



Macromolecular Nanotechnology

Effect of mixing temperature on the carbon nanofiller distribution in immiscible blends of polycarbonate and polyolefin



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ABSTRACT

We studied the selective localization of carbon nanofillers, such as multi-walled carbon nanotube (MWCNT) and graphene nanoplatelet (GNP), in immiscible polymer blends composed of polycarbonate (PC) and polyethylene (PE) or ethylene-propylene copolymer (EPR). It was found that the distribution state of the carbon nanofillers in the composites is greatly affected by the mixing temperature and the species of nanofillers. MWCNTs resided in the PE or EPR phase in the composites, which cannot be explained by the difference in the interfacial tension. A similar morphology was detected in the PC/GNP/PE composite prepared at 300 °C. In contrast, the PC/GNP/PE composite prepared at the low temperature (250 °C) and the PC/GNP/EPR composites have the carbon nanofillers mostly in the PC phase. The selective localization in the PE or EPR phase is attributed to the surface adsorption of PE or EPR chains on the carbon nanofillers, which is more obvious for PE and at the high mixing temperature. These results demonstrate that both the species of carbon nanofillers and the mixing temperature affect the carbon nanofiller distribution in the immiscible blends.

1. Introduction

Polymer composites consisting of a polymer matrix with nanofillers have attracted significant interest from researchers due to their potential as high-performance materials. In particular, precise control of nanofiller distribution makes it possible to tune various properties of a composite of two or more polymer species containing nanofillers. When nanofillers are introduced into an immiscible polymer blend composed of two polymer species, three cases of filler dispersion can take place: (i) nanofillers are randomly dispersed in both polymer phases, (ii) nanofillers are unevenly distributed in each phase, and (iii) nanofillers localize at the boundary of phases [1–4]. In order to control the structure, the recipe of a polymer blend and processing conditions have to be appropriately selected.

Dispersing nanofillers uniformly in an immiscible polymer blend is difficult even with stress that surpasses a cohesive force of filler agglomerations. Moreover, in many cases uneven distribution occurs. The main factors affecting localization of nanofillers are classified into thermodynamic and kinetic effects [5]. The thermodynamic effect is determined by the difference in the interfacial tension among polymer pairs and filler [6–8]. According to Mamunya, the spatial distribution of carbon black (CB) in an immiscible polymer blend is determined by the interfacial tension Γ_{i-c} between i -polymer and CB, as well as the interfacial tension between polymer pairs Γ_{A-B} [2]. This behavior is well expressed using the wetting coefficient ω_a , defined as Eq. (1) [1], which was confirmed by previous researches [1,9,10];

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$$\omega_a = \frac{\Gamma_{A-filler} - \Gamma_{B-filler}}{\Gamma_{A-B}} \quad (1)$$

when ω_a is smaller than -1, fillers exist in phase A. In contrast, fillers exist in phase B at $\omega_a > 1$. Moreover, fillers are localized at the phase boundary at $-1 < \omega_a < 1$.

Kinetic effect [11] can be controlled by the mixing process. It has been demonstrated that the distribution state of fillers in a polymer blend is often different from the equilibrium state because of its high viscosity. In particular, nanofillers can not diffuse into a polymer with an extremely high viscosity [12,13]. In this case, nanofillers have to reside in a low viscous phase or on the surface of a high viscous phase. Such localization leads to an electrical conductive path with a small amount of conductive fillers [14–17].

In the previous paper [18], we found that multi-walled carbon nanotubes (MWCNTs) were dispersed in the continuous polypropylene (PP) phase in the blends of PP and ethylene-propylene copolymer (EPR) when the composite was prepared at low temperatures. Furthermore, nitrogen gas purging was effective to the preferential distribution of MWCNTs in the matrix, i.e., PP. Without the use of nitrogen, in contrast, more MWCNTs were distributed in the dispersed EPR phase when the mixing temperature was high, e.g., 280 °C. This is attributed to the adsorption of EPR molecules on the MWCNT surface during melt mixing. These results significant for the material design of rubber-toughened plastics, although it is still unknown whether nanofiller distribution is controlled by the mixing condition for other blend systems.

In this research, we investigated the effect of the mixing conditions, specifically temperature, on the nanofiller localization using immiscible blends composed of polycarbonate (PC) and polyethylene (PE) or EPR. The effect of shape and species of carbon nanofillers on the distribution state was studied using MWCNT and graphene nanoplatelet (GNP).

2. Experimental

2.1. Materials

The polymers used in this study were commercially available bisphenol A polycarbonate (PC) (Panlite L-1225Y, Teijin, Japan, MFR = 11 [g/10 min]), high-density polyethylene (PE) (HJ590N, Japan Polyethylene, MFR = 40 [g/10 min]), and ethylene-propylene copolymer (EPR) (EP11, JSR, Japan) with an ethylene content of 52 wt%. The Mooney viscosity $ML_{(1+4)}^{100}$ °C of EPR is 40. Because of the large amount of propylene, the EPR is fully amorphous at room temperature. The number- and weight-average molecular weights, characterized by a size exclusion chromatograph (SEC) (HLC-8020, Tosoh, Japan) using chloroform as an eluent, are $M_n = 1.9 \times 10^4$ and $M_w = 9.7 \times 10^4$ for PC and 4.0×10^6 and 4.7×10^6 for EPR, as a polystyrene standard. Moreover, M_n and M_w of PE were also characterized by SEC using 1,2,4-trichlorobenzene at 140 °C and found to be 8.7×10^3 and 4.9×10^4 , respectively, as a polyethylene standard. The density of PE is 960 [kg/m³] at room temperature.

Multi-walled carbon nanotubes (MWCNTs) were produced by a catalytic chemical vapor deposition method using a floating reactant method and subsequent thermal treatment up to 2600 °C. Typical diameters of the MWCNTs range from 40 to 80 nm, while the lengths are between 10 and 20 μ m. The density is approximately 2300 [kg/m³]. PC/MWCNT (80/20, in weight fraction) was provided by Hodogaya Chemical (Japan) in pellet form. Graphene nanoplatelets (GNPs) were produced by Graphene Platform in powder form. The average diameter ranges from 3 to 30 μ m, and the thickness is between 0.3 and 1.5 nm. PC/GNP (80/20, in weight ratio) was prepared by adding the GNP powder to the PC-chloroform solution at room temperature. After drying, the composite was kneaded at 280 °C for 15 min using a co-rotating twin-screw mixer with a disclosed condition (ULT15TWNANO-15MG-NH, Technovel, Japan). The screw rotation speed was 250 rpm.

2.2. Sample preparation

Prior to melt blending, PC/MWCNT (80/20) and PC/GNP (80/20) were dried at 120 °C for 8 h in a vacuum oven in order to remove the moisture. PC/Nanofiller (80/20) and PE or EPR were mixed using a 30 cc internal batch mixer (IMC-1891, Imoto, Japan) at various temperatures for 20 min with a blade rotation speed of 50 rpm. The blend ratio of PC/Nanofiller (80/20) to PE or EPR was 50/50 in the weight fraction, i.e., PC/Nanofiller/PE or EPR (40/10/50) with 10000 ppm of a thermal stabilizer (Sumilizer-GP, Sumitomo Chemical, Japan). In addition, PC/PE (40/50, in weight ratio) and PC/EPR (40/50, in weight ratio) were also prepared as reference samples under the same condition, but without carbon nanofillers.

The obtained samples were compressed into flat sheets with a thickness of 1 mm using a laboratory compression-molding machine at 250 °C under 10 MPa for 3 min and subsequently cooled at 25 °C for 3 min.

2.3. Measurements

Flat sheets of PC/PE, PC/MWCNT/PE, and PC/GNP/PE with a thickness of about 1 mm were immersed in chloroform to eliminate PC fraction at room temperature for one day. Then, the insoluble portion in chloroform, which was collected by using a paper filter with 110 μ m of a pore size, was further immersed into hot-xylene at 140 °C for 6 h. Prior to the measurements, it was confirmed that PE is fully dissolved in hot-xylene under the same condition. The chloroform and xylene solutions containing dissolved polymers were collected and dried to characterize the polymer species. Another immersion experiment was performed using dichloromethane at room temperature for three days using the blend and composites comprising EPR, because dichloromethane selectively dissolves the PC fraction. Moreover, the insoluble portion collected by the paper filter was dried and weighed to calculate the soluble fraction.

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