Phase Separation Behavior of Cyanate Ester Resin/Polysulfone Blends

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ABSTRACT: The composition of the blends and the curing temperature affect the morphology of the blends and the phase separation mechanism. The phase separation mechanism depends on the viscosity of medium at the initial stage of phase separation determined by the amount of thermoplastics and the curing temperature, and is closely related with the final morphology. When the homogeneous bisphenol A dicyanate (BADCy)/polysulfone (PSF) blends with low content of PSF (less than 10 wt %) were cured isothermally, the blends were phase separated by nucleation and growth (NG) mechanism to form the PSF particle structure. On the other hand, with more than 20 wt % of PSF content, the BADCy/PSF blends were phase separated by spinodal decomposition (SD) to form the BADCy particle structure. With about 15 wt % of PSF content, the blends were phase separated by SD and then NG to form a combined structure having both the PSF particle structure and the BADCy particle structure. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 74: 33–45, 1999

Key words: phase separation; cyanate ester; polysulfone; viscosity

INTRODUCTION

Cyanate ester resins are currently attracting attention as an important thermoset material for encapsulants of electronic devices, high-temperature adhesives, and structural materials of aerospace because they have excellent mechanical, thermal, and adhesive properties.[1–8] The cyanate ester monomers undergo polycyclotrimerization to form highly crosslinked polycyanurates.[6–8] However, the inherent brittleness limits their use in high-performance applications.

To toughen the highly crosslinked thermosets many researchers have employed rubber toughening without success, because the yielding of thermoset matrices cannot be induced owing to their highly crosslinked structures.[9–12] Therefore, toughening of highly crosslinked thermosets was explored by physical blending with thermoplastics such as poly(ether imide) (PEI),[13–15] poly(ether sulfone) (PES),[16,17] and polysulfone (PSF).[18] Recently, McGrath et al.[19–21] investigated the toughening and morphology change of cyanate ester resin with reactive or nonreactive poly(arylene ether sulfone)s.

In thermoset/thermoplastic blending, the fracture toughness is determined by the morphology formed by the consequence of phase separation. The morphology of thermoset/thermoplastic blends

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is dependent on the amount of thermoplastic and the temperature of curing the thermoset. Thermoplastic particle structure in the thermoset matrix was obtained with about 10 parts per hundred resin (phr) of thermoplastic content and thermoset particle structure in the thermoplastic matrix was obtained with more than 20 phr of thermoplastic content. With 15–20 phr of thermoplastic content, the combined morphology of both thermoplastic particle and thermoset particle structure was obtained.[22–29]

Although the morphology is mainly determined by the amount of thermoplastic content, the curing temperature of the thermoset also affects the morphology of thermoset/thermoplastic blends. Because the curing temperature affects the rate of phase separation and curing reaction, phase separation and curing of thermosets are two competing factors determining the morphology of the thermoset/thermoplastic blends. The various elements that influence the thermodynamics and kinetics of phase separation during curing of thermosets need to be understood to achieve an optimum heterogeneous structure. Phase separation is induced by increasing the molecular weight of thermosets during curing, i.e., the miscibility of thermoset/thermoplastic blends is altered continuously because the phase diagram moves downward or upward, depending on the chemical structures of the blends with the progress of thermoset curing. Therefore, to control the morphology of the blends, the alternation of the phase diagram and the phase separation mechanism should be studied.

In this study, the phase-separation behavior of cyanate ester resin and polysulfone blends was investigated using the scanning electron microscope and light scattering, and the criterion to determine the morphology was examined.

EXPERIMENTAL

Materials

The bisphenol A dicyanate (BADCy) monomer with a purity of 99.9% as a crystalline powder was supplied by Ciba-Geigy under the trade name of AroCy B-10. The PSF was a commercial grade (Udel P-3500) of Rhone Poulenc in pellet form having an M_n of 8200, with a polydispersity of 5.6 determined by gel permeation chromatography and low-angle laser light scattering (GPC/

Bisphenol-A dicyanate (BADCy), AroCy-B10

Polysulfone (PSF), Udel P-3500

Figure 1   Chemical structures of BADCy and PSF.

LALLS). Figure 1 shows the chemical structures of BADCy and PSF.

Sample Preparation of BADCy/PSF Blends

PSF was dissolved in methylene chloride and mixed with BADCy at room temperature. The solution was heated in an oil bath for 1 h at 120–130°C to drive off most of the methylene chloride, and then the residual solvent and air bubble were removed under vacuum for 30 min above 130°C. The blended resin was poured in a Teflon-coated aluminum plate and then was cooled down to be solid at room temperature.

Conversion Measurement of BADCy Resins

Isothermal and dynamic DSC studies were conducted using a Perkin-Elmer DSC 7 to examine the conversion of BADCy resins. To obtain the relation of curing time and conversion for BADCy/PSF blends at a certain curing temperature, isothermal curing studies were performed by ramping the sample at 100°C/min from 100°C to the curing temperature, curing the BADCy for a certain period of time, and then quenching to room temperature. After the isothermal DSC studies of BADCy/PSF blends, dynamic DSC studies were performed from 50 to 400°C at 20°C/min to determine the residual heat of reaction. The cyanate group conversion, p, was calculated from the residual heat of reaction by the following equation:

$$p = 1 - (\Delta H_R / \Delta H_T)$$

where ΔH_T is the total heat of reaction for neat BADCy resin determined to be 790 J/g, and ΔH_R

is the residual heat of reaction for partially cured BADCy resin.

Phase-Separation Behavior

Phase-separation behavior of BADCy was investigated at three different curing temperatures of 200, 230, and 250°C during the cure process of BADCy by light scattering and a scanning electron microscope (SEM). The PSF content was varied as 10, 15, 20, and 30 parts per hundred resins (phr). To examine the morphology change, BADCy/PSF blends with various contents of PSF were cured for a given curing temperature and time, and then quenched into liquid nitrogen. SEM, Hitachi S-570, was used to observe the morphology of partially cured specimens fractured at the liquid nitrogen temperature. After the isothermal curing, the conversions of all partially cured samples were examined by the dynamic DSC. The cloud points and the phase-separation diagram of BADCy/PSF blends were measured by light scattering.

Viscosity Measurements

The viscosity changes of neat BADCy and BADCy/PSF blends during cure were measured at the curing temperatures of 200, 230, and 250°C by a Rheometrics Dynamic Spectrometer (RDS II) using 50-mm circular disks in an oscillating parallel plate with the rate of 10 radians/s. The conversion of all the samples was examined using a DSC after measuring the viscosity.

RESULTS AND DISCUSSION

Curing Kinetics of BADCy/PSF Blends

The melting temperature and the onset curing temperature of BADCy monomer are 79 and 330°C, respectively. Figure 2 shows that the conversions of neat BADCy and BADCy/PSF blends increased with the increase of reaction time at the curing temperature of 200 and 250°C. The cyclotrimerization of neat BADCy is autocatalytic reaction. The curing rate of BADCy/PSF blends is faster than that of neat BADCy, and increases slightly with the increase of PSF content. This means that the PSF acts as a catalyst for curing BADCy resins. It is widely recognized that the curing of BADCy resins are accelerated by the impurities such as water and phenolic com-

Figure 2 Conversion of BADCy versus time for BADCy/PSF blends at (a) 200°C and (b) 250°C isothermal curing: neat BADCy (●), and BADCy/PSF blends with 5 phr (○), 10 phr (▼), 15 phr (▽), 20 phr (■), and 30 phr (□) of PSF.

pounds. Therefore, the curing reaction of BADCy resins can be catalyzed by the phenolic compounds included in commercial PSF resin.

Morphology of BADCy/PSF Blends

Thermoplastic content and curing temperature are two main factors to determine the morphology of thermoset/thermoplastic blends.[13] Figure 3 shows the morphologies of BADCy/PSF blends with various PSF contents and curing tempera-

Figure 3 Effects of PSF content and curing temperature on the morphology of BADCy/PSF blends.

tures. In the case of the 250°C curing temperature, the morphology of blend is the PSF particle structure with 10 phr of PSF content, and changes to the combined structure containing both the PSF particle structure and the BADCy particle structure with 15 and 20 phr of PSF content, and the BADCy particle structure with 30 phr of PSF content. It is already reported that the morphology changes as above with the in-

crease of the thermoplastic content.[15,28–35] For the curing temperatures of 200 and 230°C, the morphologies of BADCy/PSF blends change to the BADCy particle structure with only 20 phr of PSF content, and the BADCy particle size is reduced with 30 phr of PSF. For the low curing temperature of 80°C, the combined structure is formed even with 10 phr of PSF content, and the BADCy particle structure are formed with more than 15

Figure 4 Scanning electron micrographs of BADCy/PSF(10 phr) blends at 200°C isothermal curing as the BADCy polymerizes: the conversion of BADCy is: (a) 0.45, (b) 0.55, (c) 0.58, (d) 0.60, (e) 0.62, and (f) 0.68.

phr of PSF content. From the morphologies of BADCy/PSF blends varying with PSF content and curing temperature, it is observed that the combined structure and the BADCy particle structure are formed with less of an amount of PSF by decreasing the curing temperature from 250 to 80°C, and the BADCy particle size is reduced with the increase of PSF content and the decrease of curing temperature. It appears that the growth of BADCy particle is constrained because the viscosity of BADCy/PSF blends increases with the increase of PSF content and the decrease of curing temperature.

PSF-Particle Structure

As the cyclotrimerization of BADCy proceeds and the molecular weight of BADCy resins increases, the miscibility between the BADCy resins and PSF decreases and then phase separation starts. Figure 4 shows the scanning electron micrographs of BADCy/PSF(10 phr) blends at 200°C isothermal curing as the BADCy polymerizes. Figure 5 is the schematic diagram of Figure 4, showing the nucleation and growth procedure of PSF particle. The PSF nuclei begins to be formed in the BADCy matrix with the BADCy conversion of 0.45, the PSF nuclei agglomerate to form spher-

Figure 5   Schematic diagrams of the nucleation and growth procedure of BADCy/PSF blend (10 phr) blends at 200°C isothermal curing; the conversion of BADCy is: (a) 0.45, (b) 0.55, (c) 0.58, (d) 0.60, (e) 0.62, and (f) 0.68.

ical structure with the BADCy conversion of 0.55. Then, the concentration of PSF in the PSF nuclei increases, as indicated in black with the BADCy conversion of 0.58, and the black PSF nuclei converge into one solid particle, as shown in Figure 5.

Because the BADCy/PSF(10 phr) blend is laid on *meta*-stable region in the phase diagram, the blend is phase separated through the nucleation and growth to form PSF particle structure. When the curing temperature is higher than 200°C, the nucleus is easily generated and the formed nuclei rapidly grow to become a PSF particle. However, with the low curing temperature of 80°C, the nucleus formation is difficult because the low viscosity of the blend retards the phase separation of PSF more than the curing reaction of BADCy: as the curing reactions proceed without phase separation, the BADCy/PSF(10 phr) blend is laid on unstable region and phase separated via spinodal decomposition to form a combined structure.

BADCy-Particle Structure

Figure 6 shows the scanning electron micrographs of BADCy/PSF(20 phr) blends at 200°C

isothermal curing. Figure 7 is the schematic diagram of Figure 6 showing the spinodal decomposition procedure of BADCy/PSF(20 phr) blends as the BADCy polymerizes: although the phase contrast of spinodal decomposition at the initial stage is weak, the morphology of Figure 6(a) appears to be the cocontinuous structure consisting of the continuous BADCy rich phase and the PSF rich phase, as shown in Figure 7(a). As the conversion of BADCy increases slightly from 0.46 to 0.48, the cocontinuous structure abruptly changes to the BADCy particle structure [Fig. 6(a) and Fig. 7(a)]. Because the viscosity of the BADCy rich phase is higher than that of the PSF rich phase at the conversion of 0.46, further phase separation induces the spherical domains of the BADCy rich phase and the matrix of the PSF rich phase. The spherical BADCy particle structure grows until the BADCy particles meet each other to form a honeycomb-like structure [Fig. 6(d) and Fig. 7(d)], then the shape of the BADCy particles changes to the spherical structure [Fig. 6(e) and Fig. 7(e)].

When the PSF content is more than 20 phr, the BADCy/PSF blends are laid on an unstable region

Figure 6 Scanning electron micrographs of BADCy/PSF(20 phr) blends at 200°C isothermal curing as the BADCy polymerizes; the conversion of BADCy is: (a) 0.46, (b) 0.48, (c) 0.50, (d) 0.52, (e) 0.57, and (f) 0.96.

in the phase diagram and the blends are phase separated through spinodal decomposition to form the BADCy particle structure.

Figure 8 shows the time-resolved light scattering profile for BADCy/PSF (20 phr) at 200°C isothermal curing. The scattered light intensity is shown as a function of the magnitude of scattering vector q defined by $q = (4\pi/\lambda)\sin(\theta/2)$, where λ is the wavelength of light in the specimen, and θ is the scattering angle between the incident and scattered ray. The intensity increases very fast within only 4 min, which is consistent with the SEM results: the phase separation begins at the conversion of 0.46, and ends at the conversion of 0.57 (Fig. 6). The locations of the maximum intensity decreases with curing time, i.e., the interparticle distance increases with the increase of curing of BADCy. The interparticle distance can be calculated from the maximum scattering angle by using Bragg's equation, and means the average distance between the same neighboring phases.

Figure 9 shows the time resolved light scattering profile for BADCy/PSF(30 phr) blends at 200°C isothermal curing. The maximum intensity increases exponentially with curing time [Fig.

Figure 7 Schematic diagrams of the spinodal decomposition procedure of BADCy/PSF(20 phr) blends at 200°C isothermal curing; the conversion of BADCy is: (a) 0.46, (b) 0.48, (c) 0.50, (d) 0.52, and (e) 0.57.

10(a)], and the interparticle distance calculated from the location of maximum intensity is constant at the initial curing time and then increases linearly after that [Fig. 10(b)]. These light-scattering experimental results clearly confirm that the phase separation mechanism is the spinodal decomposition, with more than 20 phr of PSF content.

Figure 8 Time-resolved light scattering profile for BADCy/PSF(20 phr) at 200°C isothermal cure.

Figure 9 Time-resolved light scattering profile for BADCy/PSF(30 phr) at 250°C isothermal cure.

Figure 10 Maximums of Intensity (a) and interparticle distance (b) versus curing time for BADCy/PSF(30 phr) during cure at 200°C.

Combined Structure

Figure 11 shows the scanning electron micrographs of BADCy/PSF(15 phr) blends at 200°C isothermal curing. Figure 12 is the schematic diagram of Figure 11 showing the combined structures of the thermoplastic particle structure and the thermoset particle structure. It appears that BADCy/PSF(15 phr) blends initially does not phase separate in the region of nucleation and growth (*meta*-stable region) because of the high viscosity of the blends and phase separate in the region of spinodal decomposition (unstable region) like the BADCy/PSF(20 phr) blends. As shown in Figure 12, the cocontinuous structure of the initial stage of spinodal decomposition agglomerates by macrocoalescence to reduce the surface energy, and forms an irregular-shaped large domain, larger than 100 μm. This different phase-separation behavior compared with BADCy/PSF(20 phr) blends (Fig. 7), i.e., formation of irregular-shaped large domain, comes from the lower viscosity of BADCy/PSF(15 phr) blends than that of BADCy/PSF(20 phr) blends. After formation of the large domain, each phase has a different concentration ratio of BADCy and PSF. The PSF-rich phase indicated by dark colors in Figure 12 is separated via a spinodal decomposition to be a BADCy particle structure, and the BADCy-rich phase (white matrix) is separated via nucleation and growth to be a PSF particle structure, as shown in Figures 11 and 12.

Figure 13 shows the time-resolved light scattering profile for BADCy/PSF(15 phr) blends at 200°C isothermal curing. Figure 14 shows the variation of maximum intensity and interparticle distance obtained form the time-resolved light scattering profile in Figure 13. The relative intensity increases as the phase separation proceeds. It is very clear that the phase separation occurs by two steps, as observed with SEM (Figs. 11 and 12). Initially, the interparticle distance increases from 3 to 12 μm, which corresponds to the formation of the large domain. In the second stage of phase separation, a new phase with a smaller interparticle distance generates, and the interparticle distance increases slightly, which corresponds to the formation of the BADCy particle structure in the PSF-rich domain.

Phase Diagram

In thermoset/thermoplastic blends, the miscibility between the thermoset and thermoplastic decreases as the curing of the thermoset increases. When the blends become metastable or unstable by curing of thermoset, the blends phase separate to form various structures such as thermoplastic particle structure, thermoset particle structure, combined structure, etc., depending on the thermoplastic content and the curing temperature. Therefore, the phase diagram of thermoset/thermoplastic blends is useful to predict the phase-separation behavior.

Figure 11 Scanning electron micrographs of BADCy/PSF(15 phr) blends at 200°C isothermal curing as the BADCy polymerizes: the conversion of BADCy is: (a) 0.49, (b) 0.54, (c) 0.57, and (d) 0.76.

Figure 15 shows the phase diagrams of BADCy/PSF blends cured isothermally at 200, 230, and 250°C. The phase diagrams were plotted by measuring the cloud point using a light-scattering apparatus. The extent of conversion of BADCy at the cloud point was investigated with the quenched blends in the liquid nitrogen by DSC. As the curing temperature increases, the extent of conversion of BADCy for the phase separation increases. This represents that the phase-separation behavior of the BADCy/PSF blends is the upper critical solution temperature (UCST) behavior, and the critical content of PSF is around 15 phr.

Viscosity

The viscosity was monitored by RDS as the curing reaction of the blends proceeds. The extent of conversion of BADCy was also measured simultaneously by DSC. In this way, the viscosity versus the extent of conversion of BADCy was obtained as shown in Figure 16.

From Figures 15 and 16, the viscosity of BADCy/PSF blends at the onset point of phase separation was obtained as shown in Table I. With less than 10 phr of PSF content the BADCy/PSF blends have low viscosity (less than 7 Pa · s) at the onset point of phase separation, and are

Figure 12 Schematic diagrams of the spinodal decomposition procedure of BADCy/ PSF(15 phr) blends at 200°C isothermal curing; the conversion of BADCy is: (a) 0.48, (b) 0.49, (c) 0.54, (d) 0.57, and (e) 0.76.

phase separated by nucleation and growth to form a PSF particle structure. With more than 20 phr of PSF content, the BADCy/PSF blends have high viscosity (more than 27 Pa · s) except the one (the BADCy/PSF blend with 20 phr of PSF content cured at 250°C), and are phase separated by spinodal decomposition to form a BADCy particle structure. However, when the BADCy/PSF blends have the intermediate viscosity between 7 and 27 Pa · s at the onset point of phase separation, the blends with about 15 phr of PSF content are initially phase separated by spinodal decomposition and followed by macrocoalescence, and then the second phase separation to form a combined structure.

From the results of Figure 3 and Table I, the viscosity of BADCy/PSF blends at the onset point of phase separation appears to be a critical parameter to determine the phase-separation mechanism and the cured structure. Therefore, the final morphology of BADCy/PSF blends determining the fracture toughness of the blends can be estimated by the viscosity of the blends, depending on the PSF content and the curing temperature.

CONCLUSIONS

The morphologies of BADCy/PSF blends depend on the content of PSF and the curing tempera-

Figure 13 Time-resolved light scattering profile for BADCy/PSF(15 phr) during cure at 200°C.

Figure 15 Phase diagrams of BADCy/PSF blends at the isothermal curing temperature: (●) 200°C, (■) 230°C, and (▲) 250°C.

and then macrocoalescence. As a second stage of phase separation, the BADCy particle structure is formed in the large irregular domain by spinodal decomposition, and the PSF particle structure is formed in the matrix by nucleation and growth. The different morphologies appear to come from the different viscosity of the BADCy/PSF blends

Figure 14 Maximums of intensity (a) and periodic distance (b) versus curing time for BADCy/PSF(15 phr) during cure at 200°C.

ture. With less than 10 phr of PSF content, the morphology of the blends is the PSF particle structure formed by nucleation and growth. With more than 20 phr of PSF content, the BADCy particle structure is formed by spinodal decomposition. With about 15 phr of PSF content, the morphology of the blends is the combined structure having both the PSF particle structure and the BADCy particle structure. First, a large irregular domain is formed by spinodal decomposition,

Figure 16 The viscosity versus the extent of conversion of BADCy for BADCy/PSF blends depending on the content of PSF at the curing temperature of 230°C: neat BADCy (●), BADCy/PSF(5 phr) blends (○), BADCy/PSF(10 phr) blends (▼), BADCy/PSF(15 phr) blends (▽), BADCy/PSF(20 phr) blends (■), and BADCy/PSF(30 phr) blends (□).

Table I   The Viscosity Values of BADCy/PSF Blends at the Onset Point of Phase Separation

Content of PSF (phr)	Curing Temperature (°C)		
	200	230	250
5	4.59	2.56	0.431
10	6.94	5.35	3.00
15	24.8	19.4	8.84
20	36.4	27.8	20.7
30	115	151	178

Unit: Pa · s.

at the onset point of phase separation, depending on the content of PSF and the curing temperature. When the viscosity of the BADCy/PSF blends is less than 7 Pa · s at the onset point of phase separation, the blends are phase separated by nucleation and growth to form a PSF particle structure. When the viscosity of blends is more than 27 Pa · s, the blends are phase separated by spinodal decomposition to form a BADCy particle structure. With the intermediate viscosity between 7 and 27 Pa · s, the combined structure is formed by a two-step processes of phase separation.

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