ORIGINAL ARTICLE

Enhanced microwave shielding and mechanical properties of multiwall carbon nanotubes anchored carbon fiber felt reinforced epoxy multiscale composites

B. P. Singh · Preetam Bharadwaj · Veena Choudhary · R. B. Mathur

Abstract Multi-walled carbon nanotubes (MWCNTs) were grown on carbon fiber (CF) felt by chemical vapor deposition that resulted into strongly anchored carbon nanotubes (CNTs) on the CF surface. These multiscale preforms were used as the reinforcement in epoxy resin to develop multi scale CF felt-MWCNT/epoxy composites. The flexural strength (FS) and the flexural modulus (FM) of the composites were found to increase with increasing amount of CNTs grown on CF felt surface. FS improved by 37 %, i.e. 119 MPa compared to 87 MPa for CF felt/epoxy composites prepared under identical conditions. The FM also improved by 153 %, i.e. 15.7 GPa compared to 6.2 GPa for the CF/epoxy composites. The incorporation of MWCNTs on the CF felt produced a significant change in the electromagnetic interference shielding of these composites which improved from −21 to −27 dB for CF felt-MWCNT/epoxy multiscale composites in the Ku band (12.4–18 GHz) and indicates the usefulness of these strong composites for microwave shielding.

Keywords Multiwalled carbon nanotubes · Microwave shielding · Carbon fiber felt · Multiscale composites

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Introduction

Electromagnetic interference (EMI) is a complex and technical challenge these days which adversely affects and degrades the effective performance of a system. It is the undesired conducted or radiated electrical disturbance from an electrical or electronic apparatus which can interfere with the operation of other electrical or electronic apparatus. Today shielding against EMI is practiced by government, military, medical, private industries and R&D laboratories. The importance of EMI shielding relates the high demand of speedy communication and reliability of electronic equipment, digital devices and wireless technology. Shielding is considered acceptable if it attenuates the intensity of electromagnetic field to an adequate level established by regulation of the electronic devices. It is also needed for deterring electromagnetic form of spying. It is therefore vital to develop new materials with enhanced EMI shielding capacity. It is well known that the shield should be conducting, therefore usually the shielding techniques focus on the use of standard metals and their composites which have shortcomings of limited mechanical flexibility, heavy weight, corrosion, physical rigidity and difficulty in tuning the shielding efficiency (Pande et al. 2009; Mathur et al. 2010; Qin et al. 2010). Further, metals mainly reflect the radiation and cannot be used in applications where absorption is the prime requisite.

Polymer composites containing carbon-based fillers [e.g. graphite, carbon black (CB), carbon fiber (CF), carbon nanofiber (CNF) and carbon nanotubes (CNTs)] have been extensively investigated for use as EMI shields owing to their unique combination of electrical conduction, polymeric flexibility and light weight. The high conductivity, small diameter, high aspect ratio and superior mechanical properties of CNTs make them an excellent choice to

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create conductive composites for high performance EMI shielding materials (Pande et al. 2009; Mathur et al. 2008b).

Epoxy resins are well established as thermosetting matrices for advanced structural composites, displaying a series of promising characteristics for a wide range of applications owing to their excellent mechanical properties, low cost, ease of processing, good adhesion to many substrates, and good chemical resistance (Garg et al. 2011; Meenakshi and Sudhan 2011; Ruban et al. 2013). Several fibrous reinforcements, e.g. CF, glass fibers, aramid fiber (Kevlar fibers), etc. (Barrau et al. 2003; de Morais et al. 2003) have been used with epoxy resin as matrix in structurally strong composite materials for commercial applications such as in aerospace industry.

It is suggested that CNT reinforced epoxy composites can also be structurally strong EMI shielding materials. For a high value of EMI shielding, high loading of CNTs were used in the past. In one such study, Huang et al. (2007) have reported EMI-SE of 18 dB with 15 wt% small single walled carbon nanotubes (SWCNTs) and 23–28 dB with 15 wt% long SWCNTs in the frequency band of 8–12.4 GHz. Li et al. (2006) observed the SE of 49.2 dB at 10 MHz with 15 wt% long CNTs. They reported that at higher frequencies, the composites exhibited shielding effectiveness of 20 dB at 1 GHz for both 10 and 15 wt% SWCNT loadings. It is quite evident from the earlier reports that the reinforcement effect of CNT can be realized only for low loading level (<1 %) and afterward it starts to decrease dramatically due to several issues, e.g. dispersion, alignment and interface (Du et al. 2007; Lau and Hui 2002). It has been reported that beyond 0.6 wt% of MWCNTs, CNTs tend to agglomerate (Yang et al. 2009) resulting in poor bending strength and modulus of the composites.

Therefore, different routes have been adopted, notably a combination of CNTs and CF (Qian et al. 2010; Thostenson et al. 2009; Kim et al. 2007) that involves dispersion of CNTs into epoxy matrix followed by impregnation in carbon fabric. However, the above compositions had the limitation of reinforcing <1 % CNT beyond which the viscosity of the CNT-epoxy mix was raised so much that it became impossible to impregnate the fabric completely (Singh et al. 2012). In a recent review article, by Lubineau and Rahaman (2012), studies conducted on the role of nano reinforcements in mechanical properties were reviewed. It was concluded in the review that compressive strength was improved up to 39 %. According to them, there is a real potential for improving the compressive properties but the clear improvement in the flexural behavior was not realized. There is a further scope to study and clarify the issue. In our previous study, PAN-based CF fabric was used as a substrate to grow MWCNT and it was used as

reinforcement in the epoxy resin (Singh et al. 2012; Mathur et al. 2012). Using the technique of CNT-grown CF fabric, 11.5 wt% CNT of CF fabric was incorporated as one of the reinforcements in the epoxy composites. Beyond 11.5 wt% of CNTs, it was not possible to grow CNTs on the PAN-based CF fabric. Here, we used three-dimensional porous CF felt and up to 18 wt% of MWCNT of CF felt was grown using chemical vapor deposition and these were used as reinforcements in epoxy resin. We also show that this multiscale composite can also serve as an efficient EMI shielding material with the combination of light weight, high strength and high electrical conductivity.

Experimental

Growth of CNTs on CF felt

CF felt (3D CF network) was chosen as a carbon substrate for the growth of CNTs. CNTs were grown on CF felt using the setup and the experimental conditions described earlier (Mathur et al. 2008a). Different amounts of CNTs were grown on the substrate by varying the deposition time. The substrate was removed after each experiment and weighed to determine the amount of CNT grown. The substrate was then sonicated in alcohol for 30 min to remove the loosely bonded CNTs on it. The weight uptake of the CNT grown on to the CF felt, selected for preparing composites, was calculated only after sonication. Different such batches were prepared with CNT uptake of 7.2, 8.4, 10, 11.6, 13.4, 14.7 and 17.9 %.

Preparation of CF felt-CNT/epoxy multiscale composites

The CNT-CF felt preforms were impregnated with epoxy matrix (Huntsman LY-556 resin and HY 5,200 hardener) to keep the reinforcement (CF + CNT) to the matrix ratio always as 40:60 by volume. The impregnated preforms were dried at room temperature to form the prepregs. These prepregs were stacked together to form a laminated assembly of the CNT-CF felt multiscale preforms. The laminates were then compression molded in a mold at 80 °C followed by a curing cycle, i.e. 2 h at 80 °C followed by 4 h at 150 °C to obtain composite bars of size 60 mm × 20 mm × 2.5 mm. The multiscale composite batches prepared with above different CNT uptake, i.e. 7.2, 8.4, 10, 11.6, 13.4, 14.7 and 17.9 % were designated as FLCNT7, FLCNT8, FLCNT10, FLCNT12, FLCNT13, FLCNT15 and FLCNT18, respectively. In a separate experiment, laminates of as received CF felt were also compression molded under similar condition to prepare CF felt/epoxy composites designated as FLCNT0. Here we are

able to grow CNTs up to 18 wt% because of high porosity in the felt and a large exposed surface area of the CF as compared to other CF networks as CF tows or CF fabric (Mathur et al. 2008a). For normal commercial CF-based composites for structural applications, the fiber volume content is generally kept between 50 and 60 % by volume. It was not possible to use that high volume percent of the CF along with MWCNTs because of the large surface to volume ratio of the nanotubes. It was found that beyond 40 vol % of CF in such cases the epoxy resin would not completely wet the reinforcement and the samples showed delamination under loading during flexural tests. The properties are compared on the basis of 40 vol% CF felt-based epoxy composites.

Characterization

Scanning electron microscope (SEM, Leo S-440) was used to observe the morphologies of reinforcements (CNT and CF felt) and the fractured surface of the composites after mechanical testing to correlate the fracture behavior with the mechanical properties. The morphological

details of CNTs were also observed under high-resolution transmission electron microscope (HR-TEM, Tecnai G20-stwin, 200 kV). A Raman spectrum of the CNTs was recorded using Renishaw Raman Spectrometer with green laser (514 nm) as an excitation source. The FS and FM were measured on Instron universal testing machine (model 4,411) under a three point flexural loading as per the ASTM standard D790. The span to depth ratio of the samples was kept as 20. At least five samples were tested for each batch and average is reported. The electrical conductivity of the composite samples was measured by four point contact method (Singh et al. 2008, 2011) using a Keithley 224 programmable current source for providing current. The voltage drop was measured by Keithley 197A auto ranging digital microvoltmeter. The values reported in text are averaged over five readings of voltage drops at different portions of the sample. The EMI shielding effectiveness measurements of the composites, i.e. FLCNT0, FLCNT8 and FLCNT18 were carried out on an Agilent E8362B vector network analyzer in the frequency range of 12.4–18 GHz (Ku band).

Fig. 1 SEM image showing a uniform growth of CNTs (18 wt%) over the surface of the CF felt preform, b, c multiscale preform showing intra-filamentary growth of CNTs, d masking of CF monofilament with CNTs
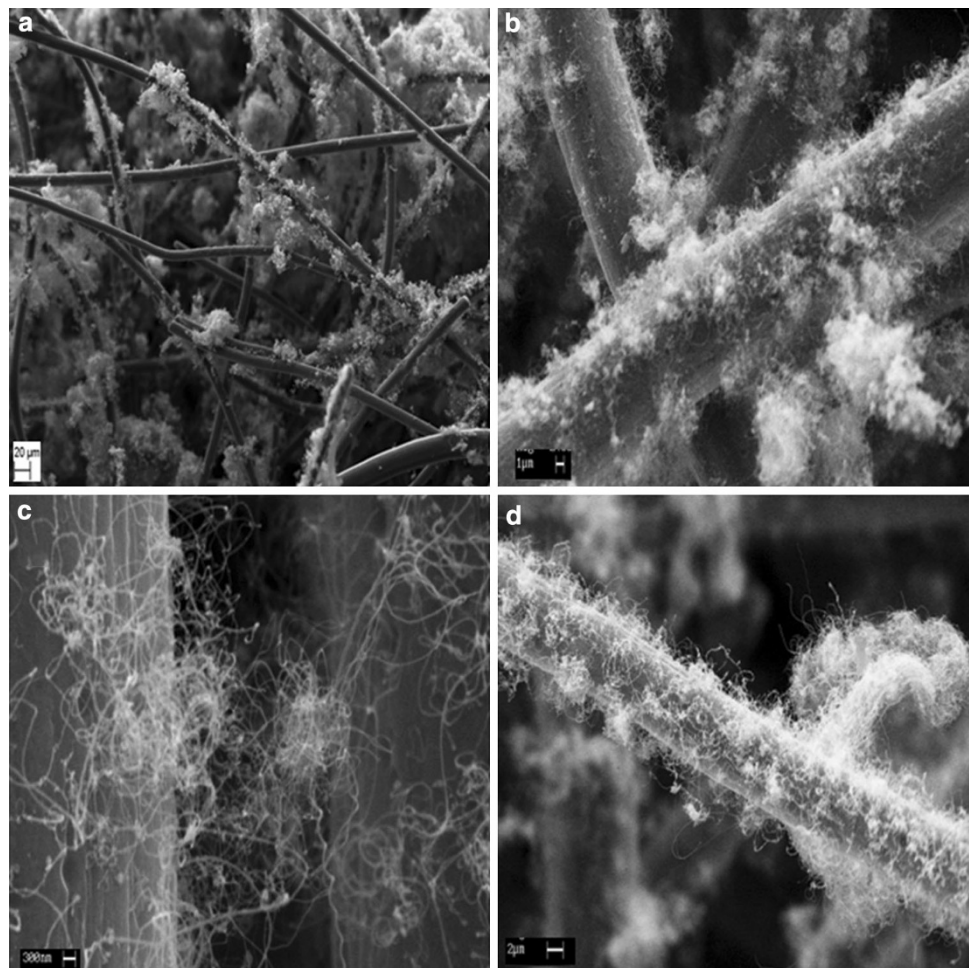
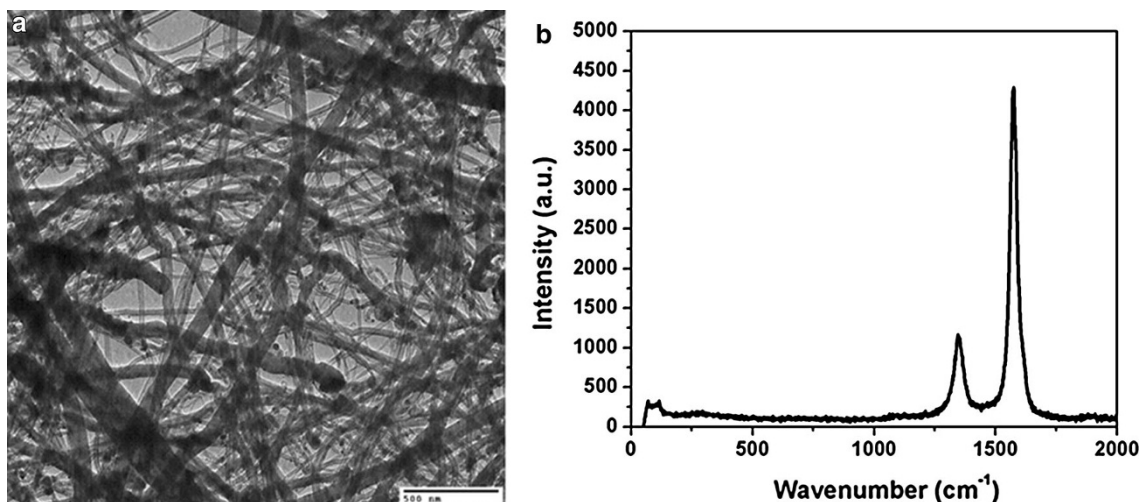Fig. 2 **a** TEM and **b** Raman spectra of the scraped material from the CF felt

Results and discussion

Morphological features of the CF-CNT felt preforms

The SEM images of the multiscale preform show a uniform growth of CNTs throughout the surface of the felt (Fig. 1a). These tubes seem to be anchored strongly in the intra-filamentary spaces and the porosity between the cross-over spaces created by the CF as shown in Fig. 1b and c and Fig. 1d shows a copious growth of CNTs on the carbon monofilament virtually covering its entire surface. The tubes are multiwalled in nature with metallic impurities as revealed by TEM (Fig. 2a). This is also confirmed by the Raman spectra of the scrapped material shown in Fig. 2b. We do not observe any peak in the region 150–300 cm^{-1} corresponding to radial breathing mode in single walled carbon nanotubes. The large value of the intensity ratio of

the G to D band also suggests that these tubes are of reasonably good quality. The preform thus forms a nice combination of micro and nano reinforcement. The growth mechanism of CNTs on the surface of the CF felt has already been discussed in our previous study (Mathur et al. 2008a).

Mechanical properties of the composites

The results on the mechanical properties of the multiscale composites with different concentrations of MWCNTs grown on the CF felt are presented in Fig. 3a, b. The flexural strength (FS) is increased to 119 MPa for the FLCNT18 as compared to only 87 MPa for the FLCNT0. An overall improvement of 37 % is therefore recorded for FLCNT18. The flexural modulus of the multiscale composites also shows gradual improvement with increasing concentration
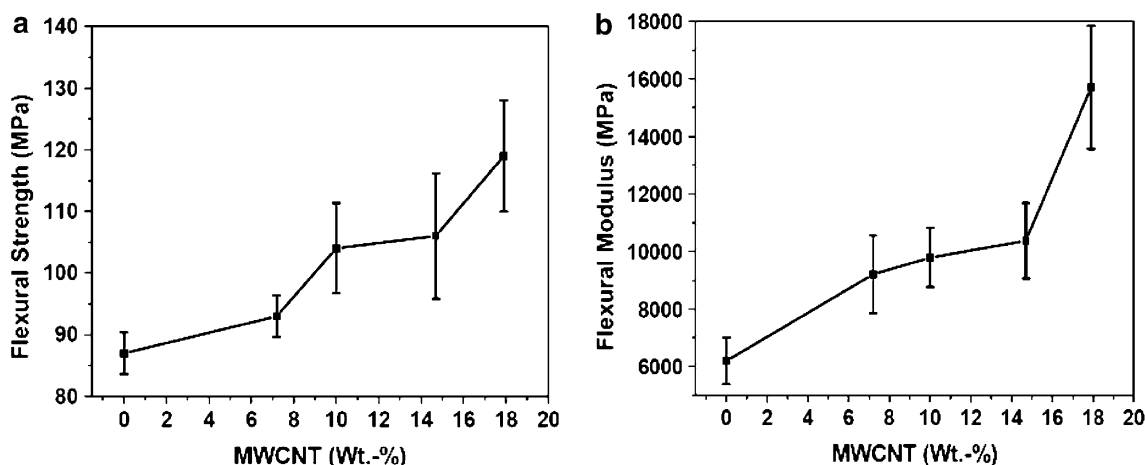

Fig. 3 **a** Variation in the FS of the CF felt-MWCNT/epoxy composites with increasing CNTs content in the multiscale preform. **b** Variation in the FM of the CF felt-MWCNTs/epoxy composites with increasing CNT content in the multiscale preform
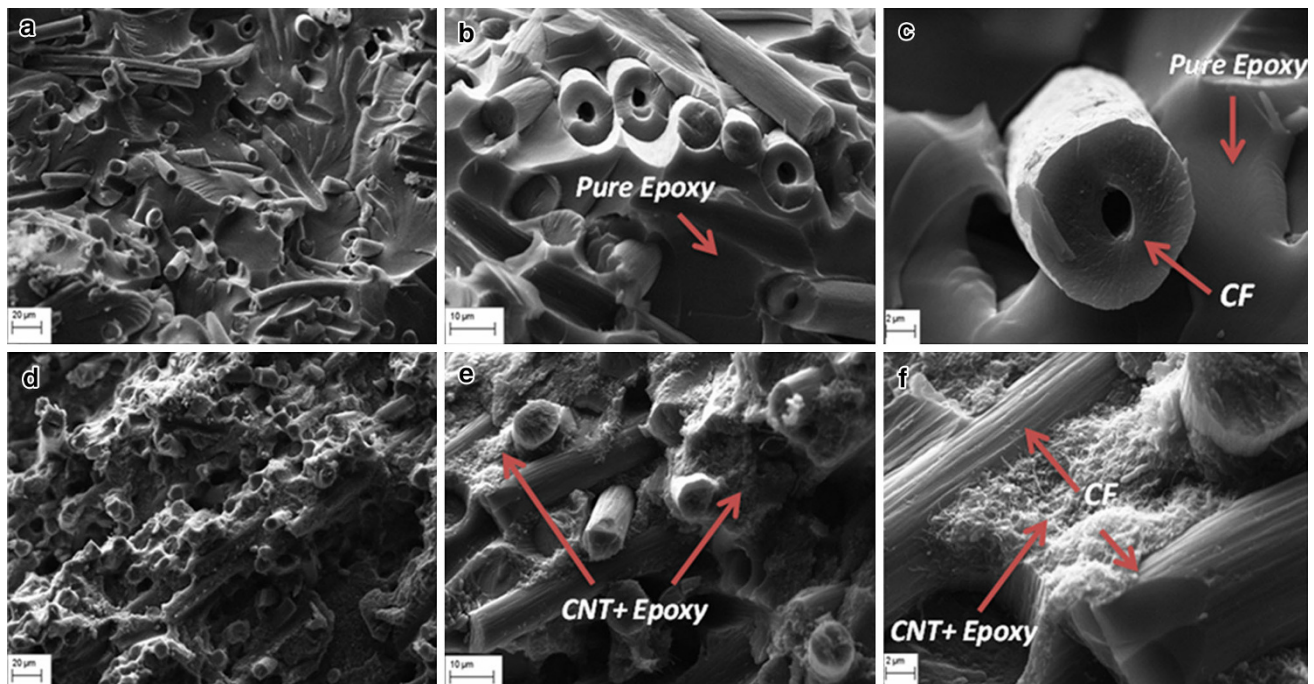
Fig. 4 SEM image of the **a–c** fracture surface of FLCNT0 at different magnifications, **d–f** fracture surface of FLCNT18 at different magnification

of MWCNTs (Fig. 3b). The flexural modulus (FM) of FLCNT18 shows a value of 15.7 GPa, an improvement of $\sim$153 % over FLCNT0. These results show that by incorporation of the MWCNTs in the CF felt as reinforcement ($\sim$18 wt% of CF felt), it is possible to achieve higher values of FS and FM compared to CF felt/epoxy composites.

The reason for the improvement in the mechanical properties of the multiscale composite can be understood by the SEM images of the cross section of their fractured surfaces. The fractured surface of the FLCNT0 (without CNT) is shown in Fig. 4a–c and the fractured surface of FLCNT18 is shown in Fig. 4d–f. It is observed that the MWCNTs are uniformly distributed in the entire volume of the epoxy matrix and interlocking between two CF. The CNTs-epoxy composite matrix seems to make strong bonds with the fiber showing less CF pull outs. The presence of MWCNTs would arrest the crack propagation in the matrix resulting into enhanced shear transfer stress for FLCNT18 as compared to FLCNT0. The fracture surface of FLCNT0 (Fig. 4a–c) shows brittle failure with fiber pull outs and uninhibited crack propagation through the matrix. The mechanical properties of FLCNT0 are comparatively lower therefore.

Electrical conductivity of the CF felt-MWCNTs/epoxy multiscale composites

Figure 5 shows the dependence of room temperature dc electrical conductivity (σ_{dc}) of the multiscale composites on the loading level of MWCNT. Pure epoxy is an electrical insulator with dc electrical conductivity value of $\sim$10^{-15} S cm^{-1} (Barrau et al. 2003). The observed rise in σ_{dc} with increased MWCNT content may be attributed to the increase in the number of conducting links within electrically insulting epoxy matrix. The added MWCNTs not only form the new links within epoxy matrix but also form conducting bridges between CF (shown in Fig. 4d–f). Such links are missing for FLCNT0 as shown in Fig. 3a–c. When the CNTs loading is increased to 18 wt%, the electrical conductivity increased to 18.3 S cm^{-1} for FLCNT18 compared to 3.2 S cm^{-1} for FLCNT0. The achieved conductivity value was near the prescribed range for microwave shielding. Therefore, good microwave shielding response is expected.

EMI shielding effectiveness measurement

EMI shielding was measured on rectangular strips of composites inside the sample holder. The holder matches the internal dimensions of Ku band (12.4–18 GHz) waveguide placed between the two ports of Vector Network Analyzer (VNA E8263BAgilent Technologies).

The EMI shielding is a direct result of reflection, absorption and multiple internal reflection losses at the existing interfaces, suffered by incident electromagnetic (EM) waves. EMI shielding effectiveness can be expressed as (Saini et al. 2009, 2011; Singh et al. 2013).
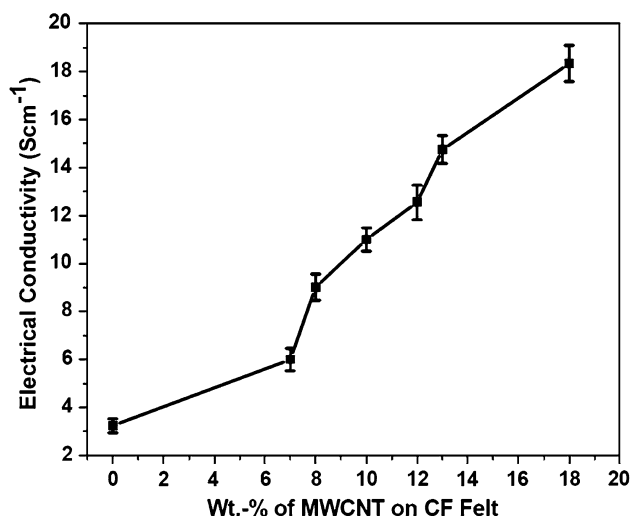
Fig. 5 Variation in the electrical conductivities of the CF felt-MWCNT/epoxy composites with increasing MWCNTs contents in the preforms

$$SE_T(dB) = 10 \log\left[\frac{P_T}{P_I}\right] = 20 \log\left[\frac{E_T}{E_I}\right] = 20 \log\left[\frac{H_T}{H_I}\right] \quad (1)$$

where P_I (E_I or H_I) and P_T (E_T or H_I) are the incident and transmitted electromagnetic powers (electric or magnetic field), respectively. The total shielding effectiveness can further be divided into three components:

$$SE_T = SE_R + SE_A + SE_M \quad (2)$$

where SE_R, SE_A and SE_M are shielding effectiveness due to reflection, absorption and multiple reflections, respectively. The scattering parameters S_{11} (or S_{22}) and S_{21} (or S_{12}) of a two port network analyzer can be related with reflectance and transmittance as:

$$T = \left|\frac{E_T}{E_I}\right|^2 = |S_{21}|^2 = |S_{12}|^2 \quad (3)$$

and

$$R = \left|\frac{E_R}{E_I}\right|^2 = |S_{11}|^2 = |S_{22}|^2 \quad (4)$$

The absorbance (A) can be written as:

$$A = (1 - R - T) \quad (5)$$

Here, it should be noted that the absorption coefficient is given with respect to the power of the incident EM wave. When SE_A is ≥ 10 dB, SE_M is negligible (Saini et al. 2009). Therefore, third term in Eq. 2 (i.e. SE_M) vanishes and SE_T can be conveniently expressed as:

$$SE_T = SE_R + SE_A;$$

Further, the relative intensity of the effectively incident EM wave inside the materials after first reflection is based on the quantity $(1 - R)$. The effective absorbance (A_{eff}) can be described as $A_{eff} = (1 - R - T)/(1 - R)$ after normalization with respect to the power of the effectively incident EM wave inside the shielding material. Therefore, it is convenient to express the reflection and effective absorption losses in the form of $-10\log(1-R)$ and $-10\log(1-A_{eff})$, respectively, i.e.

$$SE_R = 10 \log(1 - R) \quad (6)$$

$$SE_A = 10 \log(1 - A_{eff}) = 10 \log\frac{T}{1 - R} \quad (7)$$

The term skin depth (δ) is the depth of penetration at which the intensity of the electromagnetic wave is reduced to 1/e of its original strength (Kumar et al. 2013). The δ is dependent upon various parameters like angular frequency (ω), real relative permeability (μ') and total conductivity ($\sigma_T = \sigma_{dc} + \sigma_{ac}$), i.e. $\delta = \sqrt{2/\omega\mu\sigma_T}$.

Further, the angular frequency (ω) dependence of reflection and absorption losses can be expressed in the terms of total conductivity (σ_T), real permeability (μ'), skin depth (δ) and thickness (t) of the shield material as (Colaneri and Shacklette 1992).

$$SE_R(dB) = -10 \log_{10}\left(\frac{\sigma_T}{16\omega\varepsilon_o\mu'}\right) \quad (8)$$

$$SE_A(dB) = -20\frac{t}{\delta}\log_{10} e = -8.68\left(\frac{t}{\delta}\right)$$
$$= -8.68t\left(\frac{\sigma_T\omega\mu'}{2}\right)^{\frac{1}{2}} \quad (9)$$

Variation of SE_T with frequency of samples designated as FLCNT0, FLCNT8 and FLCNT18 is shown in Fig. 6. The SE_T value improved from -21 dB (FLCNT0) to -27 dB (FLCNT18). When we further investigated the reflection and absorption components of these composites as a function of CNTs content at a particular frequency in the Ku band region, it was found that as the MWCNTs content increases, both SE_R and SE_A increases as shown in Fig. 7. Therefore, the addition of MWCNT resulted in the enhancement of SE_T, i.e. from -21 to -27 dB. The primary mechanism of EMI shielding is usually a reflection of the electromagnetic radiation incident on the shield, which is a consequence of the interaction of EMI radiation with the free electrons on the surface of the shield. As a result, the shield has to be highly electrically conducting. Therefore, increase in the electrical conductivity from 3.2 to 18.3 S cm^{-1} resulted in the enhancement in the SE_R. Absorption is usually a secondary mechanism of EMI shielding where electric dipoles in the shield interact with the electromagnetic fields in the radiation. The increasing difference between SE_R and SE_A with increase in CNT loading may therefore be due to interfacial polarization of epoxy by CNT which increases the absorption component.
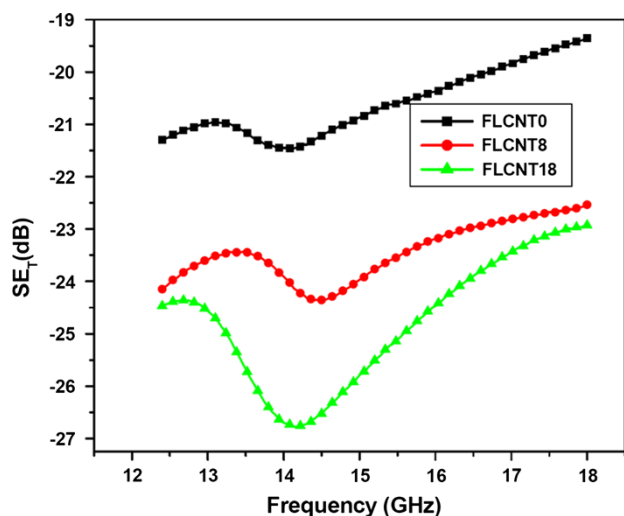
Fig. 6 Variation of total EMI shielding effectiveness with frequency and growth of MWCNTs on CF felt

Fig. 7 Variation of reflection and absorption loss with frequency and growth of MWCNTs on CF felt

The low skin depths and high conductivity (σ_T) values in the microwave region often result in contribution of SE_A becoming much more compared to SE_R. Therefore, absorption acts as dominant shielding mechanism with nominal reflection values. The enhanced absorption can be explained in terms of reduction in skin depth with the increase in both permeability (due to presence of iron as shown in Fig. 2a) and electrical conductivity (due to available highly electrical conducting MWCNTs) (Singh et al. 2012). Equation 9 predicts that reduction in skin depths can improve the contribution of absorption loss in the EMI shielding. In other words, at same thickness, the material with lowest skin depth is expected to give the maximum absorption loss. The variation of frequency-dependent skin depth of different materials, i.e. FLCNT0, FLCNT8 and FLCNT18 is shown in Fig. 8. It reveals that as the amount of growth of MWCNTs increases, the skin depth decreases from FLCNT0 to FLCNT18. Therefore, enhanced SE_A value improved the SE_T value of the composites.

Fig. 8 Variation in skin depth with frequency and the growth of MWCNTs on CF felt

Conclusions

A process has been developed to grow MWCNTs on CF felt as substrate. This multiscale substrate proved to be an effective reinforcement for producing high strength low weight strong composites with improved mechanical properties compared to those established by CF felt/epoxy composites (0 %CNT). The addition of CNTs showed considerable improvement in the FS (37 %) and FM (153 %) of the resulting multiscale composite relative to the CF felt/epoxy composites. In addition, with the incorpo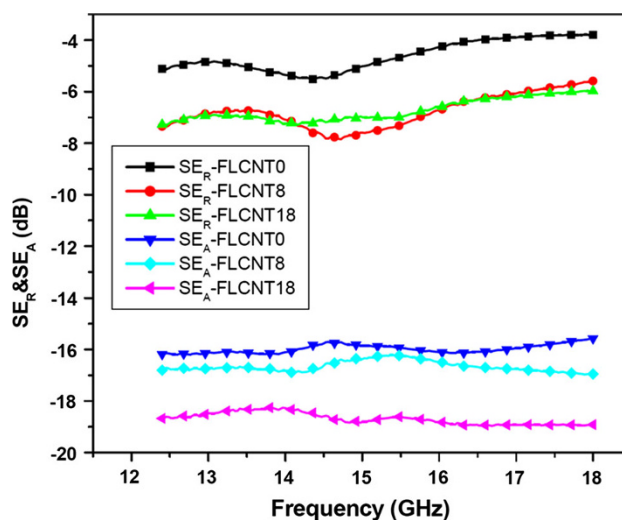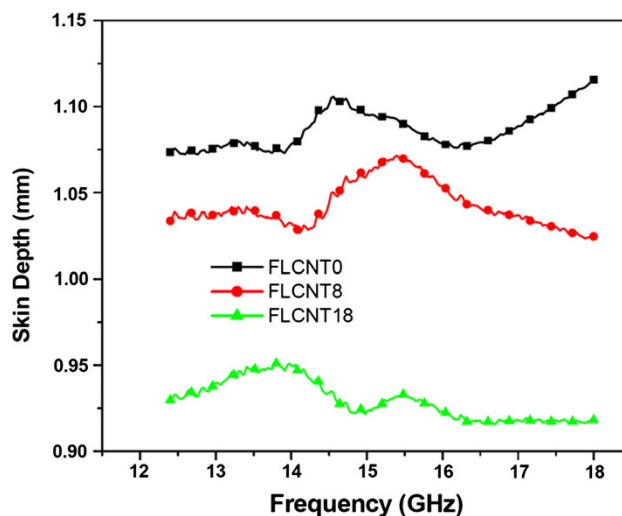ration of MWCNTs in the CF felt, EMI shielding effectiveness was also found to be enhanced from −21 (FLCNT) to −27 dB (FLCNT18). The high value of shielding effectiveness demonstrates the potential of these materials as microwave shielding materials in Ku band (12.4–18 GHz).

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