Binary Blends of Poly(ether ether ketone) and Poly(ether imide). Miscibility, Crystallization Behavior, and Semicrystalline Morphology

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ABSTRACT: Miscibility and crystallization behavior of blends of poly(ether ether ketone) (PEEK) and poly(ether imide) (PEI) were examined. The blends exhibit miscibility in the amorphous state over the entire composition range. Crystallization of PEEK is slower in the blends than in the pure compound. Upon crystallization, PEEK segregates from the miscible amorphous state, causing a shift in the composition of the amorphous phase. PEI is found to be located outside the crystalline lamellae of PEEK. Correspondingly, the crystallinity of PEEK in the crystallized blends does not change largely with composition.

Introduction

Poly(ether ether ketone) (PEEK) and poly(ether imide) (PEI) are completely miscible in the amorphous state, as recently reported by Harris and Robeson[1,2] and Goodwin et al.[3] PEEK is well-known for its excellent mechanical properties, good environmental resistance, and high continuous-use temperature and is used in engineering applications.[4-8] It is especially suitable as matrix material for thermoplastic composites because of its good adhesion to glass and carbon fibers, ascribed to the formation of transcrystalline regions at the fiber surface.[9-14] PEEK is a semicrystalline thermoplastic polymer with a T_g of 145 °C and a melting point of the main crystallites around 340 °C, depending on the thermal history. PEI on the contrary is an amorphous polymer with a high T_g of 215 °C; it has a lower chemical resistance than PEEK, however, and cannot be used above its glass transition temperature.

Therefore, blending of PEEK and PEI seems an interesting route to combine the complementary properties of both polymers. Miscibility and morphology of PEEK/PEI blends are also interesting subjects of study because PEI may be used as a joining agent for PEEK and PEEK composite parts.[15,16] The present paper will address in detail the miscibility in the amorphous state, the crystallization behavior of PEEK, the segregation phenomena occurring upon crystallization of PEEK, and the semicrystalline morphology of these blends.

Experimental Section

Materials and Blending. Granular PEEK-grade Victrex 450G was purchased from ICI and used as received (after drying). PEI-grade Ultem 1000 was obtained from General Electric Plastics Europe. Blending was performed on a corotating twin-screw extruder ZSK at 370 °C, although the actual temperature of the melt at the die appeared to be much higher (around 440 °C). The strand leaving the extruder was quenched in a water bath, air dried, and chopped into granulate. Blends containing 25, 50, and 75 wt % PEEK were prepared. The blends were either used in granular form or subsequently injection molded into square plates (60 × 60 × 3 mm³); the injection-molding temperature was 380 °C. Prior to compounding and injection molding, all materials were dried overnight at 80 °C under vacuum.

Crystallization Experiments. Compounding and injection molding of the blends resulted in amorphous samples (as judged by their transparency and the crystallization exotherm that occurred in DSC scans), with the exception of pure PEEK, which was semicrystalline as received and crystallized too fast from the melt to obtain amorphous samples in injection molding. Amorphous strands of PEEK could be obtained, however, by extrusion and subsequent quenching in a water bath.

In order to study the influence of thermal history on the semicrystalline morphology and on the melting behavior of pure PEEK and the blends, both granular and injection-molded samples were isothermally crystallized in the temperature range of 180–320 °C. Samples were placed in a preheated oven (with an accuracy of approximately 1 °C); all samples were dried prior to annealing, and the annealing was carried out under nitrogen atmosphere for 6–7 h.

Some of the annealing procedures involve a relatively long exposure of the samples to high temperatures; therefore, thermogravimetric analysis (TGA) was performed. Exposure of a PEEK/PEI (75/25) blend to 300 °C under constant air flow for 20 h resulted in a weight loss of only 0.2%. Annealing times at the higher temperatures were considerably shorter, and annealing was done under nitrogen atmosphere. However, TGA would not give information about intermolecular processes, such as crosslinking, that might occur at these temperatures, so thermal degradation cannot be ruled out completely.

Sample Characterization Methods. Differential Scanning Calorimetry. Differential scanning calorimetry (DSC) was performed on a Perkin-Elmer DSC 7 for heating scans and on a Perkin-Elmer DSC 2 for isothermal measurements. Heating scans for the analysis of the melting behavior were carried out at a heating rate of 10 °C/min, unless indicated otherwise. Samples for isothermal crystallization measurements were heated from room temperature to the desired crystallization temperature at a heating rate of 200 °C/min for crystallization temperatures below 260 °C; for crystallization temperatures above 260 °C, the samples were first molten at 400 °C in the DSC cell, held at this temperature for 1 min, and subsequently cooled down to the crystallization temperature at a cooling rate of 200 °C/min. The measurement was started as soon as the heat flow in the DSC cell had stabilized.

Dynamic Mechanical Thermal Analysis. Dynamic mechanical thermal analysis (DMTA) was performed on a Polymer Labs DMTA in bending mode, usually in dual cantilever. DMTA temperature scans were made at one or multiple frequencies and at a heating rate of 2 °C/min; storage and loss moduli (E' and E'') were recorded. The temperature at which the maximum in the tan δ (tan $\delta = E''/E'$) versus temperature curve occurred was regarded as the glass transition temperature.

Small-Angle X-ray Scattering. Small-angle X-ray scattering (SAXS) was used to determine the periodicity of crystalline lamellae in isothermally crystallized samples. SAXS patterns were recorded on flat film in a Kratky camera (slit collimation), using Cu Kα radiation. In order to minimize air scattering, the camera was kept under reduced pressure. The smeared SAXS intensity was quantitatively recorded from the developed film by using an optical densitometer. After background subtraction, the intensity profile was desmeared by using the program FFSAXS, created by C. G. Vonk,[17] and Lorentz corrected. Thus the final

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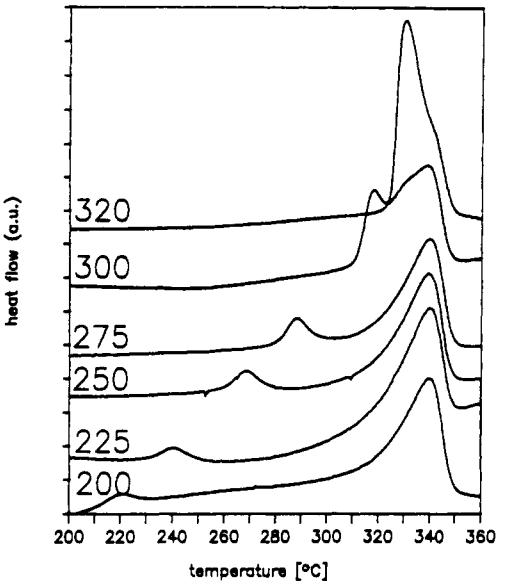


Figure 1. DSC traces for PEEK isothermally crystallized at indicated temperatures. Heating rate 10 °C/min. For easy comparison all curves are normalized to 1 mg of sample.

curve of $I(s)\,s^2$ versus s was obtained, $I(s)$ being the desmeared intensity and s the scattering vector in reciprocal space ($s = 2 \sin(\theta)/\lambda$). The long period L was determined by applying Bragg's law to the maximum of the desmeared and Lorentz-corrected SAXS curve. In some cases it was found convenient to apply a moving-average smoothing procedure to the desmeared and Lorentz-corrected scattering profile.

Results and Discussion

Melting Behavior of Pure PEEK. Because pure PEEK displays complicated melting behavior, which also occurs in the crystallized blends with PEI, it is helpful to discuss pure PEEK first. Typical DSC traces of PEEK, isothermally crystallized at different temperatures, are shown in Figure 1. PEEK is seen to exhibit two melting endotherms; the first endotherm is strongly dependent on the crystallization temperature, and the second one appears at approximately 340 °C and remains unaffected by the crystallization conditions.

The melting behavior of PEEK has extensively been described in the literature.[5,18-27] Lee and Porter[18,19] and Blundell and Osborn[20] attribute the double-melting behavior to partial melting and recrystallization of the lower melting lamellae during the DSC scan. Bassett, Olley, and Al Raheil[21] conclude from DSC and electron microscopy experiments that the higher melting lamellae are formed prior to the lower melting crystallites that lay between the primary lamellae. Cebe and Hong[27] support this conclusion. Cheng, Cao, and Wunderlich[22] also find that the higher melting crystal population crystallizes first; they also mark that the lower melting crystallites reorganize to some extent during the DSC scan and that they eventually can become part of the higher melting crystals at high temperature. Kenny et al.,[23] finally, describe the double-melting behavior in terms of a gelation process.

In parts a and b of Figure 2 DSC traces of isothermally crystallized PEEK are shown for different heating rates. For the samples crystallized at 225 °C, the area under the low-temperature peak is seen to increase slightly with respect to the area under the high-temperature peak, as the heating rate increases. For the samples crystallized at 300 °C, the lower and higher melting endotherms overlap too much to determine their respective enthalpies properly, but a similar trend is observed; the ratio of enthalpies of the low- and high-temperature peak increases substantially with DSC scanning rate. These observations are indicative

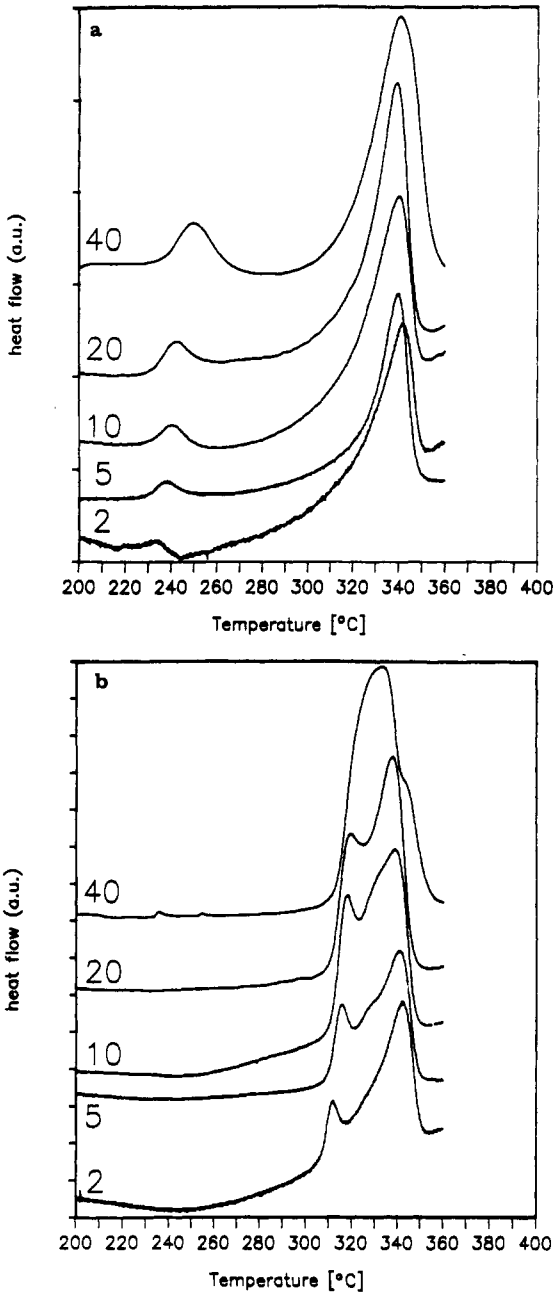


Figure 2. DSC traces of PEEK, isothermally crystallized at 225 °C (a) and 300 °C (b), at indicated heating rates (in °C/min).

of the occurrence of a recrystallization process, although it cannot be concluded exclusively from these results that continuous melting and reorganization of lamellar crystals account for the entire double-melting behavior as observed. Lee and Porter[18,19] found a similar effect of the heating rate on the heat of fusion of the two melting peaks, although the recrystallization seems to be much faster in our samples.

PEEK/PEI Miscibility in the Amorphous State. All extruded and injection-molded PEEK/PEI blends (with exception of injection-molded pure PEEK) were transparent, as would be expected for an amorphous, miscible system. Figure 3 shows tan δ versus temperature curves for injection-molded samples of different blend composition. In Figure 4 the T_g of the amorphous PEEK/PEI blends measured both with DSC and DMTA (frequency 3 Hz) is plotted as a function of blend composition. The solid line represents the Fox equation

$$1/T_g = w_1/T_{g1} + w_2/T_{g2} \qquad (1)$$

where w_1 and w_2 represent the weight fractions of the blend

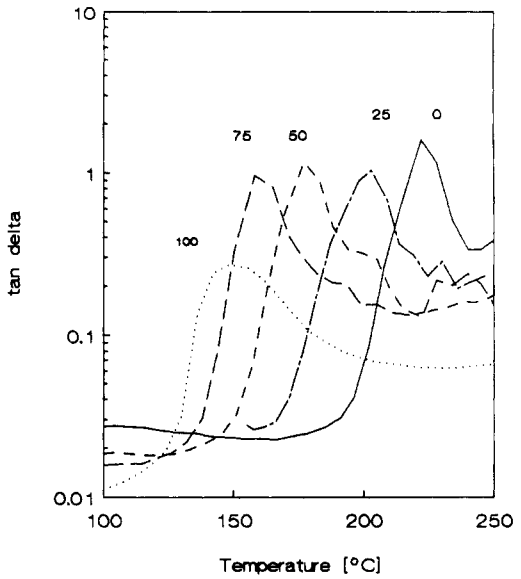


Figure 3. tan δ versus temperature curves of PEEK/PEI blends measured in DMTA at a heating rate of 2 °C/min and a frequency of 3 Hz (wt % PEEK indicated in the figure).

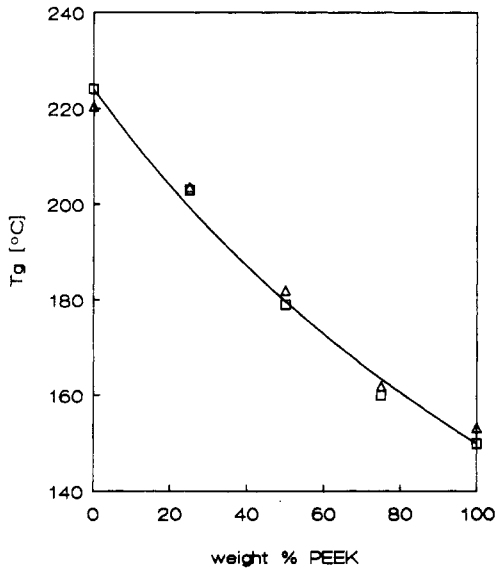


Figure 4. T_g of PEEK/PEI blends versus composition: (□) DMTA (conditions as in figure 3), (△) DSC with heating rate 10 °C/min. Drawn line represents the Fox equation.

constituents and T_{g1} and T_{g2} their respective glass transition temperatures. The single T_g that is observed for the samples indicates that PEEK and PEI are miscible in the amorphous state over the entire composition range and that the blend T_g obeys the Fox equation within experimental accuracy. The DSC and DMTA results are in satisfactory agreement (note that the heating rate was 10 °C/min for the DSC scans and 2 °C/min for the DMTA runs).

Crystallization Kinetics of PEEK in the PEEK/PEI Blends. Half-value times for isothermal crystallization of amorphous PEEK/PEI blends, obtained from isothermal DSC measurements, are plotted versus crystallization temperature in Figure 5. For the 75/25 PEEK/PEI blend and pure PEEK, crystallization half-value times at temperatures below 270 and 290 °C, respectively, could not be determined because crystallization had started before thermal equilibrium could be reached in the DSC cell. The isothermal crystallization curves are comparable to those reported by Harris and Robeson.[2] The existence of a maximum crystallization rate for pure PEEK and for the blends can be explained in terms of competition

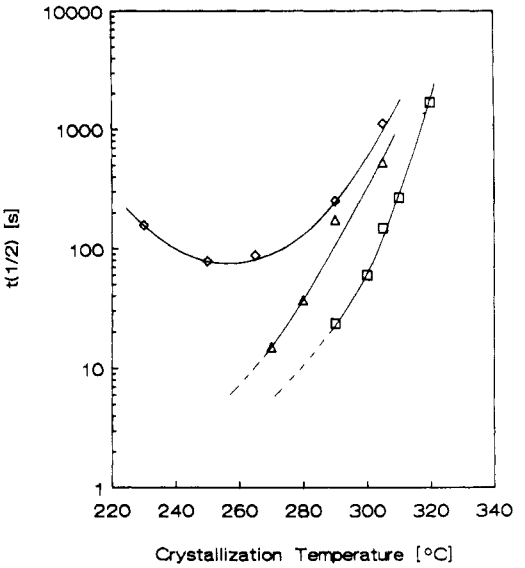


Figure 5. Isothermal crystallization half-value times ($t_{1/2}$) of amorphous PEEK/PEI blends in DSC versus crystallization temperature: (□) PEEK, (△) PEEK/PEI (75/25), (◇) PEEK/PEI (50/50).

between diffusion-controlled and secondary nucleation controlled crystal growth.[28] As can be seen from Figure 5, the crystallization rate of PEEK is substantially reduced by the addition of PEI. A practical example of this is that pure PEEK could not be injection molded completely amorphous (into 60 × 60 × 3 mm^3 plaques), since crystallization was faster than cooling in the mold. Blends containing 25 or more wt % PEI could easily be injection molded to yield completely transparent (and thus amorphous) specimens. So blending of PEEK with PEI appears to be a useful tool to control the crystallization rate of PEEK.

Blend Morphology, Melting Behavior, and PEEK Crystallinity in Crystallized Blends. DSC heating traces of PEEK/PEI blends isothermally crystallized at different temperatures are shown in Figure 6. The double-melting behavior observed for pure PEEK is seen to occur in the blends in quite a similar fashion as in pure PEEK. Remarkable is that for the blends isothermally crystallized at lower temperatures (i.e., up to 250 °C): a strong exotherm is observed between the two melting endotherms. The occurrence of this exotherm unambiguously indicates reorganization of crystals during the DSC scan. The effect is stronger for blends with higher PEI content, suggesting that the reorganization of PEEK crystals is hindered by the presence of PEI. The peak position of the melting endotherms of the isothermally crystallized blends is plotted in Figure 7 versus the crystallization temperature for different blend compositions. No significant changes of melting temperatures are observed with respect to pure PEEK. The lower melting peak for the PEEK/PEI (25/75) blends could not be determined because of the very strong appearance of the recrystallization exotherm discussed above. Basically, Figure 7 shows that the melting behavior of PEEK remains merely unaffected by the amount of PEI present in the blend, indicating that the lamellar thickness, which is known to determine the melting point of the crystallites,[28] is independent of blend composition. Figure 7 is in good agreement with the melting temperatures reported by Lee and Porter for pure PEEK.[18]

Normalized PEEK crystallinity data estimated from the DSC scans of samples isothermally crystallized at 300 and 320 °C are given in Table I. The crystallinity of the blends that were isothermally crystallized at lower tem-

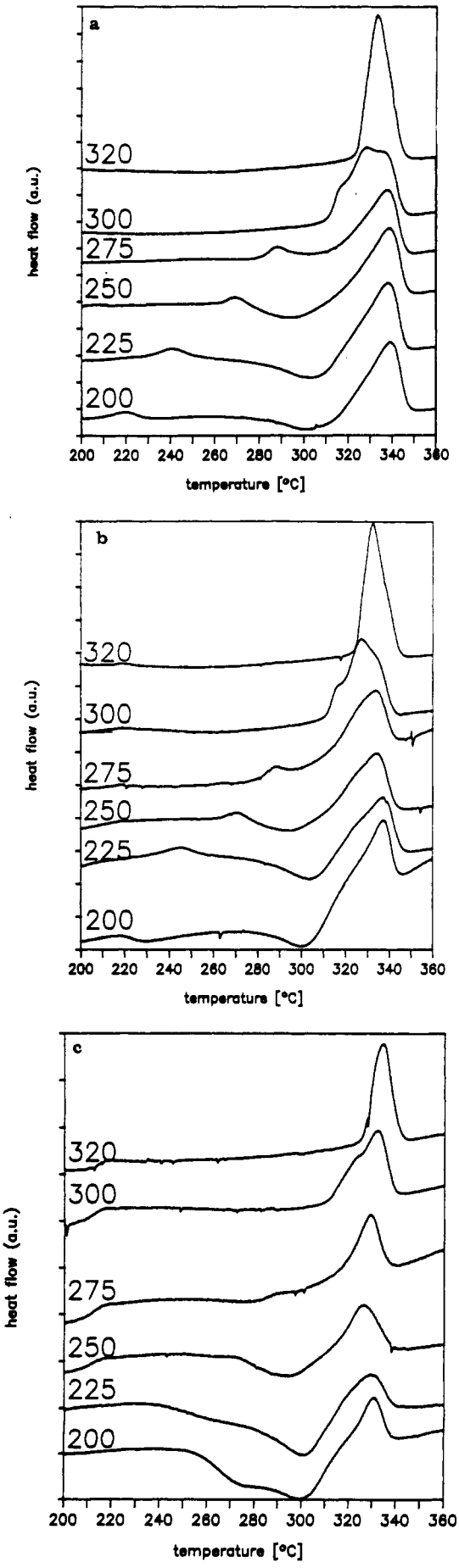


Figure 6. DSC traces for PEEK/PEI (75/25) (a), (50/50) (b), and (25/75) (c) blends isothermally crystallized at indicated temperatures. For easy comparison all curves are normalized to 1 mg of sample.

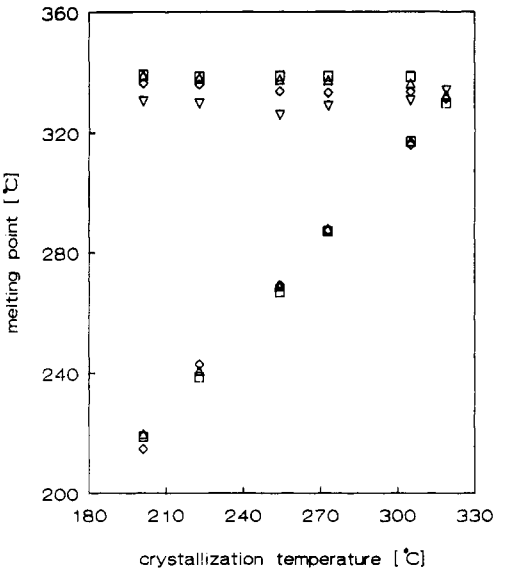


Figure 7. Temperatures of DSC upper and lower melting endotherms for isothermally crystallized PEEK/PEI blends versus crystallization temperature: (□) PEEK, (△) PEEK/PEI (75/25), (◇) PEEK/PEI (50/50), (▽) PEEK/PEI (25/75).

Table I
Normalized Heat of Fusion ($\Delta H/w_1$) and Crystallinity X_c of PEEK in PEEK/PEI Blends Isothermally Crystallized at 300 and 320 °C, Estimated from DSC Heating Scans

	T_c = 300 °C		T_c = 320 °C	
PEEK, wt %	$\Delta H/w_1$, J·g^{-1}	X_c	$\Delta H/w_1$, J·g^{-1}	X_c
25	55.6	0.43		
50	53.7	0.41	55.0	0.42
75	50.5	0.39	52.5	0.40
100	47.9	0.37	49.1	0.38

peratures could not be determined, because the recrystallization exotherm makes a reliable integration of the melting peak impossible. For the calculation of the crystallinity, a heat of fusion of 100% crystalline PEEK of 130 J·g^{-1} [20] was used. As can be seen from Table I, the crystallinity slightly increases with PEI content, and the crystallinity of the blends annealed at 320 °C is somewhat higher than that of the blends crystallized at 300 °C. It should be noted, however, that the accuracy of the crystallinity data is not expected to be better than a few percent. Harris and Robeson[2] found that the crystallinity of PEEK/PEI blends isothermally crystallized at 300 °C was between 0.31 and 0.36 and exhibited a maximum at a composition of 60–70 wt % PEEK.

Because PEEK crystallizes, it must somehow separate from the miscible amorphous phase, resulting in an increased content of PEI in the remaining amorphous phase of the blend. In order to monitor this change in composition, the T_g of the isothermally crystallized samples was determined by using DMTA. tan δ versus temperature curves for different blend compositions of samples isothermally crystallized at 250 °C are shown in Figure 8. The shoulder in the tan δ curve for the PEEK/PEI(50/50) blend presumably originates from interlamellar amorphous PEEK. Figure 9 contains the glass transition temperatures of both amorphous and isothermally crystallized samples, as determined with DMTA, versus blend composition. The T_g is seen to have shifted substantially up to higher temperatures, indicating that the composition of the amorphous phase has changed indeed upon crystallization of PEEK. The isothermal crystallization temperature does not have a significant effect on the T_g and thus on the composition of the amorphous phase. It is remarkable that the T_g of crystallized blends containing up to 50 wt % PEI approximately equals that of pure PEI,

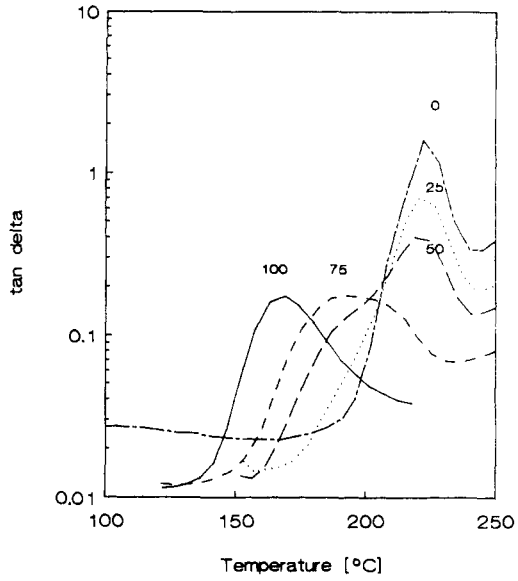


Figure 8. tan δ versus temperatures curves for PEEK/PEI blends isothermally crystallized at 250 °C. Heating rate 2 °C/min, frequency 3 Hz (wt % PEEK indicated in the figure).

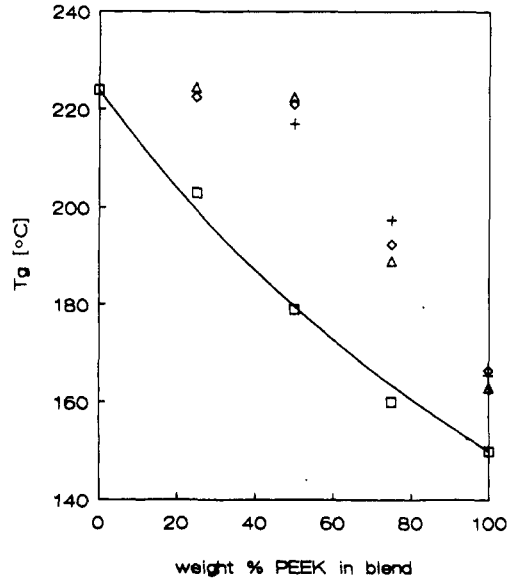


Figure 9. T_g as measured in DMTA (conditions as in Figure 8) for isothermally crystallized PEEK/PEI blends versus blend composition: (□) amorphous samples, (+) T_c = 220 °C, (◇) T_c = 250 °C, (△) T_c = 280 °C.

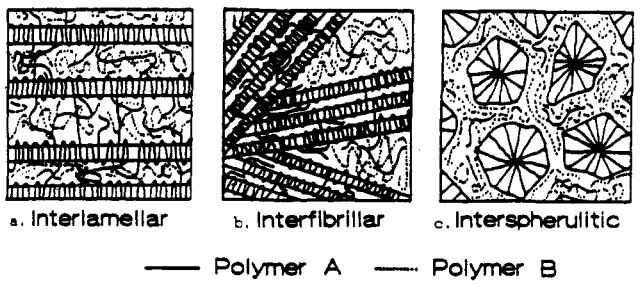
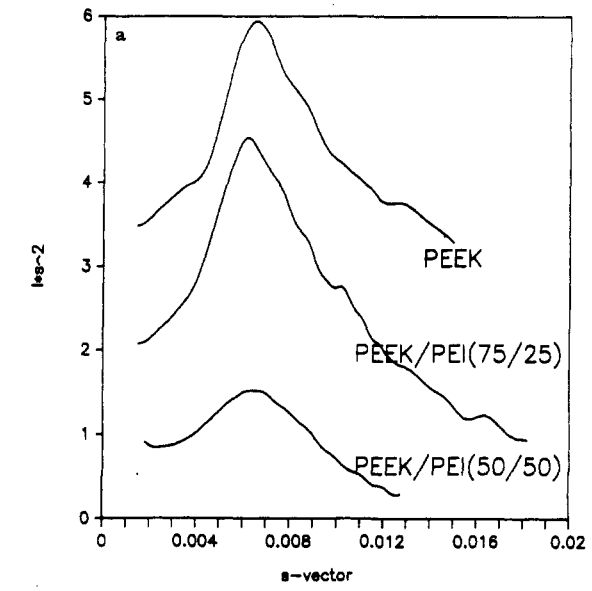


Figure 10. Possible modes of segregation in a binary blend that is miscible in the amorphous state and contains one crystallizing component.

suggesting that the PEI fraction is excluded almost completely upon crystallization of PEEK.

The T_g of pure PEEK has increased as well, as may be expected for a semicrystalline polymer. When PEEK crystallizes, molecules may be incorporated in one or more crystallites, resulting in a decreased mobility of the remaining amorphous parts of the chain. Therefore,



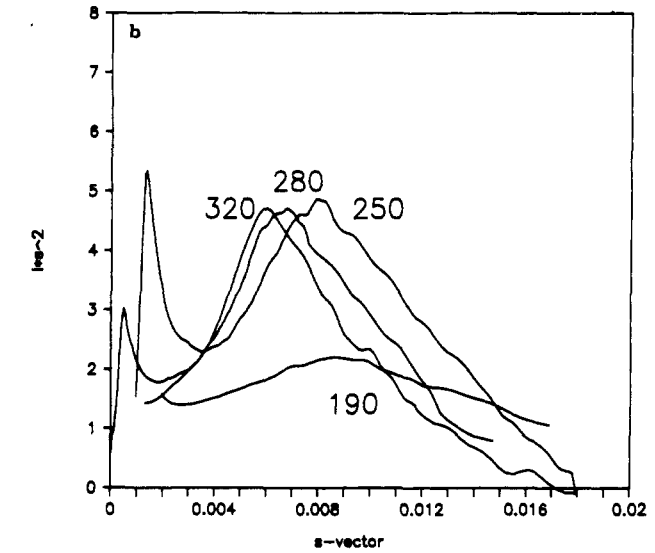


Figure 11. (a) Desmeared and Lorentz-corrected SAXS curves for PEEK/PEI blends isothermally crystallized at 320 °C. Blend compositions indicated in the figure. (b) SAXS curves for PEEK/PEI (75/25) blends isothermally crystallized at indicated temperatures.

molecular motion of the amorphous phase requires a higher energy, and the glass transition temperature raises. An increase in T_g of 10 °C has been reported for PEEK after isothermal crystallization at temperatures above 180 °C.[5]

One more question remains to be answered: how does the segregation of PEI during crystallization of PEEK affect the morphology of the blends? When PEI is rejected as PEEK crystallizes, there are basically three options: interlamellar segregation (i.e., PEI, eventually mixed with amorphous PEEK, is located between lamellae of crystalline PEEK), interfibrillar segregation (i.e., stacks consisting of crystalline PEEK lamellae with the amorphous PEI phase in between the lamellar stacks), or interspherulitic segregation (spherulites of crystalline PEEK in an amorphous matrix that mainly consist of PEI).[29,30] These segragation mechanisms are schematically illustrated in Figure 10. Diffusion and segregation phenomena during crystallization in miscible blends containing a crystallizable and an amorphous polymer have recently been studied in our laboratory.[30] Since the lamellar thickness is not expected to depend on the blend composition (DSC melting points are independent of composition), interlamellar segregation (Figure 10a) of PEI would result in an increase of periodicity (i.e., the sum of the lamellar

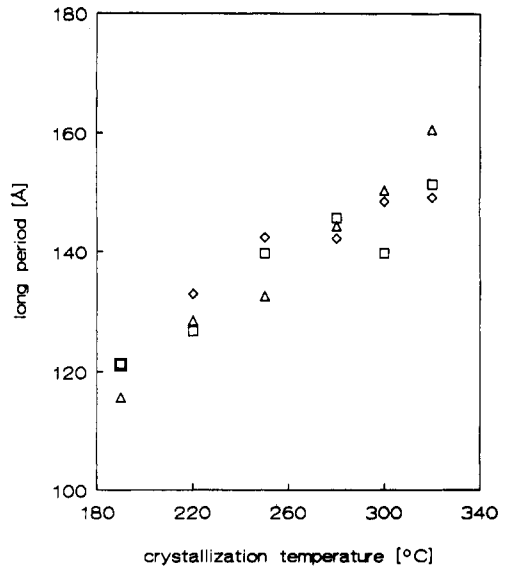


Figure 12. SAXS long period versus crystallization temperature: (□) PEEK, (△) PEEK/PEI (75/25), (◇) PEEK/PEI (50/50).

thickness and of the amorphous layer between the lamellae) with increasing PEI content. Desmeared and Lorentz-corrected SAXS curves of PEEK/PEI blends isothermally crystallized at 320 °C are depicted in Figure 11a. Figure 11b shows the SAXS patterns for the PEEK/PEI (75/25) blend isothermally crystallized at different temperatures. The long spacing, as deduced from the SAXS maximum, is plotted in Figure 12 as a function of crystallization temperature, for different blend compositions. From these results it is unambiguously concluded that interlamellar segregation does not occur in the PEEK/PEI blends, since no change in the long period with blend composition is observed. The long period increases with increasing crystallization temperature; this is in agreement with the increase in lamellar thickness as suggested from Figure 7.

Conclusions

Miscibility and crystallization behavior in blends of PEEK and PEI have been investigated. These blends are completely miscible in the amorphous state over the entire composition range. The blends exhibit a single glass transition temperature that obeys the Fox equation for miscible systems.

PEEK has the capability to crystallize, both in its pure form and in blends with PEI. The rate of crystallization of PEEK, however, is substantially lowered by the addition of PEI to the blend. Since PEEK crystallizes very fast, this may be of practical interest in controlling the crystallization kinetics of PEEK or its blends.

Upon crystallization of PEEK in the blends, phase segregation occurs, and for blends containing up to 50 wt % PEEK, this segregation is nearly complete (i.e., the segregated amorphous phase consists of almost pure PEI). PEI is segregated from the crystallizing PEEK outside the lamellae of crystalline PEEK. In the blends PEEK crystallizes in a very similar fashion as in its pure form. The same double-melting behavior, at least partly due to recrystallization during the DSC scan, is observed, and the peak temperatures of both the lower and upper melting endotherms remain unchanged for different PEI contents. PEEK crystallinity in the blends, estimated from the DSC scans, tends to be around 40% and does not change largely with blend composition.

All these findings lead to the conclusion that PEEK is segregated from the miscible amorphous phase, and crystallizes independently of it, in quite the same manner as in its pure state.

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