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Electrodeposited carbon fiber and epoxy based sandwich architectures suppress electromagnetic radiation by absorption

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A R T I C L E I N F O

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A B S T R A C T

Functional polymer composites are in huge demand in electronic industry in general and for electromagnetic interference shielding in particular, due to ease of processing, design flexibility and lightweight. Herein, efforts are made to enhance electromagnetic interference shielding effectiveness in epoxy/carbon fiber composite, by electrodepositing magnetic particles on the surface of carbon fiber. This approach results in 100% enhancement in shielding effectiveness with respect to epoxy/Carbon fiber composites. Electrodeposition, an industrially viable and a scalable technique, is adopted here to obtain nickel decorated carbon fiber. Various nickel deposited carbon fiber morphologies are obtained by varying the applied current. Various microstructures of nickel deposited carbon fiber are obtained and the final parameters are fixed. Further, X-ray diffraction confirms the presence of nickel on the carbon fiber surface. In addition magnetic, electrical, thermal behaviour of nickel deposited carbon fiber is evaluated systematically. Epoxy/carbon fiber composites are fabricated using vacuum assisted resin transfer moulding technique. 2-Layered sandwich structure is prepared with layer 1 as nickel deposited carbon fiber and layer 2 as only carbon fiber. EMI shielding effectiveness is measured in the frequency range of 12–18 GHz. Epoxy with nickel deposited carbon fiber and bare carbon fiber sandwich architecture showed excellent shielding effectiveness up to −50 dB and with maximum absorption of up to −40 dB at 15 GHz. Thermal studies are also carried out to understand the materials response at higher temperature and frequency. Such thin, light-weight, excellent EM absorbers can be used as EMI enclosures for battery casings of hybrid electric vehicles, communication systems etc.

1. Introduction

Any electromagnetic disturbance that interrupts, obstructs, or degrades or limits effective functioning of electronics and electrical equipments can be termed as 'electromagnetic interference' (EMI). As the density of the electromagnetic environment continues to increase due to digitalization, the concern about its effect from sources producing EMI also increases. So, in other words, EMI is a disturbance caused by an electromagnetic field which impedes the proper performance of an electrical or electronics device. EMI shielding refers to the reflection and/or adsorption of electromagnetic radiation by a material, which thereby acts as a shield against the penetration of the radiation through the material. The purpose is to avert EMI from affecting sensitive electronics and hence retain its properties [1–4]. EM waves can be attenuated via three mechanisms, i.e., reflection, absorption and multiple reflections. By reflection mechanism, the incoming EM waves are reflected from the shield surface due to mismatch in impedance. In absorption mechanism, the incoming EM waves are suppressed when it enters the shield material due to impedance matching and losses its energy. Multiple reflection is caused due to reflections of EM waves within the material [5,6].

In the past, metals were preferred as EMI shields, but they are dense and nonflexible, with greater susceptibility to oxidation and require energy intensive processing methods. Metal EM shields are available as sheets obtained from copper, zinc, aluminium etc. In contrast, polymers or polymer blends, are corrosion resistant, light weight, and easier to process [7–10]. But polymers does not have the ability to suppress EM waves, conducting polymers are an exception [11]. Moreover polymer composites are preferred choice as a shield material, since tailored properties are easier to achieve desired EM suppression in broadband frequency range. From literature it is evident that polymer composites based EMI shielding materials are available in various forms such as thin coating, adhesives, flexible films, fabrics, thin enclosures, etc. [12–15] Now a days significant amount of research work is being carried out in the field of polymer composites filled with carbon based material such as carbon fiber, carbon nanotubes, graphene oxide,

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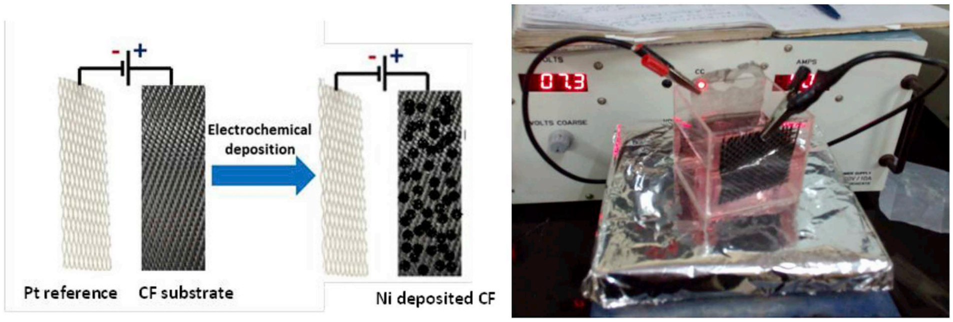


Scheme 1. Schematic representation of lab-scale electrodeposition setup.

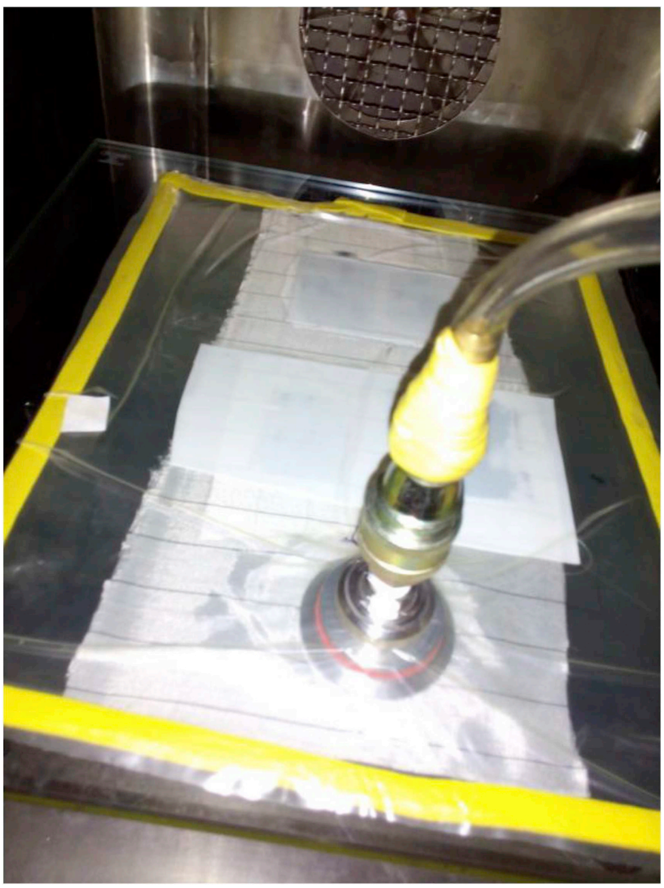


Fig. 1. Fabrication of carbon fiber composites using VARTM technique.

carbon nanofiber, to obtain excellent EM waves suppression [16–23].

There have been several studies which suggest CF can be employed as an efficient EMI shielding material. CF shows high electrical and thermal conductivity, due to its inherent graphitic structure [24]. CF is known to improve electrical, mechanical and thermal and EMI shielding properties of polymer. Hwang et al. worked with foamed and un-foamed PBT/CF composites and showed that with 30% CF loading, the foamed composites showed −11.16 dB of shielding effectiveness (SE). This behaviour was attributed to the random orientation of CF [25–29]. Park et al. [30]. studied foamed and solid polypropylene/carbon fiber (PP-CF) composites for EMI shielding application. It was observed that with 10 vol% CF, foamed PP-CF composites exhibited 24.9 dB of SE which corresponds to 99.7% EM wave blockage in the frequency range of 8–12.4 GHz. Lu and co-workers prepared ultra-thin, flexible, multilayer CF incorporated in PC matrix. This composite exhibited excellent EMI shielding effectiveness of 30.1 dB in the frequency range of 0.03–1.5 GHz [31]. Tzeng et al. [32]. investigated Cu- and Ni-coated carbon fiber/ABS composites for EMI shielding application. Electro-less technique was adopted for the deposition purpose. They observed that Ni showed better adhesion with CF surface compared to Cu, and eventually exhibited enhanced EMI shielding capability.

In the present work, efforts have been made to enhance EMI shielding effectiveness of CF composites, by electrodepositing magnetic particle (here nickel) on its surface. The purpose of this work was to thoroughly analyse and optimize the morphology of functionalized CF, best suited to achieve excellent absorption of EM waves. Another advantage of this process is that CF woven fabric integrity is intact and we need not compromise its properties caused due to fiber destruction. We optimized the morphology of Ni deposited CF (Ni-CF) by varying current and deposition time. Functionalized CF was analysed thoroughly using various characterization technique. Scanning electron microscopy was used to assess the morphology of the CF surface. X-ray diffraction pattern was obtained to confirm the presence of Ni on the surface. Vibrating sample magnetometer was used to analyse magnetic behaviour of Ni-CF. Thermogravimetric analysis was performed to understand thermal stability of epoxy/Ni-CF composites. Electrical conductivity measurements of CF and Ni-CF surface were carried out. After thorough evaluation of Ni deposited CF, we prepared 2-layered sandwich structure of Ni-CF and CF in epoxy matrix. For the current work, Ni-CF was selected as layer 1, since from literature we know that magnetic particles assist in absorption of EM waves, and layer 2 was bare CF surface. Epoxy composite with 2 layer of CF woven mat was used as the control sample. This 2-layered architecture exhibited 200% increment in EMI SE with respect to control epoxy/CF composites.

2. Experimental details

2.1. Materials

Carbon fibers used in this study are bidirectional woven PAN based with diameter of 5 μm, procured from Hindustan Technical Fabrics. Epoxy resin (bisphenol-*F*-epichlorohydrin, EPOLAM 8052) and amine based hardener (2,2′-dimethyl-4,4′methylene bis(cyclohexylamine)) were kindly provided by Axson technologies (France). Nickel(II) chloride hexahydrate, boric acid, solvents were commercially procured.

2.2. Electrochemical deposition set up

Electrochemical deposition was carried out at room temperature under ambient condition. For this, firstly, metal salt solution was prepared with 0.5 g of $NiCl_2.6H_2O$ and DI water. Boric acid (H_3BO_3) was added to maintain pH of the solution. 0.5 g of the metal salt and 0.5 g of boric acid (1:1 ratio) was dissolved in 100 ml water. This solution was used as an electrolyte for electrodeposition. CF mat was used as cathode and platinum mesh was used as anode (inert electrode). Scheme 1 depicts the experimental set-up for electrodeposition process; Scheme 1 shows the lab scale setup that was used. This technique is industrially viable and scalable.

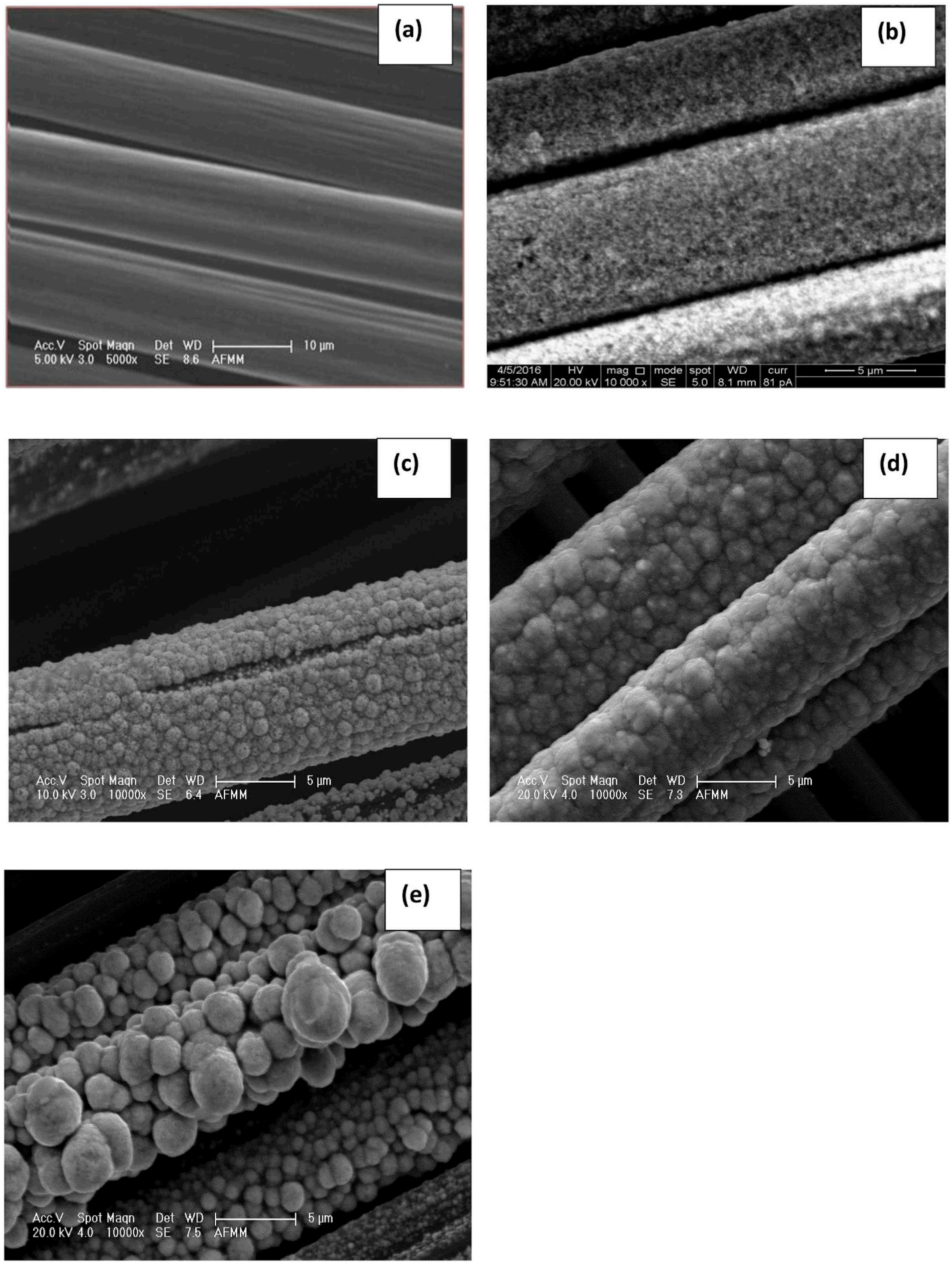


Fig. 2. SEM micrographs of Ni-deposited CF (a) 0 mA; (b) 30 mA; (c) 60 mA; (d) 90 mA; (e) 150 mA at constant deposition time of 30 min.

2.3. Sandwiched composite fabrication

In this work, our intention was to achieve maximum absorption of EM waves and provide effective EMI shielding efficiency. Sandwich composites were prepared with two layers, layer 1 as Ni-functionalized CF (Ni-CF) and layer 2 as CF, with epoxy as matrix. Composites were prepared using vacuum assisted resin transfer moulding (VARTM) technique with resin/filler ratio of 50:50 by weight. Samples were cured at 120 °C and post curing was carried out at 140 °C. 2-Layered CF/epoxy composite was used as control sample. Fig. 1 shows the fabrication of epoxy composites with VARTM setup. To fabricate sandwich structures a layer of CF mat was placed (layer 1) followed by a layer of Ni-CF (layer 2). These layers were covered with peel ply and flow media for uniform and easy flow of resin into the fiber. Then vacuum bag was laid with silicon vacuum tape at the edges to ensure efficient vacuum. Resin was infused through the inlet and vacuum was created using pump. Vacuum helps to avoid formation of any void or wrinkle on the final finished composite. Presence of such defects in the composite reduces the mechanical properties of composite. Composite with VARTM setup was kept in oven for curing at 120 °C, followed by post curing at 140 °C. After curing, sample thickness was measured to be 0.4 mm. Composites were polished slightly to carry out different experiments.

2.4. Characterization

The morphological features of the various Ni-deposited CF surfaces were characterized using ESEM Quanta 200, FEI scanning electron microscope (SEM). The structure was analysed using X-Pert PRO X-ray diffractometer with Cu target and pattern were analysed using. NETZSCH STA 409 thermogravimetric analyser (TGA) was used to study the degradation of functionalized CF in the temperature range of 30–1200 °C in air. Lakeshore Vibrating Sample Magnetometer was used to study magnetic behaviour of functionalized CF. Surface electrical conductivity of laminates were measured on Agilent probe station using 4-probe method. EMI shielding behaviour of thin laminates were

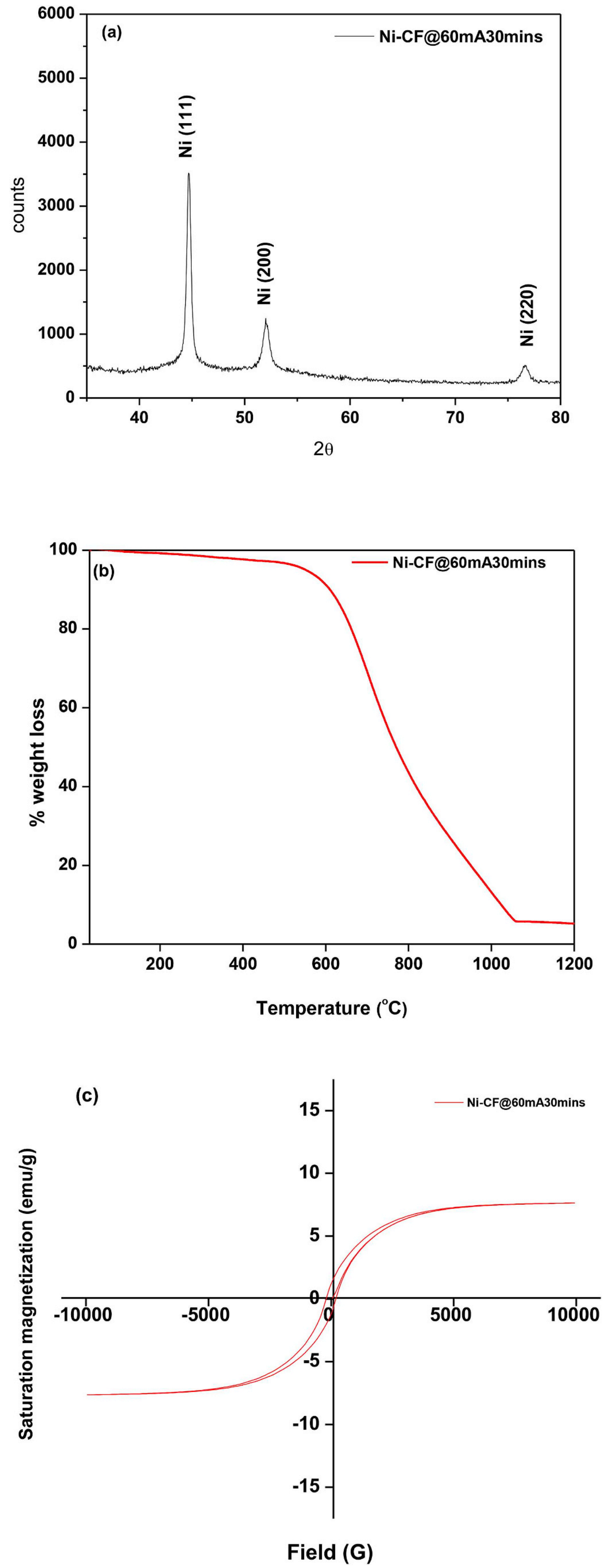


Fig. 3. a) XRD profile of the Ni-CF@60mA30mins; b) TGA for Ni-CF@60mA30mins; c) Magnetic hysteresis loop of Ni-CF@60mA30mins.

analysed using Vector Network Analyser (VNA) in the frequency range of 12–18 GHz at room temperature. Thermal scans of laminates were obtained using IR analyser, when exposed to 18 GHz EM waves.

3. Result and discussion

3.1. Microscopic analysis of functionalized carbon fiber

To observe the microstructure of nickel deposited CF, micrographs were obtained using ESEM Quanta 200 FEI scanning electron microscope. Fig. 2a-e shows SEM micrographs (at magnification of ×10000) of electrodeposited Ni on the surface of CF at varying current but constant deposition time. Fig. 2a shows the bare surface of uncoated CF with no surface defects. Fig. 2(b–e) illustrates the deposition of Ni particles on the CF surface at different applied current, i.e., 30 mA, 60 mA, 90 mA and 150 mA respectively, at constant deposition time of 30 min. We observed that with increase in applied current, electrodeposition of Ni was more uniform throughout the CF surface. Spherical shaped Ni particles uniformly deposited was observed at 60 mA. While at 90 mA, there was obvious coarsening of Ni particles and the deposition was non-uniform. At higher current of 150 mA, the growth rate of Ni was increased and micro-cones of Ni was formed on CF [33]. At higher current, we observed hydrogen evolution which is detrimental to the property of Ni-CF, as this increases hydrogen embrittlement [34,35]. Hence, from above observations applied current of 60 mA was assigned as the optimum current required for the functionalization of CF. For further discussion, we will refer this functionalized CF as Ni-CF@60mA30min. We also carried out electrodeposition with Fe and Co salts, which are discussed in supporting information. From SEM micrographs of Fe-CF and Co-CF (Figure. S1 & S2), we observed that deposition of these magnetic particles were non-uniform at various applied current value (30–150 mA). Such microstructure might not provide consistent EMI shielding behaviour in a given frequency range. Figures S3 and S4 show total shielding effectiveness of Co-CF@60mA30min and Ni-CF@60mA30min measured in the frequency range of 12–18 GHz. From fig.S3 it is clear that non-uniform deposition of Co on CF fiber gives uneven EMI shielding response at higher frequency, unlike Ni-CF@60mA30min, such material is not desirable from application point of view. Hence our studies were focussed on Ni functionalized CF.

XRD pattern was obtained for Ni-CF@60mA30min in order to confirm the presence of Ni and further to assess the phase structure of the deposited Ni. Fig. 3a shows the presence of three characteristic peaks at 2 theta value of 44.6, 52.0 and 76.6, which correspond to miller indices of (111), (200) and (220) as depicted in the XRD plot. From JCPDS reference we can assign the structure as face centred cubic [33,36]. There were no additional peaks, which suggest that there is no formation of Ni oxides during the electrodeposition process. This is important in order to achieve high saturation magnetization, which can further enhance EMI shielding effectiveness in the composites.

Thermal analysis was carried out to further confirm the absence of any impurity such as Ni oxides or hydroxides during the preparation of Ni-CF. Fig. 3b shows thermal degradation profile of Ni-CF@60mA30mins. The experiment was carried out in the temperature range of 30–1200 °C in air at a heating rate of 10 °C/min. From the curve it was clearly evident that there was degradation at 650 °C, due to the decomposition of CF structure.

In order to understand the key role of Ni-CF on EMI shielding mechanism, it is pivotal to study the magnetic and the electric conductivity of functionalized CF. It is known that magnetic particles enhance absorption of the incoming EM radiation through various mechanisms and highly electrically conducting surface shield primarily by reflection. Vibrating Sample Magnetometer (VSM) was employed to study M-H curve of functionalized CF at an external applied field of 10000 G. Fig. 3c shows magnetic property of Ni-CF@60mA30mins sample at room temperature. Carbon fiber does not show any magnetic response

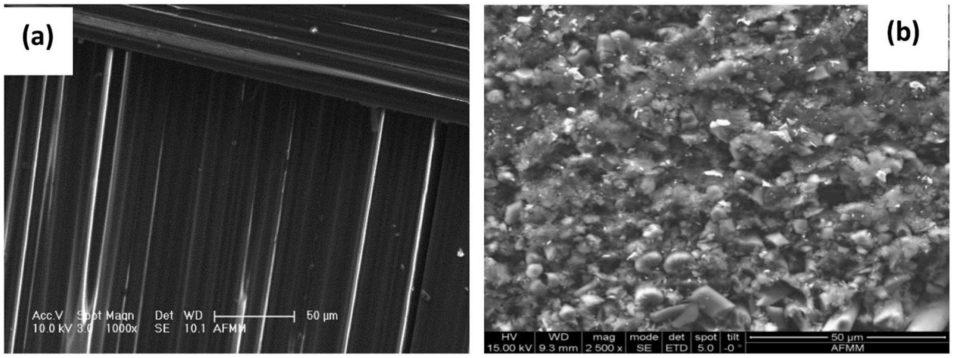


Fig. 4. SEM micrographs of Ep/CF and Ep/Ni-CF/CF composites.

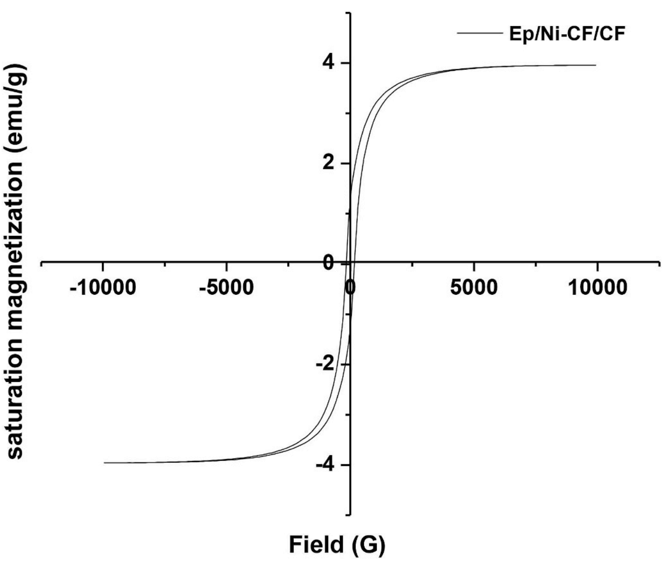
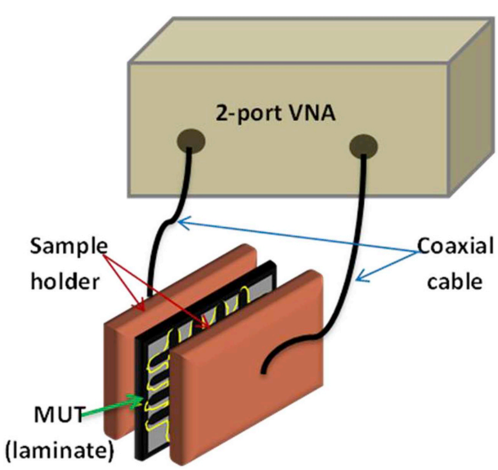


Fig. 5. M-H hysteresis curve of Ep/Ni-CF/CF composites.



Scheme 2. EMI shielding experiment set-up.

in presence of external magnetic field and is paramagnetic in nature [37]. The saturation magnetization (M_S) value for Ni-CF@60mA30mins was found to be 7.55 emu/g at 10000 G of external field. Although M_S value of Ni-CF is quite less than that of Ni particles. [37], its presence in the composite samples will provide magnetic dipoles that can interact with the incoming EM radiation and help in attenuating it. The M-H curve exhibits small value of coercivity and retentivity. From the hysteresis curve, coercivity (H_C) and residual magnetism was measured as 151 G and 1.7 emu/g respectively. Electrical conductivity is another important parameter that dictates EMI shielding behaviour. The electrical conductivity values of neat CF and Ni-CF@60mA30mins was calculated from Van der Pauw method. This method provides average surface resistivity of CF surfaces. It is also suitable for anisotropic surfaces. From results it was clear that there was negligible change in electrical conductivity values. Electrical conductivity of CF and Ni-CF was found to be in the order of 10^4 S/m. It also suggests that there is no formation of Ni oxides or hydroxides, which could inhibit electrical properties of Ni-CF.

4. Analysis of the sandwiched composite structure

After careful and thorough evaluation of CF and Ni-CF, 2-layered composites were fabricated as discussed earlier. Composites were examined for morphology, electrical, thermal and EMI shielding behaviour. Fig. 4a-b show SEM micrographs of Ep/CF and Ep/Ni-CF/CF composite structure. SEM micrograph of Ep/Ni-CF/CF exhibit rough or uneven surface due to Ni deposition on CF, while Ep/CF shows smooth surface. Such hybrid structure of Ep/Ni-CF/CF composite can provide surface to impede EM radiation and screen the devices from malfunctioning.

As discussed earlier, for a given shield material it is important to analyse its electrical and magnetic behaviour since shielding ability depends on the presence of electric/magnetic dipoles. M-H hysteresis curve for Ep/Ni-CF/CF composite was obtained using VSM at room temperature as shown is Fig. 5. Saturation magnetization (M_s) was found to be 4 emu/g at an external applied field of 10000 G. There was decrease in M_s value when compared to functionalized CF, i.e., Ni-CF@60mA30mins, due to the impregnation of non-magnetic epoxy resin in functional CF woven mat. The coercivity (H_c) and residual magnetization for Ep/Ni-CF/CF was measured as 130 G and 1.3 emu/g respectively. This presence of Ni in Ep/Ni-CF/CF composite structure would interact with the incoming EM radiation and attenuate it further. The electrical behaviour of Ep/CF and Ep/Ni-CF/CF was analysed using four probe method. Samples were cut in square shape ($10 \times 10\,cm^2$) and electrical contacts were made with probes at four corners. This method was preferred to reduce effect of contact resistance. Van der Pauw relation was used to determine surface conductivity. We observed that there was marginal change in electrical conductivity values of epoxy composites. Surface electrical conductivity of Ep/CF and Ep/Ni-CF/CF was found to be 14 and 1.3 S/cm respectively, which was sufficient to ensure effective EMI shielding. This also suggests that conducting network was not hindered due to the electrodeposition process.

EMI shielding effectiveness was measured using 2-port Rhodes and Schwarz Vector Network Analyser in the frequency range of 12–18 GHz at room temperature. Scattering parameters were measured using VNA across port 1 and 2. Epoxy composites were cut in rectangular shape to fit the waveguide. Short-open-load-through (SOLT) calibration was performed before actual measurements. From equation (1), we know SE_{total} is the summation of absorption (SE_A), reflection

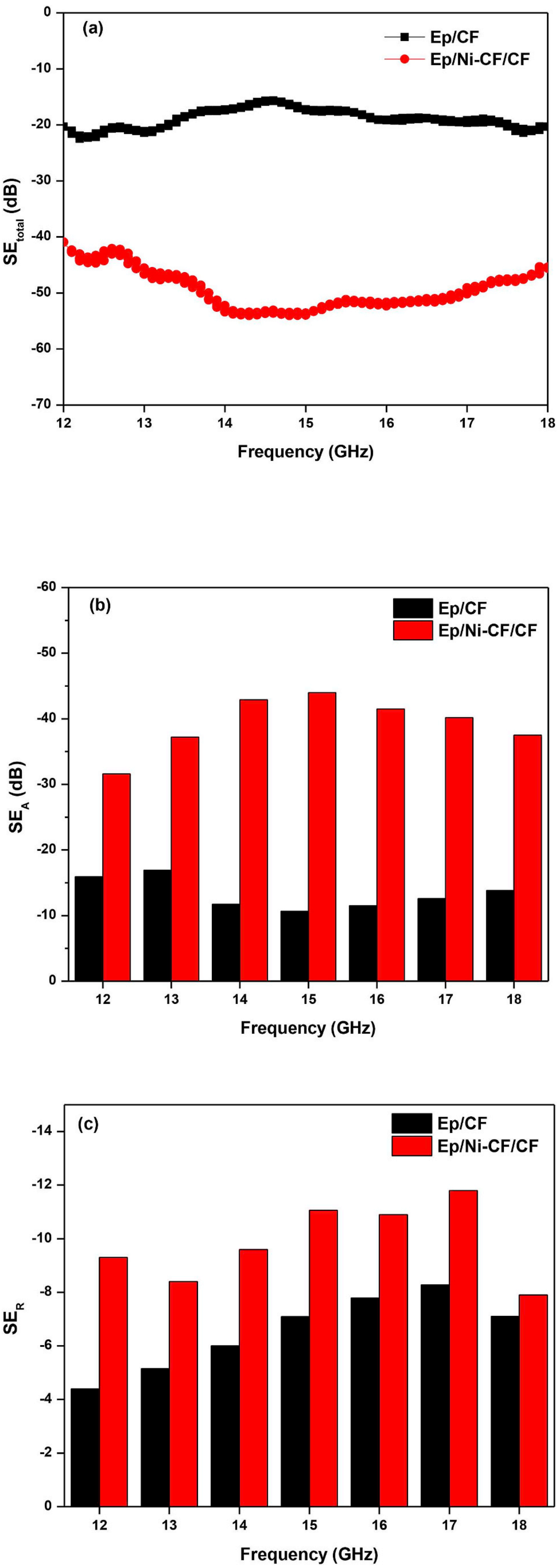


Fig. 6. a) SE_{total}; b) SE absorption and; c) SE reflection of epoxy composites as a function of frequency in Ku band.

(SE_R) and multiple reflections (SE_M). Multiple reflections is negligible since sample thickness is more than the skin depth. From equations (2) and (3) we calculated SE_A and SE_R respectively, which was obtained from scattering parameters. Scheme 2 shows the experimental set for EMI shielding measurements.

$$SE_{Total} = SE_R + SE_A + SE_M \tag{1}$$

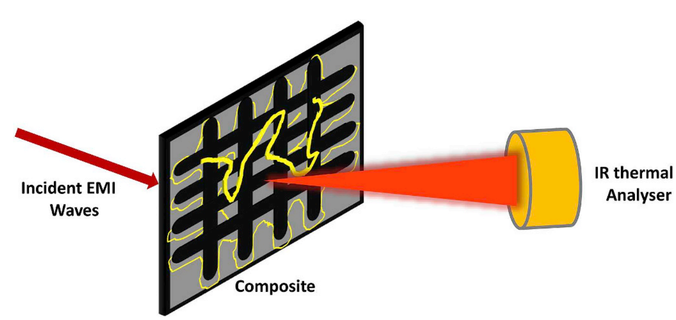
$$SE_A = 10 \times \log_{10}\left(\frac{1 - S_{11}^2}{S_{21}^2}\right) \tag{2}$$

$$SE_R = 10 \times \log_{10}\left(\frac{1}{1 - S_{11}^2}\right) \tag{3}$$

Fig. 6a shows total shielding effectiveness (SE_{total}) of epoxy composites reinforced with CF and Ni-CF. Ep/CF composite showed SE_{total} value of −20 dB at 12 GHz while Ep/Ni-CF/CF exhibited almost 100% increment in SE_{total} value and it was found to be −41 dB at the same frequency. At higher frequency of 18 GHz, Ep/CF again showed −20 dB of shielding effectiveness and for Ep/Ni-CF/CF SE_{total} was found to be −45 dB. We further investigated the mechanism involved in effective EMI shielding behaviour. Fig. 6b-c shows SE_A and SE_R plot as a function of frequency respectively. From Fig. 6b it is clear that functionalization of CF with Ni has improved the absorption ability of Ep/CF composites significantly. Ep/CF composite showed maximum SE_A value of −15 dB, while Ep/Ni-CF/CF exhibited up to −40 dB. This can be attributed to the hybrid structure formation comprising Ni and CF. Ni-CF surface provides impedance matching, which assist in the absorption of incident EM ways and further reduce its energy along the thickness. Hence it provides excellent absorption behaviour of Ep/Ni-CF/CF composites. Although there was no significant change in SE_R, since reflection is dominant in the presence of electrically conducting materials. But we observed from electrical responses of composites that there was only slight change in conductivity value and hence SE_R did not alter much. For Ep/Ni-CF/CF composite, SE_R was found to be approximately −11 dB, while Ep/CF showed SE_R -7 dB at 17 GHz (see Fig. 6c). Such materials can be used as excellent EM absorbers.

From above discussion, it is clearly evident that absorption mechanism is dominant in EMI shielding behaviour of Ep/Ni-CF/CF. Hence it becomes important to study the thermal response of the material when exposed to high frequency EM radiation. Scheme 3 shows the experimental set-up adopted to measure thermal response of laminates under the influence of EM radiation. For this analysis epoxy composites were exposed to 18 GHz frequency for 10 min and then thermal response of these composites were captured using infrared scanner. Fig. 7 show thermal scans of Ep/CF and Ep/Ni-CF/CF captured after exposure of high frequency EM waves for 10 min. We observed that there was no significant temperature rise within the material. Ep/CF showed average temperature of 28 °C, while Ep/Ni-CF/CF showed temperature of 30 °C.

Further we carried out thermal degradation study of these composites which is important from application point of view. Thermal degradation curve of composites were studied using TGA in the



Scheme 3. Thermal scans of composite obtained using IR thermal analyser under the influence of high frequency EM waves.

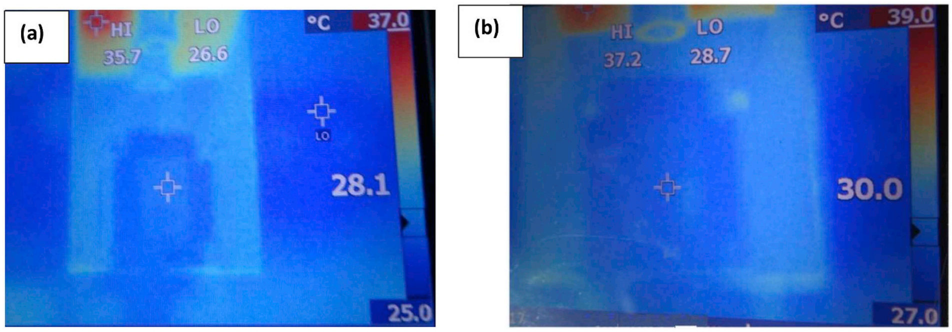


Fig. 7. Thermal scans of (a) Ep/CF; (b) Ep/Ni-CF/CF when exposed to EM radiation of 18 GHz.

temperature range of 30–800 °C. Fig. 8 shows thermal decomposition behaviour of Ep/CF and Ep/Ni-CF/CF composites. TGA scans of Ep/CF and Ep/Ni-CF/CF show two step degradation. The step 1 degradation (as indicated in Fig. 8) between 250 and 400 °C indicates decomposition of epoxy matrix; while step 2 corresponding to weight loss in the temperature range of 450–600 corresponds to the onset of degradation of carbon fiber. There is no additional degradation observed in both samples, which indicates that during processing of composites Ni particles did not react to produce oxides or hydroxides. The % residual weights of Ep/CF and Ep/Ni-CF/CF at 800 °C were found to be 45% and 50% respectively.

5. Conclusions

Herein, we have clearly demonstrated a strategy to suppress EM radiation by using thin, light-weight and easy to fabricate and integrate epoxy/CF based sandwich architecture. By electrodeposition, a strategy which is scalable industrially, Ni was deposited onto CF to fabricate functional epoxy/CF sandwich architecture. This strategy was optimized by varying the applied current (30–150 mA) and time. The electrodeposited CF was thoroughly characterized and the laminates were tested for electrical, thermal and magnetic properties. 2-Layered epoxy composites were prepared with CF and Ni deposited CF which exhibited excellent shielding effectiveness of −50 dB and attenuated the incoming radiation mostly by absorption. Such lightweight architectures can be used for battery casings and other electronic components of automobiles.

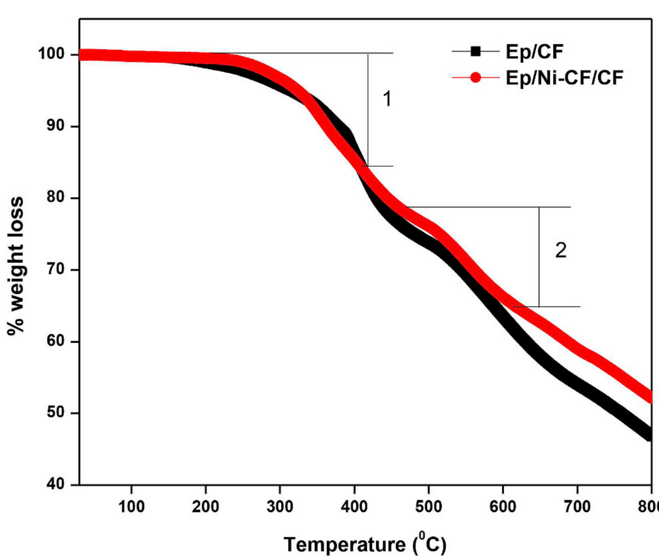


Fig. 8. TGA curve of epoxy composites as a function of temperature.

Data availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an on-going study.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.compositesb.2018.12.123.

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