

Elevated conductivity and electromagnetic interference shielding effectiveness of PVDF/PETG/carbon fiber composites through incorporating carbon black

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Abstract Along with the electronic products entering people's life, the electromagnetic radiation is becoming a serious problem threatening human health. Consequently, the aim of this paper is fabricating the electromagnetic shielding materials containing carbon fiber (CF), carbon black (CB), poly(vinylidene fluoride) (PVDF) and poly(ethylene terephthalate-co-1,4-cyclohexylenedimethylene terephthalate) (PETG). By adjusting CB content, the composite with high electromagnetic interference shielding effectiveness (EMI SE) was achieved. Additionally, the effects of CB on the rheological, dynamic mechanical properties, and electrical resistivity of PVDF/PETG/CF composites were investigated in detail. That CB formed the conductive networks in the PVDF/PETG/CF/CB composite at 5 % of CB and above led to the reduction in electrical resistivity and the augment of the modulus as well as the glass transition temperature of PETG. From the electrical resistivity and storage modulus points of view, the short CF exhibited the better synergistic effect with CB than the long CF did. But the lowest electrical resistivity (0.39 Ω .cm) occurred in the long CF based composites containing 15 % of CB, and its EMI SE is determined to above 30 dB over a frequency of 0.1 to 1500 MHz.

Keywords PVDF · PETG · Carbon fiber · Carbon black · EMI SE

Introduction

The electromagnetic radiation, emitted by the electrical and electronic equipments (cellular phone, computer, microwave oven etc.), has been regarded as the one of the main pollution source. The electromagnetic radiation not only leads to the operational malfunction of electric devices and the loss of important information, but also endangers human health and induces various kinds of illness [1–3]. Hence, many researchers tried their best to develop new materials to shield the electromagnetic radiation to protect people from being harmed.

Generally, the electromagnetic shielding materials should own low electrical resistivity or high magnetic permeability. For now, the two types of materials are used to fabricate the electromagnetic shielding materials, the metal/metal-based materials and polymer-based materials [3]. The metal materials exhibit the strong ability to reflect the electromagnetic wave owing to their low electrical resistivity, whereas the shortcoming of easy corrosion largely degrades the EMI SE [4]. The polymer-based materials are considered to be promising EMI materials due to the resistance to corrosion, low cost, light weight, easy processing etc. However, polymer is nonconductive by nature, and this fault requires the polymer to be incorporated a large amount of conductive fillers, such as carbon black, carbon fiber, graphite, carbon nanotubes and graphene [5]. Thus the materials can acquire the ideal conductivity, EMI SE as well as the higher elastic modulus. Song et al. have prepared the cyclic butylene terephthalate/graphite/carbon black composite and its EMI SE reached more than 50 dB in the frequency of 30~3000 MHz [6].

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The co-continuous phase polymer blends can make the conductive fillers form the conductive networks in the matrix by selective location of fillers in one component or the interface in the blends [7, 8]. The co-continuous immiscible polymer blends have a lot of merits, such as decreasing the conductive fillers loading, reducing the product cost and improving the mechanical properties. This technology has been successfully used in proton exchange membrane fuel cells, EMI materials, antistatic materials fields and others [9–12]. Sun et al. reported that the CNTs preferentially located in the PC phase of the PC/SAN blends, and the electrical resistivity was improved largely [7]. Xu et al. showed that the carbon black preferentially located in the interface between PMMA and PP in the PMMA/PP/CB composites; and in PA6 phase in PS/PA6/CB, PP/PA6/CB, PMMA/PA6/CB and PAN/PA6/CB composites, and these co-continuous immiscible composites presented the high electric conductivity [13].

Here, in order to obtain the co-continuous blends with good conductivity, the two kinds of polymers are used: PVDF and PETG. Carbon fiber and carbon black are the conductive fillers. The melt compounding method is adopted to fabricate the PVDF/PETG/CF/CB composites. The objective of this paper is to produce the electromagnetic shielding materials and study the effects of CB on the rheological, electrical resistivity and dynamic properties of PVDF/PETG/CF composites. In addition, the conductive mechanism of the co-continuous polymer blends was discussed.

Experimental

Materials and preparation

PVDF (FR907) was bought from the Shanghai 3 F New Material Co., Ltd (China); PETG (GN071) was purchased from the Eastman Chemical Company; Carbon black (Printex XE-2) with specific surface area of 1000 m²/g was the product of the Degussa-Hu's Company of Germany; Carbon fiber with the diameter of 7 μm was produced by the Nanjing Weida Composite Material Co., Ltd (China). It is noted that the two types of carbon fiber are used in our study: one is the 10 mm-long carbon fiber, and the other is 150 μm long. Before experiments, all materials were dried at 70 °C in a vacuum drying oven for 24 h.

The PVDF/PETG/CF/CB composites were prepared by melt compounding technology, where the mass ratio of PVDF to PETG is controlled to be 60:40. First, PVDF, PETG, carbon black and carbon fiber with a certain mass ratio were compounded at 230 °C for 20 min using an internal mixer (Changzhou Suyan Technology Co., Ltd, China) with a mixing speed of 60 rpm; the achieved mixtures were then pressed into the disc (12 cm in diameter and 1 mm thick) and the strip specimens (60 mm×10 mm×4 mm) using a block

press (230 °C, 10 min) with 10 MPa. For the convenient study, the long carbon fiber-based PVDF/PETG/CF/CB composites are labeled as the LCF composites and the short carbon fiber-based PVDF/PETG/CF/CB composites are marked as the SCF composites. All component contents are listed in Table 1. The CF content in composites is fixed to be 5 % since this level of loading exceeds the electrical resistivity percolation threshold.

Characterization

Rheological test

The rheological properties of the LCF and SCF composites were measured using a parallel-plate rheometer (MARS III, produced by Thermo, Germany). The storage modulus and complex viscosity of the composites were collected in the frequency range from 0.1 to 10 Hz; the temperature and shearing stress were fixed to be 200 °C and 300 Pa, respectively.

SEM observation

The morphology observation of fracture surface of PVDF/PETG/CF/CB composites was carried out using a scanning electron microscope (SEM, S4800, Hitachi Corporation) with an accelerating voltage of 10 kV. First, composites were dipped into liquid nitrogen and fractured, and then the fracture surface was coated with gold particles.

Dynamical mechanical behaviors

The dynamical mechanical properties were measured using Dynamic Mechanical Analyzer (DMA 242E), produced by the NETZSCH company, Germany. The composites were measured by the bending mode with the temperature from 30 to 150 °C. The heating rate was 2 °C min⁻¹ and the measuring frequency was fixed to be 1 Hz.

Table 1 The contents of PVDF/PETG/CF/CB composites

		PVDF (wt%)	PETG (wt%)	CF (wt%)	CB (wt%)
LCF composites	LCF 0	57	38	5	0
	LCF2	55.8	37.2	5	2
	LCF5	54	36	5	5
	LCF10	51	34	5	10
	LCF15	48	32	5	15
SCF composites	SCF0	57	39	5	0
	SCF2	55.8	37.2	5	2
	SCF5	54	36	5	5
	SCF10	51	34	5	10
	SCF15	48	32	5	15

Electrical resistivity

The electrical resistance R (Ω) of PVDF/PETG/CF/CB composites was measured using a DC low resistance tester TH2512, produced by the Southeast University of China. The electrical resistivity, ρ ($\Omega\cdot\text{cm}$), is calculated using the following relation:

$$\rho = R \times S/L \tag{1}$$

where L is the sample thickness and S (cm^2) represents the surface area of the electrodes.

Electromagnetic interference shielding effectiveness

The EMI shielding measurements were conducted at room temperature over a frequency range of 0.1 MHz to 1500 MHz using electromagnetic shielding measuring instrument (E7401A), produced by the Agilent Company, America.

Results and discussion

Rheological properties

Rheological technology is a good tool to investigate the polymer composites since it can make us not only understand the viscoelasticity performance of the materials, but also evaluate the dispersion of fillers in the matrix and the microstructure variations [7]. Different from the SEM technology, the rheological technology may be used to master the filler dispersion in the composites in a relative large scale. The storage modulus, loss modulus, and viscosity are the important parameters of rheological technology.

Figure 1a and b show the changes of storage modulus of the LCF and SCF composites as a function of CB content. Without CB, the storage modulus of the LCF and SCF composites shows a strong dependence on the frequency used. However, as CB is incorporated, such dependence gradually becomes weaker and weaker as CB content increases. For example, at 15 % of CB, the storage modulus of the LCF and SCF composites is observed to keep constant by and large in spite of frequency. Importantly, the changes of the storage modulus in the low frequency can provide us with useful information on the filler dispersion in the matrix. Generally speaking, the “platform” at the low frequency indicates the good fillers dispersion in the matrix and the formation of effective filler networks [5]. For the composites without CB and with 2 % of CB, there is no difference on the plots of the storage modulus versus frequency, and this implies that the viscoelasticity of the composites did not change. But when CB content reaches 5 %, a platform appears at the low frequency (below 1 Hz), indicative of the formation of CB networks, as can be seen in Fig. 1a and b. This case confirms that the rheological

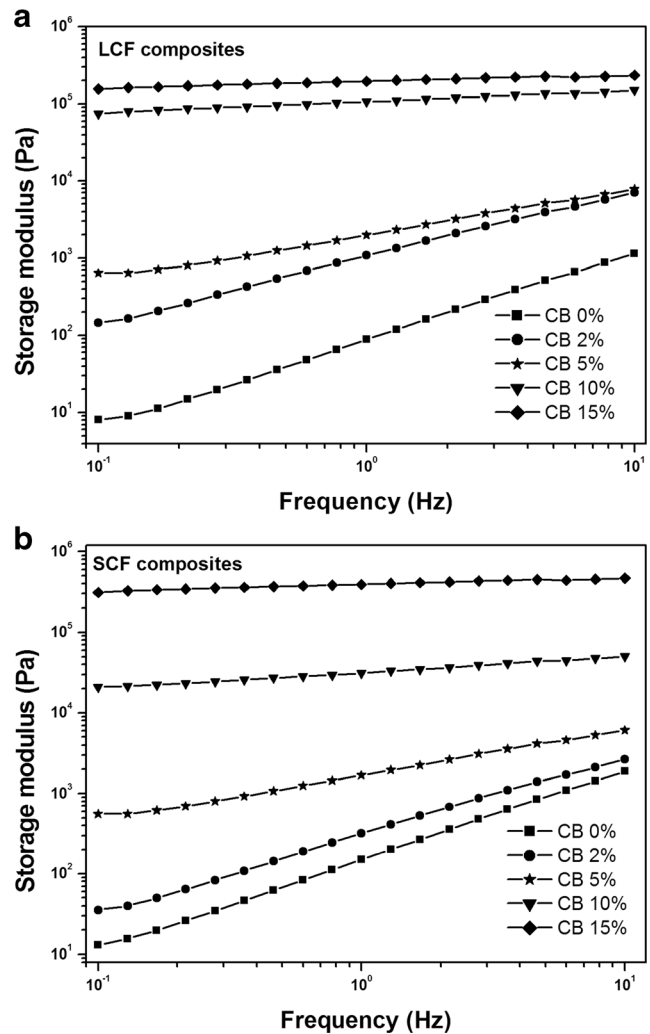


Fig. 1 The variation of the storage modulus of the LCF and SCF composites as a function of frequency. **a** the LCF composites; **b** the SCF composites

percolation threshold is around 5 % of CB for the LCF and SCF composites. These behaviors may be attributed to the establishment of CB conductive networks in the composites, which inhibits the movement of the polymer segments. Similar results have been also found in cyclic olefin copolymer/CF/CB composites [15]. Besides, such platform in the low frequency grows taller and taller with increasing CB content, and this is reflection of enhancement in the rigidity of the composites. When CB content reaches 10 % and above, the storage modulus presents the solid-like behaviors, i.e., the storage modulus is independent on the frequency, which is attributed to the fact that the relaxation of polymer chains is inhibited [14, 15].

Figure 2a and b describe the variations of the complex viscosity of the LCF and SCF composites as a function of CB content. Normally, the complex viscosity of the composites has a fierce relation with the intermolecular interaction, molecules entanglement and the networks in the matrix. Clearly, at the same frequency, the viscosity of the LCF and

SCF composites increases with increasing CB content, which means the internal structure of the composites changed. Also, it proves that the processing property of the LCF and SCF composites become poor. As shown in Fig. 2, the viscosity of the LCF and SCF composites without CB as well as viscosity of composite with 2 % of CB only shows a slight decline with increasing frequency, which is ascribed to the stable CF networks in the composites. However, when CB content attains 5 % and above, the viscosity of the composites decreases as frequency increases and this behavior is related to the formed CB networks. According the above results of the storage modulus, one can know that the CB (beyond 5 %) starts to contact with each other and fabricate the networks in the matrix. The formed CB networks are sensitive to the frequency and are easy to be destroyed by the shearing force, and the destroyed CB networks have no time to re-establish at higher frequency. Therefore, the viscosity exhibits a decreasing trend with increasing frequency. Also, this case suggests that the CF networks were more stable than the CB networks.

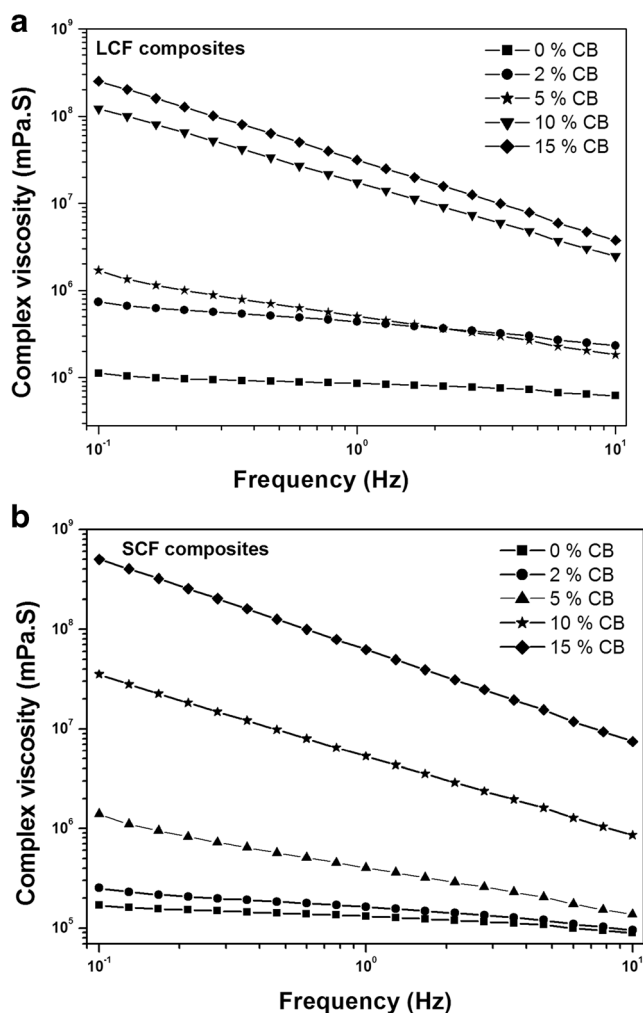


Fig. 2 The variation of the complex viscosity of the LCF and SCF composites as a function of frequency. **a** the LCF composites; **b** the SCF composites

Morphology of PVDF/PETG/CF/CB composites

Figure 3 showed the SEM photos of fracture surface of composites with 15 % of carbon black. Figure 3a and b belong to the LCF composite and SCF composite, respectively. Clearly, the carbon black could disperse uniformly in PVDF/PETG blends and no obvious aggregation appeared, as indicated in white arrow. In the LCF composite (Fig. 3a), the carbon fiber surface was very smooth, indicating the poor interfacial properties between CF and polymer, but. However, in SCF composite (Fig. 3b), the carbon fiber was coated with a thick layer of polymer, suggesting the good interfacial properties between CF and polymer. These results meant that the SCF composites presented better mechanical properties than the LCF composites did. This was uniformed with the results of rheological properties.

Dynamic mechanical analysis

DMA is widely used to investigate the miscibility of polymer blends, mechanical properties and heat resistance of the materials. Figure 4a and b show the variation of the loss angle factor ($\tan \delta$) and storage modulus of the LCF and SCF composites as a function of temperature. A big relaxation peak appears at 80 °C in the composites (shown in Fig. 4a), which is ascribed to the glass transition temperature (T_g) of PETG. For the SCF composites, the T_g increases from 80 to 85 °C when CB content changes from 0 to 15 %. In the same CB content range, the T_g for the LCF composites is also elevated about 5 °C (81 to 86 °C). An increase in the T_g indicates that the movement of polymer chain segments was inhibited. The reason for this is due to carbon black having high specific surface area largely increase intermolecular force between filler and polymer. In addition, for the LCF or SCF composites, one can notice that after CB is incorporated, the height of T_g peaks is observed to decline significantly, suggesting the augment in the rigidness. This is due to the formed CB networks enhance the elastic modulus and inhibit viscous deformation of the materials.

Figure 4b shows the storage modulus of the composites as a function of temperature. At the same temperature, the storage modulus of the SCF composites without CB is the lowest, and it begins to decline at above 60 °C or so. The LCF composite without CB presents the similar behaviors like the SCF composite without CB, but it owns the higher storage modulus.

The storage modulus of the composites is observed to increase significantly after CB is added. e. g., for the SCF composites, the storage modulus of the SCF composite with 15 % of CB is 6100 MPa at 50 °C, increasing by 220 % compared with the SCF composite without CB. But for the LCF composites, the storage modulus of the LCF composite with 15 % of CB only increases by 49 %, compared with the LCF composite without CB. Such results mean that the short carbon fiber had the better synergistic effect with CB than long carbon fiber did. In addition, the carbon black also improves the

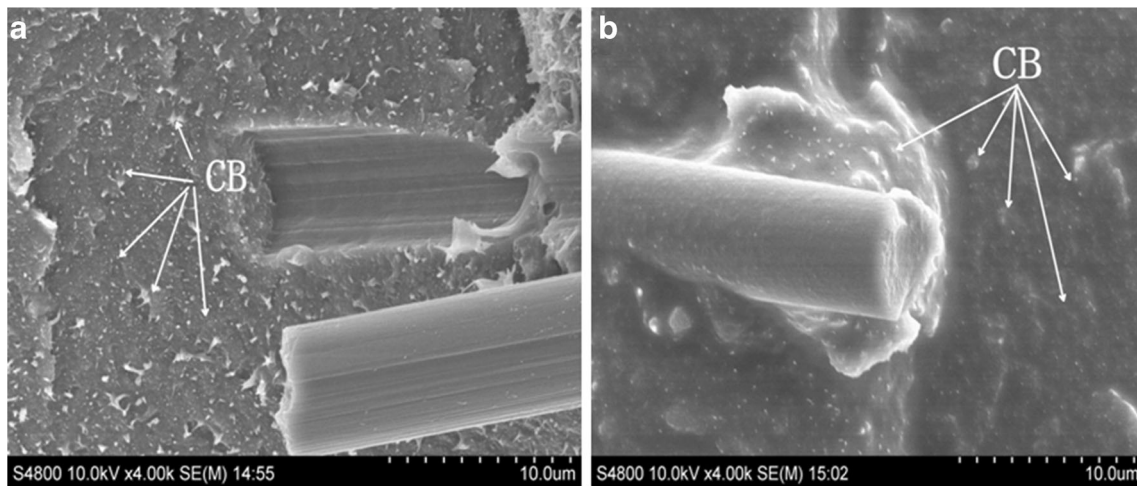


Fig. 3 The SEM photos of PVDF/PETG/CF/CB composites. **a** the LCF composites with 15 % of CB; **b** the SCF composites with 15 % of CB

heat resistance of the composites. For instance, the storage modulus of the SCF composite without CB at 60 °C is 1832 MPa, while the SCF composite with 15 % of CB

presents the same modulus at 112 °C, indicative improved heat resistance.

Electrical resistivity

Generally, the materials with low electrical resistivity have good EMI performance. Only in this way can the incident electromagnetic wave collide with the free electrons in the matrix, and then is reflected or absorbed. Figure 5 exhibits the changes of electrical resistivity of the LCF and SCF composites as a function of carbon black content. It is expected that the electrical resistivity of the LCF or SCF composites decreases as carbon black content increases due to the more conductive networks. Without carbon black, the 5 % of long carbon fiber or short carbon fiber make the PVDF/PETG blends transform from the insulator into the semiconductor, which implies that this level of CF loading (5 %) in the PVDF/PETG blends was beyond the electrical percolation threshold. The LCF composite without CB displays the lower electrical resistivity than the SCF composite without CB, this is because the long carbon fiber has the higher aspect ratios (143000) than short carbon fiber (aspect ratios: 21). The long carbon fiber is easier to contact with each other and establish the conductive networks in the matrix.

In order to reduce the electrical resistivity further, carbon black should be added into the composites. For the SCF composites, the electrical resistivity decreases from 14,400 to 4.79 Ω.cm when CB content changes from 0~15 %. However, the lowest electrical resistivity appears at the LCF composites, where the electrical resistivity decreases from 72.34 to 0.39 Ω.cm in the CB content range of 0~15 %. Evidently, at a given CB content, the electrical resistivity of the LCF composite is lower than that of the SCF composite, as shown in Fig. 6. The reason is attributed to the fact that the short carbon fiber has higher specific surface area than long carbon fiber, which means more polymer resin was needed to wet short

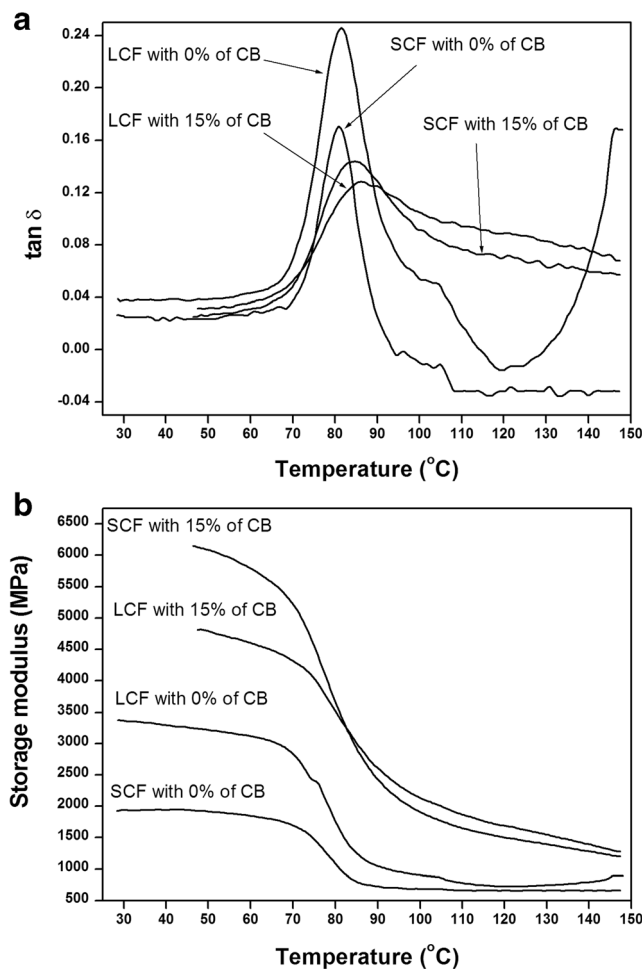


Fig. 4 The variation of the tan δ and storage modulus of the LCF and SCF composites as a function of temperature. **a** tan δ ; **b** storage modulus

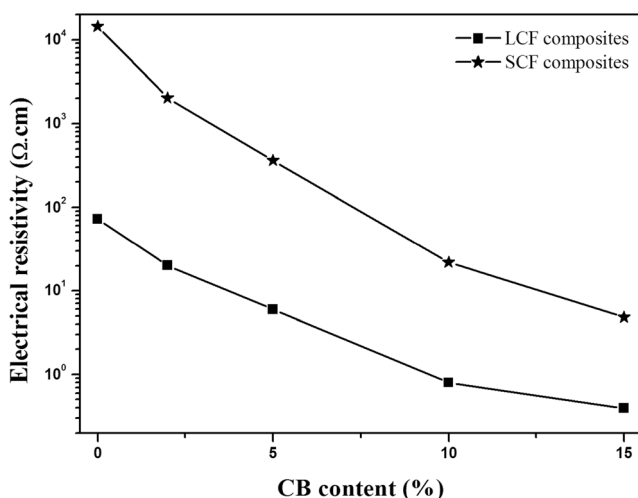


Fig. 5 The variation of the electrical resistivity of the LCF and SCF composites as a function of CB content

carbon fiber. This case not only increases the viscosity (as shown in Fig. 2a and b), but also causes the conductive fillers not to be wetted by polymer resin completely, leading to the aggregation of conductive fillers or gaps between the fillers and polymer. Finally, the electrical resistivity increases.

Importantly, the results of electrical resistivity may confirm that the short carbon fiber exhibits better synergistic effect with carbon black than the long carbon fiber does since the electrical resistivity of the SCF composites decreases by 3000 times or so within the 15 % of CB, while it changes only 180 times for the LCF composite. This conclusion is consistent with the results from DMA.

The above results are attractive and competitive since the achieved electrical resistivity is far lower than the composites reported. e. g., the PP/CB composites, fabricated by compression molding, showed the electrical resistivity of 148 Ω.cm [15]. Al-Saleh et al. observed that the co-continuous PS/PE/CB composites displayed the minimum electrical resistivity of 2.6 Ω.cm [16, 17].

EMI SE

The EMI SE is referred to the ability to reflect and absorb the electromagnetic wave. The materials with the low electrical resistivity or high magnetic permeability, usually exhibit a good EMI SE. In general, the electromagnetic shielding includes the three parts: reflection, absorption and the multi-reflection [16].

$$SE = 10 \log \frac{P_0}{P_t} \quad (2)$$

Where P_0 is the incident electromagnetic power and P_t is the transmitted electromagnetic power. The EMI SE is expressed in decibel (dB) units. For example, if the EMI SE is equal to 20 dB, it means that 99 % of electromagnetic wave

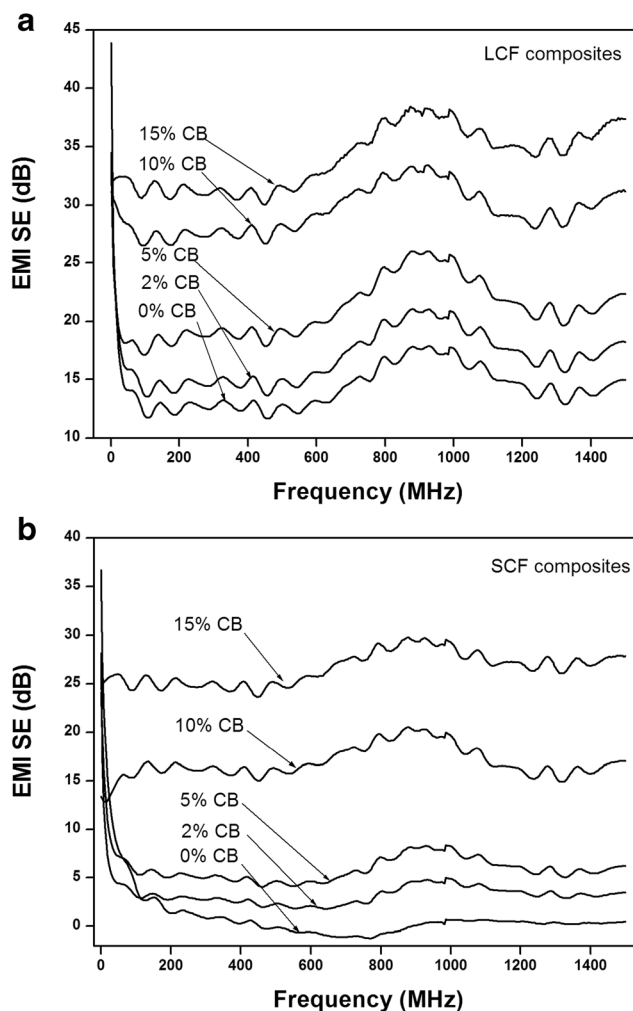


Fig. 6 The variation of EMI SE of the LCF and SCF composites as a function of CB content. **a** the LCF composites; **b** the SCF composites

was shielded, and a EMI SE of 30 dB suggests that 99.9 % of electromagnetic wave power was dissipated. From a commercial point of view, only the EMI SE of the materials exceeds the 30 dB, the materials have the good business sense [6].

Figure 6a and b present the EMI SE of the PVDF/PETG/CF/CB composites with different carbon black content over the frequency range from 0.1 to 1500 MHz. The EMI SE of all composites increase as CB content increase owing to the reduction in the electrical resistivity. The LCF composite without CB presents the EMI SE of above 10 dB over the frequency range of 0.1 MHz to 1500 MHz. At below 5 % of CB, the EMI SE is observed to increase slowly with CB content, but when at above 5 % of CB, the EMI SE rises significantly, indicative of a EMI SE percolation threshold. This result is similar to the rheological results. The SCF composites also exhibit the same behaviors. Especially, one can observe that the SCF composite without CB does not almost shield the electromagnetic wave at all frequency investigated, which is due to the high electrical resistivity. It is seems that the materials can present the EMI function when the electrical

resistivity exceeds a certain value. As CB is added, the EMI SE of the SCF composite with 15 % of CB is elevated to more than 25 dB. Unfortunately, the SCF composites still does not satisfy the commercial requirement where an EMIS SE of 30 dB is needed. Accordingly, the EMI SE of the LCF composite with 15 % of CB is raised to 30 dB and above in the range of 0.1 MHz to 1500 MHz, which can satisfy the commercial requirement. These results are better than that reported. e. g., Li et al. prepared an epoxy composite with 15 % single-walled CNTs, where the maximum EMI SE was 20 dB in the frequency of 500 to 1.5 GHz [18]. Yang et al. reported that the PS/CNTs composite exhibited the EMI SE of 20 dB in the frequency range of 8.2–12.4 GHz [19]. Compared with the above results, the PVDF/PETG/CF/CB composites not only own the higher EMI SE, but also have the lower production cost.

Double conductive pathway

Compared with the single polymer system, the co-continuous immiscible polymer blends can make the conductive fillers preferentially disperse in one component of the composites. Such structure may cause conductive fillers to get to the electrical percolation at lower filler content, forming the conductive network in one component in the blends, which is called the *Single Conductive Pathway*. e. g., The electrical resistivity of the ABS/PA6/MWCNTs composites is lower than that of PA6/MWCNTs or ABS/MWCNTs composites [19]; the PEEK/PI/CNTs composites present the higher conductivity than that of single polymer-based CNTs composites [11]. This characteristic is very important for the conductive polymer materials since it not only reduces the product cost, but also decreases the viscosity and improves process and the mechanical properties of the materials.

However, this level of conductive fillers cannot satisfy the practical needs in some case. Consequently, in practice, the conductive fillers content is far beyond the electrical percolation threshold. In our previous experiments, we have found that the highest loading level of CB in the PVDF or PETG was 15 % or so, and the CB preferentially located in the PET phase of the PVDF/polyethylene terephthalate (PET) blends due to the CB has good compatibility with PET. Here, the PETG has the similar molecular structure with the PET, one can therefore presume that the CB preferentially exists in the PETG phase in the PVDF/PETG blends. Obviously, for the co-continuous PVDF/PETG blends, the PETG phase only accommodates the 7.5 % of CB. It means that the superfluous 7.5 % of CB must disperse in the PVDF phase, and thus the *Double Conductive Pathways*, not a *Single Conductive Pathway* in the PETG phase, are established in the PVDF phase and PETG phase in the PVDF/PETG blends. It is clear that the composite with the *Double Conductive Pathways* displays lower electrical resistivity than the composite with *Single*

Conductive Pathway does since it can accommodate more conductive networks.

Conclusion

The co-continuous PVDF/PETG/F/CB composites were prepared by melting compounding method. The effects of CB on the rheological properties, morphology, dynamic mechanical properties, electrical resistivity and EMI SE of PVDF/PETG/CF/CB composites have been studied. The rheological experiments showed that the storage modulus and complex viscosity of the composites increased as CB content increased. At 5 % of CB and above, a platform appeared in the low frequency on the plots of storage modulus versus frequency, indicative of the formation of the CB networks. Such CB networks caused the T_g to increase about 5 °C and the elastic modulus to augment largely. The electrical resistivity of the SCF composites decreased from 14,400 to 4.79 Ω .cm as CB content changed from 0 to 15 %, whereas it varied from 72.34 to 0.39 Ω .cm for the LCF composites. Accordingly, the EMI SE of the SCF composites was raised to above 25 dB over a frequency of 0.1 MHz~1500 MHz, whereas the EMI SE of the LCF composites was elevated to more than 30 dB. The double conductive pathway, existing in the PVDF and PETG phase in the continuous polymer blends, was proposed to elucidate the conductivity mechanism of the composites.

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