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High-performance conductive materials based on the selective location of carbon black in poly(ether ether ketone)/polyimide matrix

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ABSTRACT

A novel high performance conductive material with excellent comprehensive properties was prepared by melt-blending, and its performances were adjusted by controlling the selective location of carbon black (CB) in poly(ether ether ketone) (PEEK)/thermoplastic polyimide (TPI) matrix. With increasing the CB loadings, the morphology of PEEK/TPI blends changed from sea-island to co-continuous structure, which was owing to the selective location of CB in TPI phase. Notably, with the selective location of CB in the induced co-continuous PEEK/TPI matrix, the electrical percolation threshold was reduced to 5 wt%, which was significantly lower than that of binary PEEK/CB (9 wt%) and TPI/CB (10 wt%) composites. And the electrical conductivity of ternary PEEK/TPI/CB composites was 10^4 to 10^6 times higher than that of binary composites at identical 7.5 wt% CB loading, which was attributed to the double percolation effect. Moreover, the incorporation of CB could improve the thermal and mechanical properties effectively.

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1. Introduction

Currently, conductive materials have attracted more and more attention in various areas, such as electro-chromic display, electromagnetic interference shielding and photovoltaic conversion [1–4]. Compared with traditional metal conductive materials, polymer-based conductive materials have increasing popularity in recent years because of their light weight, resistance to corrosion, flexibility, and processing advantages [5–8]. In the developed fields of polymer-based conductive materials, super-engineering plastics are of great interest. They are sought as an alternative for metals in the field of aerospace technology and energy industry because of their outstanding properties [9–12].

Poly(ether ether ketone) (PEEK) is a semicrystalline super-engineering plastic with outstanding performance such as excellent solvent resistance, superior mechanical properties, ease of injection molding [9,13]. But the modulus of PEEK decrease evidently at elevated temperature because of a relatively low glass transition temperature (T_g) around 140–145 °C, which limits its applications at high temperature. On the other hand, YZPI™ thermoplastic polyimide (TPI) is another high performance engineering polymer with a T_g as high as 255 °C. However, TPI has a lower chemical resistance and worse processability than that of PEEK because of its amorphous structure. Therefore, blending PEEK/TPI together is an efficient strategy to combine the complementary properties of both polymers, it is proven to improve the modulus at elevated temperature of the former and the chemical resistance and processability of the latter [12,14]. And PEEK/TPI blend is believed to be an ideal polymeric matrix for high performance conductive materials. Meanwhile, as PEEK and TPI are immiscible with each other, the performance of PEEK/TPI blends is determined not only by properties of each component, but also by the morphology formed [15,16]. Nowadays, much effects have been devoted to polymer blends with a co-continuous structure due to a synergistic improvement of the blend properties, including electrical and thermal conductivity, elastic modulus, and heat resistance [17–19]. How to form and stabilize a co-continuous domains in PEEK/TPI blends during the melt processing remains a challenge.

The addition of nanoparticles into immiscible polymer blends is an effective method to extend the phase co-continuity and obtain a co-continuous morphology [20–23]. While carbon black (CB), which has been considered as an ideal filler for polymer composites, is the most widely used conductive filler due to its abundant source, low density, permanent conductivity and low cost for industrial usage. Wu et al. clarified the mechanism of CB induced co-continuity in acrylonitrile-butadiene-styrene (ABS)/polyamide 6 (PA6) blends [19]. They found that CB was preferentially localized in the PA6 phase, CB self-assembly played a key role not only in

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forming the co-continuous structure but also in stabilizing the co-continuous polymer domains.

However, due to required high CB loadings to obtain excellent conductivity and relatively high melt viscosity of PEEK and TPI matrix, few work has been focused on the PEEK or TPI based CB composites, and how to reduce the electrical percolation threshold of PEEK/CB and TPI/CB composites remains challenging [24]. While selective location of CB in immiscible polymer blends is one of the most promising methods to reduce the percolation threshold and enhance the electrical conductivity [25]. Low CB loading means relatively low melt viscosity and improved impact resistance, as well as low cost to the final commercial products. An electrically conductive material with very low filler contents can be achieved by creating a co-continuous structure in the blend, which is called double percolation phenomenon [26–28]. Double percolation refers to the percolation of CB within one phase of the continuous polymer blend (first percolation), which itself percolates in the blend (second percolation). The reduction of percolation threshold in the double percolation system could be explained by the difference in the affinity of CB to the polymer components, which results in the CB selectively located in one of the two polymer phases. Li et al. reported that PP/epoxy/CB composites exhibited a highly inregular morphology in which CB was selectively distributed in the epoxy phase [25]. The ternary PP/epoxy/CB composites showed reduced percolation threshold compared with binary CB composites, which was attributed to the double percolation effect. CB-filled immiscible polymer composites with lower percolation threshold and enhanced conductivity have various applications in positive temperature coefficient (PTC) materials, electromagnetic interference shielding and composite bipolar plates [29–31].

In this work, PEEK/TPI and CB were respectively chosen as the polymer matrix and the filler to prepare a high performance conductive material with low percolation threshold and excellent comprehensive properties. Different ratios of CB were added into the PEEK/TPI matrix, and PEEK/TPI/CB composites were fabricated by melt extrusion. The selective distribution of CB in PEEK/TPI matrix and the morphology changes induced by adding CB were investigated. Moreover, the effects of CB on the electrical, thermal and mechanical properties of PEEK/TPI blend were systematically discussed.

2. Experimental section

2.1. Materials

Poly(ether ether ketone) (PEEK) was supplied by Changchun Jilin University Super Engineering Plastics Research Co., Ltd. (P.R. China), the chemical structure of PEEK is shown in Fig. 1a. YZPI™ thermoplastic polyimide (TPI) was purchased from Nanjing Yuezi Chemical Col., Ltd. (P.R. China), and its chemical structure for reference is shown in Fig. 1b. The inherent viscosity of PEEK is 0.63 dL g^{-1} in H_2SO_4, and the inherent viscosity of TPI is 0.33 dL g^{-1} in DMAc, determined with an Ubbelohde viscometer at 25 °C. The melt index of PEEK is 102 g·10 min^{-1}, and the melt index of TPI is 15 g·10 min^{-1}, which is measured by a melt flow speed-indicator at 355 °C. CB nanoparticles with diameters of 80–140 nm, iodine number of 90 mg g^{-1}, and dibutyl phthalate number (DBP) of 120 mL·(100 g)$^{-1}$ were obtained from Tianjin Jinqiushi Chemicals Co., Ltd. (P.R. China) and used as received.

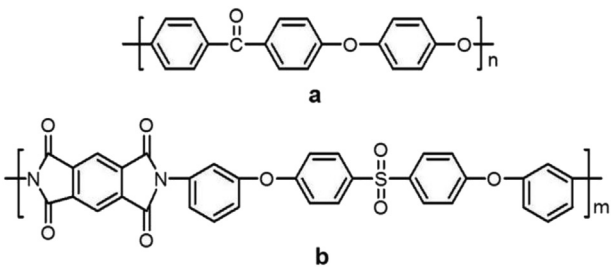

Fig. 1. Structure of (a) PEEK and (b) TPI.

2.2. Sample preparation

PEEK/TPI/CB composites were prepared by melt blending. First, PEEK, TPI and CB were pre-mixed with a high-speed mixer for 2 min. Polymers and CB were dried in a vacuum oven at 120 °C for 24 h before use. Then, the pre-dispersion mixtures were blended using a Mini-Haake co-rotating twin-screw extruder at a screw speed of 80 rpm and a temperature of 355 °C. The detailed sample names and corresponding components for PEEK/TPI/CB composites are listed in Table 1. Moreover, binary PEEK/CB and TPI/CB composites with the same CB contents were also prepared by the same procedures for comparative experiments. Finally, the obtained samples were cut into granules, dried at 120 °C for 6 h, and then molded at 355 °C by compression or injection molding for different measurements.

2.3. Characterization

Scanning electron microscopy (SEM) observation was performed using a HITACHI-SU8020 cold field emission scanning electron microscope operating at a 3.0 KV accelerating voltage. The selective extraction by DMAc for 24 h was firstly achieved to remove the TPI from the samples. The samples were dried and surfaces were gold-sputtered before scanning.

Transmission electron microscopic (TEM) images were taken on a JEM-1200EX electron microscope operated at an acceleration voltage of 100 KV. The sample preparation involved embedding membranes in an Epon 812-Araldite mixture followed by ultramicrotomy with a diamond knife to obtain 80 nm thin sections which placed on copper grides for TEM analysis.

The alternating current (AC) conductivity were measured at room temperature in the frequency range between 10^2 and 10^6 Hz using an Aglient 4294A precision impedance analyzer. The samples were cut into small round specimens with a diameter of 10 mm, and two opposite surfaces of samples were coated with silver conductive glue to reduce the contact resistance between samples and electrodes.

Thermal gravimetric analysis (TGA) was conducted under air atmosphere at a heating rate of 10 °C·min^{-1}. Samples of 3–5 mg were contained within open platinum pans of a PerkinElmer TGA-7 instrument.

Tensile tests were performed on a Shimadzu AG-1 universal testing machine with a tensile rate of 5 mm·min^{-1} at room

Table 1
Sample names and corresponding components for PEEK/TPI/CB composites.

Sample names	PEEK/TPI/CB (wt%)
P/T	50/50
P/T/3%-CB	48.5/48.5/3
P/T/5%-CB	47.5/47.5/5
P/T/6%-CB	47/47/6
P/T/7.5%-CB	46.25/46.25/7.5
P/T/9%-CB	45.5/45.5/9
P/T/10%-CB	45/45/10
P/T/12.5%-CB	43.75/43.75/12.5
P/T/15%-CB	42.5/42.5/15

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