Scheming of microwave shielding effectiveness for X band considering functionalized MWNTs/epoxy composites

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Abstract. Present typescript encompasses an extraordinary electrical and mechanical behaviors of carboxylic (–COOH) functionalized multiwall carbon nanotube (MWNTs)/epoxy composites at low wt.% (0.5, 0.75, 1 wt.%). Functionalization on the surface of the nanotube assists MWNTs in dispersing it into epoxy polymer in a respectable manner. Fabricated composites are exposed to different characterization techniques in order to examine the overall physical properties. Microwave shielding effectiveness (SE) for X band (8-12 GHz) and the flexural properties have been premeditated to predict the electrical and mechanical performances. It was found that the total SE of the nanocomposites was increased with the positive gradient of MWNT contents. The best result was recorded for 1 wt.% MWNT loading (SE of about 51.72 dB). In addition, incorporation of nanofillers enhanced the flexural modulus, flexural strength and micro-hardness of the resulting composites while comparing with neat epoxy. Nanocomposites with 0.75 wt.% MWNT loading demonstrated an increment of 101% in modulus than that of neat epoxy. The increment in mechanical properties was due to achievement of good dispersion quality, effective bonding between MWNTs and epoxy polymer analyzed by micrographs of fracture surfaces.

Keywords: MWNT/epoxy composite; functionalization; shielding effectiveness; flexural modulus; hardness

1. Introduction
Polymer-based composites are gaining more importance compared to metals and other commercial materials due to their light weight, low cost and easy processing [1]. Since the discovery of carbon nanotubes (CNTs), polymer-based composites find a new dimension for the application of this material in various structural parts, automobiles, civil engineering, electrical circuits etc. CNTs being such nanofiller combined with exceptional material properties like high specific strength and stiffness, high aspect ratio, high flexibility and high electrical conductivity. Further CNTs possesses diameter dependent specific surface area that offers available surface for interaction while incorporated into polymer matrix [2]. This interaction in fact is responsible in order to modulate the matrix properties in a significant manner.

However, using CNTs(MWNTs) as reinforcing agents in polymer matrix can be a pretty challenging task. Because, the presence of strong inter-tube interaction offered by the van der Waals’ forces creates a genuine difficulty to achieve qualitative dispersion of MWNTs into polymer. Till date, a number of techniques are available to overcome the problem of dispersion of CNTs into polymer. These include mechanical stirring, magnetic stirring, Ball milling, Calendering process, stir and extrusion, ultra sonication and high shear mixing etc. [3, 4]. The method of sonication among them...
has been most commonly used to disperse MWNTs in polymer like epoxy resin. Now the fact is that like dispersion interfacial adhesion between the CNTs and polymer like epoxy is also a critical issue tailoring which one can have sufficient stress transfer from the matrix to the MWNTs. That is, a strong interfacial adhesion between MWNTs and polymer is highly desirable for the effective performance of the resulting composites. A number of researches have been worked since a decade to improve this interfacial adhesion between MWNTs and polymer. According to them, attachments of some functional groups on the surface of MWNTs not only improve the adhesion rather helps in achieving uniform dispersion of MWNTs into polymer [4].

Now, in the present era, careful investigation reveals a critical need to develop versatile and effective novel materials that would suit to applications like EMI (Electromagnetic Interference) shielding and ESD (Electrostatic Discharge). Current material options that provide active SE are metals, metal-fibre filled plastics, polyacrylonitrile (PAN) nickel coated reinforced polymers, aluminium structures, nickel and copper metalized fabrics, and more recently, nano reinforced polymer composites (NRPCs) [5, 6]. Typical limitations found with materials used for shielding to prevent EMI are associated with corrosion susceptibility, lengthy processing times, high equipment cost for production and difficulty of material utilization mainly as conductive layers. NRPCs, like carbon nanotube (CNTs) reinforced polymer matrices seem to overcome some of these limitations because they are lightweight materials with design flexibility, corrosion resistant and suitable for mass production through conventional plastic manufacturing technologies such as extrusion and injection moulding [1].

Though a quantitative number of literatures are available on EM shielding i.e. SE of the non-functionalized MWNTs/polymer composites, a little work has been reported on SE of functionalised MWNTs/polymer composites. For example, polyaniline /functionalized coated MWNTs have been added to polymer matrices for EMI shielding materials and tested in the frequency range of 8.2-12.4 GHz (X band). According to their report, SE was about 23.3 dB for 7% MWNTs loading in polystyrene (PS) [7]. S.H. Park et al. [8] worked on the –COOH attached single walled CNT/RET polymer and they got 9-25 dB of SE taking 0.5–4.5 wt.% of fillers content. Grimes et al. [9] reported that SWNT-polymer composites possess high real permittivity as well as imaginary permittivity in the 0.5-2GHz range. They found that the permittivity decreases rapidly with increase in frequency.

In this present schoolwork the novelty lies with a different curing mechanism at the time of fabrication compared to the conventional techniques. Fabricating very low wt.% –COOH functionalized MWNT/epoxy composites, a details study of the EMI shielding has been performed in the X band frequency range (8-12 MHz). In addition, mechanical studies have been performed to observe the overall strength of the fabricated composites. So the prime goal here is to produce an effective shielding material along with the significant mechanical properties that can be used in marine, military, scientific electronic devices and communication instruments.

2. Materials and methods

In this present study, –COOH functionalised MWNT as reinforcing component and epoxy resin (Ciba-Geigy, araldite LY-556 based on bisphenol A, India) as matrix material have been used for the synthesis of the nanocomposites. Functionalised MWNTs were commercially purchased from NanoAmour, USA; having diameter of around 20–30 nm and lengths of about 10–30 μm with a carbon purity of > 95%. During fabrication epoxy resin (5.3-5.4 equiv/kg) has been cured with an aliphatic primary amine hardener (HY-951) in the wt. ratio 100/12.

Functionalised MWNTs with 0.5, 0.75, and 1 wt.% were taken for the fabrication of MWNTs/epoxy nanocomposites. The technique adopted for preparing the composite was solution reduction following ultra-sonication. Prior to the mixing of nanotubes to the epoxy, MWNTs were sonicated with ethanol solution to remove entanglement of the MWNTs bundle. Ethanol was added to epoxy resin also and mixed manually for reducing the viscosity of the epoxy. The obtained untangled solution of MWNTs was then dispersed with epoxy mixture using an ultrasonic processor. This procedure has been adopted following sufficient optimisation. After 6 hours of continuous sonication, the mixture was allowed to keep under vacuum chamber for removing air bubbles, generated due to sonication process for 12 hours. The suspensions were then mixed with the curing agent in the specified wt. ratio and mixed manually for 10 minutes. Thereafter, the final mixture were poured into open moulds and
kept at low temperature (4°C) for 2-3 days for the effective settling purpose. Neat epoxy samples without adding nanotubes were also fabricated in the same method as above for comparison purposes. The obtained cured composite in this process was then post cured at a temperature around 90°C for a few hours. A detailed description on the processing of the nanocomposites is given in our earlier work [10]. Prepared composites were designated as follows:

Neat epoxy composite: E_p
Nanocomposite with 0.5 wt.% of functionalized MWNTs loading: FC_{0.5}
Nanocomposite with 0.75 wt.% of functionalized MWNTs loading: FC_{0.75}
Nanocomposite with 1 wt.% of functionalized MWNTs loading: FC_1

3. Characterisation details

Shielding effectiveness (SE) of all the nanocomposites was experimentally investigated over a frequency range 8-12 GHz (X band), using rectangular waveguide with the help of Agilent PNA-L Network Analyzer (N5230C) and Agilent software 85071E. The operational range of the analyser was 10 MHz-20 GHz. Using S-parameters generated from the instrument, SE of the nanocomposites has been calculated. Method used for real and imaginary parts of the dielectric permittivity was ‘Transmission Epsilon Fast’ method with the accuracy of 1-2%. Flexural tests were accompanied according to ASTM D790–02 ‘Standard Test Methods for Flexural Properties of Un-reinforced and Reinforced Plastics and Electrical Insulating Materials’ on a universal testing machine (Instron 5967). The mode of test was three point bending and the rate at which the load was imposed was 2.0 mm/min. The span between two supports, width and thickness of the specimen were 60 mm, 5 mm and 12.5 mm respectively. In order to reproduce the results five specimens were tested in each group. The hardness of all composite specimens was measured using a micro-hardness tester. The measurement was done by the Vickers hardness test method, which is all about indenting the test material with a diamond indenter. In this method, the hardness is calculated by measuring the load applied and the average diagonal length of the indentation. A total of 10 points on each of the specimens were measured in order to get the average readings. Finally, field emission scanning electron microscope (Nova NANOSEM 450) was used to observe the fracture surface topography of all the composites specimens.

4. Results and discussions

4.1. Electromagnetic shielding effectiveness

The shielding effectiveness SE (dB) is defined as the ratio of power of the incident to the transmitted electromagnetic (EM) wave. It is expressed as [11],

\[ SE_T (dB) = 10 \log_{10} \left( \frac{P_T}{P_I} \right) = 20 \log_{10} \left( \frac{E_T}{E_I} \right) = \log_{10} \left( \frac{H_T}{20 \Omega} \right) \]

where, \( P_I \) (E_I or H_I) and \( P_T \) (E_T or H_T) are the power (electric field intensity) of the incident and transmitted EM wave, respectively.

When a microwave radiation is incident on a material surface, its energy is partly reflected, absorbed, and transmitted. The MWNTs/epoxy composites can attenuate the microwave radiation and absorb its energy. According to the classic transmission line theory, the reflectance (R), absorbance (A) and transmittance (T) can be expressed with the scattering parameters S_{11}, S_{12}, S_{21}, and S_{22} of vector network analyser (VNA) i.e.,

\[ T = \frac{|S_{11}|^2 + |S_{12}|^2 + |S_{21}|^2}{|S_{22}|^2}, \quad R = \frac{|S_{21}|^2}{|S_{11}|^2 + |S_{12}|^2 + |S_{21}|^2}, \quad A = \frac{|S_{11}|^2}{|S_{11}|^2 + |S_{12}|^2 + |S_{21}|^2} \]

The total electromagnetic interference shielding effectiveness (EMI SE) i.e., \( SE_T \) is the sum of contributions from absorption (SE_A), reflectance (SE_R), and multiple reflection (SE_M); for example, \( SE_T = SE_A + SE_R + SE_M \). When \( SE_T < 15 \) dB, it is usually assumed that \( SE_T = SE_A + SE_R + SE_M \) and when \( SE_T > 15 \) dB then \( SE_T = SE_A + SE_R \) (i.e., SE_M is negligible). These two attenuation factor i.e., SE_R and SE_A can be conveniently expressed as:

\[ SE_R = 10 \log_{10} (1-R), \quad SE_A = 10 \log_{10} (1-A) = 10 \log_{10} \left( \frac{P_I}{P_T} \right) \]

Here in the fig. 1, EMI SE of various –COOH functionalised MWNTs/epoxy with different loadings of MWNTs (0.5, 0.75, 1 wt.%) in the measured frequency region (8 to 12 GHz, X band) have been portrayed. The neat epoxy showed very less attenuation with \( SE_T \approx 0.4 \) dB that is why not shown in
this fig. It is clearly visible that addition of functionalised MWNTs to the epoxy increased the total shielding effectiveness. In addition, the SE was increased upon increasing the wt.% of MWNTs. The highest EMI SE ≈51.72 dB was provided by the nanocomposite FC₁, whereas, FC₀.5 and FC₀.75 showed maximum SE of 51.03 dB and 51.55 dB respectively. Sung-Hoon Park et al. [8] also reported the increment of SE for the gradual increment of contents of CNTs. They have obtained SE of low wt.% functionalised CNT epoxy composites maximum up to 30 dB in the same frequency range. Saib et al. [12] reported the EMI SE of CNT/polyethylene composites with low wt.% of CNT loading at 11 to 28 dB in a frequency range from 20–40 GHz. Singh et al. [13] measured the EMI SE in the range ≈11–20 dB with greater wt. % of CNTs (4.2, 15.1, 20.4 wt.%). So it can be concluded that, in our case, inclusion of lower wt.% of MWNTs to the epoxy polymer greatly improved the EM wave attenuation and hence contributed profoundly to the total SE. This kind of enhancement of SE may occur mainly for the achievement of qualitative dispersion of the MWNTs in the epoxy. In addition, formation of strong secondary bonds between MWNTs and the induced functional group may be the cause of such enormous increment.

On the line of investigation, fig. 2 revealed that the contribution of absorption attenuation in total SE was intense than the reflection attenuation for all the cases. This was due to the fact that MWNTs has free electrons in its skeleton, so it can absorb energy and helps in attenuating the microwaves [14]. Further the high surface area of MWNTs also may be a reason for its good microwave absorption behaviour. In addition, the individual MWNT and MWNT bundles in the epoxy composite samples can absorb the microwave energy and attenuate the radiation via the interaction between interior electrons and exterior microwave radiation. It is also well established that compared with any other CNTs like single and double walled CNTs, MWNTs have more defects due to their complicated structures which facilities in greater absorption of EM waves [15]. Apart from these, even if covalent attachment of functional groups to the surface of MWNTs can improve the efficiency of load transfer, it must be noted that these functional groups may introduce defects on the walls of the perfect structure of nanotubes [16]. These defects in turn may help in absorption of EM waves acting as polarization centres.

When EMI shielding materials are exposed in an external EM field, the radiation would induce two kinds of electric currents. One is the displacement current due to the presence of polarization effect including unpaired point defects and polarized interfaces. The displacement current contributes to the real part of permittivity (ε'). The other one is the conduction current due to the presence of free electrons in the material. The conduction current contributes to the imaginary part of permittivity (ε") and causes dielectric loss [17]. The frequency dependence of permittivity in the current investigation is represented in fig. 3. This frequency dependence reflects the fact that a material's polarization does not respond instantaneously to an applied field. The response must always be causal (arising after the applied field) which can be represented by a phase difference. For this reason, permittivity is treated as a complex function of the frequency of the applied field. The real and imaginary part represents the stored energy in the material and energy dissipation respectively by the material itself. Fig. 3(a) and (b) showed the real and imaginary permittivity spectra of the epoxy as well as nanocomposites.
Fig. 2: Shielding effectiveness of (a) FC_{0.5} sample (b) FC_{0.75} sample, (c) FC_1 sample samples respectively. The spectra showed that $\varepsilon'$ (Fig. 3a) value decreased with increase in frequency. This can be ascribed to the inability of the polarization vector to maintain in phase movement with incident high frequency electromagnetic radiation. Further $\varepsilon''$ increases with loading of higher amount of CNT which may be due to the Maxwell-Wegner interfacial polarization [18].

Fig. 3: Real part of permittivity vs. frequency (a) imaginary part of permittivity vs. frequency (b) of epoxy and nanocomposites

4.2. Flexural properties

Fig. 4 illustrates the flexural properties of epoxy and functionalized MWNTs/epoxy composites. The addition of any kind of nanofiller increases the stiffness and strength of the matrix material, as long as a proper impregnation with the matrix and a sufficient adhesion is ensured. Concordantly, in this case, epoxy containing 0.5 and 0.75 wt.% of MWNTs i.e., FC_{0.5} and FC_{0.75} were showing 89% & 101% modulus enhancement in comparison to pure epoxy with modulus 4501 MPa while relatively lower (40%) improvement in modulus was observed in FC_1. The strength values were in the order like FC_1 < FC_{0.5} < FC_{0.75} with 84, 93, 148 MPa respectively in comparison to epoxy having strength of 66 MPa (Fig. 4). The functionalized nanotubes turned out to be more efficient concerning an improvement of the flexural modulus, thus again proving the relevance of surface functionalization. Good dispersion of MWNTs and cross-linking of functional groups present on the surface of MWNTs with polymer that restricts the mobility of polymer chains in turn enhanced strength and modulus [2].
In addition, good interfacial adhesion between the functionalized MWNTs and matrix, high aspect ratio and high modulus and strength of MWNTs, also contributed to the increment in overall properties. However, decrease in strength at higher MWNTs content can be attributed to the following effects: firstly, creation of voids and agglomeration of MWNTs in the sample that may have decreased the strength; secondly, improper dispersion of MWNTs at higher loading can reduce the strength. In addition, Agglomerations of MWNTs act as areas of weakness and can cause stress concentration leading to lower flexure properties of epoxy samples modified with MWNTs at higher loading.

4.3. Hardness

The strength of all the composites is studied using a micro hardness test er. The harness value is calculated using the Vickers hardness (HV) method is given by $HV = \frac{2F \sin (136.5/2)}{D^2} = 1.854 \frac{F}{D^2}$ (approximately). Where, $F$ = load in kgf and $d$ = arithmetic mean of the two diagonals, $d_1$ and $d_2$ in mm.

The obtained hardness value of epoxy matrix was 12 MPa while addition of functionalized MWNTs increased it to a great extent. An increment of 75%, 166% and 133% hardness values were observed respectively for FC$_{0.5}$, FC$_{0.75}$, FC$_1$ nanocomposites with respect to neat epoxy. There may be a number of reasons for which hardness of the nanocomposites can be increased. Firstly, materials having higher crystallinity have greater hardness. Secondly, the reinforcement effect which is caused due to the addition of high aspect ratio, high modulus, and high strength nanofillers (MWNTs). These reinforced fillers contributed to the increment in hardness value. Third is the homogeneous dispersion of the fillers (MWNTs). Since CNTs are resilient materials, homogeneously dispersed CNTs can add rigidity and hardness via interfacial interaction with the epoxy matrix. Thus, the soft polymer matrix holds together when challenged by an applied force and the mechanical properties like modulus, hardness are improved [19]. Fourth is the formation of long range ordered structure which can be accomplished by the functionalization of MWNTs. So, all of these said reasons are equally accountable for the dramatically enhancement in hardness values in this case. Hardness results agreed well with flexural test results with the finding that among the specimens FC$_{0.75}$ showed the maximum improvement. However, for higher concentration in case of FC$_1$, the hardness value got deteriorated than FC$_{0.75}$ though it was still higher than other specimens. Reduction in hardness value at FC$_1$ may be
due to the formation of voids due to addition of higher nanoparticle contents [20]. In addition, higher concentrated MWNTs may act as stress concentrator in the composite which can be the cause behind the decrement in mechanical properties.

4.4. Fracture surface analysis

The fracture surfaces of composites give first information about fracture mechanisms and the influence of particle modification on the fracture behaviour. To investigate the failure mechanism of epoxy as well as functionalized MWNTs/epoxy composites, the fracture surfaces of failed specimens were examined. The micrograph of neat epoxy in fig.6(a) demonstrates a typical feature of brittle fracture behaviour, thus accounting for the low fracture toughness of the unfilled epoxy. The surface is smooth, with very directionally deformation lines, which means that the crack propagated is not interrupted. However, the fracture surfaces of composites in fig.6(b) seem to be comparatively rougher. The ridge patterns and rivers marks indicate that the composites presented a more ductile fracture. This means that the energy required for the propagation of the crack increased. This is in accordance to the obtained results on mechanical characterisation (Fig.4), where it was concluded that the sample with best mechanical properties (with highest strength and modulus) was with 0.75 wt. % functionalized MWNTs.

Fig.6: Comparison of fracture surfaces between (a) neat epoxy, (b) nanocomposite (FC0.75)

The degree of functionalization has a certain influence on the mechanical properties of this resulting composite. The presence of carboxylic acid groups on the surface enables a better dispersion of MWNTs and reduces the amount of impurities. These introduced functional groups lead to covalent bonds with the epoxy resin. Higher magnification in fig.7 (a),(b) shows that in FC0.5 and FC0.5 system, nanotubes were well separated and uniformly embedded in the epoxy resin. Stronger adhesion at the MWNTs/epoxy interface was obtained due to the reaction between carboxyl group and DGEBF (hardener used in present work). Although, several deboned interface was observed in fig.7(b) superior adhesion at the functionalized MWNTs/epoxy interface resulted in the failure of nanotubes without pulling out from epoxy matrix. A spatial bonding of the reactive groups at the interface, enables a partial debonding of the interface, but allows a crack bridging.

When the MWNTs loading increased to 1 wt%, the failure mode of nanocomposite changed (Fig. 8). In most locations, the crack damage started from the interface due to which MWNTs were pulled out. In these areas, MWNTs suffered from external force leaving the smooth resin matrix exposed weak interfacial bonding. This type of stress concentration could be due to agglomeration of several MWCNTs and formation of bubbles leading to voids due to viscous mixture of epoxy and MWCNTs. However, strong bonding was noticed in some areas that may be attributed to functionalization process. Agglomeration facilitates network formation or CNT-to-CNT interactions through surface contact (or tunnelling) and hence facilitates the electrons flow through the formed CNT network [21]. Hence, best shielding effectiveness obtained in this specimen.
Fig. 7: Fracture surfaces of (a) FC$_{0.5}$, (b) FC$_{0.75}$

Fig. 8: Low and high resolution of the fracture surface of FC$_1$

5. Conclusion

Due to the low temperature curing and incorporation of functionalized MWNTs to the epoxy matrix an improved dispersion as well as strong interfacial bonding was realized in the final composites. This facilitates significantly the total shielding effectiveness and flexural properties of epoxy. The highest EMI SE of 51.72 dB was provided by the nanocomposite FC$_1$. According to the flexural data FC$_{0.5}$ and FC$_{0.75}$ were showing 89% & 101% modulus enhancement in comparison to pure epoxy while relatively lower (40%) improvement in modulus was observed in FC$_1$. The strength values were in the order FC$_1$<FC$_{0.5}$<FC$_{0.75}$ with 84, 93, 148 MPa respectively in comparison to epoxy having 66 MPa. Nanocomposites containing 0.5, 0.75 and 1wt.% of CNTs were demonstrated an increment of 75%, 166% and 133% hardness values respectively with respect to neat epoxy. Micrographical analysis confirms better dispersion in FC$_{0.75}$ resulting maximum enhancement in mechanical properties and found agglomerates in FC$_1$ resulting maximum improvement in electrical properties.

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