

Facile Preparation of Electromagnetic Interference Shielding Materials Enabled by Constructing Interconnected Network of Multi-walled Carbon Nanotubes in a Miscible Polymeric Blend

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Abstract Electromagnetic interference (EMI) shielding materials are in great demand in electronic equipment and our surrounding environment to resist the increasing serious radiation pollution. Compared with their metal counterparts, conductive polymer composites (CPCs) have unique advantages of lightweight, corrosive resistance, low cost, and excellent processability, and are therefore an ideal choice for developing high-performance EMI shielding materials. However, CPCs based EMI shielding materials are limited to high filler loading, which caused poor mechanical properties and processability. Here, we demonstrate a facile and highly scalable approach to develop high-performance EMI shielding materials with low filler loading by using miscible poly(phenylene oxide)/polystyrene (PPO/PS) blend as the matrix. In contrast to PS/carbon nanotubes (CNTs) composites, PPO/PS/CNTs composites show much higher EMI shielding effectiveness caused by good dispersion of CNTs and highly interconnected conductive network. An excellent EMI shielding effectiveness of 23–25 dB is achieved for PPO/PS/10% CNTs composites with a thickness of only 375 μm , which is superior to most of reported polymer/CNTs composites prepared by melt-compounding. In addition, the results show that although absorption is the major shielding mechanism, the contribution of reflection is also important and closely related to the connectivity of conductive network, as well as the electrical conductivity of the CPCs.

Keywords Conductive polymer composites; Polymer blend; Electromagnetic interference shielding; Conductive network; Dispersion

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INTRODUCTION

With the rapid development of modern electronic devices that get smarter and smaller by packing with large integrated circuits, electromagnetic interference (EMI) has become a new kind of pollution which has detrimental impacts on electronic equipment performance, surrounding environment as well as the human health.^[1–3] Therefore, it is emergent to develop high-performance EMI shielding materials that can effectively shield radio frequency radiation.

Reflection and absorption are two main mechanisms for EMI shielding.^[4] Reflecting radiation occurs when the shielding materials have mobile charge carriers which interact directly with the electromagnetic fields. As a result, shielding materials tend to be electrically conductive.^[5] Absorption of electromagnetic radiation occurs when the shielding materials have electric and/or magnetic dipoles which can interact with the radiation. Hence, high electrical conductivity is one of the most important factors that determine reflectivity and absorption characteristics of the shielding materials.^[1,2]

Metal shrouds or covering the metal powder on the surface of bulky material are the earliest choice for the EMI shielding materials due to their excellent electrical conductivity.^[1] However, these materials all suffer from poor mechanical flexibility, susceptibility to corrosion and high cost for practical applications; especially, exceedingly high weight of metal shrouds is not appropriate for the smaller devices and components. Hence it is of great significance for the development of lightweight, flexibility, low-cost, and easy-to-fabricate shielding materials. Electrically conductive polymer composites (CPCs),^[6–9] prepared by incorporating conductive fillers into polymer matrix, have become a popular kind of EMI shielding materials due to their unique advantages such as low density, high processability, corrosive resistance and low cost. Carbon fillers, particularly carbon nanotubes (CNTs) and graphene, have been widely used as conductive fillers to form electrically conducting networks for their large aspect ratio and high electrical conductivity.^[10–12]

However, high loading of carbon fillers is required to get target EMI-shielding effectiveness (SE) of shielding materials prepared by melt-compounding, which inevitably causes high cost, serious processing difficulties, and brittleness of the composites.^[13] Although some efforts have been paid to decrease the loading of carbon fillers, none of the ap-

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proaches is cost-effective and highly scalable for large-scale production. For example, forming a segregated structured conductive network can decrease the filler loading and improve the EMI-shielding performance. However, this network structure decreases the mechanical properties of the CPCs, not to mention the high cost and low production efficiency.^[14,15]

From engineering point of view, it is of great interest to develop strategies which can significantly improve the EMI-shielding performance of CPCs based shielding materials prepared by melt-compounding which is a facile method and compatible with current industrial processes. As mentioned before, high electrical conductivity is an important factor determining the EMI shielding performance of the shielding materials. It is well known that the conductivity of CPCs is closely related to the dispersion of the conductive fillers, as well as the connectivity of the conductive network for a given system.^[16] Recently, our research group found that using miscible polymer blend, *i.e.*, poly(phenylene oxide)/polystyrene (PPO/PS), as the matrix is a low cost and effective method for improving the electrical conductivity of the composites by improving the dispersion of CNTs enabled by tuning matrix viscosity through blend ratio.^[17] The best dispersion state of CNTs was found at the intermediate viscosity matrix with the blend ratio 35/65 in weight that had balanced effects on infiltration of polymer molecules and breakage of primary CNT agglomerates. The perfectly dispersed CNTs formed interconnected network and resulted in the best electrical performance.

In this work, we further studied the benefits of using miscible polymer blends as the matrices in fabricating EMI shielding materials. In details, the miscible PPO/PS blend with weight ratio 35/65 is used as the matrix and CNTs are used as the conductive fillers to fabricate EMI shielding materials by melt-compounding, which is then compared with PS/CNTs composites in terms of CNT dispersion and EMI shielding performance. On the other hand, developing high performance PPO/PS based EMI shielding materials has great practical values, because PPO/PS blends are commercial miscible alloys and widely used in electric and electronic components. The results demonstrate that the EMI shielding effectiveness (SE) of PPO/PS/CNTs composites is much higher than that of PS/CNTs, which mainly results from increased contribution of reflection for the EMI radiation obtained by much better dispersion of CNTs and highly interconnected conductive network, even though absorption is the major shielding mechanism and reflection is the secondary shielding mechanism. PPO/PS/CNTs composites with 10 wt% CNTs exhibit excellent EMI SE value of 23.4 dB, which is much higher than those of the most reported polymer/CNTs composites with the same CNTs content prepared by melt-compounding and sufficient for the commercial applications as EMI-shielding materials. This work confirmed that the dispersion of the conductive fillers and the connectivity of conductive network, as well as the electrical conductivity, are significant factors to obtain high EMI-shielding performance of CPCs. This work also offers a highly scalable and facile method for the preparation of high-performance EMI-shielding materials.

EXPERIMENTAL

Materials

Poly(phenylene oxide) (PPO, PX-100 L) was supplied by Mitsubishi Engineering-Plastic Corporation (Japan). Polystyrene (PS, 158K) was purchased from BASF YPC Company Limited (China). Multi-walled carbon nanotubes (CNTs, FT-9001) were purchased from Cnano Technology Ltd. (China). As provided by the manufacturer, the average diameter and length of CNTs were about 10–15 nm and 10 μm , respectively. According to the manufacturer, the purity of CNTs is about 95%. All of the materials were used as received.

Composite Preparation

PPO/PS/CNTs composites were prepared by melt-compounding with a torque rheometer (RM-200A, Harbin Hapro Electrical Technology Co.) at a rotation speed of 60 r/min for 8 min; the compounding temperature was 270 $^{\circ}\text{C}$. As a comparison, PS/CNTs composites were also prepared with compounding temperature of 200 $^{\circ}\text{C}$ under the same other conditions. The specimens for the electrical conductivity and the EMI SE measurements were prepared by hot-pressing with a hot press machine (Labtech Inc) at 5 MPa. A schematic representation of the fabrication of the polymer composites is shown in Fig. 1(a).

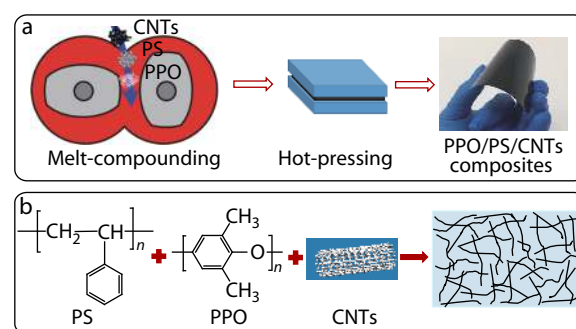


Fig. 1 (a) Schematic diagram for preparation of PPO/PS/CNTs composites. (b) Molecular structure of PS, PPO and CNTs and schematic representation of the composites.

Characterization

The EMI SE of the samples was measured using the waveguide method in 8.2–12.4 GHz (X-band) by a vector network analyzer (VNA, Agilent Technologies, E5071C). The wave-guides were linked to the VNA with two cables. The calibration of the VNA was made according to the through-reflect-line method. Samples with thickness of 375 μm were clamped between two coupling flanges of the input and output wave guides. The input power used in the present research was 0 dBm, which corresponds to 1 mW. Electrical conductivity of the samples was measured by using a low resistivity test (KYD-1, Guangzhou Kunde Technology Co.) based on four-point probe method. The dispersion of CNTs within the composites was analyzed using field emission scanning electron microscopy (FESEM, JSM-7041, JEOL Instrument) and transmission electron microscopy (TEM, H-7650B, Hitachi High-Technologies Co.). Samples were cryo-fractured in liquid nitrogen and the fractured surfaces were gold sputtered and then observed by FESEM with accelerating voltage of 5 kV. For TEM observation, thin sections of 100 nm were prepared using an ultratome with an ultrasonic glass knife at room temperature.

RESULTS AND DISCUSSION

The dispersion states of CNTs in PPO/PS/CNTs and PS/CNTs composites were observed using FESEM and TEM (Fig. 2). As can be seen in Figs. 2(a) and 2(c), the CNTs were uniformly distributed in the PPO/PS blend matrix as individual nanotubes and intertwined with each other, forming an interconnected network for electron transport in the composites. However, PS/CNTs composites show CNT-rich and CNT-deficient zones, in which most of the CNTs agglomerate together and some are isolated without interconnection, but not a continuous and interconnected CNT network (Figs. 2b and 2d). The mechanism for the much better dispersion of CNTs in PPO/PS blend than that in PS matrix is attributed to the intermediate matrix viscosity of the PPO/PS blend matrix that had balanced effects on infiltration of polymer molecules and breakage of primary CNT agglomerates, which has been discussed in detail in our previous article.^[17]

Inspired by this remarkable dispersion of CNTs, we measured the EMI-shielding properties of the PPO/PS/CNTs composites in the X-band frequency range (8.2–12.4 GHz) and compared them with those of PS/CNTs composites (Fig. 3). The EMI SE values of the two types of composites both increase with increasing CNT loading, implying that the EMI SE is mainly attributed to the formed conducting network in the polymer matrix. The EMI SE values of the PPO/PS/CNTs composites are always greater than those of the PS/CNTs composites with the same CNT contents, which are more obvious when the CNT content is between 2 wt% and 5 wt%. This is because the absence of continuous percolation CNT network in PS/CNTs composites results in a lower electrical conductivity, whereas interconnected network is already formed when CNT content is only 1.5 wt% in PPO/PS/CNTs composites.^[17] Fig. 3(d) presents the electrical conductivities of two types of

composites. A higher electrical conductivity in PPO/PS/CNTs was observed compared to PS/CNTs composites with the same CNT content, which is in agreement with previous reported results.^[17] This indicates that an interconnect conductive network and the high conductivity are important factors for the high electromagnetic shielding values. With 5 wt% and 10 wt% of CNTs, the EMI SE values of PPO/PS/CNTs increase to 11.7 and 23.4 dB at a sample thickness of only 375 μm , respectively, which are much higher than the reported values for polymer/CNTs composites, especially prepared by melt-compounding, as shown in Table 1. It should be noted that 23.4 dB achieved in PPO/PS/10% CNTs composites is sufficient for the commercial applications as EMI-shielding materials which recommended limit of 20 dB.^[18]

To identify the shielding mechanisms of the PPO/PS/CNTs composites, the contributions of reflection (SE_R) and absorption (SE_A) to the overall shielding are presented in Fig. 4. The SE_R and SE_A of the PS/CNTs are also presented for comparison. The results suggest that the SE_R and SE_A of the PPO/PS/CNTs composites are 8.3 and 15.1 dB at around 10 GHz, respectively, indicating that absorption contributes significantly (64.5%) to the overall EMI SE compared to the contribution of reflection (35.5%). This means that absorption is the dominant shielding mechanism, rather than reflection in the composites. However, the contribution of reflection is not so small compared to the reported results, which is only 11% in epoxy/CNT sponge nanocomposites^[13] and 4% in the polystyrene (PS)/reduced graphene oxide (rGO) composites.^[14] Thus, reflection is the secondary shielding mechanism of the PPO/PS/CNTs composites. On the other hand, although both SE_R and SE_A of the PPO/PS/CNTs composite are larger than those of the PS/CNTs, the increment of SE_R is much larger than that of SE_A . This implies that the improvement of EMI shielding performance of PPO/PS/CNTs compared to PS/CNTs

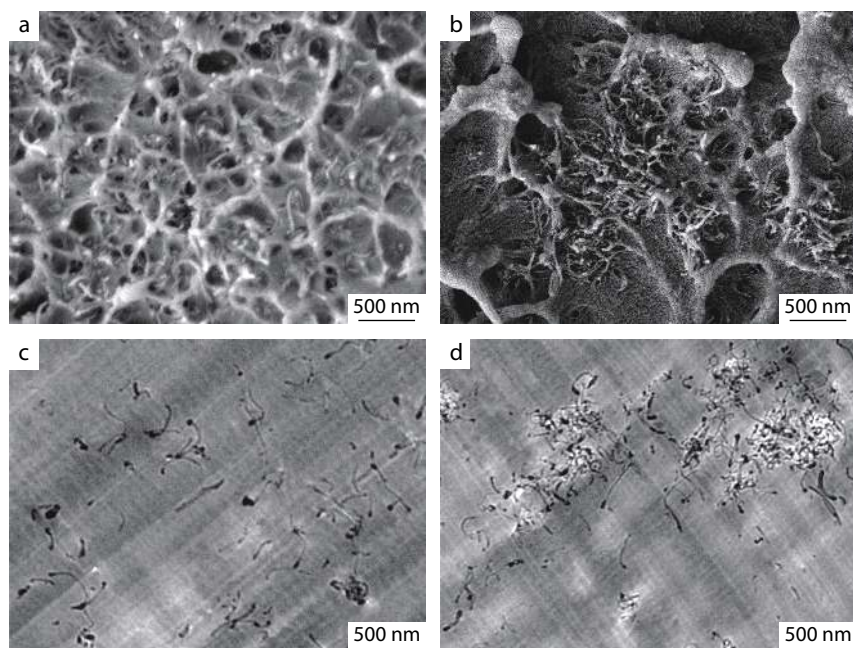


Fig. 2 FESEM images of (a) PPO/PS/5%CNTs and (b) PS/5%CNTs, and TEM images of (c) PPO/PS/5%CNTs and (d) PS/5%CNTs.

is mainly contributed by the increased contribution of reflection. As discussed before, reflecting radiation occurs when the shielding materials have mobile charge carriers which interact directly with the electromagnetic fields. This further proves that the improvement of EMI shielding performance resulted from much better dispersion of CNTs and higher conductivity in PPO/PS/CNTs composites which increase the contribution of reflection compared to PS/CNTs composites. As for the dependences of the SE_R and SE_A as a function of frequency, both types of composites show slight decrease of SE_R and increase of SE_A with the increase of frequency, which is consistent with the theoretical predictions^[19] for mono-

lithic conductive materials. The results demonstrate that the connectivity of the conductive pathways, and thereby the conductivity, is required to obtain high EMI SE value of CPCs based shielding materials, especially for the shielding by reflection.

Fig. 5 shows the reflected (P_R), absorbed (P_A), and transmitted (P_T) powers of PPO/PS/CNTs, and PS/CNTs composites. For both types of composites, it is apparent that the amount of energy blocked by reflection is higher than that blocked by absorption. It should be noted that this observation does not indicate that reflection is the dominant shielding mechanism, as the lower amount of power blocked by absorption

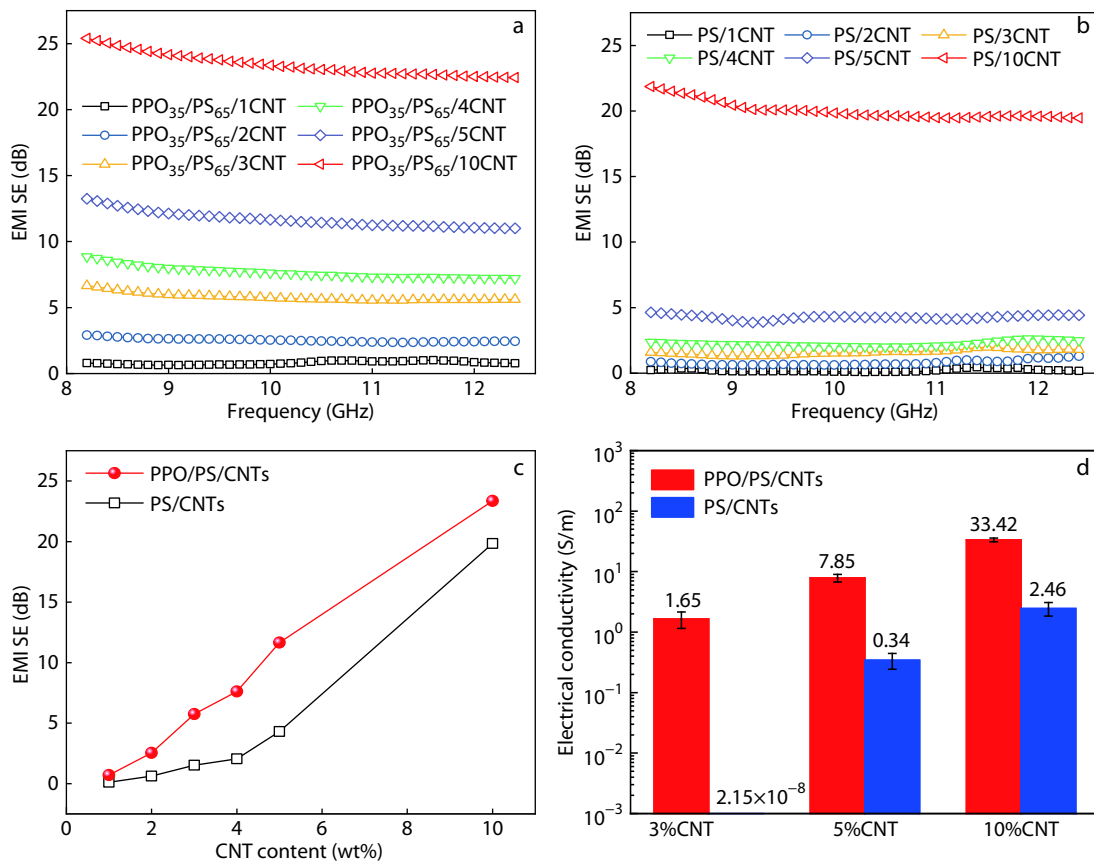


Fig. 3 Measured total EMI SE of the (a) PPO/PS/CNTs and (b) PS/CNTs composites with various CNT contents in the X-band frequency range. (c) The EMI SE at 10 GHz as a function of CNT contents. The thickness of the samples was 375 μ m. (d) Electrical conductivity of PPO/PS/CNTs and PS/CNTs composites with different CNT contents.

Table 1 Comparison of EMI shielding performance of polymer/CNTs composites.

Polymer matrix	CNTs content	Thickness (mm)	EMI SE (dB)	Frequency (GHz)	Method	Ref.
PPO/PS	10 wt%	0.375	23.4	10	Melt	This work
PS	10 wt%	–	14.5	10	Solution	[20]
PS	10 vol%	300	17–18	8.2–12	Solution	[21]
PMMA	10 vol%	300	16–18	8.2–12	Solution	[21]
PP	9.3 wt%	1.0	16	8–12	Melt	[4]
PU	10 wt%	–	12	10	Melt	[22]
HDPE	10 wt%	3.0	28	0.5–1.5	Melt	[23]
BR	8 wt%	1.0	9–12	8.2–18	Melt	[24]
LDPE	8 wt%	1.65	20	13	Solution	[25]
PC	10 wt%	3	19	13	Melt	[26]
Epoxy	15 wt%	–	15–20	0.5–1.5	Solution	[27]
Epoxy	15 wt%	2	17	8.2–12.4	Solution	[28]

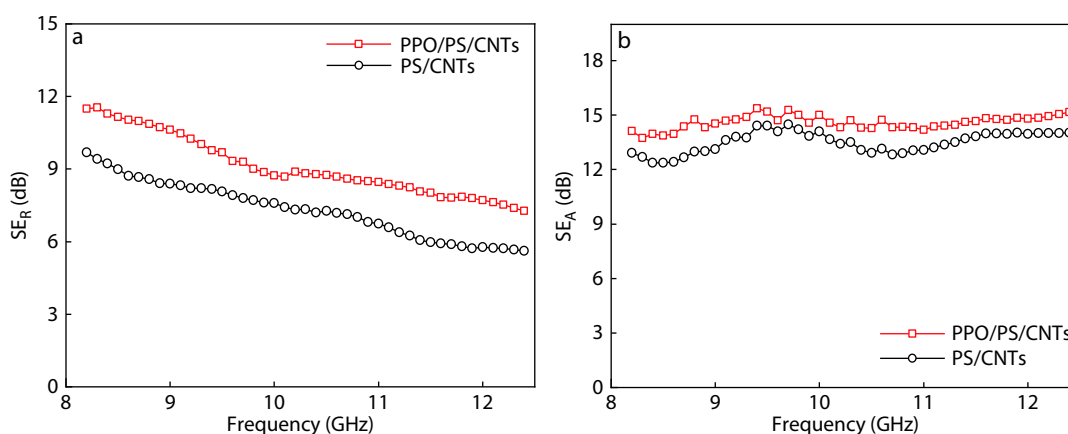


Fig. 4 (a) SE_R and (b) SE_A as a function of X-band frequency range for the PPO/PS/10%CNTs and PS/10%CNTs composites.

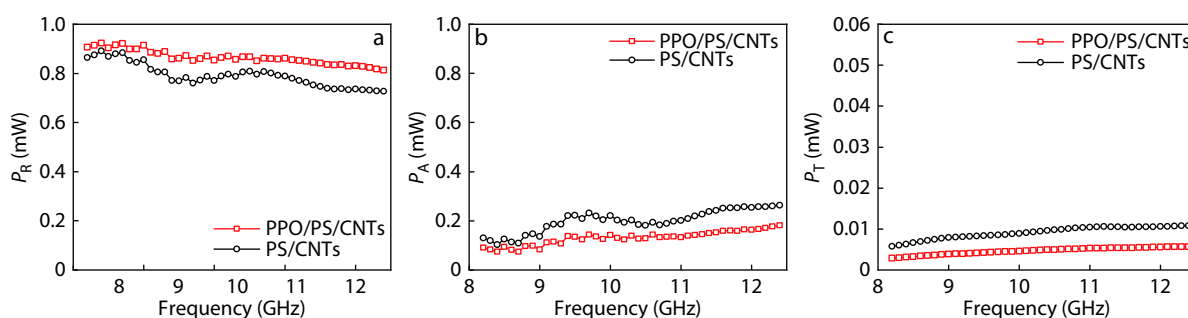


Fig. 5 Dependence of (a) P_R , (b) P_A , and (c) P_T of PPO/PS/10%CNTs and PS/10%CNTs composites as a function of X-band frequency range.

is because the lower amount of power transmitted into a shield as a result of the better reflection and EMI SE is a relative quantity and is not much related to the absolute power values. Actually, the contribution of absorption to the overall shielding should be based on the ability of the sample to attenuate the power that has not been reflected. Fig. 5 also exhibits that, the P_R values of PPO/PS/CNTs composites are higher than those of PS/CNTs composites, but the P_A values of PPO/PS/CNTs composite are lower than those of PS/CNTs composites, even though the P_T value of PPO/PS/CNTs composite is negligible due to good shielding performance and much lower than that of PS/CNTs composite. The results reveal that more electromagnetic waves are reflected by PPO/PS/CNTs composite, which leads to a much lower P_T value. This is consistent with aforementioned conclusion that the highly interconnected conductive network composed of much better dispersed CNTs improves the shielding effectiveness contributed by reflection.

On the basis of the above discussion, a potential mechanism can be proposed to explain the higher EMI SE of PPO/PS/CNTs composites and shown in Fig. 6. The EMI shielding originates from highly interconnected CNTs network and excellent electrical conductivity of the composites. Absorption is the dominant shielding mechanism and reflection is the secondary shielding mechanism. In details, when the electromagnetic waves strike the surface of PPO/PS/CNTs composites, including the interface between CNTs and polymer matrix, abundant free electrons with strongly increased mobility induced by highly interconnected conductive network composed of good dispersed CNTs interact directly with

the electromagnetic fields, resulting in immediate reflecting of some electromagnetic waves. Then the remaining waves pass through the internal structure within the composites, that is, the interconnected CNTs network, and are absorbed by interaction with the high electron density of CNTs which convert the EM waves energy into heat. The surviving waves repeatedly reflect back and forth and are absorbed among the CNTs network in the composites.

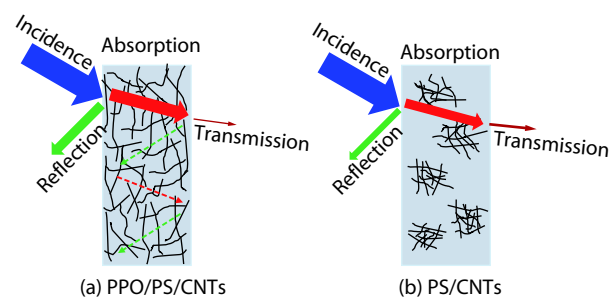


Fig. 6 Schematic of EMI shielding mechanism in the PPO/PS/CNTs composites and PS/CNTs composites.

CONCLUSIONS

This study presents that using miscible polymer blends as the matrix to tune the dispersion of CNTs is a highly scalable method and compatible with current industrial processes for developing high performance EMI-shielding materials. The EMI shielding effectiveness of PPO/PS/CNTs composites is much higher than that of PS/CNTs, since the good dispersion of CNTs

and highly interconnected conductive network improve the contribution of reflection mechanism for the EMI shielding. In other words, shielding by reflection is closely related to the dispersion of the conductive fillers and the connectivity of conductive network, as well as the electrical conductivity of the CPCs. Absorption is the major shielding mechanism and reflection is the secondary shielding mechanism in the composites. PPO/PS/CNTs composites exhibit excellent EMI SE value of 23–25 dB in X-band frequency range with 10 wt% CNTs and at a sample thickness of 375 μm , which is much higher than those of the most reported polymer/CNTs composites at the same CNTs content and sufficient for commercial applications as EMI-shielding materials.

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