ポリカーボネート/ポリエチレンテレフタレート ブレンドのモルフォロジー

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Morphological Studies of Polycarbonate/ Poly (ethylene terephthalate) Blends

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1. Introduction

Blends of various polyesters with bisphenol A polycarbonate (PC) have become a very important subject for the modification of polymer properties such as impact resistance, fatigue behavior, and heat resistance. The solvent resistance of PC, which is its main defect, is improved by mixing it with poly (ethylene terephthalate) (PET). This PC/PET blend system in particular has been of great interest, based on commercial and scientific points of view^{1)~9)}.

Based on the improvement of the properties of PC/PET blends, it was first assumed that PC/PET is miscible over the whole composition range. However, Nassar *et al.*¹⁾ showed that PC/PET blends containing more than 60 wt%-70 wt% PET formed a single amorphous phase but formed two amorphous phases below this range on the basis of differential thermal analysis and dynamic mechanical behavior. From experimental results on solvent extraction, infrared spectroscopy, differential scanning calorimetry (DSC), and dynamic mechanical thermal analysis, Chen *et al.*⁶⁾ reported that PC/PET blends contained two amorphous phases over the whole composition range. Hanrahan *et al.*⁹⁾ in their investigation on PC/PET blends using DSC and dielectric loss spectroscopy showed that the blends are almost completely immiscible over the range of compositions studied. Nishi *et al.*⁹⁾ concluded from DSC, phase contrast microscopy, and infrared spectroscopy that PC and PET were immiscible over the whole composition range, for both extruder-mixed and solvent-cast blends.

Although the miscibility and mechanical behavior of this blend have been well studied in the past as mentioned above, little has been known about the morphology of this PC/PET blend system. Makarewicz *et al.*¹⁰⁾ demonstrated the fracture surfaces of melt-mixed PC/PET blends containing PET as the major component (90 wt%) by scanning electron microscopy. They revealed that no distinct phase separation between these two polymers occurred. Exposing these blends to benzene and acetone also demonstrated the thoroughness of mixing between the two components. However, these experiments are not sufficient to clarify the morphology of the blends, because the composition range studied was limited.

When melt-mixed blends are formed, different phase separation and homogeneous phase in the blends are assumed to depend on molecular characteristics, composition, interfacial tension, viscoelasticity of the blend components, and thermal history, etc. It is important to clarify

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the morphology of the blends since the morphology exerts a significant influence on the various properties of the blends.

In this study, we investigated the change in the microstructure of melt-mixed PC/PET blends by Fourier transform ir attenuated total reflectance (FTIR ATR) spectroscopy, Fourier transform ir microscopy (Micro FTIR) and scanning transmission electron microscopy (STEM).

2. Experimental

2.1 Materials

The bisphenol A polycarbonate (PC) used was supplied by Teijin Chemicals Limited. (number-average molecular weight $M_n=25,000$). The poly (ethylene terephthalate) (PET) used was provided by Mitsubishi Rayon Co. Limited. ($M_n=36,000$). Both polymer powders were dried at 120°C in a vacuum oven for 24 hrs. The pre-dried powders were mixed in a various compositions by a Toyoseiki Labo-Plastomill twin screw extruder with T-die. The diameter of the screw was 20 mm and its length-to-diameter ratio was 25. Temperatures of the extruder were set at 280°C (die), 275°C (barrel zone 3), 270°C (barrel zone 2), and 250°C (barrel zone 1). The screw speed was in the range of 10 to 15 r.p.m. The retention time of the extruder was within *ca.* 10 min. Film thickness was *ca.* 100 μm . Polymer blends in weight percentage of PC of 10, 20, 30, 40, 50, 60, 70, 80, and 90 are hereafter called PC/PET =10/90, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30, 80/20, and 90/10, respectively.

2.2 Fourier Transform ir Spectroscopy

Fourier transform ir attenuated total reflectance (FTIR ATR) spectra of PC/PET blend surfaces were obtained on a Nicolet 20 DXB FTIR spectrometer equipped with an ATR attachment (Spectra-Tech Inc.) using KRS-5 (45° facecut angle) and germanium (Ge; 45° facecut angle) crystals, which are hereafter called simply KRS-5 45° and Ge 45°, respectively. All spectra were collected by co-adding 500 scans at 4 cm^{-1} resolution with Happ-Genzel apodization with a long-range HgCdTe (MCT) detector. Reproducible sample contact was achieved with the use of a torque wrench¹¹.

Micro FTIR spectra of PC/PET blend films were obtained on a Perkin Elmer 1650 FTIR microscope (Micro FTIR). All spectra were collected by co-adding 256 scans at 4 cm^{-1} resolution with a long-range HgCdTe (MCT) detector. Thin cross-section samples (*ca.* 0.5 μm in thickness) were analyzed through a mask window of 10×30 μm area.

2.3 Scanning Transmission Electron Microscopy

For the electron microscopy observations, thin films were stained with osmium tetroxide and examined by Topcon DS-130 S scanning transmission electron microscopy (STEM). Thin sections (*ca.* 0.07 μm) were cut in a direction perpendicular to the film surface using a RMC MT6000—XL ultramicrotome and observed.

3. Result and Discussion

3.1 Miscibility

From the Flory-Huggins theory¹², the Gibbs' free energy of mixing, ΔG_m , for a mixture of two polymers in the absence of solvent can be written as follows^{13,14}:

$$\Delta G_m/(RT) = n_1 \ln \phi_1 + n_2 \ln \phi_2 + \chi_{12} \phi_1 \phi_2 (m_1 n_1 + m_2 n_2) \quad (1)$$

where n_1 and n_2 are the number of moles of polymers 1 and 2, ϕ_1 and ϕ_2 are the volume fraction of polymers 1 and 2, respectively. χ_{12} is the Flory-Huggins polymer-polymer interaction parameter of the mixture. m_1 and m_2 are essentially the degree of polymerization of polymers 1 and 2, respectively.

By differentiating eq. (1) with respect to n_1 and n_2 ¹³, the chemical potential of mixing is shown as,

$$\Delta \mu_1/(RT) = \ln \phi_1 + (1 - m_1/m_2) \phi_2 + m_1 \chi_{12} \phi_2^2 \quad (2)$$

$$\Delta \mu_2/(RT) = \ln \phi_2 + (1 - m_2/m_1) \phi_1 + m_2 \chi_{12} \phi_1^2 \quad (3)$$

Using eqs. (2) and (3), and the chemical potential relations in the two conjugate phases, the Flory-Huggins polymer-polymer interaction parameters (χ_{12}) of PC/PET blends were determined by Kim *et al.*^{14,15} and found to vary from 0.054 to 0.037 with composition for extruded PC/PET blends at 275°C. It can be assumed that these χ_{12} values for PC/PET blends are applicable to our present study since our experimental condition such as the extrusion temperature is similar to Kim *et al.*'s.

By equating the first and second derivatives of either eqs. (2) or (3) to zero, the χ_{12}

Fig. 1 FTIR ATR spectra of the carbonyl stretching bands for (a) PC/PET=70/30 and (b) PC/PET=30/70 (wt%) surfaces. The ATR crystals of Ge 45° and KRS-5 45° were used

Fig. 2 Plot of the frequencies of the carbonyl bands of (a) PC and (b) PET against the PET weight fraction

value under the critical condition is obtained as shown below :

$$\chi_{12}^c = (1/m_1^{0.5} + 1/m_2^{0.5})^2/2 \quad (4)$$

From eq. (4) and the degree of polymerization of PC and PET used in this study, we obtained $\chi_{12}^c = 0.015$. Based on these values of χ_{12} and χ_{12}^c , it is assumed that melt-mixed PC/PET blends in this study are immiscible for all the compositions.

3.2 FTIR ATR

Figure 1 shows representative expanded ATR spectra from 1820 to 1650 cm^{-1} of PC/PET=70/30 and PC/PET=30/70 surfaces. KRS-5 45° and Ge 45° crystals were used. Two characteristic absorption bands of the carbonyl groups of PC (-O-CO-O-) and PET (-CO-O-) were observed in this region. For PC and PET homopolymers using a KRS-5 45° crystal, these bands were observed at 1769 and 1717 cm^{-1} , respectively, and no other peak was found in this region^{16), 17)}. In the case of PC/PET=70/30, the carbonyl stretching bands were found at 1771 and 1720 cm^{-1} using KRS-5 45° and shifted to 1775 and 1726 cm^{-1} using Ge 45°, respectively. In the case of PC/PET=30/70, the carbonyl stretching bands were found at 1774 and 1715 cm^{-1} using KRS-5 45° and shifted to 1775 and 1723 cm^{-1} using Ge 45°, respectively. It was therefore proved that the frequency of the carbonyl stretching bands for PC/PET blends varies with the blend composition and the ATR crystal. The absorbance of the ATR spectra is well known to be obtained as a function of a refractive index of a sample. Jones *et al.*¹⁸⁾ showed that the values of the refractive index (n_2) for polymer films were dispersed depending on the values of the absorption index (k). The anomalous dispersion for n_2 values should be responsible for the change of the substantial ATR spectra and the peak shifts, etc. To avoid the effects of the anomalous dispersion, an ATR crystal whose refractive index and facecut angle are larger than those of other crystals is preferred to be used. Unfortunately, the k value for PET has not been reported, but the k value for PC at 1774 cm^{-1} was found to be 0.277, which may be rather low compared to 0.727 at 1194 cm^{-1} ¹⁸⁾. This result implies that the effect of the anomalous dispersion of n_2 for PC on the ATR spectra in this region is supposed to be very little and/or negligible.

As is evident from Figure 1, the spectra of PC/PET blends showed two peaks which correspond to that of the respective polymer components, and no other peak was found in

the region due to aromatic esters and aromatic aliphatic carbonates. This result means that the transesterification in these blends did not take place^{9),19)}. And this was confirmed over the whole composition range studied in this blend system.

A plot of the frequencies of the carbonyl bands of (a) PC and (b) PET against the PET weight fraction is shown in Figure 2. The peak positions obtained are averages on at least 3 samples prepared independently. With respect to ATR spectroscopy, the depth of ir beam penetration d_p has been estimated²⁰⁾ as a function of a wave length λ and a refractive index of an ATR crystal (n_1) and a sample (n_2). According to the definition, we obtain ir spectra in the outer (shallow) surface layer using a Ge 45° crystal, while those in the inner (deep; the order of a few μm) surface layer are obtained using a KRS-5 45° crystal.

Figure 2 shows that the peak position of the aromatic carbonate band for PC shifted to higher frequencies with increasing PET weight fraction. It is also shown that the peak position of the carbonyl stretching of the aliphatic ester band for PET shifted to higher frequencies with increasing PC weight fraction. These shifts are considered as an evidence of intermolecular interactions. Several FTIR studies of polymer blends have revealed the characterization of specific intermolecular interactions and conformational changes in blend systems. Coleman *et al.*²¹⁾ reported that in the amorphous poly (vinyl chloride) (PVC)/aliphatic polyester blends, the carbonyl band ascribed to the polyester shifted to lower frequency and broadened in the presence of an increasing concentration of PVC. This result was interpreted as evidence for a specific intermolecular interaction involving the carbonyl group. Based on further experimental results, they concluded²²⁾ that a hydrogen bonding type of interaction between the polyester carbonyl group and the methine proton of PVC exists in these blend systems. Pearce *et al.*²³⁾ manifested the hydrogen-bonding interaction between the substituted phenol condensation resins and poly(methyl methacrylate) by a shift of $\sim 25\text{ cm}^{-1}$ in the FTIR stretching frequency of the carbonyl group. Karasz *et al.*²⁴⁾ showed that for FTIR spectra of aromatic polybenzimidazole/aromatic polyimide (PBI/PI) blends, the frequency shifted up to 55 cm^{-1} in the N-H stretching band of PBI and up to 6 cm^{-1} in the carbonyl stretching bands of PI, depending on the blend composition. They also suggested that the miscibility of these blends derives from specific intermolecular interactions involving the $>\text{NH}$ and carbonyl groups.

In the case of a KRS-5 45° crystal for both PC and PET in Figure 2, the frequency varied in the range of about 20% to 50% of the PET weight fraction, i.e., the peak position of the carbonyl bands of both PC and PET shifted to higher frequencies with increasing the other component. It is well known that the self-association force holding PET molecules together is strong because of the molecular packing structure due to the intermolecular interaction between the main-chain methylene groups and carbonyl groups²⁵⁾. Consequently, these peak shifts are assumed to imply the decrease of self-association forces of each component polymer and therefore the improvement of phase separation.

The peak shift in the case of a Ge 45° crystal for both PC and PET was relatively small compared to a KRS-5 45° crystal. This result suggests that a self-association state of each component is relatively kept in the outer surface layer since the information in the outer surface layer is obtained using a Ge 45° crystal compared to a KRS-5 45° crystal on the basis of the d_p value, as mentioned above.

From these results on FTIR ATR, the domain structure in the surface layer of PC/PET blend films may be speculated. Thus, it can be presumed that the domain structure transformed in the range of ca. 20 to 50 wt% of PET because the frequency varied in this range, as mentioned above. Based on the peak shift data using a different ATR crystal, it was also suggested that the domain dimension is larger in the outer surface layer than in the inner surface layer of PC/PET blend films.

3.3 Surface Morphology

In order to obtain information on the domain structure in the surface layer of PC/PET blend films, we have also studied the morphology by STEM observations. Figure 3 shows STEM photographs of the microstructure in the surface layer of PC/PET blend films. The sample surface is observed at the bottom of these photographs. The dark portions in these photographs correspond to the domains composed of PET stained with osmium tetroxide. It is clear from these photographs that the change in domain structure depends on both the blend composition and the depth of PC/PET surfaces. The PET domains are spherical in the

Fig. 3 STEM photographs of the microstructure in the surface layer of PC/PET (a) 80/20, (b) 70/30, (c) 60/40, (d) 50/50, (e) 40/60, and (f) 30/70 (wt%) blend films. The sample surface is observed at the bottom of these photographs. The dark portions correspond to PET domains

Fig. 4 Schematic representation of cross sectional view of PC/PET blend films. Thin cross-section samples were analyzed by Micro FTIR through a mask window of $10 \times 30 \mu\text{m}$ area (dark portions) in both the outer bulk ($\text{ca. } 20 \mu\text{m}$ in depth from the surface) and the inner bulk ($\text{ca. } 50 \mu\text{m}$ in depth from the surface) layers

Fig. 5 Micro FTIR spectra of the carbonyl stretching bands in the outer bulk layer of PC/PET blend films as a function of the weight fraction

matrix of the PC component in the outer surface layer of PC/PET=80/20 and 70/30, while the PC domains are spherical in the matrix of the PET component in the outer surface layer of PC/PET=40/60 and 30/70. The PC domains are spherical in the matrix of the PET component in the outer surface layer of PC/PET=60/40, but lamella-like structure is also observed in the inner surface layer of the same sample. The PET domains are spherical in the matrix of the PC component in the outer surface layer of PC/PET=50/50, while the PC domains are spherical in the matrix of the PET component in the inner surface layer of the same sample. Thus the phase inversion and the disorder of the domain structure was observed in PC/PET=60/40 and 50/50 blends. This result should support our conclusion that the domain structure transformed in the range of $\text{ca. } 20$ to $50 \text{ wt}\%$ of PET. In PC/PET=80/20, 70/30, 40/60, and 30/70 blend films, the dimension of the spherical domains is larger in the

Fig. 6 A plot of the frequencies of the carbonyl bands of PET in both the outer bulk and the inner bulk layers for PC/PET blend films against the PET weight fraction

Fig. 7 STEM photographs of the microstructure in the bulk layer of PC/PET (a) 80/20, (b) 70/30, (c) 60/40, (d) 50/50, (e) 40/60, and (f) 30/70 (wt%) blend films. The upper layer and the lower layer of the photographs correspond to the outer bulk (*ca.* 20 μm in depth from the surface) layer and the inner bulk (*ca.* 50 μm in depth from the surface) layer, respectively

outer surface layer than in the inner surface layer. This result was in accord with our presumption from experimental results on FTIR ATR. However, it is somewhat difficult to account for the fact that the domain dimension increases along the vertical direction of the sample surface from the inner surface layer to the outer surface layer.

3.4 Micro FTIR

Schematic representation of cross sectional view of PC/PET blend films is shown in Figure 4. For Micro FTIR measurements, thin cross-section samples (*ca.* $0.5\ \mu\text{m}$ in thickness) were analyzed through a mask window of $10\times 30\ \mu\text{m}$ area (dark portions in Figure 4) in both the outer bulk (*ca.* $20\ \mu\text{m}$ in depth from the surface) and the inner bulk (*ca.* $50\ \mu\text{m}$ in depth from the surface) layers. Figure 5 shows Micro FTIR spectra from 1820 to $1680\ \text{cm}^{-1}$ of the carbonyl stretching bands in the outer bulk layer of PC/PET blend films as a function of the weight fraction. Two peaks which correspond to that of the respective polymer components were observed, and no other peak was found in the region due to aromatic esters and aromatic aliphatic carbonates, as well as ATR spectra obtained from PC/PET blend surfaces. This means that the transesterification in these blends did not take place^{9), 19)}. And this was confirmed over the whole composition range studied in this blend system. Moreover, the peak position of the carbonyl stretching of the aliphatic ester band for PET shifts to higher frequencies with increasing PC weight fraction, which is considered as another evidence of intermolecular interactions.

A plot of the frequencies of the carbonyl bands of PET in both the outer bulk and the inner bulk layers for PC/PET blend films against the PET weight fraction is shown in Figure 6. The peak positions obtained are averages on at least 3 samples prepared independently. The frequency was found to vary in the range of about 20% to 50% of the PET weight fraction. Thus the transformation of the domain structure, such as the phase inversion and the disorder of the microstructure, was supposed to occur as well.

The peak shift in the outer bulk layer was larger than that in the inner bulk layer. This result suggests that a self-association state of PET component is relatively kept in the inner bulk layer, that is, the dimension of the PET domains is larger in the inner bulk layer than in the outer bulk layer of PC/PET blend films.

These resulting Micro FTIR spectra of the bulk layer of PC/PET blend films suggest that the domain structure is transformed in the range of *ca.* 20 to 50 wt% of PET, and that the domain dimension is changed with the depth.

3.5 Bulk Morphology

Information on the domain structure in the bulk layer of PC/PET blend films was obtained from STEM observations of the morphology. Some of STEM photographs are shown in Figure 7. The dark portions in these photographs correspond to the domains composed of PET stained with osmium tetroxide. The upper layer and the lower layer of the photographs correspond to the outer bulk (*ca.* $20\ \mu\text{m}$ in depth from the surface) layer and the inner bulk (*ca.* $50\ \mu\text{m}$ in depth from the surface) layer, respectively. It is shown that the change in domain structure depends on both the blend composition and the depth of the bulk layer. The PET domains are spherical in the matrix of the PC component in the outer bulk layer of PC/PET=80/20 and 70/30, while the PC domains are spherical in the matrix of the PET component in the outer bulk layer of PC/PET=30/70, and are both spherical and cylindrical for PC/PET=40/60. In the case of PC/PET=60/40, spherical, cylindrical, and lamella-like structures of the PET domains are observed. In the outer bulk layer of PC/PET=50/50, spherical, cylindrical, and lamella-like structures of the PC domains are observed in the matrix of the PET component, while lamella-like structure is predominantly observed in the inner bulk layer. It is also proved that the domain dimension is larger in the inner bulk layer than in the outer bulk layer over the whole composition range studied. These results are consistent with our presumption from experimental results on Micro FTIR.

Figure 8 shows STEM photographs for the overall microstructure of (a) PC/PET=70/30, (b) PC/PET=40/60, and (c) PC/PET=30/70 (wt%) blend films. In Figure 8 (a) and (b), the central area corresponds to the center (as shown in Figure 4) of the cross section for the blend film. The domain dimension clearly varies from the outer bulk (*ca.* $20\ \mu\text{m}$ in depth from the surface) layer to the other outer bulk (*ca.* $20\ \mu\text{m}$ in depth from the other surface) layer through the inner bulk (*ca.* $50\ \mu\text{m}$ in depth from the surface) layer. Figure 8 (c) shows the overall change of the domain dimension in the surface layer and the bulk layer for PC/PET=30/70 blend films. Thus these STEM observations revealed that the

Fig. 8 STEM photographs for the overall microstructure of PC/PET (a) 70/30, (b) 40/60, and (c) 30/70 (wt%) blend films. The surface of PC/PET=30/70 is observed at the bottom of the photograph

domain dimension decreases along the vertical direction from the inner bulk layer to the outer bulk layer, while increases when approaching the outer surface layer.

4. Conclusions

It was proved by STEM observations that the morphology change of melt-mixed PC/PET blends could be presumed by the peak shifts of the carbonyl stretching bands for FTIR ATR and Micro FTIR spectra. Figure 9 illustrates schematic representation of the relationship between the morphology of the melt-mixed PC/PET blends and the observations by FTIR ATR, Micro FTIR, and STEM. As shown in Figure 9, it is concluded that the transformation of the domain structure, such as the phase inversion and the disorder of the microstructure, occurred in blend compositions less than 50 wt% of PET. The self-association force of PET component may be supposed to account for the transformation of the domain structure in the PC-rich region. It was also elucidated that the domain dimension decreases along the vertical direction from the inner bulk (*ca.* 50 μm in depth from the surface) layer to the outer bulk (*ca.* 20 μm in depth from the surface) layer, while increases when approaching the outer surface layer, as shown in Figure 9. This result may be somewhat surprising based on the values of the surface tension of two polymers, viz., $\gamma=42.9$ dyn/cm for PC and $\gamma=44.6$ dyn/cm for PET²⁶⁾. It is therefore considered that this morphological change results from the self-association force of PET component, molecular weight, melt viscosity, and the condition for the extrusion molding. It will be interesting to investigate to what extent the morphological change in depth exerts an influence on the surface characteristics and the mechanical properties of melt-mixed PC/PET blends.

Fig. 9 Schematic representation of the relationship between the morphology of melt-mixed PC/PET blend films and the observations by FTIR ATR, Micro FTIR, and STEM

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日本語要旨

溶融混練したビスフェノール A ポリカーボネート (PC)/ポリエチレンテレフタレート (PET) ブレンドのモルフォロジーを、フーリエ変換赤外全反射分光法 (FTIR ATR)、顕微鏡フーリエ変換赤外分光法 (顕微 FTIR) および走査透過型電子顕微鏡 (STEM) により解析した。FTIR ATR と顕微 FTIR 測定の結果、PC と PET に基づくカルボニル吸収ピーク位置は、PET 組成の約 20~50 wt% で変化した。すなわち、相分離構造のドメインサイズが、PC/PET ブレンドフィルム断面の深さに依存して変化するが示唆された。また、STEM 観察の結果からも、ドメインサイズがブレンドフィルム断面の内部から表面に近づくに従って小さくなり、表面近傍では逆に大きくなる傾向が認められた。また PET が 50 wt% 以下の組成で、相分離構造の反転や相構造の乱れが観察された。STEM により観察されたこれらの相分離構造は、FTIR ATR および顕微 FTIR 測定の結果から予想されたモルフォロジーと一致した。