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Electromagnetic shielding effectiveness of polycarbonate/graphene nanocomposite foams processed in 2-steps with supercritical carbon dioxide

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ABSTRACT

The electromagnetic interference (EMI) shielding properties of polycarbonate/graphene composites foamed with supercritical carbon dioxide were investigated as a function of cellular morphology and graphene particle dispersion. The 2-step foaming method used was found to improve graphene dispersion and led to a different cellular structure compared to traditional 1-step foaming. Reflection was found to be the dominant EMI shielding mechanism and EMI shielding effectiveness was improved with large cell morphology that promoted isotropic/random orientation of graphene particles. A maximum EMI specific shielding effectiveness of ~78 dB cm^3/g was achieved in foams, which was more than 70 times higher than that of the unfoamed polymer (1.1 dB cm^3/g). The study shows that by controlling foaming process conditions and nanoparticle characteristics, it is possible to improve multiple properties while achieving lightweight materials suitable for various applications.

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1. Introduction

The preparation of electromagnetic interference (EMI) shielding materials has obtained an increased attention in the academic and industrial fields compared to conventional metal-based EMI materials [1]. Materials with this property are needed for protecting electronics from unwanted radiated signals which can cause unacceptable system performance. The malfunction of electronics can be hazardous, as electronics can be associated with strategic systems such as aircrafts, nuclear reactors, transformers, control systems, communication systems, among others [2]. Nowadays the main goal is to prepare lightweight materials with electromagnetic protection properties [3]. Therefore weight reduction increases the importance for foaming polymers for these types of applications. In the current study we present the effect of the cellular structure promoted by foaming on graphene nanoplatelets orientation and their role on electromagnetic interference shielding behavior.

2. Experimental

Bisphenol A polycarbonate (melt flow index of 17.5 dg/min) and graphene nanoplatelets (GnP) (with average thickness of 6–8 nm, average platelet diameter of 15 μm, and bulk density of 2.2 g/cm^3) were used. Polycarbonate/graphene (PC/GnP) composite samples were prepared by melt compounding using an internal mixing with a graphene concentration of 0.5% (by weight). The pelletized composites were compression-molded in a hot-plate press at 220 °C at a constant pressure of 4.5 MPa [4]. Foaming was done with the use of supercritical carbon dioxide (CO_2) via a 2-step method as follows: First the samples were saturated in a high-pressure vessel at 80 °C and 14.0 MPa for 210 min, then they were cooled to room temperature in approximately one hour, followed by slow depressurization. Samples were then removed from the vessel and left to stabilize at room temperature for 120 min. Finally, the samples were heated in a compression press to 165 °C for 40, 60, 80 or 100 s at a constant pressure of 6.0 MPa, after which the applied pressure was quickly removed leading to free

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expansion of the sample [5].

Small and wide angle X-ray scattering experiments were carried out at room temperature on a Nanostar-U instrument (sample distance of 105 cm). Composite morphologies were previously characterized using a JEOL JSM-5610 scanning electron microscope (15 kV, working distance of 30 mm) [4]. The average cell sizes (ϕ) along the disc thickness (vertical direction, ϕ_{VD}) and disc width (radial direction, ϕ_{WD}) were measured using the intercept counting method [6]. The transmission electron microscopy (TEM) images were acquired on a JEOL JEM-2011 LaB6 TEM operating at 200 kV. The electromagnetic interference shielding effectiveness (EMI-SE) measurements were carried out in the X-band frequency range (8.0–12.4 GHz) using an Anritsu 37397C vector network analyzer. The electromagnetic interference shielding effectiveness (EMI-SE) measurements were carried out in the X band frequency range (8.0–12.4 GHz) using an Anritsu 37397C vector network analyzer (VNA), which consisted of two test fixture ports connected to two WR 90 coaxial waveguides and a sample holder that was placed between the two waveguides. Samples were cut to fit into waveguide sample holder (22.9×10.2 mm^2) with thicknesses of 2 mm. A two port VNA calibration was performed before data collection. Scattering parameters S_{11} (forward reflection coefficient) and S_{21} (forward transmission coefficient) were collected to calculate the electromagnetic interference shielding effectiveness.

3. Results and discussion

The change of particle morphology after foaming was investigated via TEM. Unfoamed composite (PC–GnP, Fig. 1a) presented thicker platelets when compared to foamed samples (Fig. 1b) suggesting that there is a partial exfoliation of graphene platelets after foaming.

The cellular morphological features of foamed composites, which were discussed in detail in our previous publication [7], were found to depend on the presence of graphene, amount of dissolved supercritical CO_2, and CO_2 saturation/foaming conditions (see Table 1 for a summary).

SAXS experiments showed the presence of structural anisotropy as a function of heating time (Fig. 2a–e). Unfilled PC foamed under the same conditions also showed a similar dependence of anisotropy on heating time suggesting that chain stretching due to cell growth might have an influence on he anisotropy. However, the anisotropy is stronger in GnP-filled composites suggesting that there might also be a contribution coming from graphene platelets. Two different sets of peaks were observed in SAXS intensity vs. azimuthal angle plots (one pair of peaks at 0° and 180° and the second pair at 90° and 270°) for PC80–GnP4. The locations of these sets of peaks suggest the presence of bimodal orientation [8], which might be explained by the stretching of polymer chains and re-alignment of graphene platelets due to foaming [9] particularly if the cell growth or sample expansion is not isotropic, which is the case in the current study. TEM experiments showed different graphene platelet orientations between samples heated for 100 s (Fig. 2f) and 40 s (Fig. 2g), which supports that graphene platelets might be orienting along the direction of greater expansion (vertical direction); the ratio of vertical expansion (ER_{VD}) to radial expansion (ER_{WD}) was 1.12 for heating time of 40 s and 1.33 for heating time of 100 s. It is, therefore, possible that unequal sample expansions led to both polymer chain stretching and graphene platelet orientation along the vertical direction during foaming (regardless the isotropic-like cellular structure displayed). It is important to note that because of the plasticizing effect of CO_2, the glass transition temperature of PC would be lowered leading to increased mobility [10,11], which would enable chain stretching and orientation of the graphene platelets. However, what we probably observe in azimuthal SAXS intensity profiles is the contribution coming from structures that have a component along the radial direction (given that the X-ray source was directed along the vertical direction, see inset). Therefore, any anisotropy observed in 2D SAXS patterns observed in Fig. 2e should be attributed to graphene orientation due to unequal expansion of the sample.

The EMI shielding effectiveness (EMI-SE) of solid and foamed PC/GnP composites as a function of frequency are presented in Fig. 3a. In general, foamed samples showed up to 10 times enhancement in EMI-SE compared to unfoamed composite (PC–GnP). It has been suggested that specific EMI-SE (EMI-SE normalized by density) might be more appropriate when comparing

Table 1
Foaming process parameters and structural features of polycarbonate–graphene composite foams.

Label	t_{heat} (s)	ρ (g/cm^3)	ρ_{rel}	N_f (cell/cm^3)	ϕ_{VD} (μm)	ϕ_{WD} (μm)	AR
PC80–GnP1	40	0.18	0.14	6.07×10^8	26	21	1.3
PC80–GnP2	60	0.23	0.19	6.19×10^7	46	34	1.4
PC80–GnP3	80	0.29	0.24	7.88×10^8	20	14	1.4
PC80–GnP4	100	0.33	0.28	1.56×10^9	11	11	1.1

t_{heat}: Heating time; ρ: density; ρ_{rel}: relative density (normalized by the unfoamed composite density of 1.14 g/cm^3); N_f: cell density; ϕ_{VD}: Average cell size along the vertical direction (sample thickness); ϕ_{WD}: Average cell size along the sample width (radial direction); AR: aspect ratio ($=\phi_{VD}/\phi_{WD}$).

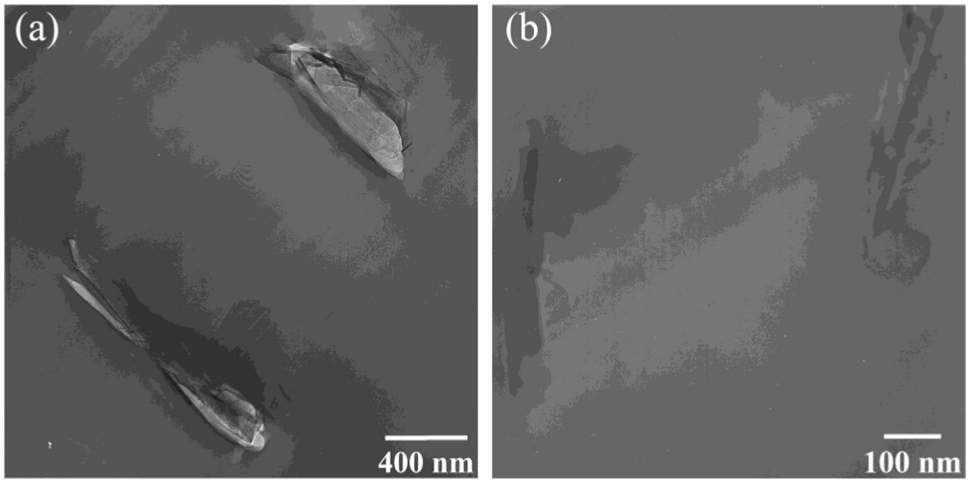

Fig. 1. TEM micrographs of (a) unfoamed (PC–GnP) and (b) foamed (PC80–GnP1) composites.

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