



ISSN: 2350-0328

**International Journal of Advanced Research in Science,
Engineering and Technology**

Vol. 3, Issue 3, March 2016

Microwave Absorption Properties of MWCNT and NiZn Ferrite Nano-fillers/TPU based Nano-Composites

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ABSTRACT: Toroidal shaped nano-composite samples having bi-filler nano-particles (filler 1: MWCNT & filler 2: Ni Zn nano-ferrite) with varying weight contents thoroughly mixed in thermo-plastic poly-urethane (TPU) matrix have been successfully prepared. Microwave absorption properties (stealth properties) of prepared nano-composite have been studied. Simulation studies for metal backed single layered absorbers have been carried out for studying the electromagnetic (EM) absorbing properties for 2.0 mm thicknesses of the nano-composites. The vector network analyser (Model PNA E8364B, Software module 85071E) attached with coaxial measurement set up has been utilized to investigate the complex permittivity and permeability. Microwave absorbing properties were examined by utilizing the measured values of complex permittivity and complex permeability of the absorber in the frequency range of 2-18 GHz. Reflection loss R_L (dB) vs. frequency variation have been also determined for 2.0 mm thicknesses of the nano-composites employing the simulation code. SEM and TGA were performed to analyse the morphological and thermal behaviour of the nano-composite. The complex permittivity & permeability of the nano-composites have been found to be frequency dependent. Higher reflection loss (R_L , dB) have been reported in C band (higher frequency side) and X band (higher frequency side) for the sample thicknesses of 2.0 mm.

KEYWORDS: MWCNT, Ni Zn Ferrite, Reflection loss, Microwave absorber, RCS, Permittivity and Permeability.

I. INTRODUCTION

Radar absorbing materials (RAM) have been initially used for low observable objects and further have been used for stealth technologies [1]. During the Second World War, RAM have been used as a counter measure to RADAR detection system [2]. Various techniques have been devised to achieve Stealth (against air-craft detection). These techniques are classified as (a) Geometry (Radar Absorbing Structures) [3], (b) Active loading [4], (c) Passive loading [5] and (d) distributed loading (Broad band) [6]. For effective stealth, Radar Cross Section (RCS) of the object must be minimised i.e. less than a critical value [7] to hide from enemy RADAR and the above techniques have been proven to be effective. Many researchers have worked on development of Microwave absorbing materials [8-9]. Currently, Abbas et al. are working on NiZn nano-ferrite, Carbon black and MWCNT filler based PU nano-composites since 2005 [10, 11]. In this present paper, we are discussing some aspects of the conducting and magnetic nano fillers (multi-wall Carbon nano tubes (MWCNT) and NiZn nano-Ferrite) dispersed in thermoplastic Poly-urethane matrix based nano-composites.

II. EXPERIMENTAL

A. Materials and method of synthesis

Nano-composite samples preparation has been carried out by using bi-fillers (multi-wall carbon nano tubes and Nickel Zinc nano-ferrite) nano-particles in thermo-plastic poly urethane (TPU). The multi-wall carbon nano tubes (MWCNT) was procured from M/s. Sigma Aldrich and Ni Zn nano-ferrites ($Ni_{0.5}Zn_{0.5}Fe_2O_4$, APS-50nm) from M/s. MK Nano, Canada. These fillers in different proportions have been thoroughly homogenised in thermoplastic poly-urethane (TPU) matrix as shown in table 1 below:

Sample Code	Filler 1 (Ni Zn Nano-ferrite) Wt %	Filler 2 (MWCNTs) Wt%	Matrix (TPU) Wt %
10F40C	10	40	50
15F35C	15	35	50
20F30C	20	30	50
25F25C	25	25	50
30F20C	30	20	50
35F15C	35	15	50
40F10C	40	10	50

Table 1: Nano-composite samples Compositions

The two pack thermoplastic poly-urethane (TPU) consists of polyol-8 (Ciba-Geigy, Switzerland) and hexamethylene di-iso-cynate (E-Merck, Germany) mixed in 50–50 ratios. The mixture (fillers +TPU) was homogenized and then put in the mould followed by curing it under heat and pressure for a definite time in a hydraulic press. The samples were prepared in toroidal shaped (figure 1 (b)) with an outer diameter of 7.0 mm, an inner diameter of 3.0 mm to fit in coaxial waveguide sample holder. The materials and their sources are listed in table 2 below:

MATERIAL	SOURCE
1. Multi wall Carbon Nano-tube, 95% pure	M/s. Sigma Aldrich
2. Nickel Zinc Ferrite (Nickel Zinc Iron Oxide) Nano-powder $NiZnFe_2O_4$ (Ni: 0.5 Zn: 0.5), 99.9% Pure, APS: 50 nm	M/s. MK Nano, Canada
3. Two pack thermo-plastic polyurethane (TPU) matrix consists of (a) Polyol-8 (b) Hexa-methylene di- isocynate mixed in ratios 50:50	M/s. Ciba-Geigy, Switzerland M/s. E-Merck, Germany

Table 2: Materials & Sources

B. Microwave measurements

Microwave absorbing properties of the nano-composite samples have been studied using coaxial line method. Electromagnetic parameters (complex permittivity and Complex permeability) of composite were investigated using AGILENT vector network analyser (figure 1 (a)) Model PNA E8364B in the frequency range of 2–18 GHz.

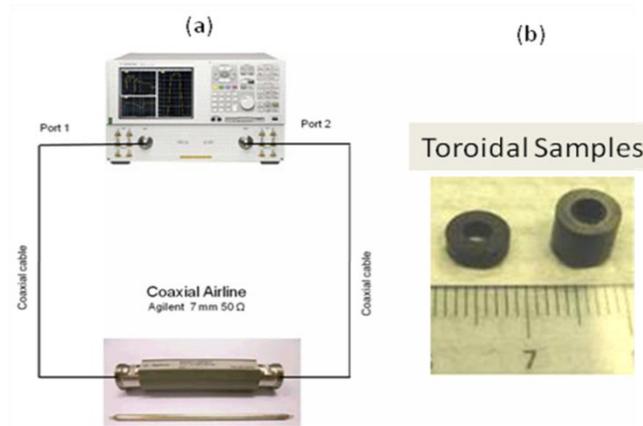


Figure 1: (a) Vector Network Analyser (VNA) for micro-wave measurements, (b) Samples

The reflection loss (R_L) with different thicknesses (t) have been calculated by utilising the well established equations (1) and (2) given below:

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

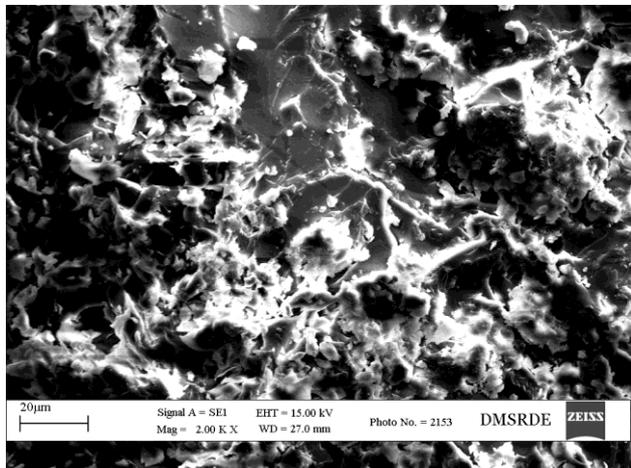
$$Z_{in} = \left(\frac{\mu_r}{\epsilon_r} \right)^{\frac{1}{2}} \tanh \left[j \left(\frac{2\pi f t}{c} \right) \sqrt{(\mu_r \epsilon_r)} \right] \quad \dots\dots\dots(2)$$

where Z_{in} is the normalized input impedance at free space and material interface, $\epsilon_r = \epsilon' - j\epsilon''$ and $\mu_r = \mu' - j\mu''$ are the complex permittivity and permeability respectively of the material. Real part is a measure of the extent to which the material will be polarized or magnetized by the application of electric or magnetic field respectively while imaginary part is a measure of the energy loss incurred in re-arranging the alignment of the electric or magnetic dipoles as according to applied ac fields, t is the thickness of the absorber, and c and f are the velocity of light and the frequency of microwave in free space, respectively.

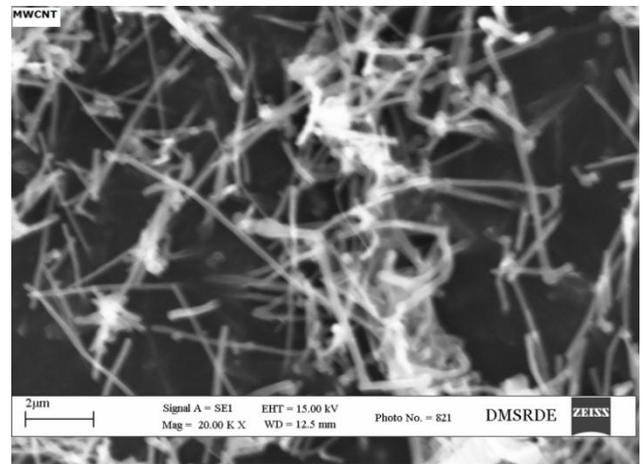
III. RESULT AND DISCUSSION

A. Morphological Properties

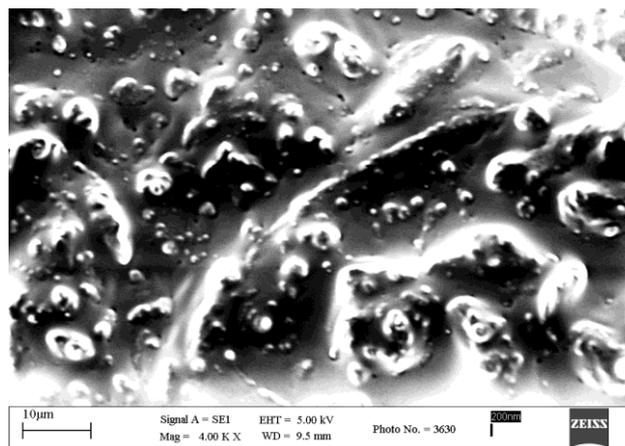
The surface morphologies have been studied with the help of Scanning Electron Microscope (SEM) images. The SEM images of Thermoplastic polyurethane (TPU), neat MWCNT, Nano spherical NiZn Ferrite filler in TPU are shown in figure 2 (a), 2(b) & 2 (c) respectively.



2(a)



2(b)



2 (c)

Fig. 2. Scanning Electron Micrographs (a) SEM of Thermoplastic Poly Urethane (TPU) matrix (b) Mutli-wall Carbon Nano-Tubes (MWCNT) (c) NiZn ferrite nano-filler in TPU matrix

Figure 2(a) shows the rubberised nature of virgin thermoplastic polyurethane (TPU) matrix. SEM micrograph in figure 2(b) shows that the needle shaped Mutli-Wall Carbon Nano-Tubes (MWCNT). Figure 2 (c) shows dispersion of spherical $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ (NiZn Nano-ferrite) in TPU matrix.

Thermal Properties

Thermo gravimetric analysis (TGA) has also been carried out (not shown) to study the thermal stability of the prepared nano- composite. The prepared nano-composites are found to have a thermal stability of at least up to 250 °C.

B. Permittivity Spectra and Permeability Spectra

The dielectric parameters (ϵ' , ϵ'') of MWCNT +Ni Zn Ferrite /PU nano-composites are shown in the figure 3 (a) and figure 3(b). The figure 3(a) shows that the variation of dielectric constants (ϵ') of the prepared nano-composites (MWCNT + NiZn ferrite in TPU matrix) in 2-18 GHz frequency range.

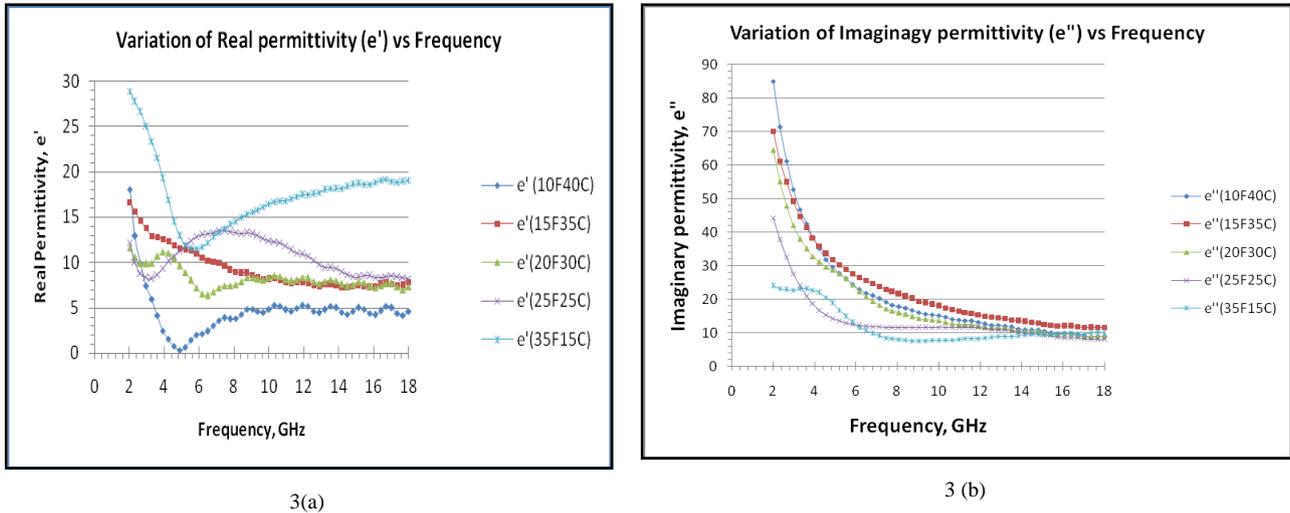


Fig. 3. Variation of the (a) Dielectric constant (ϵ') with frequency (GHz) (b) Dielectric Loss (ϵ'') with frequency (GHz) of Nano MWCNT and nano NiZn ferrite /TPU matrix based nano-composites

Figure 3 (a) shows as the ferrite content increases the dielectric constants (ϵ') increases. Also it is observed that the stichometry of the MWCNT is varying appropriately. In the figure 3 (b) the dielectric loss (ϵ'') parameter is found to vary (decrease) exponentially.

Figure 4 (a) shows the real permeability (μ') is frequency dependent in 2-18 GHz frequency range. Figure 4 (b) shows the variation of magnetic loss (μ'') with frequency in 2-18 GHz. The magnetic loss factor is found to vary with applied frequency in 2-18 GHz. With the increasing ferrite content & decreasing MWCNT content, the magnetic loss (μ'') shows a frequency dependent complicated behaviour.

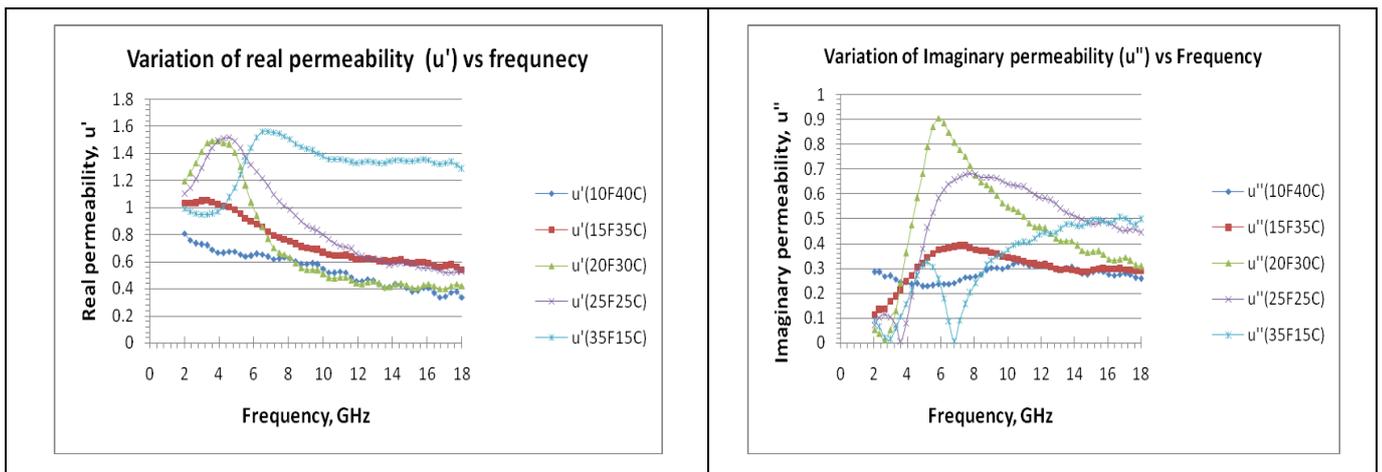


Fig. 4. Variation of (a) Real permeability (μ') and (b) magnetic loss (μ'') with frequency (GHz) of nano-composite samples

C. Microwave absorbing properties

The reflection loss (dB) of the prepared nano-composite samples for thickness (t) 2.0 mm have been calculated using experimentally obtained values of ϵ_r and μ_r . Figure 5 depicts the variation of the reflection loss (dB) with frequency of the prepared nano-composites with different compositions (table-1) of nano-fillers in the frequency range of 2 GHz - 18 GHz. It is evident from the figure 5, as the NiZn ferrite nano-filler content is increasing, the reflection loss is also increasing simultaneously. The optimum MWCNT content is the measure contributor in the reflection loss.

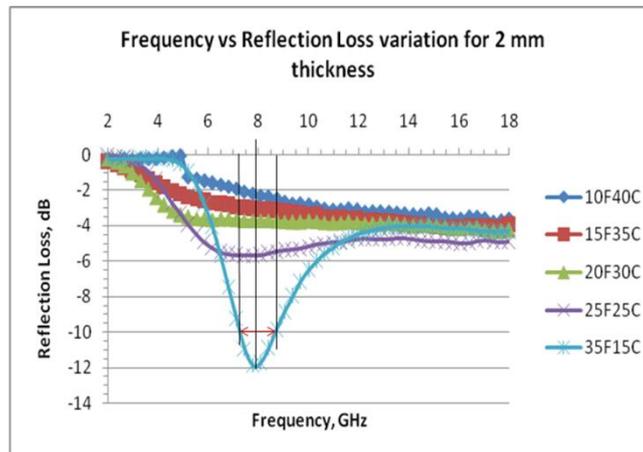


Fig. 5. Reflection loss (R_L , dB) vs. frequency (GHz) for $t = 2.0$ mm for various compositions of Nano-Composites

The maximum reflection loss observed for thicknesses are shown in the table 3.

Sample	Maximum Reflection Loss (R_L , Max)	Matching frequency (GHz)	Thickness t (mm)	Band width for reflection loss greater than -10 dB loss
35F15C	-12dB	7.9	2.0 mm	7.25 GHz to 8.75 GHz

IV. CONCLUSION

Toroidal shaped Nano-composites with bi-filler (MWCNT+Ni_{0.5}Zn_{0.5}Fe₂O₄) nano-particles in different proportions mixed in TPU (wt. 50%) matrix have been successfully prepared. The electromagnetic properties (complex permittivity & permeability) have been measured using vector network analyser (VNA). The reflection losses R_L (dB) for single layer metal backed condition have been successfully measured for 2.0 mm thickness of prepared nano-composites. For the nano-composite sample with 35% NiZn ferrite nano-particles (filler 1) and 15 % Multi-wall Carbon Nano-tubes (MWCNT) i.e. 35F 15C nano-composite, the maximum reflection loss (R_L , Max) of -12 dB has been obtained at the matching frequency of around 7.9 GHz. The Scanning electron micrograph shows the microstructure of TPU, MWCNT and NiZn ferrite in TPU. By TGA study, it is found that RAM was thermally stable upto 250 °C. The Electromagnetic parameters are found to be frequency dependent by Microwave analysis. Hence the above mentioned nano-composite may prove a promising Radar absorbing material for stealth application.

V. ACKNOWLEDGEMENT

Authors are grateful to Dr. Namburi E. Prasad, Director DMSRDE, Kanpur for permitting this work. The authors are also thankful to Dr. T.C. Shami and Mr. Alok Dixit of DMSRDE Kanpur for their support in microwave measurements. The authors also extend their thanks to Prof. (Dr.) Sangeeta Kale of DIAT Pune for her inspiration and support.

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ISSN: 2350-0328

International Journal of Advanced Research in Science, Engineering and Technology

Vol. 3, Issue 3, March 2016

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