Radar Absorbing Nanocomposites Based MultiLayered Graphene Platelets/Epoxy

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Abstract
Graphene nanostructures were synthesized by Hummer method. 1, 3, 5 and 7 wt% of graphene nanostructures were suspended in certain amount of acetone on a mechanical stirrer and stirred then added to epoxy resin. After 4 hours, solution and Graphene platelets (GPs) were prepared. Nanostructures were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transform infrared (FT-IR) spectroscopy. The electromagnetic interference shielding was studied by reflection loss (RL). According to the results, the multilayered graphene 3% wt of has a completely smooth surface and its absorption average and maximum are reported as -13.5 dB and -30.3 dB.

1. Introduction
Polymer composites with carbon nanofillers have many potential applications, including thermal management, electronics, green energies, and transportation [1] also their morphology as well as their dispersion state within the polymeric matrix [2-5]. Radar absorbing materials (RAM) have gained a fundamental role in civil and military applications requiring the control of the electromagnetic (EM) environment. Such materials are characterized by high shielding properties against EM fields at radio frequencies (RF), but at the same time by a low reflection coefficient over a defined frequency range [6]. Nanoscale materials based on single layered 2-D graphene sheets have attracted much attention recently due to many unusual properties predicted [7]. That These novel graphene materials may offer another intriguing nanoscale filler material with low density for various composite applications [8]. The wide use electromagnetic interference (EMI) shielding of radio frequency radiation continues to be a serious concern for modern society. Electrical conductivity of graphene can be up to 6000 S cm⁻¹ [9].

The scope of this paper is to demonstrate the feasibility of new bilayer absorbing screens, entirely
made with nanocomposites filled with different types of carbon micro and nanostructures, like graphene the new multilayer graphene platelets (GP) synthesized in our Nanotechnology Lab. It is demonstrated that the proper choice of the filler and of the polymeric matrix allows one to design and realize nanocomposites with the desired properties.

2. Experimental procedure

2.1 Materials

All the reagents for the synthesis of graphene nanosheets such as nitric acid, sulfuric acid, potassium permanganate, deionized water, hydrogen peroxide, hydrochloric acid and hydrazine. All the reagent for prepared of GP such as acetone, LY5052 resin epoxy and hardner and GR synthetic.

2.2 Methods

FT-IR spectra were recorded on Magna-IR, spectrometer 550 Nicolet in KBr pellets in the range of 400–4000 cm\(^{-1}\). Powder X-ray diffraction (XRD) patterns were collected from a diffractometer of Philips Company with X'pertpro monochromatized Cu K\(\alpha\) radiation (\(\lambda = 1.54\ \text{Å}\)). Microscopic morphology of products was visualized by SEM (Cam Scan, MV2300). Radar absorption were measured using HP network analyzer 8720C, X-Band Horn in the frequency range of 8-12 GHz.

2.3 Synthesis of graphene nanosheet

First, 5 g of graphite was added into a beaker containing 500 ml. Then, 30 ml of HNO3 and 80 ml of H2SO4 (98%) were stirred together in an ice bath. Next, 10 g of KMnO4 was slowly added over 30 min, and the mixture was stirred for about 30 min. Then the ice bath was removed, 175 mL of deionized (DI) water was added and the solution was stirred for 30 min at 35 °C, while the colour of the solution changed from black to yellowish brown. Then, 30 ml of H2O2 (30%) at 40 °C was added then stirred the mixture for 30 min. The resulting mixture of HCl 5% and deionized water rinse several times and was centrifuged. Filter paper on deposits was filtered and dried at 60 °C under vacuum about 1 h. Then hydrazine used for reduction, and solution was washed, centrifuged and dried. Finally the graphene nanostructures were prepared.

2.4 Fabrication of GP/ Epoxy nanocomposites

A calculated amount (1, 3, 5 and 7) wt% of graphene nanosheets was suspended in certain of acetone on a mechanical stirrer at 500 rpm for 20 minutes. Then, with respect to graphene, a certain amount of resin added to the solution and was stirred for 2 hours. Stirring was continued for 1 hour at a speed of over 1100 rpm to yield a uniform suspension. Agitation was continued to make a stiff mixture and remove acetone in solution. Then, the resulting mixture stayed in the dark environment for 24 hours and was vacuumed in an oven at 50 °C with a pressure of 300 mbar for 45 minutes to evaporate acetone completely. After that, considered ratio of hardener was added and stirred for 20 minutes. The mixture with the certain thickness of 3 mm were cast. After smoothing the surface of the mold at 50 °C for 1 hour, and at 110 °C for 2 hours, it was placed to cover the desired form.

3. Results and discussion

Fig. 1 Contains FT-IR spectra of graphite and graphene. For the graphite spectrum, besides the O-H stretching of water and the vibration from carbon bonding, no obvious vibration peak is observed. For the graphene include two absorption bands at 2348 cm\(^{-1}\) and 3304-3500 cm\(^{-1}\) can be distinguished, implying the presence of O-H groups.
Fig. 1. FT-IR spectra of graphite and graphene.

Fig. 2 contains XRD patterns of S2 and S6 samples. A sharp peak at 2θ of 19.68° was observed for GP.

Fig. 3 shows the SEM images of graphene nanosheets, S2, S5 and S6 samples. As shown in Fig. (3-a) graphene nanosheets are individually exfoliated. According to Fig. (3-b) SEM images surface of sample S2 indicates that the surface is almost flat but uneven. Fig. (3-c) shows the SEM images surface of sample S5 indicates that the unevenness is less than smooth surface. Fig. (3-d) shows the SEM images surface of sample S6 indicates that the surface smoother and smoother than its predecessor.

Fig. 2. XRD patterns of S2 and S6 samples.

Fig. 3. SEM images of graphene nanosheets, S2, S5 and S6 samples.

**Nanocomposite RL Characterization**

Since measurements of the complex effective permittivity were performed according to the ASTM 5568-01 standard using the waveguide method, samples were directly poured into the flanges and then machined to the desired thickness upon complete cross-linking.
Table 1. Configurations and performances of single layer and multi layer absorbing samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Layer no.</th>
<th>Material</th>
<th>Thickness (mm)</th>
<th>Max. Abs. (dB)</th>
<th>Average Abs. (dB)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>1</td>
<td>GR(1%) + epoxy</td>
<td>3</td>
<td>-8.3</td>
<td>-3.9</td>
</tr>
<tr>
<td>S2</td>
<td>1</td>
<td>GR(3%) + epoxy</td>
<td>3</td>
<td>-14.4</td>
<td>-7.3</td>
</tr>
<tr>
<td>S3</td>
<td>1</td>
<td>GR(3%) + epoxy</td>
<td>3</td>
<td>-11.1</td>
<td>-5</td>
</tr>
<tr>
<td>S4</td>
<td>1</td>
<td>GR(3%) + epoxy</td>
<td>3</td>
<td>-6.6</td>
<td>-2.8</td>
</tr>
<tr>
<td>S5</td>
<td>1 2</td>
<td>GR(3%) + epoxy</td>
<td>1 2</td>
<td>-26.2</td>
<td>-11.4</td>
</tr>
<tr>
<td>S6</td>
<td>1 2 3</td>
<td>GR(3%) + epoxy</td>
<td>1 2 3</td>
<td>-30.3</td>
<td>-13.5</td>
</tr>
</tbody>
</table>

Effective complex permittivity of the composites was extracted from the measurement of the scattering parameters according to the aforementioned standard, using sets of waveguides, respectively for the X bands (8-12 GHz) [10].

The reflection loss (RL) of single absorb layer was calculated as follows:

\[
RL \ (dB) = 20 \log \left( \frac{Z_{in} - 1}{Z_{in} + 1} \right)
\]  \hspace{1cm} (1)

while the normalized input impedance \( Z_{in} \) was calculated by

\[
Z_{in} = \frac{\sqrt{\mu_r \varepsilon_r} \tanh \left( \frac{2 \pi f d}{c} \sqrt{\mu_r \varepsilon_r} \right)}{\sqrt{\varepsilon_r}}
\]  \hspace{1cm} (2)

where \( f \) is the microwave frequency, \( d \) is the thickness of the absorb layer, \( c \) is the velocity of electromagnetic wave in vacuum, and \( \varepsilon_r \) and \( \mu_r \) are the complex relative permittivity and permeability, respectively. RL is expected to be as low as possible at a given sample thickness [11]. The characteristics of the designed samples are reported in Table 1.

![Fig. 4. Radar absorption spectra of S2, S5 and S6 samples.](image)

4. Conclusion

Graphene nanostructures were synthesized via a Hummer method. Then graphene nanostructures were added to epoxy resin. The influence of graphene nanostructures on the reflection loss of PS matrix was studied. The results show that the graphene nanostructure can enhance the absorption of the PS matrix. The SEM images of S6 sample indicates that the surface is smoother than its predecessor. The RL curves shows that two types of single layer and multilayered GP/epoxy nanocomposite production and its performance was to absorption radar waves. The GP/epoxy composites (S6 sample) had high permittivity and
very low microwave reflection loss, with great potential to be used in the field of protecting people from microwave radiation.

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References