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Influence of CVI treatment of carbon fibers on the electromagnetic interference of CFRC composites

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Abstract

Short carbon fibers were treated at temperatures around 1100 °C in a furnace through chemical vapor infiltration technology. The fiber surface was observed by scanning electron microscopy. The reflectivity of electromagnetic radiation by the composites that were reinforced by surface-treated carbon fibers and by as-received ones was measured in the frequency range of 8.0–18.0 GHz. The reflectivity for different carbon fiber contents of 0.2%, 0.4%, 0.6% and 1.0 wt% was investigated. Results showed that the reflectivity of the composites that were reinforced by untreated carbon fibers tended to increase with the increasing fiber contents. The minimum reflectivity was −19.3 dB, far less than −10 dB, when the fiber content was 0.4% and there were wave-absorbing properties. However, after surface treatment, the minimum reflectivity was −8.1 dB for the same fiber content of 0.4%, indicating significant wave-reflecting properties. The achieved reflectivity values after surface treatment were generally greater than those without treatment.

Keywords: Carbon fibers; Chemical vapor infiltration; Cement matrix; Electromagnetic characteristics; Reflectivity

1. Introduction

Electromagnetic interference (EMI) shielding is increasingly needed for electronics and radio frequency sources, due to the interference of radio frequency waves to electronic devices [1–3]. EMI shielding involves the reflection, absorption and/or multi-reflection of such electromagnetic radiation. EMI may contribute to environmental pollution owing to the wide applications of electrical and electronic devices, such as computers, calculators, automobile communication systems, televisions, mobile telephones and other electronic products [2]. Electromagnetic waves can damage living organisms and, therefore, EMI problems are attracting the attention of various scientific and research communities. Shielding materials have been developed to reduce electromagnetic pollution by reflecting and/or absorbing electromagnetic waves effectively [3,4]. A common way of eliminating EMI problems is to use materials that have a high conductivity as a shielding package [5–8]. Typical metals, such as copper and aluminum, have been used for EMI shielding materials due to their high conductivity [6,7]. However, there is an increasing interest in the use of carbon fiber-reinforced cement-based composites (CFRCs) in the field of EMI shielding. Compared with metals, conductive carbon fibers have the advantage of low density, high modulus, high strength, variety in shape design, better appearance, light weight and low cost [1,6].

Cement composites are usually poorly conductive materials. Carbon fibers exhibit good electrical conductivity and consequent low resistivity ($9 \times 10^{-3}\ \Omega\,\mathrm{cm}$), and also high elasticity and resistance to corrosion [9–11]. A carbon fiber reinforced composite (CFRC) can be conveniently prepared by adding carbon fibers into a cement matrix. Conductive currents can be formed in it when electromagnetic waves are incident upon its surface. Carbon fibers usually reflect microwaves. They seldom absorb microwaves, so

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they are often used as reflection base plates for absorbing materials. According to the literature [9,11,13], materials exhibit good radar wave-absorbing properties when their reflectivity is less than −10 dB, which means that the electromagnetic wave is more than 90 % attenuated.

Many researchers have used different ways to treat carbon fibers to improve the electrical and mechanical properties of the CFRCs [14,15]. However, little has been reported, so far, on the chemical vapor infiltration (CVI) treatment of carbon fibers before they were incorporated into a cement matrix to prepare the CFRCs. This paper, therefore, is concerned with the influence of CVI treatment and carbon fiber contents on the electromagnetic characteristics of the CFRC composites.

2. Experimental

2.1. Major raw materials

The short carbon fibers used were PAN-based 5 mm in length, provided by *Jiyan* Carbon Ltd., Co. (*Jilin*, China). They were used in the amount of 0.2%, 0.4%, 0.6% and 1.0 wt%, respectively. The major parameters are shown in Table 1. The matrix was 42.5 R Portland cement with the execution standard GB175-1999 (*Yaoxian* Cement Plant, *Shaanxi*, China). The dispersant used was hydroxyethyl cellulose (HEC) (a viscosity of 30,000 Pa, *Shandong*, China). Silica fume was of 300 mesh with the purity 99.7% (*Shanghai*, China) and it was used in 10%. HEC was used in the amount of 0.6%. The water/cement weight ratio was 0.46. A highly efficient water-reducing agent naphthalenesulfonate formal condensate (FDN) was used in 0.5%. All the ingredients were used by weight of cement. The defoamer was liquid tributyl phosphate (TBP) (*Tianjin* Chemical Reagent Plant, China) in 0.03 vol.%. Sand used was China ISO Standard, with the execution criterion of GSB08-1337-2001 (*Xiamen* Standard Sand Co., Ltd., China). The sand/cement mass proportion was 2.9.

2.2. CVI surface treatment of short carbon fibers

Short carbon fibers were wrapped loosely with a piece of carbon cloth. The package was placed on a carbon/carbon preform in a conventional medium-sized deposition furnace. Propylene was used as precursor to introduce into the furnace at ambient pressure. The diluted nitrogen gas (N_2) was put into the furnace to prevent carbon fibers from oxidation at high temperatures. The temperature in the furnace was around 1100 °C. Propylene was decomposed and a thin layer of pyrocarbon was deposited on the fiber surface. The deposition speed and time should be kept moderate [16]. If the treatment time was too long, a thicker layer of pyrocarbon would be produced such that fibers were hardened and were easily broken. If the treatment was too short, little pyrocarbon would be formed. In this experiment, the treatment time was 96 h. Carbon fibers were observed under a SEM-JSM6700F. Apparently, the surface before treatment was smooth and straight, as shown in Fig. 1a and b, whereas that after CVI treatment was rough owing to the uneven deposition of pyrocarbon, as shown in Fig. 1c and d. By treating at high temperatures, the surface structure of carbon fibers had been reformed. The colloidal substances on the fiber surface had been removed and the chemical functional groups such as C=O and C–OH on the surface were converted into –COOH. This functional group was more active to the bonding between the fibers and the cement matrix. Thus, not only the dispersion of carbon fibers in the cement matrix was improved, but the reflectivity of the CFRC composite was influenced. In addition, the rougher surface might be beneficial to the interface bonding between the cement matrix and carbon fibers due to the larger surface area and more surface grooves.

2.3. Preparation of CFRC samples

Short carbon fibers were put into a glass beaker and immersed completely into water. The beaker was vibrated by ultrasonic waves for 10 min for pre-dispersion, during which there was no breakage of fibers. HEC was dissolved into water and the beaker was continuously vibrated for another 10 min. The mass fraction of HEC in the aqueous solution was 1.68%. The mixture was stirred intermittently during vibration. A defoamer TBP was added to drive out air bubbles. The mixture was stirred by hand for 1 min and was vibrated again for 5 min. Meanwhile, the temperature of the mixture was maintained between 38 and 42 °C. The ultrasonic power was 250 kW.

Cement, silica fume, standard sand and FDN, all in their dry states, were mixed and stirred in a JJ-5 rotary mixer with a flat beater for 1 min. The above prepared fiber mixture was then added and stirred for 2 min. After pouring the mix into the oiled 180 mm × 180 mm × 10 mm square moulds, an external vibrator was used to facilitate compaction and decrease the number of air bubbles. The samples were placed in a curing box and demolded after 24 h and then allowed to cure in the box for 28 days. The temperature in the box was 21 ± 1 °C and the relative humidity was more than 95%. Eight pieces of CFRC sam-

Table 1
Mechanical properties of carbon fibers

Item	Diameter (μm)	Density (g/cm^3)	Strength (GPa)	Shear strength (MPa)	Modulus (GPa)	Elongation (%)	Electrical resistivity (Ω cm)
Index	7 ± 0.2	1.76–1.78	2.5–3.0	80	200–220	1.25–1.5	$15–30 \times 10^{-3}$

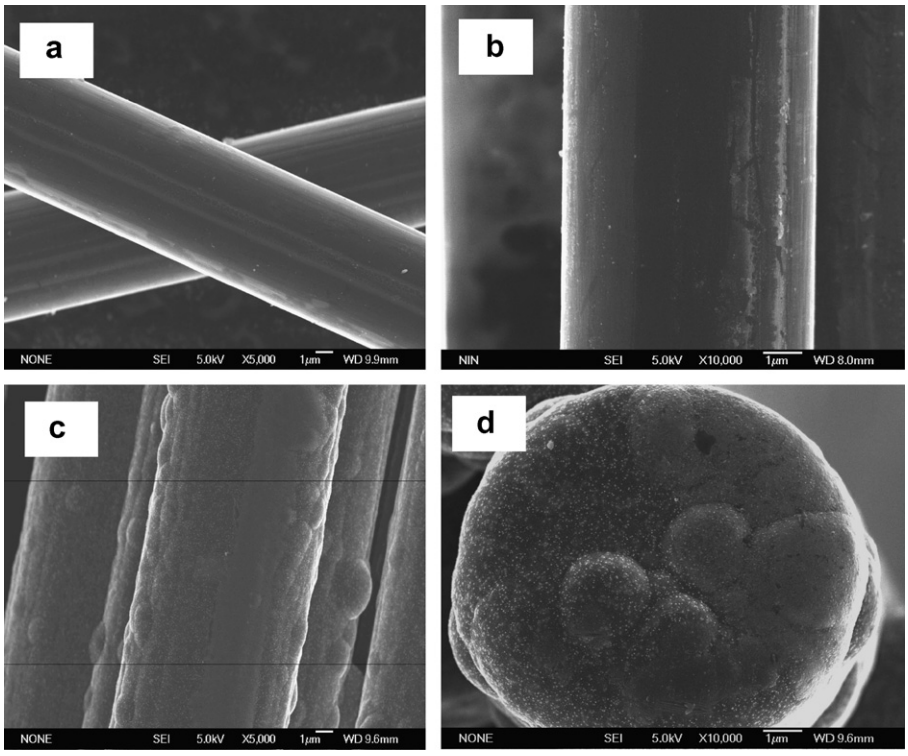

Fig. 1. SEM images of short carbon fibers. (a) and (b) Prior to CVI surface treatment; (c) and (d) after CVI surface treatment.

ples for different fiber contents of 0.2%, 0.4%, 0.6% and 1.0 wt% were prepared for measurement.

The dispersion of carbon fibers affected greatly the mechanical properties of the CFRC composites. Fig. 2a shows a SEM image of the fracture surface of a CFRC sample for good dispersion of carbon fibers. Fibers can stop the formation of micro-cracks and their expansion in the matrix, thereby enhancing the toughness of the composites. Therefore, the bending strength and the tensile strength increase with the increasing fiber contents as shown in Fig. 2b. Additionally, good dispersion is beneficial to the formation of conductive networks owing to the fiber contact one another.

2.4. Reflectivity measurements of CFRC samples

A finished sample is a square of 180 mm long, 180 mm wide and 10 mm thick. It must be dry before measurement to eliminate the possible influence of moisture on the conductivity, which will further affect the reflectivity.

The Naval Research Laboratory (NRL) testing system for the reflection loss was applied to measure the reflectivity as shown in Fig. 3. The measurement ranged between 8 and 18 GHz. The maximum measurable attenuation was −40 dB. The incident wave was transmitted from a horn antenna and was reflected by the sample. The other horn antenna received the reflected wave. The signal was then

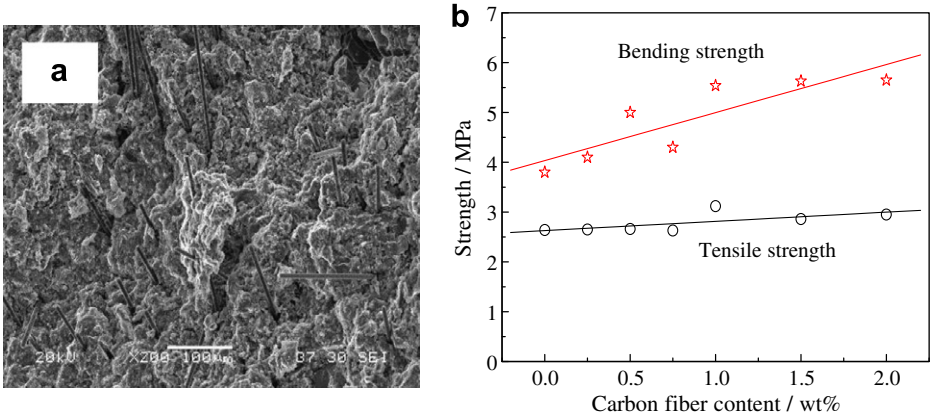

Fig. 2. (a) SEM image of the fracture surface of a CFRC sample for good fiber dispersion (0.6 wt%); (b) the bending strength and the tensile strength vary with the fiber contents.

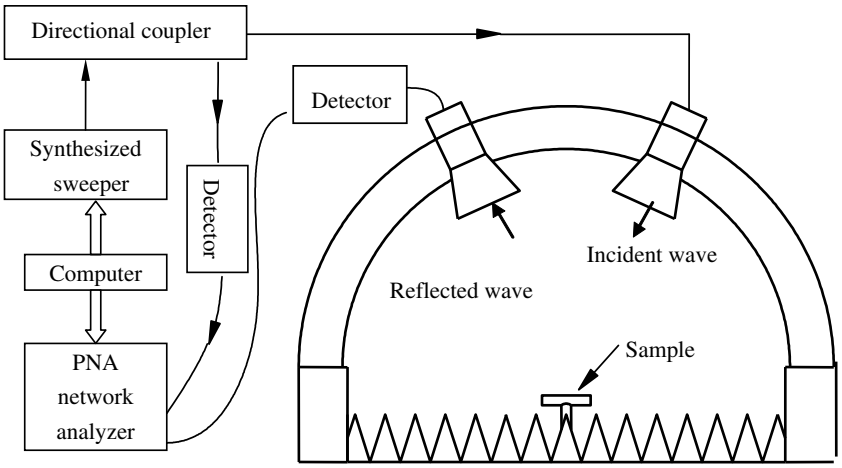

Fig. 3. Simplified block setup of the NRL reflectivity measurement system.

transmitted to the network analyzer. The minimum test distance from the sample to the horn mouth was approximately evaluated in accordance with the formula:

$$R_{\min} = \frac{D^2}{\lambda} \tag{1}$$

where D is the lateral maximum size of the antenna mouth, and λ is the wave length. In this work, the distance between the sample and the horn mouths was about 1.75 m. The floor around the sample pedestal was covered with standard pyramidal absorbers, providing a −40 dB background level relative to the 0 dB calibration [13,17].

The tester was connected to an Aglient PNA Series Network Analyzer (E8362B, 10–20 GHz; USA). The frequency was scanned from 8.0 to 18 GHz, and 401 data points were taken for reflection and also for transmission. The measurement was carried out in a laboratory in the School of Materials Science, Northwestern Polytechnical University, People's Republic of China.

3. Results and discussion

3.1. Electromagnetic interference mechanism of CFRC samples

Continuous currents can be produced in continuous carbon fibers upon the action of electromagnetic fields. Thus, electromagnetic waves will be reflected strongly. However, continuous currents are not easily produced in CFRC composites that are reinforced with short carbon fibers. When carbon fibers are uniformly distributed, they can contact one another to form conductive networks. On the other hand, electrons can leap from one fiber to another when carbon fibers are close enough for hopping conduction ($\leqslant$10 nm). In this work, the fiber length is around 5 mm. The wavelength of the incident wave lies in between 1.67×10^{-2} and $3.75\times\ 10^{-2}$ m in the frequency range of 8–18 GHz. The fiber length is close to the half-wavelength of the incident wave. Consequently, the short carbon fibers can be regarded as electric dipoles [9,11,12]. When the incident wave arrives at the surface of the composites, it interacts with the fibers. At this moment, a strong coupling occurs, producing current. This way, the electromagnetic energy turns into heat energy, attenuating in the matrix.

The variations of fiber contents mean that the distance between electric dipoles is changed. When the fiber content is lower, the interaction between electric dipoles is weaker. Group of electric dipoles are formed, which are similar to an array of discrete vibrators. Each independent group contacts through electrical impedance to attenuate waves. When the fiber content increases to some point, a multiple interference pattern between adjacent dipoles takes place. The approaching electric line of vibrator forces repel one another. Concurrently, the electric field superimposes and the stronger wave-reflection occurs [2,11].

The direction of the reflected wave is closely related to the carbon fiber orientation in the composites. When the direction of the incident wave is parallel to the fiber orientation, a large conductive current will be produced. Thus, microwaves are strongly reflected. Under such a circumstance, the behavior of the fibers looks like metals. However, when the direction of the incident wave is perpendicular or oblique to the fiber orientation, microwaves are absorbed mainly. The principle of the attenuation of microwaves is illustrated in Fig. 4. Suppose that the incident wave arrives in the sample along the x_1 axis. A θ angle appears between the direction of the incident wave and the fiber orientation then. This angle divides the incident wave into two components: one is parallel $E^{i}_{\parallel}$ and the other is perpendicular $E^{i}_{\perp}$. When the two components interact with carbon fibers, two corresponding reflected components $E^{r}_{\parallel}$ and $E^{r}_{\perp}$ are formed. The relations between the amplitudes of the parallel reflected components can be expressed as $|E^{r}_{\parallel}| \approx |E^{i}_{\parallel}|$, whereas those between the amplitudes of the perpendicular reflected components can be described as $|E^{r}_{\perp}| \ll |E^{i}_{\perp}|$ [9,13]. Accordingly, the composite reflected wave E^{r} is not parallel to the direction of the incident wave, resulting in the appearance of a component that is parallel

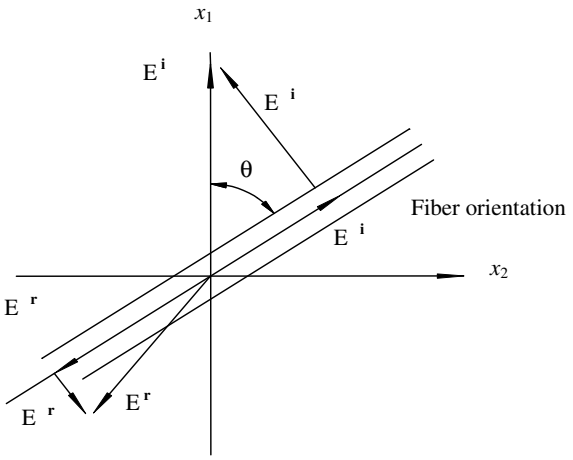

Fig. 4. Reflected electric field at fibers not parallel to the incident electric field.

to the x_2 axis. As a result, microwaves are attenuated either by reflection or by absorption.

3.2. Measurement principle of reflectivity of CFRC samples

Given the wave length, reflectivity is defined as the following when the electromagnetic wave is incident upon the plane from the same angle in the same power.

$$\Gamma = P_a/P_m \tag{2}$$

where Γ is the reflectivity of the sample, P_a is the reflection power of the sample, and P_m is the reflection power of the good conductive plane. Practically, the absolute reflection power is not measured, whereas the ratio of the reflection power of the plane and the sample against the same reference signal is measured:

$$\Gamma_m = P_m/P_i \tag{3}$$
$$\Gamma_a = P_a/P_i \tag{4}$$

where P_i is the power of the reference signal that is in direct proportion to the transmitting signal. Thus, the reflectivity of the sample can be expressed as:

$$\Gamma = P_a/P_m = \frac{P_a/P_i}{P_m/P_i} = \frac{\Gamma_a}{\Gamma_m} \tag{5}$$

When expressed in dB, the following Γ formula is usually used:

$$\Gamma_{dB} = 10\ \lg\ \Gamma_a - 10\ \lg \Gamma_m \tag{6}$$

Due to the way reflectivity is measured and calculated, the maximum reflectivity obtainable is zero and that the practical case will always result in negative values when quoted in dB's.

3.3. Analysis of reflectivity versus frequency curves of CFRC samples

The reflectivity versus frequency curves of the CFRC samples are illustrated, respectively, from Figs. 5–8. According to the literatures, a threshold value of reflectivity −5 dB for preventing electromagnetic radiation for civil use. When the measured reflectivity is over this value, the wave-reflecting properties are dominant. When the measured reflectivity is below this value, the wave-absorbing properties prevail. When the fiber content is 0.2%, two sharp absorbing peaks appear as shown in Fig. 5a. The minimum reflectivity is −14.3 dB near the frequency of 8.0 GHz. The composites exhibit wave-absorbing properties. After carbon fibers experienced thermal treatment, one absorbing peak emerges as seen in Fig. 5b. The reflectivity is −8.4 dB around 15 GHz. There are dominantly wave-reflecting properties.

When the fiber content increases to 0.4%, still two absorbing peaks appear as shown in Fig. 6a. The reflectivity data are −19.3 dB and −11.9 dB, both of which are below −10 dB, demonstrating wave-absorbing properties. After CVI treatment, no apparent absorbing peak occurs as seen in Fig. 6b. The reflectivity is narrowed between

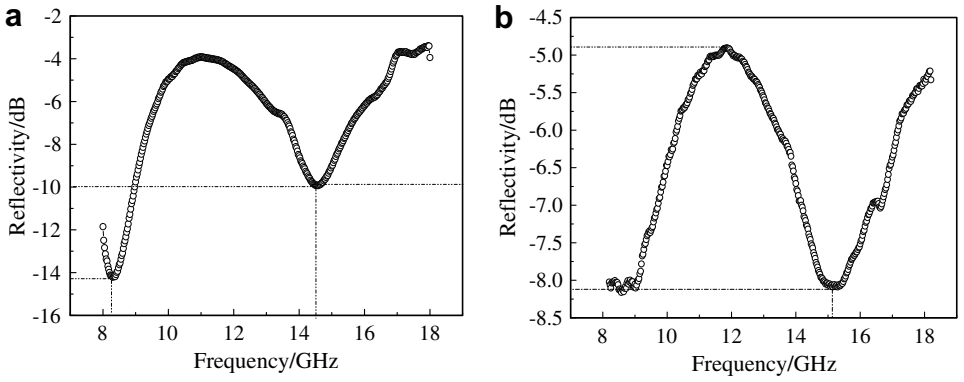

Fig. 5. Reflectivity versus frequency curves of a CFRC sample for a fiber content of 0.2 wt%. (a) A CFRC sample reinforced by untreated carbon fibers; (b) a CFRC sample reinforced by CVI treated carbon fibers.

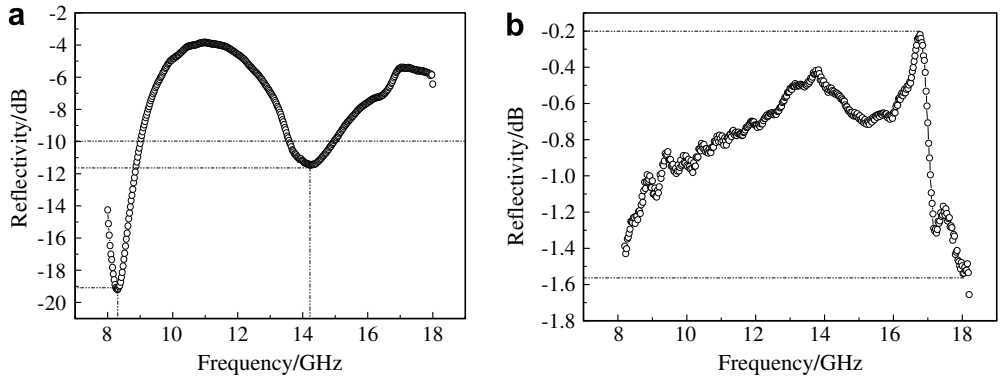

Fig. 6. Reflectivity versus frequency curves of a CFRC sample for a fiber content of 0.4 wt%: (a) a CFRC sample reinforced by untreated carbon fibers; (b) a CFRC sample reinforced by CVI treated carbon fibers.

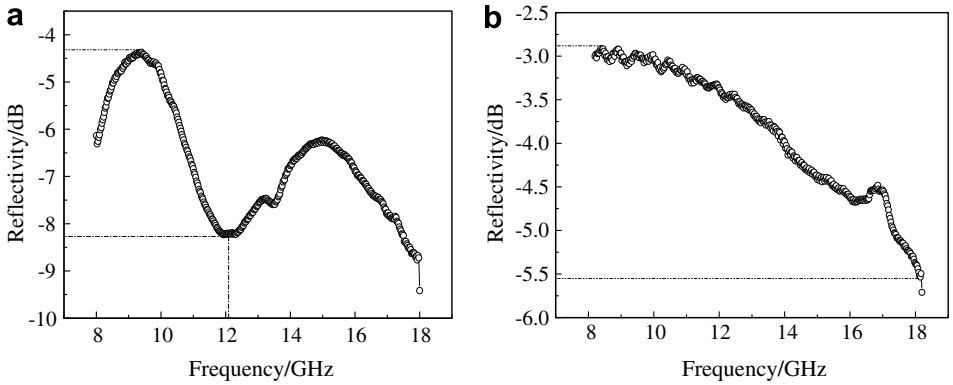

Fig. 7. Reflectivity versus frequency curves of a CFRC sample for a fiber content of 0.6 wt%: (a) a CFRC sample reinforced by untreated carbon fibers; (b) a CFRC sample reinforced by CVI surface treated carbon fibers.

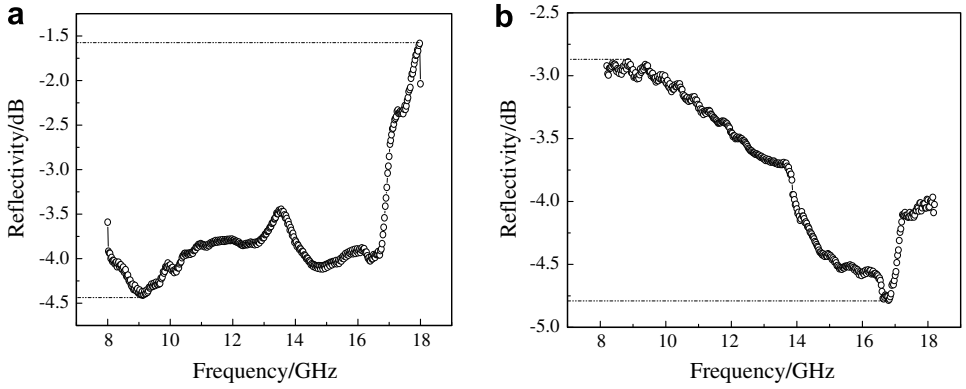

Fig. 8. Reflectivity versus frequency curves of a CFRC sample for a fiber content of 1.0 wt%; (a) a CFRC sample reinforced by untreated carbon fibers; (b) a CFRC sample reinforced by CVI treated carbon fibers.

−0.2 and −1.57 dB. Any data in this range is greater than −10 dB. There are stronger wave-reflecting properties. For convenient study, the data at the end of the curve is recognized as the minimum reflectivity.

As the fiber content rises to 0.6%, an inactive absorbing peak takes place as shown in Fig. 7a. The reflectivity is −8.2 dB around 12 GHz. After pyrocarbon was deposited on the fiber surface, the reflectivity is compressed within −2.55 to −5.52 dB as shown in Fig. 7b. Obviously, the wave-reflecting properties prevail.

When the fiber content reaches 1.0% finally, there are hardly absorbing peaks before CVI treatment as shown in Fig. 8a. The reflectivity is compressed within a scope of −1.52 to −4.48 dB. After carbon fibers were treated through CVI process, a very weak absorbing peak appears as shown in Fig. 8b. The reflectivity is −4.56 dB near

17 GHz. It can be found that whether carbon fibers are treated or not, the reflectivity data are far greater than −10 dB. The composites exhibit apparent wave-reflecting properties.

The more negative the reflectivity value, the better the material absorbs waves [11–13]. When the wave-absorbing performance of a material is emphasized, the smaller reflectivity value is desired. When the wave-reflecting property of a material is stressed, the larger reflectivity value prevails [9,13]. Larger reflectivity values mean that the material reflects dominantly electromagnetic waves and smaller values imply that the material absorbs mainly microwaves. Usually, a higher dB value, which implies that the material is a good EMI shielding material against electromagnetic waves, is desired for shielding effectiveness transmission measurements. The higher the dB is, the better the EMI shielding capabilities. A lower dB means that the material is a good wave absorber [17–19]. The electromagnetic energy can be attenuated by combination of reflection and absorption, anyway [20–22].

The measured minimum reflectivity data are listed in Tables 2 and 3, respectively. It can be seen from Table 2 that the reflectivity values tend to increase with the increasing fiber contents. Before 0.4%, the wave-absorbing property is displayed. After this percentage, the wave-reflecting property becomes dominant. When carbon fibers are treated by CVI process, all the achieved reflectivity values are above −10 dB. The wave reflections are dominant. Compared with the reflectivity values prior to CVI treatment, those after CVI treatment, as a whole, are greater. Therefore, it can be inferred that CVI treatment enhances the wave reflection capability of the CFRC composites. Chung [1,6] found that due to the skin effect (i.e. the phenomenon that electromagnetic radiation at high frequencies only interacts with the near surface region of an electrical conductor), small fillers (higher surface area) should provide better shielding. CVI treated carbon fibers are ideal reinforcing fillers owing to their high strength and modulus, large surface area and electrical properties [23].

Table 2
The minimum reflectivity of the CFRC composite reinforced by as-received carbon fibers for different carbon fiber contents in different frequency ranges

No.	Carbon fiber content (wt%)	Minimum reflectivity (dB)	Frequency range (GHz)	Reflectivity below −10 dB
1	0.2	−14.3	8.0–8.5	Existent
2	0.4	−19.3	8.0–8.5	Existent
3	0.6	−8.2	11.0–13.0	Nonexistent
4	1.0	−4.4	8.0–10.0	Nonexistent

Table 3
The minimum reflectivity of the CFRC composite reinforced by CVI-treated carbon fibers for different carbon fiber contents in different frequency ranges

No.	Carbon fiber content (wt%)	Minimum reflectivity (dB)	Frequency range (GHz)	Reflectivity below −10 dB
1	0.2	−8.1	14.3–15.8	Nonexistent
2	0.4	−1.6	8.0–18.0	Nonexistent
3	0.6	−5.5	8.0–18.0	Nonexistent
4	1.0	−4.6	16.0–17.0	Nonexistent

4. Conclusions

(1) CVI surface treatment at high temperatures has reformed the surface structure of carbon fibers by enlarging the specific surface area and by activating the chemical functional groups on the fiber surface. After CVI treatment, the wave-reflecting capability is enhanced and the composites are more suitable for the application of electromagnetic shielding materials.
(2) Before CVI process, the reflectivity data in general tend to increase with the increasing fiber contents. The wave-absorbing properties are dominant before a fiber content of 0.4%. After this percentage, the reflection prevails. The minimum reflectivity reaches −19.3 dB.
(3) After CVI treatment, the reflectivity increases except the data at 0.4%. All the achieved reflectivity values are greater than −10 dB, with the minimum data of −8.1 dB. The wave reflection properties are dominant.

Acknowledgements

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