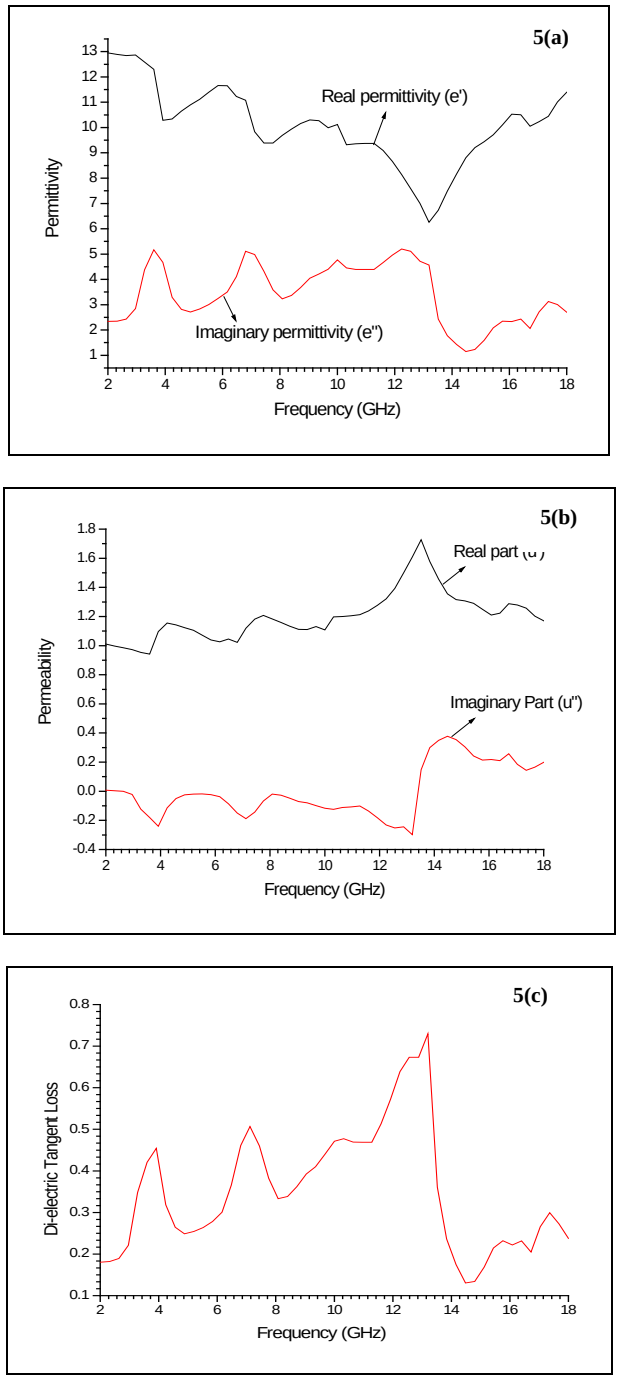

Figure 5: (a) Electric permittivity, (b) Magnetic permeability and (c) Di-electric Tangent loss (tan δ_e) vs. frequency variations of nano-composite

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This leads to formation of small localized conducting areas in the nano-composite leading to the observed pattern of the electromagnetic parameters. MWCNTs encapsulated in the CBP are considered to play an important role in improving the electric loss of composite. The electromagnetic parameter and tangent loss results in the variation of Z_{in} and thus affects the absorbing properties.

3.4. Microwave Absorbing Properties

The reflectivity of the prepared nano-composite sample having 250mg carbon black and 50 mg MWCNT in one ml polyurethane for various sample thickness has been calculated using experimentally obtained values of ε_r and μ_r. Figure 6 depicts the variation of the reflectivity with frequency regime of 2- 18 GHz.

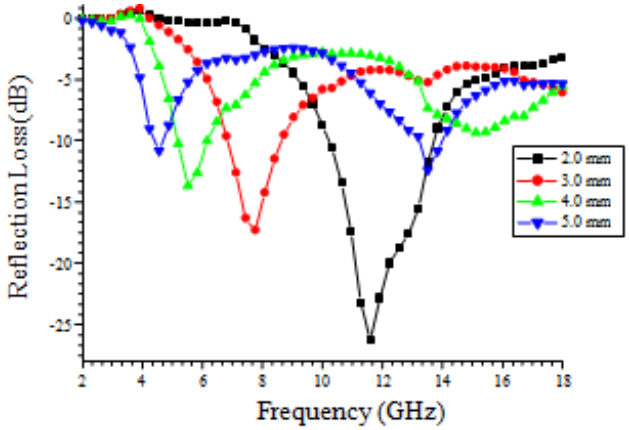

Figure 6: Frequency dependency vs. Reflection loss of composite for different thicknesses

The maximum absorbing peak is found to be -26.23 dB at 11.6 GHz which is in the range of X wave band and the corresponding value of matching thickness (d_m) is 2.0 mm. The bandwidth of the reflection loss (R_f) below −10 dB is 3.22 GHz (13.52 GHz-10.30 GHz).

4. Conclusion

We have successfully prepared the Carbon black and MWCNT PU based torroidal shaped nano-composite. Sample having 250mg carbon black and 50 mg MWCNT in one ml polyurethane with 2 mm thickness has shown more than 10 dB reflection loss in the frequency band of 10.30 GHz to 13.52 GHz with peak absorption of -26.23 dB at matching frequency of 11.6 GHz. Samples of relatively less thickness have shown relatively larger reflection loss towards higher end of frequencies. Thus the prepared carbon black and MWCNT based composite may be utilized in stealth technology as well as EMI shielding for particular band of microwave frequencies.

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