Electromagnetic Wave Shielding and Microwave Absorbing Properties of Hybrid Epoxy Resin/Foliated Graphite Nanocomposites

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ABSTRACT: The aim of this study is to prepare and characterize foliated graphite nanosheets (FGNs) reinforced composites based on epoxy resin for the electromagnetic wave shielding and microwave absorbing applications. The microstructure of as prepared FGNs and epoxy reinforced with different content of foliated graphite was examined by means of scanning electron microscopy and transmission electron microscopy. The effect of FGNs on thermal stability of composites was examined by thermal gravimetric. It is found that the inclusion of FGNs into the epoxy resin matrix enhances the microstructure core of epoxy resin composites. Static electric properties such as electrical conductivity, carrier mobility, number of charge carriers, and thermoelectric power of composites were studied in details. Dielectric properties of epoxy/FGN composites were characterized as a function of composition and frequency in the range of 1–18 GHz. The electromagnetic wave shielding as a function of frequency of composites was examined and compared with theoretical values. The highest shielding effectiveness was obtained for high foliated graphite loading sample FG40 at frequency of 18 GHz it equals to 62 dB. Finally, the electromagnetic wave properties such as absorption loss and reflection loss as a function of frequency were investigated. © 2012 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 000: 000–000, 2012

INTRODUCTION

Nanostructured composites constitute a rapidly growing field, in terms of both scientific interest and industrial application. Conducting polymeric nanocomposites are hybrid materials formed by an organic polymeric matrix, in which inorganic fillers such as carbon black, carbon nanotubes, gold, silver, graphite, and so forth are dispersed in nanometric dimensions.[1–5] On this scale, the inorganic filler can dramatically improve the physical properties of the polymer, even at very low contents.[6–10] Foliated graphite nanosheets (FGNs) are emerging as a novel nanostructured material with a potential for electrochemical energy storage device,[6] Positive temperature coefficient of resistance (PTCR) thermistors,[1] gas sensors,[8] bipolar plates,[9] and further more applications due to its unique characteristics of good chemical stability, high electrical and thermal conductivity,[10] high aspect ratio,[11]

low weight, and easy manufacturing.[12] In everyday life, people incessantly meet with a lot of devices and tools such as microwave ovens, screens, inductive heaters, alarms, mobile phones, cables supplying electric energy for receivers and wires used for information transfer and energy, TV/radio stations, radar systems, and different kinds of electronic devices, which emit electromagnetic radiation, resulting in a significant increase of electromagnetic distortion in human environment.[2,6] The development of microwave absorbing techniques continues to attract much attention because of the increasing environmental pollution from nonionizing microwave irradiation and the essential part of a stealth defense system for all military platforms.[12,13] Furthermore, a controversy is in progress worldwide regarding the potential health hazards associated with exposure to electromagnetic fields. A growing need exists for suitable materials,

which can act as a shield and limit the effects of electromagnetic energies, and it is also needed for deterring electromagnetic forms of spying.[3] To shield and limit against electromagnetic interference shielding (EMI), conductive polymer composites have begun to replace metals for various shielding applications in the electrical and electronic industries, especially for the electronic housing materials. Most of these composites are not strong enough for most structural applications.[4] So, the traditional EMI shielding materials such as permalloy, nickel, steel, and so forth are still in use.[8] However, the high density of these materials restricts some of their applications. To reduce the density of some of the metals, many researchers have developed techniques to produce metal foams and some of them have excellent properties.[10–12] To solve the above problems, in this study, we successfully fabricated a highly conducting polymer nanocomposites based on FGNs-loaded epoxy resin. The microstructure, electrical properties, EMI, and microwave absorbing of epoxy/foliated graphite were examined in details.

EXPERIMENTAL DETAILS

Materials

Polymer material used in this work was epoxy resin with a commercial type Epikote 815 with a density of 2.15 g/mL and hardener type B002W produced by Yuka Shell Epoxy Chemical, Tokyo, Japan. The resin/hardening ratio was stoichiometric according to the manufacturer's data sheets. The conducting filler used in the elaboration of composites consisting of FGNs was prepared by the hydrosulfate procedure involving the following three main steps: (1) stirring of graphite flakes 500 mesh for 96 h in a mixture of concentrated nitric and sulfuric acids (4 : 1 vol) to form an intercalation compound (graphite hydrosulfate), (2) hydrolysis of the graphite intercalation compound with water and drying of oxidized graphite to a friable state, and (3) thermal expansion of oxidized graphite by rapid heating at $1000°C$ for 50 s to form exfoliated graphite. Then, the exfoliated graphite powder was ultrasonically irradiated with power 500 W for 12 h to get FGNs with an apparent density of 0.005 g/cm^3.

Fabrication of Epoxy/FGNs

Five batches of epoxy/FGNs weight ratios are: 100/0, 90/10, 80/20, 70/30, 60/40 and abbreviated as epoxy, FGN10, FGN20, FGN30, and FGN40, respectively. The epoxy/hardener with different content of FGNs was prepared by centrifuging mixer (Matsuo, Obu, Japan) for 2 min at room temperature. The bulk samples of nanocomposite were obtained by casting the green composites on Teflon mould. Then, the nanocomposites were vulcanized in an oven at $135°C$ for 2 h and cooled down to room temperature in the oven.

Instruments Used

The surface morphology and the dispersion of FGNs in the epoxy matrix were examined using scanning electron microscope (SEM, JSM-5310 LVB; JEOL, Tokyo, Japan). Transmission electron microscopy (TEM) of as synthesized FGNs was performed using a (JEOL 2010) high resolution TEM operated at 200 kV. The thermal stability of pure epoxy and epoxy/graphite nanocomposites was investigated using nonisothermal thermogravimetric (TG) with a Shimadzu DTG-50 instrument. The meas-

urements were conducted at heating rates of $10°C$/min in air atmosphere in the temperature range of $20–600°C$ and the mass of the sample is 0.3 g. The volume electrical conductivity of the epoxy/FGN nanocomposites was performed by two probe methods.[1] The current through the sample was measured under a steady constant voltage using a digital Keithley 642 electrometer. The specimens were made in the form of discs with diameter 1 cm and thickness 30 mm. Mosaic gold–copper electrodes were embedded into samples during the preparation process to reduce the sample electrode contact resistance.[2] A computer was used to acquire data. The Hall effect was used to determine the carrier concentration and mobility carriers of epoxy/FGNs nanocomposites. The measurements were performed using the van der Pauw configuration under direct current ranging from 2×10^{-3} to 4×10^{-4} A, and the applied magnetic field was 0.33 T. The equipment used for this purpose was a Keithley source meter (model 231). The thermoelectric power (TEP) measurements were made on computer-controlled equipment by gripping the samples between two blocks of the stainless steel. Two fine thermocouples were attached on the samples to measure the temperature gradient across these samples produced by heater attached one of the stainless steel blocks. Dielectric properties such as relative and imaginary permittivity, and dielectric loss as a function of frequency in the range of 1–18 GHz were performed using an RCL bridge (3531ZHitester, Hioki, Japan). Microwave properties of nanocomposites were carried out with Hewlett–Packard waveguide line containing spectroanalyzer, power meter, coefficient of reflection meter, and coefficient of attenuation meter. Samples having a thickness of 1 mm were used during measurement. The microwave properties of nanocomposites were examined for each sample by continuously sweeping the frequency between 1 and 18 GHz.

RESULTS AND DISCUSSION

Morphology Observation

The morphology and the degree of dispersion of FGNs in the epoxy matrix were observed by SEM. SEM image of as synthesized FGNs is depicted in Figure 1(a). The TEM image inset in Figure 1(a) indicates that the as-synthesized foliated graphite composed of nanosheets with thickness of 22 nm. The FGNs possessed a high aspect ratio (width-to-thickness) of about 400. SEM of FGN40 sample is depicted in Figure 1(b). In Figure 1(b), it is seen that graphite nanosheets are well distributed homogeneously into resin matrix. In addition, there is a good intimate adhesion between the epoxy matrix and foliated graphite surfaces. Because of the high aspect ratio nanosheets, a very low weight fraction of FGNs is sufficient to make network contacts resulting in an increase of the thermodynamic stability, electrical properties, EMI, and microwave absorbing of nanocomposites as confirmed later in this article.

Thermal Stability Study

The thermal stability of the pristine epoxy resin and epoxy/FGN nanocomposites was analyzed by TG to evaluate the influence of FGNs contents on the decomposition and thermal stability of the nanocomposites. The epoxy charged with FGNs has a higher thermal stability than the uncharged epoxy. This assertion is based on the analysis of the thermal gravimetry analysis (TGA)
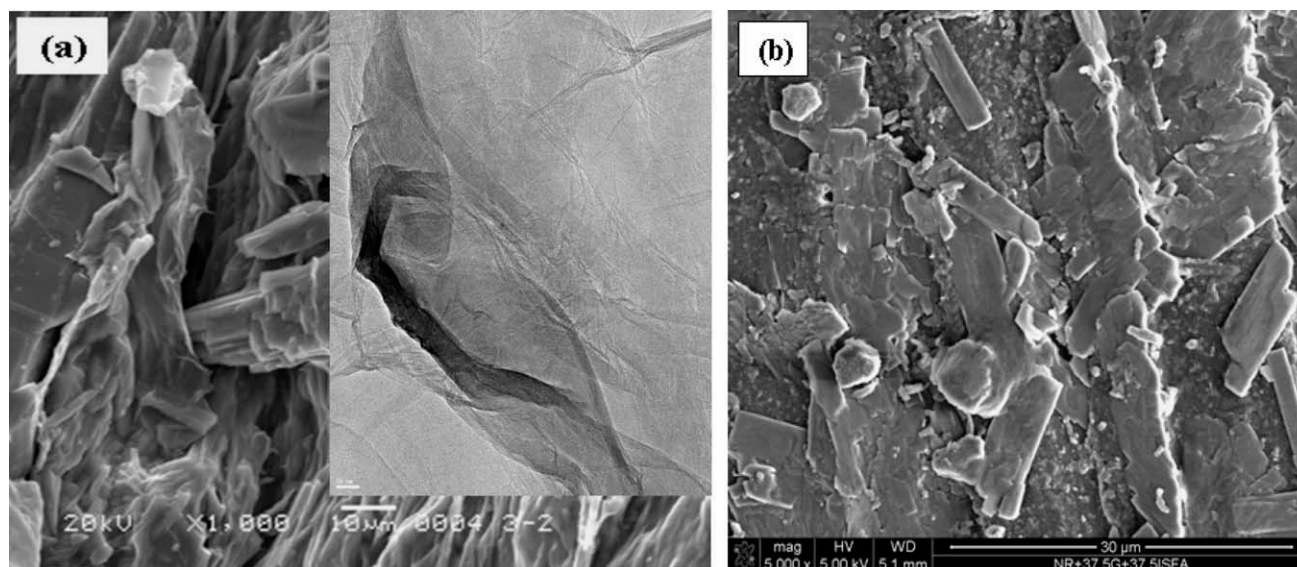
Figure 1. (a) SEM micrograph of as-synthesized FGNs obtained by ultrasonic powdering technique and the inset is the TEM image of foliated graphite nanosheets and (b) SEM image of sample FGN40.

curves of pure epoxy and epoxy/FGN nanocomposites plotted in Figure 2. It is clear that the temperature at thermal decomposition of the composites shifts to higher temperature value with the increase of FGNs content compared with pristine epoxy. The slight increase in the decomposition temperature with the increase of foliated graphite content in the composite due to the well interfacial adhesion among FGNs and epoxy resin matrix.[1,2] Pristine epoxy shows one visible weight loss at 199°C, assigned to the liberation of volatile hydrocarbons.[2,3] Nanocomposites with higher contents of the foliated graphite present complex thermo decomposition processes, and the several weight losses observed could be related to the degradation of the epoxy matrix. This improved thermal stability of epoxy/FGN nanocomposites is believed to originate from the fact that FGN, which was dispersed homogeneously in the epoxy matrix, serve as the mass transfer barriers against the volatile pyrolyzed products in the epoxy matrix, eventually retarding thermal degradation of the nanocomposites.[13,14] This supports the intermolecular interactions between epoxy matrix and foliated graphite as according to the SEM analysis.

Static Conductivity, Mobility Carriers, and TEP
Figure 3 gives the variation of electrical conductivity (σ), charge carrier's mobility (μ), number of charge carriers (N), and TEP of epoxy/FGN nanocomposites reinforced with different content of FGN nanosheets. Epoxy resin is an excellent insulating material, with electrical resistivity of about $10^{14} \Omega$ cm, whereas FGNs have similar electrical characteristics to metallic and/or semimetallic materials.[1,2] In Figure 3, the electrical conductivity increases much with increasing volume fraction of FGN nanoparticles into nanocomposites. The electrical conductivity increase is attributed to the improved FGNs dispersion within epoxy resin matrix and high aspect ratio of FGNs.[5,7] Furthermore, the enhancement of the electrical conductivities of nanocomposites is attributed to charge transfer among filler and matrix. It is established that FGNs possess high aspect ratios and

large surface area and thus gain great advantages over spherical or elliptical fillers in forming conducting networks in polymer matrices. The effect of FGNs on mobility carriers (μ) and number of charge carriers (N) of nanocomposites are depicted in Figure 3. It is seen that both μ and N increase with increasing foliated graphite content into nanocomposites. This is attributed to the increase of the network contacts into composites with inclusion FGNs as confirmed by the SEM image in Figure 1(b). In addition, the increase of the μ and N are due to the fact that the good dispersion of FGN in epoxy matrix and good interface
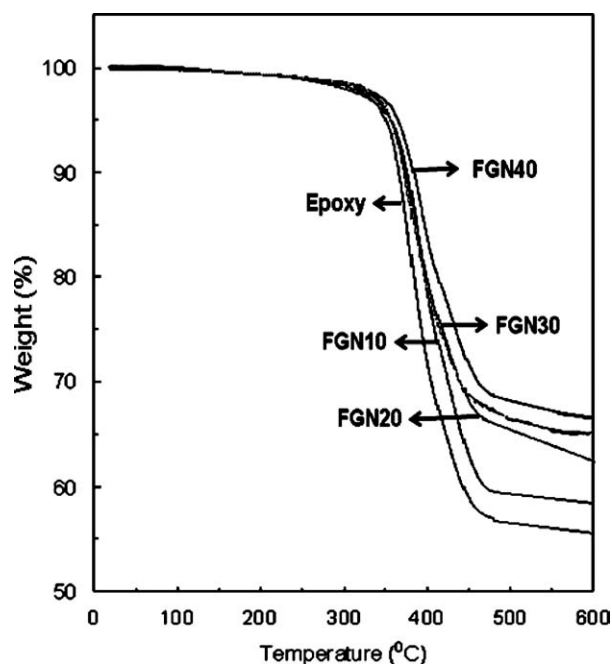

Figure 2. Thermogravimetric analysis (TGA) curves of pure epoxy and epoxy/FGN nanocomposites.
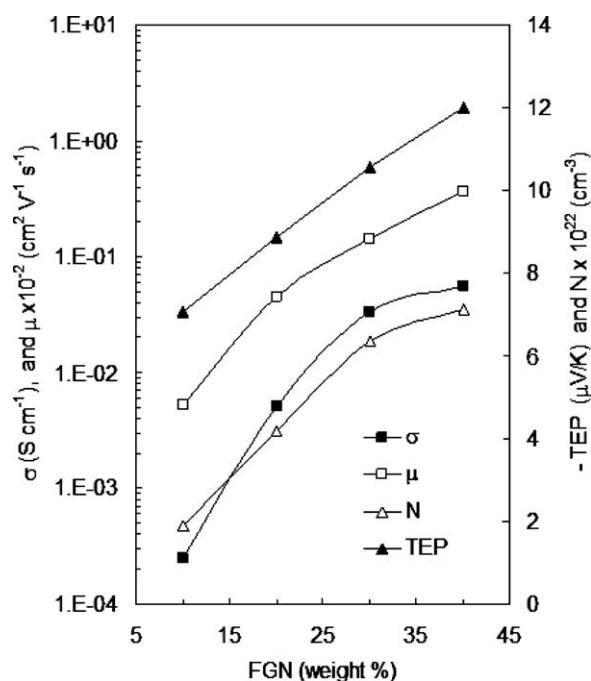
Figure 3. Variation of electrical conductivity (σ), charge carriers' mobility (μ), number of charge carriers (N), and TEP of composites.

interaction among filler and matrix. This reflects that the FGNs acts as a charge carrier reservoir in the entire epoxy resin matrix.[2,3]

The TEP of epoxy/FGN as a function of foliated graphite content is depicted in Figure 3. It appears that the epoxy/FGN nanocomposites have a negative value of the TEP. This reflects that electron conduction dominates the origin of the Seebeck effect in epoxy/FGN composites.[4,15] It considers that the FGNs provide free electrons which would make the TEP more negative. It is interesting to mention that, the high negative value of TEP is due to the carriers scattering at the interface among epoxy matrix and FGN.[2]

Electromagnetic Wave Shielding Effectiveness of Nanocomposites

It is well known that EMI effectiveness against electromagnetic interference is composed by sum of three parameters namely reflection loss (R) (i.e., the initial reflection loss from both surfaces of the shield), penetration loss and/or absorption loss (A) (i.e., the absorption or penetration loss within the barrier itself), and internal reflection loss (M) at the exiting interface (i.e., loss of energy by multireflection) at exiting interfaces of the incident electromagnetic waves in the test material. These three losses are interrelated by the following equation:[16,17]

$$EMI = R + A + M \tag{1}$$

where EMI is the shielding effectiveness and/or insertion loss, which represents the reduction (dB) of the power level of an electromagnetic field at a point in space after a conductive material is inserted between that point and the source. R is the sum of the initial reflection losses (dB) from both surfaces of the shield exclusive of additional reflection losses, A is the absorption loss (dB) within the barrier itself, and M is the mul-

tiple reflection loss at exiting interface (dB) and is neglected for conducting polymer composites.[1,2]

However, for plane wave radiation, the EMI can be calculated using the following equation:[16]

$$EMI = 108 + \log\left(\frac{\sigma}{\mu_r f}\right) + 1.33h(\sigma\mu_r f)^{\frac{1}{2}} \tag{2}$$

where μ_r is the magnetic permeability of the sample, f is the frequency of radiation, and h is the thickness of the tested sample.

The electromagnetic wave shielding effectiveness (EMI) of epoxy/FGN nanocomposites versus FGNs content at fixed frequency 5 GHz is depicted in Figure 4. Interestingly, EMI of nanocomposites increases with increasing foliated graphite content into composites. The reason is that increasing FGNs content can obtain the higher electric properties (i.e., conductivity and dielectric constant), which facilitates improvement of dielectric loss for microwave shielding materials. That is to say, there is a close dependence between EMI and conductivity for the composites made of conductive fillers and epoxy matrix. The EMI strongly depends on the electrical conductivity of the composite. Composites with higher electrical conductivity have higher electromagnetic shielding effectiveness.

The electric field of a plane wave penetrating a conductor drops exponentially with increasing depth into the conductor. The depth at which the field drops to $1/e$ of the incident value is called the skin depth (δ), which is given by:[17–19]

$$\delta = \frac{1}{\sqrt{\pi\sigma f\mu_r}} \tag{3}$$

Figure 4 depicts the variation of skin depth with FGNs content into composites. It is seen that the skin depth decreases with
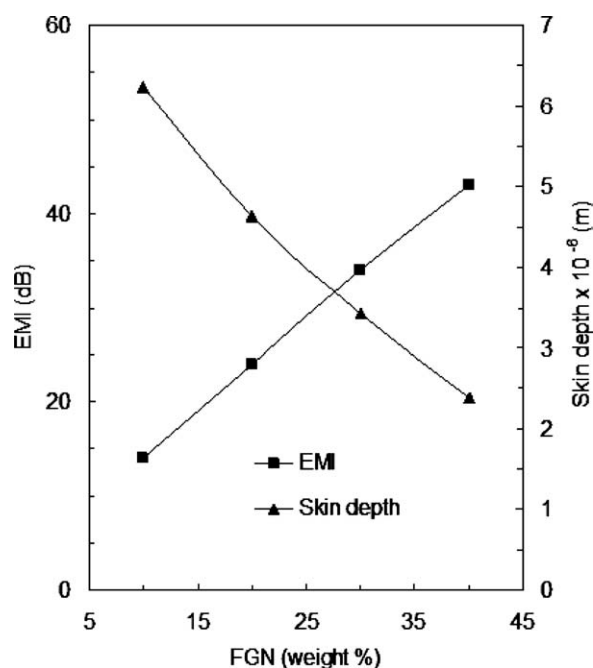

Figure 4. The electromagnetic wave shielding (EMI) and skin depth of epoxy/foliated graphite nanocomposites versus graphite nanosheets content.
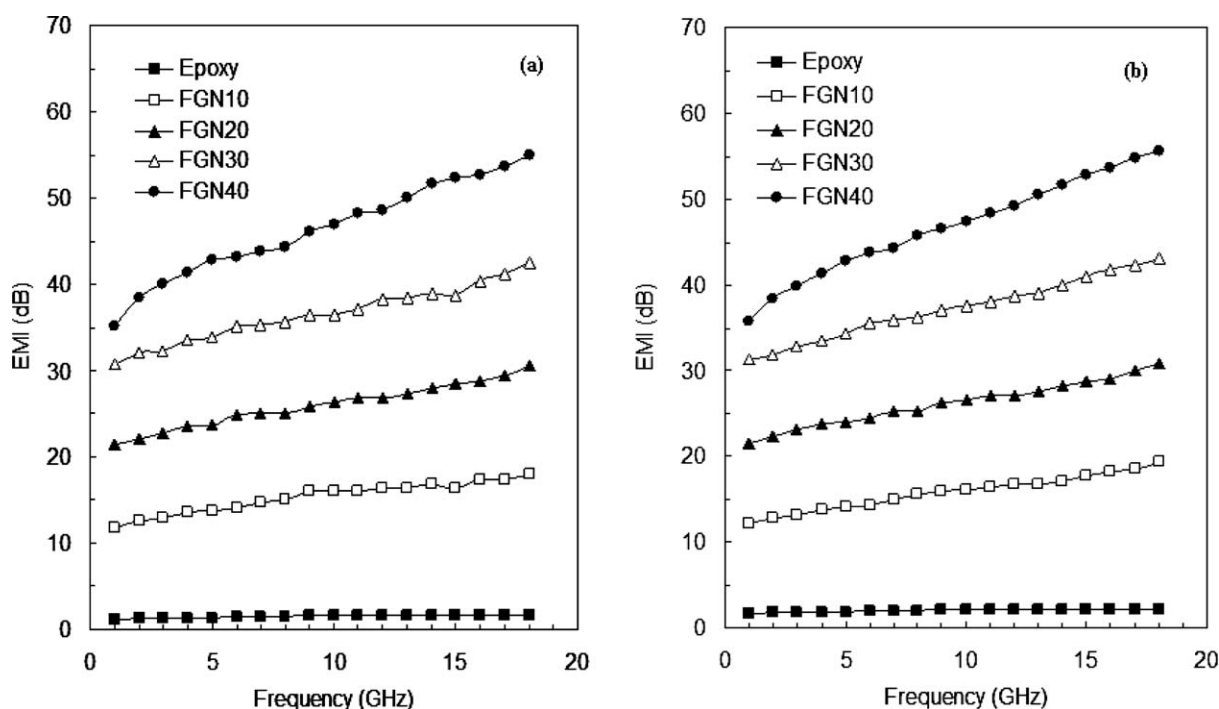
Figure 5. The measured and calculated values of EMI as a function of frequency of epoxy/foliated graphite nanocomposites (a) measured values of EMI of composites and (b) calculated values of EMI of composites.

increasing foliated graphite content (i.e., increases conductivity) into composites. This indicates that an oscillating electromagnetic signal of increasing frequency transmitted along an epoxy/foliated graphite composite.[16,17]

The measured and calculated values of EMI as a function of frequency in the range of 1–18 GHz of epoxy/FGN nanopcomposites are depicted in Figure 5(a,b), respectively. Because of their inherent insulating characteristics, the epoxy resins do not resist the penetration of electromagnetic energy.[1,9] It is obvious that the EMI increases with increasing frequency. The EMI of the 40 wt % foliated graphite loaded in epoxy matrix was 35–55 dB over the frequency range of 1–18 GHz. Because of the network linked due to distribution and interface adhesion of the entire FGNs, the epoxy matrix behaves like a conducting filament, which intercepted electromagnetic energy.[19] The above fact, the network linked into FGNs reinforced epoxy nanocomposites was observed in scanning electron micrographs Figure 1(b). Furthermore, interfacial multipoles formed between epoxy matrix and FGN will contribute to the high electromagnetic wave shielding effectiveness of composites.

In comparison between Figure 5(a,b), it is clear that a good agreement between the measured and theoretical calculated values of the EMI of epoxy/FGN nanocomposites. These results indicate that epoxy/FGN composite can be used for electromagnetic wave shielding applications at microwave frequency up to 18 GHz, especially for radar absorbing devices.

Microwave Absorbing Properties of Nanocomposites

To understand the intrinsic reasons for microwave absorbing of the nanocomposites, we measured the dielectric properties such as real and imaginary permittivity and dielectric loss of the ep-

oxy/FGN composites in the frequency range of 1–18 GHz. Real and imaginary permittivity of epoxy/FGN are plotted in Figures 6 and 7, respectively. As the weight percentage of FGN content in the composites increased, both the real and imaginary parts of the permittivity became larger. These results originate from
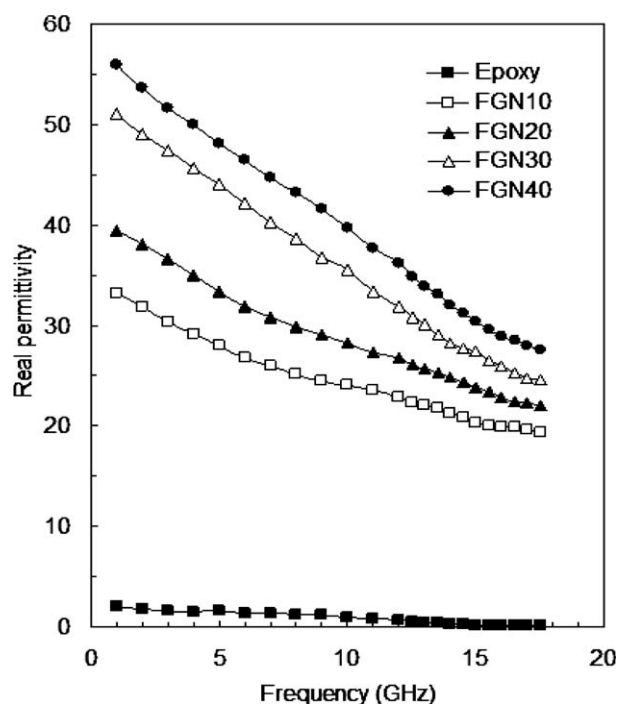

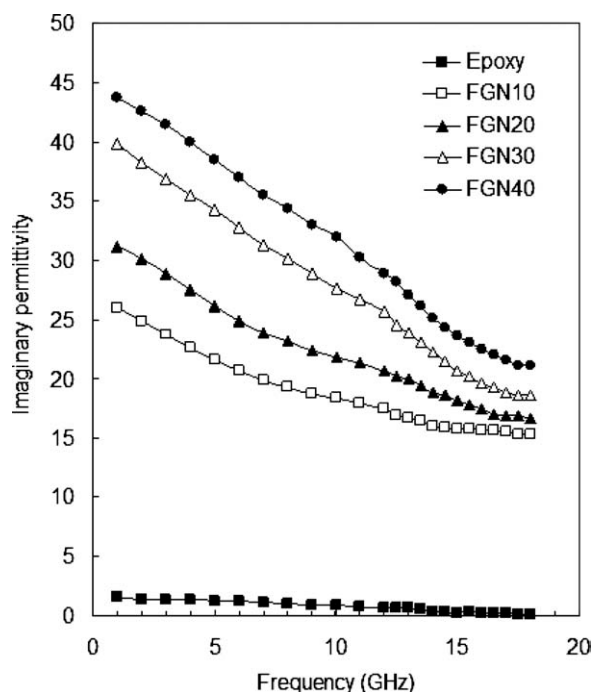**Figure 6.** Real permittivity versus frequency of epoxy/FGN composites.

Figure 7. Imaginary permittivity against frequency of epoxy/FGN composites.

the sudden buildup of the foliated graphite conductive network with increased foliated graphite contents as confirmed above.[7,18]

The variations of real permittivity with frequency at room temperature for pure epoxy and epoxy/FGN composites at different contents of FGNs are depicted in Figure 7. As can be seen from these figures, the real permittivity decreases as the frequency increases. The continuous decrease of real permittivity with increasing frequency up to 13 GHz is ascribed to the tendency of dipoles in macromolecules to orient themselves in the orientation of the applied field.[15,16] The slight decrease in the dielectric constant in high frequency is explained by the fact that as the frequency is raised, the interfacial dipoles have less time to orient themselves (i.e., the dipoles will hardly be able to orient) in the direction of the alternating field.[10,11,19]

The variation of dissipation factor as a function of frequency with different content of FGNs is depicted in Figure 8. It can be seen from these figures that as more FGNs are added to the epoxy, the dissipation factor, in general, shows an increase, and the dissipation factor decreases with an increase in frequency. The dissipation factor increases with the increase in FGNs content, which is considered a consequence of interfacial polarization.[11,19,20] Furthermore, with increasing foliated graphite content into composites new conducting channels are formed and build up a space charge polarization leading to an increase of dissipation factor.[1] It suggests that the microwave absorption enhancement of composite results mainly from dielectric loss.[21,15]

According to the transmission line theory, the reflection loss (R) relates to the frequency, thickness, complex relative permeability, and permittivity and can be obtained from the equation as follows:[18]

$$R = 20 \log 10 \left| \frac{z-1}{z+1} \right|, \qquad (4)$$

where z is the normalized impedance of a microwave absorber is given by:[19,20]

$$z = \sqrt{\frac{\mu_r}{\varepsilon_r}} \tanh\left(j\frac{2\pi}{c} f\, h\sqrt{\mu_r \varepsilon_r} \right), \qquad (5)$$

where μ_r and ε_r are the relative permeability and relative permittivity of composite medium, respectively, c is the speed of light, f is the frequency of the electromagnetic wave, and h is the thickness of the absorber and is taken to be 1 mm.

The measured and calculated reflection loss of nanocomposites in the frequency range of 1–18 GHz is plotted in Figure 9(a,b). It is seen that the theoretical calculation of reflection loss agrees well with the experimental results. It is observed that the reflection loss increases with increasing foliated graphite content into composites. From Figure 9(a), it is found that pristine epoxy almost does not absorb microwave when there is no filler in it and it can be considered as weak microwave absorber.

It is clear that the position of reflection loss moves to lower frequency with increasing FGNs content. This phenomenon is ascribed to the electric field from a short distance of resonance multidipoles with increasing FGN content into composites.[20] This reflects that the reflection peak of the epoxy /FGN nanocomposites can be adjusted easily by changing the FGN content
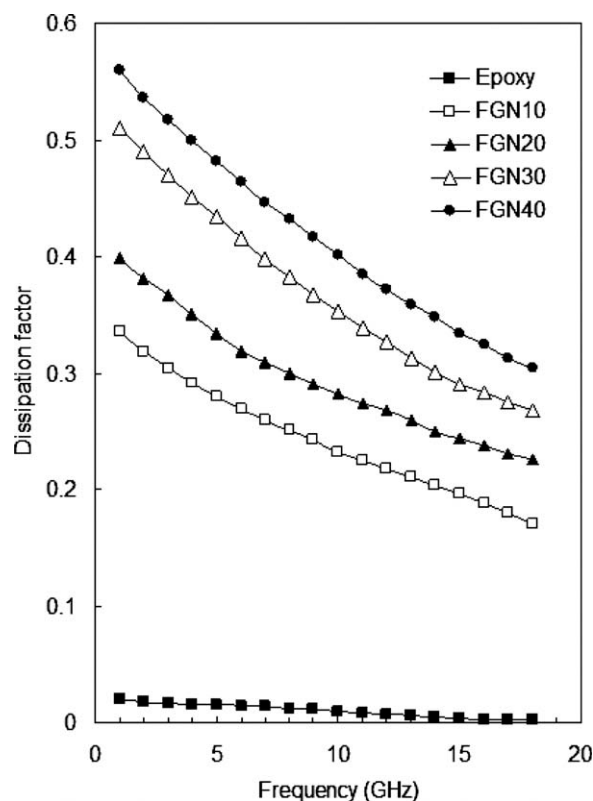

Figure 8. Dissipation factor against frequency of epoxy/FGN composites.
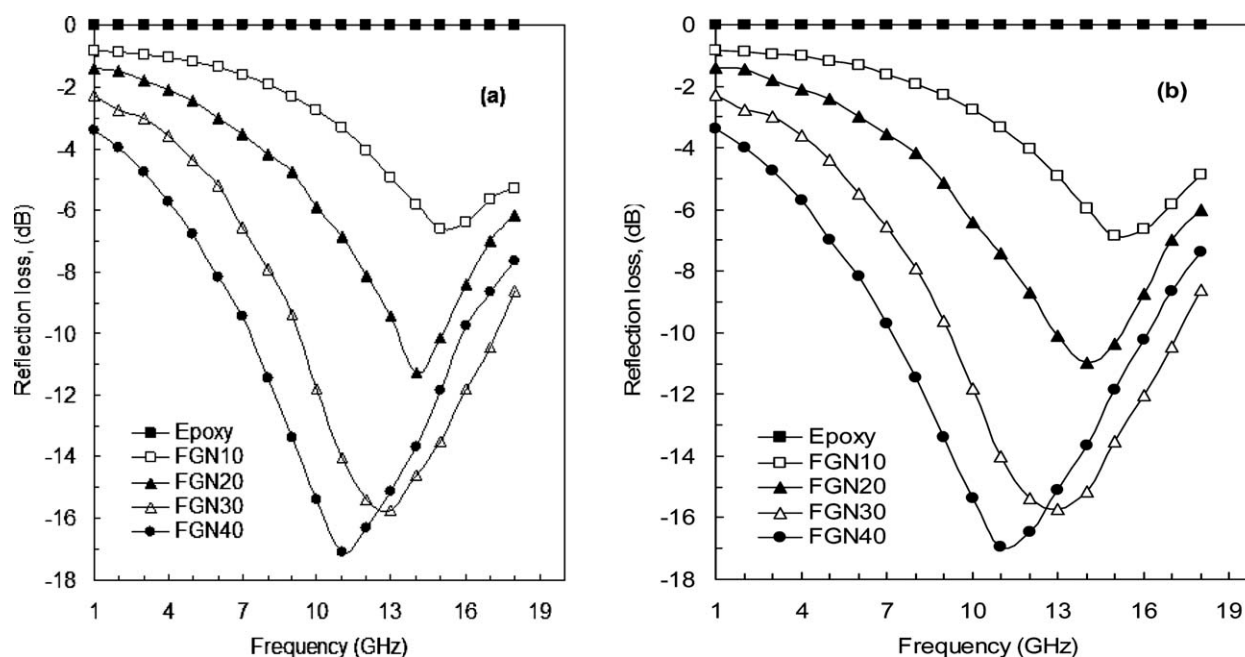
Figure 9. (a) The measured reflection loss of nanocomposites in the frequency range of 1–18 GHz and (b) the calculated reflection loss of nanocomposites in the frequency range of 1–18 GHz.

entire nanocomposites. The maximum absorption reaches 37 dB when the content of FGN 40 wt % and reduced to 22, 18 and 13 dB when the content decrease to 30, 20, and 10 wt %, respectively. This strong clue states that the as fabricated epoxy/FGN nanocomposites can be used as radar absorbing materials due to the broad frequencies absorbing properties.

CONCLUSIONS

1. The FGNs possess a special structure and are favorable to form conducting networks and good interfacial adhesion in the epoxy matrix, thus resulting in low graphite loading in the nanocomposites. The electrical conductivity, mobility carriers, and number of charge carriers of epoxy were improved by inclusion of FGNs.
2. The thermal stability was highly enhanced with increasing foliated graphite content into composites.
3. The shielding effectiveness against EMI of the nanocomposites depends on the FGN content. Nanocomposites with higher FGN content have greater EMI shielding effectiveness. The epoxy/FGN nanocomposites with 40 wt % FGN loading were technically useful materials (EMI = 35–55 dB) in the frequency of 1–18 GHz.
4. Real and imaginary permittivity and dissipation factor of nanocomposites increased with an increase in volume content of FGNs, which has been attributed to interfacial polarization. Dielectric properties of epoxy/FGN composites decreased with an increase in frequency.
5. The epoxy/FGN nanocomposites exhibit good microwave absorption performances in the frequency range of 1–18 GHz. Theoretical calculation of reflection loss agrees well with the experimental results and this nanocomposite may

become a good microwave absorbing material at microwave frequency.

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