CONTINUOUS CARBON FIBER POLYMER-MATRIX STRUCTURAL COMPOSITES FOR ELECTROMAGNETIC FUNCTIONS

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Introduction

Composite materials are well-known for structural applications, due to the combination of high strength, high modulus and low density that is characteristic of composites containing continuous fibers as the reinforcement. Composite materials are commonly designed in terms of the mechanical properties, with little or no attention to the functional properties. However, the increasing importance of non-structural applications and multifunctional structural applications has given impetus to the development of functional composite materials, such as composite materials for electrical, electromagnetic and thermal applications. This paper concerns composite materials for electromagnetic applications.

Electromagnetic functions include the shielding, reflection and absorption of electromagnetic radiation. In particular, electromagnetic interference (EMI) shielding refers to the reflection and/or absorption of electromagnetic radiation by a material, which thereby acts as a shield against the penetration of the radiation through the shield. As electromagnetic radiation, particularly that at high frequencies (e.g., radio waves, such as those emanating from cellular phones) tend to interfere with electronics (e.g., computers), EMI shielding of both electronics and radiation source is needed and is increasingly required by governments around the world. The importance of EMI shielding relates to the high demand of today's society on the reliability of electronics and the rapid growth of radio frequency radiation sources.

In contrast to shielding is the transmission of electromagnetic radiation, as needed for low-observable aircraft and radomes. Electromagnetic observability of an object refers to the ability of the object to be observed or detected by electromagnetic waves, particularly microwaves associated with a radar. Low observability is desirable for military aircraft and ships. In this work, polymer-matrix structural composites containing continuous carbon fibers have been developed for electromagnetic functions such as shielding and low observability in the radio wave frequency range.

Increasing the electromagnetic interference shielding effectiveness of carbon fiber polymer-matrix composite by using activated carbon fibers

The mechanisms of EMI shielding are reflection, absorption and multiple reflections. Reflection is usually the dominant mechanism, especially for carbon fibers. The contribution by multiple reflections is usually relatively small for carbon composites. To improve the contribution by reflection, previous work has emphasized the use of nickel coated carbon fibers, as nickel is more conductive than carbon [1,2]. However, the enhancement of the contribution by multiple reflections by fiber modification has not received previous attention. In this work, we found that appropriate activation of the carbon fibers enhances the contribution of multiple reflections without degrading the mechanical properties.

Activation is a method of fiber surface modification that involves a chemical reaction which causes increase of the specific surface area through the formation of surface pores. Activated carbon fibers with specific surface area typically exceeding 1000 m^2/g are used for adsorption, which is relevant to fluid purification. They are also used for the storage of hydrogen, natural gas and other fuels. In addition, they are used as electrodes for double-layer capacitors and for industrial processes. However, they have not been previously investigated for use in electromagnetic applications such as EMI shielding.

Epoxy (a thermoset) is the dominant matrix used for carbon fiber polymer-matrix structural composites. The epoxy used in this work was EPON Resin 862 together with EPI-CURE 3234 curing agent in the weight ratio 100 : 15.4 (Shell Chemical Co., Houston, TX).

The carbon fiber used in this work was Thornel P-25 (without sizing or twist) from Amoco Performance Products Inc., Alpharetta, GA. The diameter was 11 μm.

Carbon fiber activation was conducted by (i) washing the fiber with acetone for the purpose of surface cleansing, (ii) heating the fibers in flowing nitrogen from room temperature to 1000°C at a heating rate of 10°C/min, (iii) maintaining the temperature at 1000°C for 1 h for the purpose of removal of surface volatile components, if any, (iv) introducing CO_2 gas (0.6 vol.% in N_2) for 1 h while the temperature remained at 1000°C for the purpose of activation, and (v) cooling to room temperature at 10°C/min in flowing nitrogen.

Unidirectional carbon fiber epoxy-matrix composites with a fiber volume fraction of 34.5% were fabricated by (i) preparing the prepreg (i.e., immersing the fibers in the resin, with curing agent, and then immediately squeezing the fibers between glass tubes (to remove excessive resin) and then winding the fibers onto a mandrel), (ii) after about 30 min, cutting the prepreg into sheets, (iii) stacking eight prepreg sheets in a steel mold of size 178 x 101 mm, such that the fibers in all the layers were in the same direction, (iv) initial curing of the resin by heating from 25 to 121°C at a heating rate of 4°C/h and at a pressure of 2.0 ± 0.2 MPa, and (v) post curing of the resin by heating at 121°C and 2.0 ± 0.2 MPa for 2 h. Composites were fabricated using separately as-received fibers and activated fibers.

Electromagnetic testing using the coaxial cable method was conducted using an Elgal (Israel) SET 19A shielding effectiveness tester, which was connected to a Hewlett-Packard (HP) 8752C network analyzer. An HP 85032B type N calibration kit was used to calibrate the system. The frequency was scanned from 1.0 to 1.5 GHz, while 200 data points were taken in reflection and also in transmission. The specimens were annular, with outer diameter 97 mm, inner diameter 32 mm and thickness 3 mm.

The attenuation upon transmission (the EMI shielding effectiveness) is significantly increased by activating the fibers, whereas the attenuation upon reflection is not much affected by activation (Table 1). This means that the shielding effectiveness is increased by activation, not because of increased reflectivity, but because of increased multiple reflections and/or increased absorption. Since the activation has minor effect on the overall dipole concentration in the composite (in spite of the functional groups formed on the fiber surface after activation), the enhanced shielding effectiveness is mainly attributed to increased multiple reflections.

Table 1 Attenuation under transmission and under reflection of carbon fiber composites.

Fiber type	Attenuation upon transmission (dB)	Attenuation under reflection (dB)
As-received	29.6 ± 0.9	1.3 ± 0.2
Activated	38.8 ± 0.8	1.2 ± 0.2

Single fiber tensile testing was performed on as-received and activated fibers, using a screw-action mechanical testing system (2/D, Sintech, Stoughton, MA) at a crosshead speed of 1 mm/min. The gage length was 128 mm. Activation has little effect on the tensile strength or modulus of the fibers (Table 2). It probably increases both strength and modulus slightly, as made possible by the heating at 1000°C in step (iii) of the activation process.

Table 2 Tensile properties of carbon fibers before and after activation. Standard deviations are shown in parentheses.

Fiber type	Strength (MPa)	Modulus (GPa)
As-received[a]	665 (87)	126 (7)
Activated[b]	727 (151)	138 (15)

[a] Six specimens tested
[b] Eight specimens tested

The BET specific surface area of the fibers was determined by nitrogen adsorption and gas pressure measurement, using ASAP 2010 (Micromeritics, Norcross, GA). The specific surface area is 7.6 and 90 m^2/g before and after activation respectively. Though the specific surface area is significantly increased by the activation, the area after activation is low compared to that of typical activated carbon fibers. Thus, the absence of tensile property degradation by activation is not unreasonable. The main (or mean) pore size of the fibers is 19.3 and 20.1 Å before and after activation respectively. Hence, activation increased the specific surface area, with little effect on the pore size.

In conclusion, the use of activated carbon fibers with specific surface area 90 m^2/g as a continuous reinforcement (35 vol.%) in a polymer-matrix composite enhances the EMI shielding effectiveness of the composite due to multiple reflections. The shielding effectiveness at 1.0 – 1.5 GHz is 39 dB, compared to a value of 30 dB when untreated fibers are used. The activation treatment does not degrade the tensile properties of the fibers.

Decreasing the electromagnetic observability of carbon fiber polymer-matrix composites by using epoxy-coated carbon fibers

A way to decrease the electromagnetic observability [3] is to decrease the reflectivity. The reflectivity is related to the electrical conductivity. Hence, a way to decrease the reflectivity is to decrease the conductivity. The conductivity of a carbon fiber polymer-matrix depends not only on that of the fibers themselves, but also depends on the electrical connectivity among the fibers. This is particularly true for the conductivity in the transverse direction (direction perpendicular to the fiber direction). In this paper, we decreased the electrical connectivity by using epoxy-coated carbon fibers, thereby attaining the goal of decreasing the observability.

The carbon fiber used was Thornel P-25 (without sizing or twist) from Amoco Performance Products Inc., Alpharetta, GA. The diameter was 11 μm. The epoxy used was EPON Resin 862 together with EPI-CURE 3234 curing agent in the weight ratio 100 : 15.4, as provided by Shell Chemical Co., Houston, TX.

The coating of carbon fibers with epoxy was carried out by (i) washing the fibers with acetone for the purpose of surface cleaning, (ii) immersing the fibers in a resin solution (obtained by dissolving 2 g of 862 resin and 0.308 g of curing agent in 1000 ml of acetone) for 24 h in the presence of vibrations provided by an ultrasonic cleaner, and (iii) removing the fibers from the solution and drying in air at room temperature.

Unidirectional carbon fiber epoxy-matrix composites with a fiber volume fraction of 35.5% were fabricated by (i) preparing the prepreg (i.e., immersing the fibers in the resin, with curing agent, and then immediately winding the fibers onto a mandrel), (ii) after about 30 min, cutting the prepreg into sheets, (iii) stacking eight prepreg sheets in a steel mold of size 178 x 101 mm, such that the fibers in all the layers were in the same direction, (iv) initial curing of the resin by heating from 25 to 121°C at a heating rate of 4°C/h and at a pressure of 2.0 ± 0.2 MPa, and (v) post curing of the resin by heating at 121°C and 2.0 ± 0.2 MPa for 2 h. Composites were fabricated using separately as-received fibers and epoxy-coated fibers.

The electromagnetic transmission/reflection was measured as in the last section, except that the specimens were 2 mm thick.

Tensile testing of composites in the longitudinal direction was performed by using a hydraulic mechanical testing system (MTS Corp., Eden Prairie, MN) at a crosshead speed of 0.2 mm/min. The specimen length (excluding the end-tab regions) was 105 mm in the stress direction. Four specimens of each type were tested.

The electrical resistivity of composites in the transverse direction was measured by using the two-probe method, using silver paint in conjunction with copper wire for electrical contacts. The same results were obtained using the four-probe method. The specimen was of size about 45 x 18 x 1.6 mm for the composite with as-received fibers and about 31 x 20 x 2.0 mm for the composite with epoxy-coated fibers, such that the longest dimension is in the transverse direction. The distance between the two electrical contacts ranged from 27.2 to 34.2 mm for the composite with as-received fibers and ranged from 23.3 to 24.5 mm for the composite with epoxy-coated fibers. All dimensions were separately measured for each specimen. Four specimens of each type were tested.

Table 3 shows the attenuation upon transmission (same as the EMI shielding effectiveness) and that upon reflection. The attenuation upon transmission is decreased by coating the fibers with epoxy, whereas the attenuation upon reflection is increased by the epoxy coating. This means that the reflectivity is decreased by the epoxy/coating, thus causing the transmissivity to increase. Table 3 also shows that the transverse resistivity is increased by coating the fibers with epoxy. The increase in resistivity is consistent with the decrease in reflectivity.

Table 4 shows that both the tensile strength and modulus are decreased by about 10% by the epoxy coating of the fibers, while the elongation at break is not affected. The decreases in strength and modulus are attributed to the presence of the interface between the matrix and the epoxy coating on the fibers in the case of the composite containing epoxy-coated fibers. This interface is absent in the composite containing as-received fibers.

In conclusion, the electromagnetic observability of epoxy-matrix composites containing continuous carbon fibers was decreased by using epoxy-coated carbon fibers. The attenuation of electromagnetic waves at 1.0 – 1.5 GHz upon reflection was increased from 1.3 to 1.7 dB, while the attenuation upon transmission was decreased from 30 to 24 dB. The effect is attributed to the decreased transverse electrical conductivity of the composite due to the epoxy coating. The epoxy coating caused the tensile strength and modulus of the composite to decrease by about 10%, while the elongation at break was not affected.

Table 3 Attenuation upon transmission, attenuation upon reflection and transverse electrical resistivity of carbon fiber epoxy-matrix composites at 1.0 – 1.5 GHz.

Fiber type	Attenuation upon transmission (dB)	Attenuation upon reflection (dB)	Resistivity (Ω.mm)
As-received	29.6 ± 0.9	1.3 ± 0.2	20.7 ± 2.4
Epoxy coated	23.8 ± 0.8	1.7 ± 0.2	70.9 ± 3.8

Table 4 Tensile properties of carbon fiber epoxy-matrix composites. Standard deviations are shown in parentheses

Fiber type	Strength (MPa)	Modulus (GPa)	Elongation at break (%)
As-received	718 (11)	85.5 (3.8)	0.84 (0.03)
Epoxy coated	626 (21)	73.5 (4.4)	0.85 (0.03)

References

1. Huang CY and Pai JF. Studies on processing parameters and thermal stability of ENCF/ABS composites for EMI shielding. J. Appl. Polym. Sci. 1997;63(1):115-123.
2. Shui X and Chung DDL. Submicron diameter nickel filaments and their polymer-matrix composites. J. Mater. Sci. 2000;35:1773-1785.
3. Peterson J. Overview of low observable technology and its effects on combat aircraft survivability. J. Aircraft 1999;36(2):380-388.