Enhanced microwave absorption properties of nickel-coated carbon fiber/glass fiber hybrid epoxy composites-towards an industrial reality

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Abstract

The Nickel-coated carbon fibers (NiCFs)/glass fibers (GFs) hybrid epoxy composites were manufactured by employing papermaking technology to produce NiCFs/GFs paper followed by composites liquid molding. The microwave absorbing properties of single-layered and double-layered NiCFs/GFs hybrid epoxy composites were evaluated. The reflection loss (RL) were calculated by the measured complex permittivity and permeability using waveguide method by vector network analyzer in the X-band (8.2–12.4 GHz) range. The double-layered composites were fabricated by using matching layer and absorbing layer to enhance the microwave absorption performance. The enhanced microwave absorbing properties of double-layered composites with minimum RL of −48.1 dB and effective absorption bandwidth of 3.2 GHz in the X band can be achieved when the total thickness of the matching layer and absorbing layer is 2.5 mm, which is attributed to synergistic effect of improved impedance matching characteristic and superior microwave attenuation capability of the absorbing layer, demonstrating that the NiCFs/GFs hybrid epoxy composites can be a promising candidate of high performance microwave absorbing materials for realizing industrialization.

1. Introduction

Widespread and fast-growing usage of electronic, radar and wireless devices brings lots of convenience to people’s life while inevitably produces invisible and undesirable byproduct such as electromagnetic interference (EMI) and radiation pollution, which not only cause electromagnetic compatibility problems of sophisticated electronics, but also threaten human health when under long-time radiation exposure [1–4]. Thus, to address these issues, it is of paramount importance to explore microwave absorbing materials (MAMs) which can attenuate electromagnetic wave through converting electromagnetic wave to thermal energy by electromagnetic loss or dissipating them by interference.

Tremendous efforts have been devoted to exploit high-performance MAMs that have high absorbing capability, broad effective absorption bandwidth (bandwidth below −10 dB), low density and thin thickness. Especially, carbon materials such as carbon fiber, carbon nanotube, graphene [5–25] have drawn extensive attention by virtue of their high dielectric loss, chemical resistance and lightweight characteristic. Among them, compared with nano-sized carbon materials, carbon fiber as micro-sized additives are still the most widely used due to its abundant source, high specific and modulus as well as economic applicability.
Favorable impedance matching and high microwave attenuation capability plays a significant role in obtaining high performance MAMs, both of them are indispensable. Combination of magnetic or dielectric filler and carbon materials employing physical blending or surface decoration method have been verified to be a promising strategy to improve the impedance matching characteristics thus enhancing the microwave absorption properties. For instance, Qiang [26] filled epoxy/silicon resin with CF and carbonyl iron and the composites with 2 wt% CF and 65 wt% carbonyl iron showed effective absorption bandwidth of ~10 GHz at only 1 mm thickness. Qiang [27] plated Fe$_3$O$_4$ nanoparticles on CF to produce Fe$_3$O$_4$/CFs composites and revealed that the minimum reflection loss is ~35 dB at 4.41 mm thickness with 50 wt% content of Fe$_3$O$_4$/CFs in paraffin. Singh [28] has grown CNTs on CFs by using CVD method, indicating that the epoxy composites containing 0.35 and 0.50 wt% of CNT and CF showed minimum reflection loss of ~42 dB and effective absorption bandwidth of 2.7 GHz at thickness of 2.5 mm. Qiu [29] prepared Fe$_3$O$_4$-CNTs-hollow CFs composites, which exhibited a minimum reflection loss of ~50.9 dB at thickness of 2.5 mm when loaded with 25 wt% of the composites in paraffin. Yang [30] synthesized ZnO/Zn/CF composites and found that 60 wt% ZnO/Zn/CF in the wax showed enhanced microwave absorbing properties, a minimum reflection loss of ~39.4 dB can be achieved at 4.5 mm thickness. Movassagh-Alanagh [31] fabricated PANI@nano-Fe$_3$O$_4$@CFs heterostructures and its epoxy composites with 1 wt% loading showed minimum reflection loss of ~11.1 dB and effective absorption bandwidth of 6 GHz. Chen [32] prepared PANI/Ni/CF by in situ polymerization of PANI on nickel-coated CF, which exhibited a minimum reflection loss of ~12.4 dB with 20 wt% content in paraffin at 2 mm thickness. Nonetheless, many of the research mentioned above always needs complex synthetic process, which cannot be easily scaled up, besides, the synthesized various CF-based absorbent will be filled to polymer matrix such as epoxy for application, which brings further challenges like poor dispersibility, intertwining or flow-induced fiber orientation due to the high fiber aspect ratio.

Herein, the papermaking technology was employed to produce preformed nickel-coated carbon fibers (NiCFs)/glass fibers (GFs) paper in which the fibers were physically interconnected and dispersed randomly, and the NiCFs/GFs hybrid epoxy composites were manufactured by impregnating the paper with epoxy to prepare prepreg followed by vacuum bagging process. The microwave absorbing properties of single-layered and double-layered NiCFs/GFs hybrid epoxy composites were evaluated and the microwave absorption mechanism was also discussed, the results demonstrates the NiCFs/GFs hybrid epoxy composites can be a promising candidate of high performance microwave absorbing materials for realizing industrialization.

2. Experimental

2.1. Materials
Short NiCFs were prepared by electroless plating method in our previous research [33], the length is approximately 4 mm, short GFs were supplied by Nanjing glass fiber research institute and the length is about 6 mm. The polyethylene glycol and sodium carboxymethylcellulose were purchased from Sinopharm group chemical co. LTD, the epoxy (CYD-128) was commercially available bisphenol A type, which was provided by china petrochemical corporation, the curing agent was modified aliphatic amine synthesized by our own Laboratory.

2.2. Preparation of NiCFs/GFs hybrid epoxy composites
The preparation of NiCFs/GFs hybrid epoxy composites was divided into two steps: NiCFs/GFs paper production and composites liquid molding, as depicted in figure 1. Firstly, the NiCFs/GFs paper with areal density of 30 g m$^{-2}$ was produced by wet papermaking method (circular discs with 200 mm in diameter), details as belows: 1.5 g sodium carboxymethylcellulose and 4.5 g polyethylene glycol were dissolved in 1 l of water to form a solution, a certain amount of short NiCFs and GFs were stirred in the solution in the fiber disintegration device (Tico742, PTI, Austria) at a speed of 3000 rpm for 1 min, then the suspension was poured into the filtration unit of Rapid-Koethen sheet forming machine(RK-3A, PTI, Austria), after filtration the moist paper was removed from the copper wire mesh, then placed in the drying system, after that the NiCFs/GFs paper was obtained, the NiCFs content in the NiCFs/GFs hybrid paper was 2 wt%, 4 wt%, 6 wt% and 8 wt% respectively.

Next, the NiCFs/GFs hybrid epoxy composites was prepared by liquid molding technology. The appropriate amount of modified amines (24 wt% of epoxy) were added to the epoxy and stirred evenly, then hand lay-up method was adopted to impregnate NiCFs/GFs paper with epoxy, afterwards, the composites was manufactured by putting the prepreg into a steel mold for vacuum bagging molding. The weight ratio of NiCFs/GFs paper to the epoxy resin is 15/100. The composites were initial cured overnight and postcured at 80 °C for 2 h. The composites with varied NiCFs and GFs loadings mentioned above were denoted as NiCF2, NiCF4, NiCF6 and NiCF8, respectively.
2.3. Characterization

Scanning electron microscopy (SEM, JSM-5610LV) was used to observe the surface morphologies of CFs and NiCFs as well as fractured surface morphologies of composites. The energy dispersive spectroscopie (EDS) equipped with SEM was used to determine the element composition of nickel coatings. Digital electron microscope images of NiCFs/GFs paper were taken by HD digital microscope (UM039, Shenzhen Maike Video Electronics Co., Ltd.). The hysteresis loop of the NiCFs was tested using a vibrating sample magnetometer (BHV-525, Japan Riken Electronics Co., Ltd). The electromagnetic parameters of the NiCFs/GFs hybrid epoxy composites were measured using an N5247A PNA vector network analyzer (Agilent Technologies Co., Ltd, US) by waveguide method. The composites were carved to rectangular samples with extremely precise length and width of 22.86 mm and 10.16 mm by carving machine, while the thickness were approximately 1 mm. The scattering (S) parameters in the frequency range of X band were obtained and the complex permittivity ($\varepsilon = \varepsilon' - j\varepsilon''$) and permeability ($\mu = \mu' - j\mu''$) were also calculated by the material testing software of the PNA.

3. Results and discussion

3.1. Characterization of NiCFs

Figures 2(a) and (b) shows the pristine CFs and NiCFs, as can be seen from it, the surface of pristine CFs was smooth and a dense and uniform nickel coating was plating on the CFs after electroless plating. Figure 2(c) shows the EDS results of the nickel coatings on the surface of CFs, there were only trace concentrations of carbon and oxygen elements, revealing the coatings mainly consisted of Ni and P. Figure 2(d) Magnetization hysteresis loop of the NiCFs of room temperature, it can be seen that the saturation magnetization ($M_s$), coercivity (Hc) of nickel coatings.

3.2. Microstructure NiCFs/GFs paper

Figure 3 shows digital electron microscope images of NiCFs/GFs paper with different content of NiCFs. It can be seen that NiCFs monofilaments are distributed in the glass fibers networks and randomly orientated, and as the content of the NiCFs increases, the probability of the NiCFs monofilaments overlapping each other in the NiCFs/GFs paper becomes large.

3.3. Fractured surface morphologies of NiCFs/GFs hybrid epoxy composites

The fractured surface morphologies of NiCFs/GFs hybrid epoxy composites at different magnification are displayed in figure 4, as can be seen from it that the NiCFs and GFS are randomly oriented in the epoxy matrix, both of them are fully infiltrated by epoxy resin, the NiCFs are insulated by GFs in composites NiCF2, by increasing the content of NiCFs, a few NiCFs may interconnect with each other to form conductive pathways in composites NiCF8, however, a continuous conductive network can be obstructed due to the blocking effect of
Figure 2. SEM images of the CFs: (a) pristine, (b) NiCFs, (c) EDS of the NiCFs, (d) Magnetization hysteresis loop of the NiCFs of room temperature.

Figure 3. Digital electron microscope images of the NiCFs/GFs paper: (a) 2 wt%, (b) 4 wt%, (c) 6 wt%, (d) 8 wt%.
GFs, because dense conductive network may lead to strong reflection of EM waves, which results in decreased electromagnetic wave absorption capability.

3.4. Microwave absorbing properties of the single-layered composites

The RL of single-layer absorber with metal backup model can be calculated by following equations according to transmission line theory \[4, 12\]:

\[
RL (dB) = 20 \log_{10}\left(\frac{Z_{in} - Z_0}{Z_{in} + Z_0}\right)
\]

\[\text{(1)}\]

\[
Z_{in} = Z_0 \sqrt{\frac{\mu_r}{\varepsilon_r}} \tanh \left( j \frac{2 \pi df}{c} \sqrt{\mu_r \varepsilon_r} \right)
\]

where \(Z_{in}\) and \(Z_0\) are the impedance of free space and the normalized, \(\varepsilon_r, \mu_r\), and \(d\) are the complex permittivity, complex permeability and thickness of the composites, respectively, \(f\) is the frequency of the microwave, \(c\) is the velocity of light in vacuum. The bandwidth of RL below \(-10\) dB is called effective absorption bandwidth, which means 90% absorption of the microwave by the absorber. The real part \(\varepsilon'\) and \(\mu'\) stands for the electric and magnetic field storage capacity, while the imaginary part \(\varepsilon''\) and \(\mu''\) signify the dielectric and magnetic loss ability. The \(\varepsilon_r\), and \(\mu_r\) significantly affects two vital factors, namely impedance matching and attenuation characteristic, which jointly determine the microwave absorption properties of the composites. The impedance matching is a premise which ensures the microwave can enter into the absorber while attenuation characteristic represents the capability of converting EM energy to heat dissipation. The input impedance \(Z_{in}\) of the absorber should be as close as possible to the impedance of free space \(Z_0\).

Figure 5 shows frequency dependence of the complex permittivity of NiCFs/GFs hybrid epoxy composites, as shown in figures 5(a) and (b), both the complex permittivity of \(\varepsilon'\) and \(\varepsilon''\) increase with the increase of NiCFs content in hybrid paper, as the content of NiCFs is increased from 2 wt% to 8 wt%, \(\varepsilon'\) increases from 6.7–6.3 to 14.0–12.9, \(\varepsilon''\) increases 1.7–1.4 from to 5.7–4.3 respectively. The dielectric properties of a material mainly come from polarization under alternating electric field, which primarily consists of electronic, ionic, electric dipolar and interfacial polarization, in the microwave frequency range it is more likely that dielectric properties arise from electric dipolar orientation polarization. The interfacial polarization occurs when charges accumulate at the interfaces between which the permittivities or conductivities are different, especially in heterogeneous materials possessing plenty of interfaces, the nickel coatings and CFs or epoxy formed heterogeneous structure.
which results in space charge accumulation at the interfaces. Increasing the content of NiCFs leads to more electric dipolar orientation polarization along with higher interfacial polarization at the NiCFs/GFs hybrid epoxy composites interfaces, thus the higher $\varepsilon'$ can be obtained. As for $\varepsilon''$, aforementioned polarization associated relaxation constitute the dielectric loss mechanism, besides, increasing NiCFs loadings leads to more conductive paths forming in the epoxy resin, thus there also exists conduction loss due to the conduction current induced by electromagnetic wave. Therefore, the dielectric loss of the composites mainly stems from polarization relaxation loss and conduction loss. Furthermore, $\varepsilon'$ and $\varepsilon''$ decreases with the increase in frequency, which may be attributed to that the formation of dipoles cannot follow the enhanced applied electric field thus losses its responsiveness. It also can be seen from figures 5(c) and (d) that the complex permeability $\mu'$ and $\mu''$ of the hybrid composites increase with increasing content of the NiCFs, $\mu'$ increases from 1.02–0.99 to 1.11–1.09, $\mu''$ increases from 0.03–0.02 to 0.09–0.06, suggesting that the electromagnetic loss of the NiCFs/GFs hybrid epoxy composites comes from dielectric loss and magnetic loss.

Figure 6 shows the calculated results of absorbing properties of NiCFs/GFs hybrid epoxy composites at different thicknesses in X-band. As can be seen from figure 6(a) that composites NiCF2 has relatively poor absorption performance, the effective absorption bandwidth is 1.6 GHz (8.2–9.8 GHz) and minimum RL is $-14.8$ dB at the thickness of 3.5 mm, which is due to the lower complex permittivity and permeability. From figure 6(b), it can be seen that when the thickness of composites NiCF4 is 2.5 mm, the minimum RL is $-17.6$ dB, and the effective absorption bandwidth is 2.9 GHz (9.1–12.0 GHz). By further increasing the NiCFs content in the paper to 6 wt%, the composites shows better microwave attenuation performance, as can be seen from the figure 6(c), the minimum RL of $-48.5$ dB can be achieved when the thickness of the composites is 2 mm, and the effective absorption bandwidth is 2.3 GHz (10.1–12.4 GHz), which is attributed to the enhanced dielectric loss and magnetic loss. As for composites NiCF8 displayed in figure 6(d), which shows minimum RL of $-18.9$ dB and the effective absorption bandwidth of 3.0 GHz (8.6–11.6 GHz) at the thickness of 2.5 mm. Compared with composites NiCF6, NiCF8 exhibited deteriorated microwave absorption performance, which is due to an increased impedance mismatch caused by enhanced conductive pathways in the composites.

![Figure 5](image-url)

Figure 5. Frequency dependence of (a) (c) real part, (b) (d) imaginary part of complex permittivity and permeability of the hybrid composites as a function of NiCF content.
3.5. Microwave absorbing properties of the double-layered composites

Above obtained results reveals that single-layered absorber has limited regulating parameters, which is difficult to achieve the high attenuation capability as well as broad absorption bandwidth simultaneously, double-layered design consisted of matching layer and absorbing layer can be employed to acquire excellent microwave attenuation performance, at the same time, thinner thickness and broader effective absorption bandwidth are desirable. The schematic diagram of double-layered microwave absorbing structures is depicted in figure 7. The matching layer should meet the requirement of impedance matching and always possesses mild complex permittivity and permeability, which is beneficial for incident microwave to enter into the interior of absorbing layer at the utmost extent rather than reflection on the surface [8]. After passing through the matching layer, the incident microwave will be attenuated by the absorbing layer composed of high dielectric or/magnetic loss materials. The $Z_{in}$ of double-layer absorber backed by metal reflector can be calculated by following equations [34]:

Figure 6. Microwave absorbing properties of NiCFs/GFs hybrid epoxy composites

Figure 7. Schematic diagram of double-layered microwave absorbing structures.
Where $Z_{\text{in}2}$ is the input impedance at the free space and double-layered absorber, $Z_{\text{in}1}$ is the input impedance at the absorbing Layer 1 and matching layer 2, $Z_1$ and $Z_2$ is the characteristic impedance of each layer, $\varepsilon_{\text{r}1}$ and $\varepsilon_{\text{r}2}$, $\mu_{\text{r}1}$ and $\mu_{\text{r}2}$, $d_1$ and $d_2$ is the relative complex permittivity, complex permeability, thickness of absorbing Layer 1 and matching layer 2, respectively.

Figure 8 displays the microwave properties of double-layered composites absorber with different matching layer and absorbing layer thickness in the X band frequency range. As can be seen from figure 8(a), when using NiCF6 as the absorbing layer, the NiCF2 the matching layer, the thickness of absorbing layer stays the same, as the thickness of matching layer increases from 0.1 mm to 0.5 mm, the absorption peak shifts to lower frequency, the minimum RL also decreases, however, the effective absorption bandwidth was broadened to 3.1 GHz (9.3–12.4 GHz) when the thickness of the matching layer is 0.2 mm. As depicted in figure 8(b), the total thickness of the double layer absorber was set as a constant value of 2.5 mm, the absorption peaks shift to lower frequency while the thickness of absorbing layer increases, the minimum RL of $-48.1$ dB and the effective absorption bandwidth of 3.2 GHz (8.2–11.4 GHz) can be achieved when the absorbing layer and matching layer is 2.2 mm and 0.3 mm respectively, the minimum RL and effective absorption bandwidth shows a decreasing tendency by further increasing the thickness of the absorbing layer. Accordingly, the microwave absorption properties can be optimized by employing double-layered design, the enhanced absorption properties can be attributed to synergistic effect of improved impedance matching characteristic and superior microwave attenuation capability of the absorbing layer, which can meet the requirement of higher microwave absorption performance.

Table 1 compares the microwave absorption performance of different related composites or structures reported in the literature. By contrast, the double-layered composites in this work exhibit better absorption capability as well as effective absorption bandwidth than other reported composites or structures. The
microwave absorbing properties could be optimized by adjusting the electromagnetic parameters and thicknesses of each layer, which demonstrating that the NiCFs/GFs hybrid epoxy composites can be a promising candidate of high performance microwave absorbing materials. Furthermore, compared with other excellent absorbers which often need employing complex synthetic method, the paper-making method can be more easily realized industrialization, and double-layered design demonstrates a considerably simple way for optimizing the microwave absorbing properties.

4. Conclusions

In summary, the papermaking technology was employed to produce preformed nickel-coated carbon fibers (NiCFs)/glass fibers (GFs) paper, and the NiCFs/GFs hybrid epoxy composites were manufactured by composites liquid molding. The microwave absorbing properties of single-layered NiCFs/GFs hybrid epoxy composites were evaluated. The optimum microwave absorption properties can be obtained when the content of NiCFs in the paper is 6 wt% and the thickness of the composites is 2 mm, the minimum RL of −48.5 dB and the effective absorption bandwidth is 2.3 GHz. The double-layered composites were fabricated by using matching layer and absorbing layer to enhance the microwave absorption performance. The enhanced microwave absorbing properties of double-layered composites with minimum RL of −48.1 dB and effective absorption bandwidth of 3.2 GHz in the X band range can be achieved when the total thickness of the matching layer and absorbing layer is 2.5 mm, which is attributed to synergistic effect of improved impedance matching characteristic and superior microwave attenuation capability of the absorbing layer, demonstrating that the NiCFs/GFs hybrid epoxy composites can be a promising candidate of high performance microwave absorbing materials for realizing industrialization.

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