Static and Dynamic Mechanical Properties of Modified Bismaleimide and Cyanate Ester Interpenetrating Polymer Networks

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ABSTRACT: Interpenetrating polymer networks (IPNs) based on different ratios of modified bismaleimide (BMI) resin [BMI/2,2′-diallylbisphenol A (DBA)] and cyanate ester (CE) (b10) have been synthesized via prepolymerization followed by thermal curing. A systematic study of both static and dynamic mechanical properties of the cured BMI/DBA–CE IPN resin systems was conducted through flexural, impact testing, and dynamic mechanical analysis (DMA). The static mechanical investigation shows that the flexural strength, flexural strain at break, and impact strength of the cured BMI/DBA–CE IPN resin systems are relatively lower than that calculated by rule of mixture of two individuals: BMI/DBA and b10. However, the flexural moduli of the IPN resin systems have more consistent features compared to that calculated by rule of mixture. Single damping peaks are detected for the cured BMI/DBA–CE IPN resin systems, which suggests a substantial degree of interpenetration between two networks. The damping peaks of the cured BMI/DBA–CE IPN resin systems do tend to become broader with increasing concentration of BMI/DBA, whereas the intensity of damping peaks of the IPN resin systems decreases. The obtained results not only provide insight information about the characteristic structures of these BMI/DBA-–CE IPN resin systems, but also give guidelines for their applications. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 88: 2000–2006, 2003

Key words: bismaleimide; cyanate ester; interpenetrating networks; mechanical properties; thermodynamics

INTRODUCTION

Bismaleimide (BMI) resins are good candidates for high performance resin matrix due to their good thermal stability, good fire resistance, low water absorption, and good retention of mechanical properties at elevated temperatures, especially in hot/wet environments.[1–3] However, unmodified BMI resins suffer from brittleness and poor processability due to high crosslinking density after curing, poor solubility in ordinary solvents, and narrow processing window.[4–6] In addition, the relatively high dielectric constant and loss limit their applications in electronic industry. Mechanical properties have constantly been considered as a major concern in selecting modification method of BMI resins. Many conventional methods, which include rubber or thermoplastic modification, can improve the fracture toughness of BMI, but usually result in a significant decrease in modulus, yield strength, and glass transition temperature of the cured resin.[7,8] Therefore, it is highly desirable to exploit a new approach to modify BMI resins, which involves the formation of thermoset/thermoset blends, probably to form interpenetrating polymer network.

An interpenetrating polymer network (IPN), belonging to a unique class of polymer blends or alloys, consists of two or more distinct crosslinked polymer networks held together by permanent interpenetration. IPNs are attractive as they allow the combination, in network form, of two otherwise noncompatible polymers. This characteristic structure of IPNs may result in synergistic effect on properties, which is suggested to combine good properties of different polymers.[9–11]

Cyanate ester (CE) (b10) with inherent low dielectric constant and loss is proposed to modify the dielectric properties of BMI resins in order to broaden its application in microelectronic and aerospace industries. However, very high curing temperature (>250°C) is usually necessary to achieve crosslinking of CE b10 through cyclotrimerization of cyanate ester groups in the uncatalyzed system.[12] Previous studies showed that 2,2′-diallylbisphenol A (DBA) was one of the most effective modifiers of BMI resin to improve the toughness and processability while maintaining other good properties.[13,14] Another attractive point of DBA was their phenolic hydroxyl group, which is believed to have catalytic effect on the cyclotrimerization of cy-

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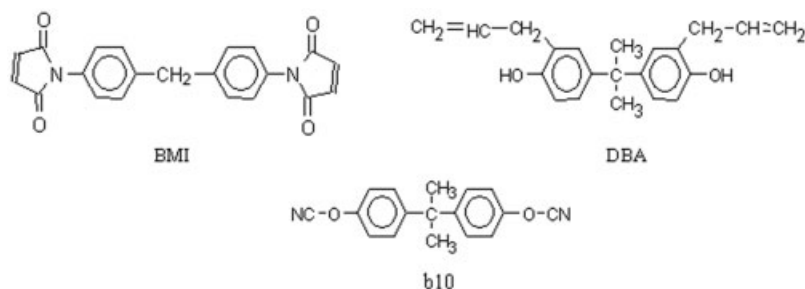
Scheme 1 Structure of raw materials.

anate ester groups.[15] Hence, it is very interesting to study the interpenetrating polymer networks based on different ratios of BMI/DBA and CE b10, in which DBA with unique dual functional groups may act as both modifier of BMI resin and the catalyst of CE b10. In these self-catalytic BMI/DBA–CE IPN resin systems, two discrete networks formed via different reactions interpenetrated or entangled with each other in the cured structure, which is expected to have the combined advantages of low dielectric constant and loss, high temperature resistance, good mechanical properties and processability.

In this study, IPNs based on different ratios of the modified BMI resin (BMI/DBA) and CE (b10) are prepared via prepolymerization followed by thermal curing. The static and dynamic mechanical properties of the cured BMI/DBA–CE IPN resin systems was systematically investigated through flexural testing, impact testing, and dynamic mechanical analysis (DMA).

EXPERIMENTAL

Materials

4,4'-Bismaleimidophenyl methane (BMI, 95%) and 2,2'-diallylbispbenol A (DBA, 85%) were obtained from Aldrich Chemicals, and were used without further purification. 2,2'-Bis(4-cyanatophenyl) isopropylidene (b10, 99%) was supplied by Ciba–Geigy of the United States and was used as received. The structure of raw materials was shown in Scheme 1. The detailed composition of BMI/DBA–CE IPN resin systems was listed in Table I.

TABLE I
BMI/DBA–CE IPN Resin Systems Formulation

Resin systems	Components	Ratio
Modified BMI (BMI/DBA)	BMI:DBA	Molar ratio = 2:1
bb72	BMI/DBA:b10	Weight ratio = 75:25
bb55	BMI/DBA:b10	Weight ratio = 50:50
bb27	BMI/DBA:b10	Weight ratio = 25:75

Prepolymers preparation

BMI and DBA with molar ratio (2:1) were mixed with stirring at 120 ∼ 150°C for 25 min to form a homogeneous mixture. Then appropriate CE b10 (according to the weight ratio stated in Table I) was added into the above mixture at 120°C and the final mixture was kept at 140°C for about 10 min before degassing. The obtained transparent, amber-colored prepolymers were stored in a freezer until use.

Preparation of the cured BMI/DBA–CE IPN resin systems

The prepolymers were degassed under vacuum (0.09 MPa) at 140°C for 10 min and poured into preheated (140°C) glass sheet molds (20 × 20 cm²). The following cure schedule was applied in air, i.e., 180°C/2h+200°C/2h+220°C/2h+250°C/5h.

Mechanical properties of the cured BMI/DBA–CE IPN resin systems

The flexural strength and modulus were measured on an Instron 5567 universal tester following the specification of ASTM D-790. The dimension of the samples was 80 × 8 × 4 mm. The span length was set to 40 mm and a 1 kN load cell was employed. A crosshead speed of 0.5 mm/min was used during the tests.

The un-notched Izod impact strength was measured in a Tinius Olsen Model 892 pendulum impact test machine. The samples with dimensions of 60 × 12 × 4 mm were used. DMA was performed using TA DMA 2980 Dynamic Mechanical Analyzer from room temperature to 400°C at a heating rate of 5°C/min at 1 Hz with a constant strain of 20 μm by single cantilever mode. The samples with dimensions of 17.5 × 12.5 × 2 mm were used.

RESULTS AND DISCUSSION

Figure 1 illustrates the typical stress–strain responses of the cured BMI/DBA–CE IPN resin systems. For the ease of comparison, the stress–strain curves of BMI/
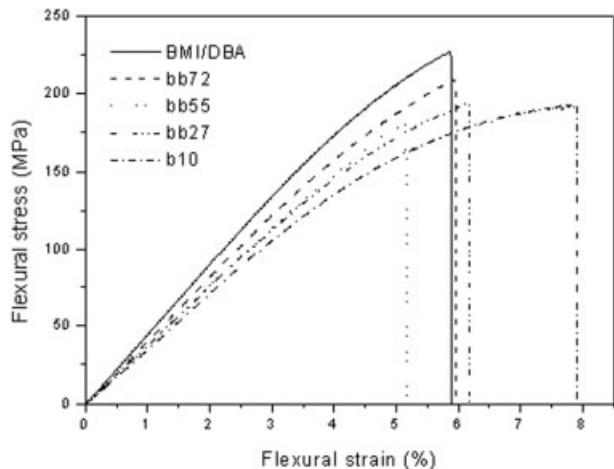
Figure 1 Effect of composition on flexural stress–strain behavior of BMI/DBA–CE IPN resin systems.

Figure 3 Effect of composition on flexural strain of BMI/DBA–CE IPN resin systems.

DBA and CE b10 are also presented in Figure 1. The composition effect on flexural strength, flexural strain at break and flexural modulus of BMI/DBA-CE IPN resin systems are summarized in Figures 2–4. From a series of plots, it can be seen that BMI/DBA has relative higher flexural strength, modulus, and lower strain at break compared to other cured resin systems, which are around 214 MPa, 4.33 GPa, and 5.58%, respectively. This could probably be correlated to the cured structure of the BMI/DBA resin. The curing reactions between BMI and DBA are proposed to take place through "Ene" and "Diels–Alder" reactions between allyl groups of DBA and maleimide groups of BMI, in which some portion of BMI self-polymerization occurred simultaneously.[16,17] All these reactions that occurred during curing belong to the addition reaction, and no by-products of low molecular weight are generated. Therefore, the cured resin structure in the final product is more compact and has fewer defects. Moreover, many aromatic and hybrid rings ex-
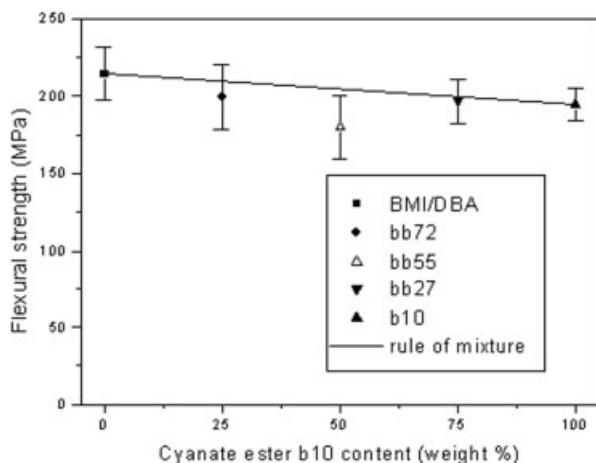
isted in the cured resin structure after curing. All these features of the cured resin can be responsible for the excellent mechanical properties of the BMI/DBA resin. The flexural properties of BMI/DBA obtained in this work is consistent with another modified BMI resin system reported by Stenzenberger,[18] which consists of commercial compimide 796 and 2,2-bis(3-allyl-4-hydroxyphenyl)hexafluoropropane, with flexural strength 214 MPa, modulus 4.6GPa, and strain at break of 5.64%, whereas the flexural properties of this modified BMI resin are much higher than that obtained by other modification methods, such as thermoplastic-[19] and rubber-modified BMI resin.[20]

However, it was noted that b10 exhibit relatively lower flexural strength, lower modulus and the highest flexural strain at break (194.5 MPa, 3.65 GPa, and 8.47%) among all the cured resin systems. CE (b10) can be cured to form triazine ring through the cyclotrimerization of cyanate ester functional groups (OCN). The crosslinking density of CE resin (b10) is substan-
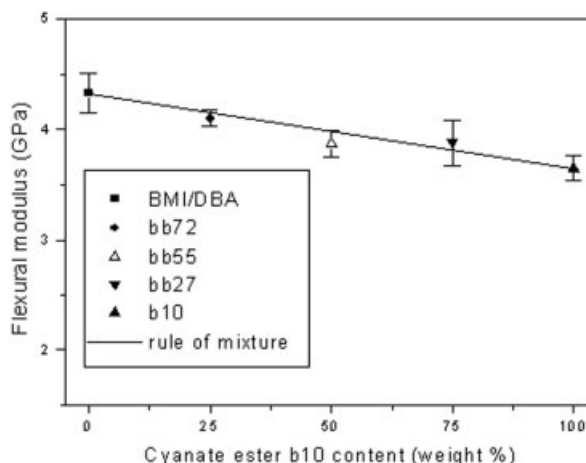

Figure 2 Effect of composition on flexural strength of BMI/DBA–CE IPN resin systems.

Figure 4 Effect of composition on flexural modulus of BMI/DBA–CE IPN resin systems.

tially lower than that of BMI resins and multifunctional epoxies—thus the polycyanurate networks are generally toughener than bismaleimides and epoxies.[21] Furthermore, the high percentage (67%) of —O— linkage between triazine rings leads to enhance the rotational freedom of the chain segments. All these can provide efficient toughening responses of the cured b10 resin.

The flexural properties of the cured BMI/DBA–CE IPN resin systems are clearly shown in Figures 2–4. The data calculated by rule of mixture are also shown in the corresponding graphs. It was found that these BMI/DBA–CE IPN resin systems basically displayed mechanical properties typical of rigid thermosets. BMI/DBA–CE IPN resin systems exhibited similar flexural properties and generally intermediate values between the two individual components, i.e., BMI/DBA and b10.

The flexural strength of bb27, bb55, and bb72 are 196, 179, and 199 MPa, respectively, which are slightly lower than that calculated by rule of mixture, especially for bb55. This could be explained by the structure of the cured BMI/DBA–CE IPN resin systems. An earlier report[22] on a heteronuclear nuclear magnetic resonance (NMR) study gave no evidence that the CE groups and maleimide groups would react to form chemical bond. In this work, BMI/DBA and CE b10 are believed to crosslink through different reactions to form two discrete networks, which interpenetrated or entangled with each other in the cured resin structure. The characteristic of such a reaction may result in two tendencies that affect the cured IPN resin structures and further flexural strength in opposite ways. On the one hand, IPNs may result in a unique crosslinked structure, which will enhance the flexural strength, due to two discrete networks interpenetrating or entangled with each other. On the other hand, there also exists an opposing effect on flexural strength due to the IPN structure. Generally, it is difficult for such IPN resin systems to attain the same level of curing as the pure resins due to a higher degree of networks interlocking and greater diffusion control effect during the curing process, especially at the end stage of curing. It is believed that in this case the lower flexural strength observed in BMI/DBA–CE IPN resin systems could be explained as due to domination of the latter effect, which shows its greatest effect on bb55. The flexural strain at break of bb27, bb55, and bb72 are 6.11%, 4.97%, and 5.85%, respectively, which are much lower than the corresponding data calculated by rule of mixture. This is presumably due to the cured resin structure resulting from domination of network interlocking and greater diffusion control effect during curing as well.

The flexural modulus of bb27, bb55, and bb72 are 3.88, 3.87, and 4.10 GPa, respectively, which are more consistent with the results calculated by rule of mix-
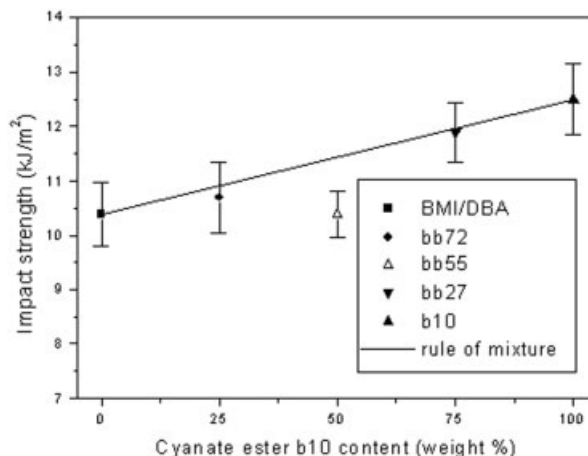

Figure 5 Effect of composition on impact strength of BMI/DBA–CE IPN resin systems.

ture. The modulus of polymeric materials in the rubbery and leathery states is directly related to the crosslinking density. However, in the glassy state, the resins are frozen to glass so that the cooperative motions of chain segments are prohibited. Consequently, the modulus is measured in the elastic region where the van der Waals attraction among the chain segments was affected. Thus, the modulus is not directly related to the crosslinking density but dependent on the packing density or concentration of the chain segments in the glassy state.[23] In these BMI/DBA–CE IPN resin systems, BMI/DBA and CE b10 are believed to crosslink through different reactions to form two discrete networks, which interpenetrated or entangled with each other in the cured resin structure. This characteristic of reaction may result in higher packing density in the cured IPN resin structure as compared to individual pure resin. It could probably compensate the aforementioned negative effect observed in flexural strength. Therefore, flexural modulus of the cured BMI/DBA–CE IPN resin systems shows more consistency with the results calculated by rule of mixture, even in bb55, which may have the highest degree of packing density due to most effective combination of both chemical crosslinking and physical entanglement.

Impact strength

The effect of composition on impact response of BMI/DBA–CE IPN resin systems and corresponding data calculated by rule of mixture are shown in Figure 5. From the observations, it can be seen that the impact strength of BMI/DBA–CE IPN resin systems also show lower values when compared with rule of mixture. This phenomenon is similar to some reports on other thermoset–thermoset IPNs, such as an interpenetrated system based on a tetrafunctional epoxy resin
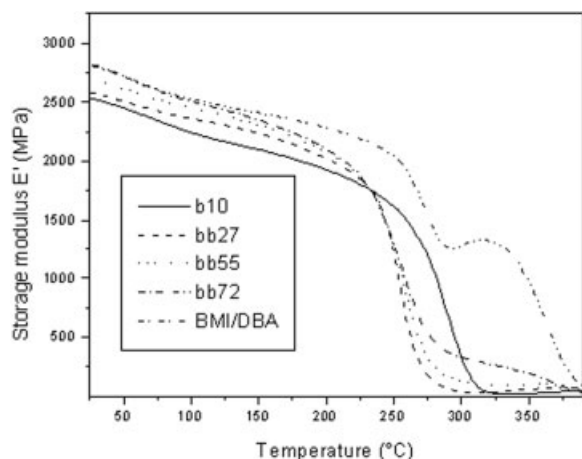
Figure 6 Overlay curves of storage modulus vs temperature of BMI/DBA–CE IPN resin systems.

and a bismaleimide,[24] in which the identical property (i.e., fracture toughness) shows a gradual decrease up to about 30% body weight of BMI. However, there are also other contradicting observations where simultaneous full-interpenetrating polymer networks of blocked polyurethane and vinyl ester exhibited higher impact strength when the blocked polyurethane content increased.[25] This indicated that there is no one particular general trend in toughness of IPN resin systems, as this would be dependent on the structure of various IPN resin systems, i.e., whether a system is based on a rigid–rigid or rigid–flexible network. The relative lower impact strength observed in these cured BMI/DBA–CE IPN resins was once again attributed to the difficulties for IPN resin systems of attaining the same level of curing as the pure resins.

Dynamic mechanical properties of the cured BMI/DBA–CE IPN resin systems

The overlay storage moduli (E') as a function of temperature for BMI/DBA–CE IPN resin systems are plotted in Figure 6. The corresponding overlay plot of tan δ vs temperature is shown in Figure 7. The results of glass transition temperature (T_g), the intensity and the half width of the damping peaks, the loss modulus peak value ,and loss modulus peak temperature of the cured BMI/DBA–CE IPN resin systems are listed in Table II. As for comparison, above information of BMI/DBA and pure b10 resin is also included.

From Figure 6, it can be seen that there are two-step decreases in storage modulus E' of the cured BMI/DBA resin, which correspond to the two damping peaks observed in tanδ vs. temperature plot (see Fig. 7). This observation is different from that reported in other allyl compounds modified BMI resins, in which only one T_g around 282–292°C was obtained for cured resins according to different backbone of allyl

compounds.[26,27] On the other hand, the glass transition temperature of the cured pure BMI resin has been reported to be about 360°C.[28] However, it should be noted that the molar ratios of BMI and allyl compounds in the first case are generally less than 2:1, which is different from the composition in this work (molar ratio BMI:DBA = 2:1). It is proposed that there exist multiple reactions during curing between BMI and DBA—for example, the "Ene" and "Diels–Alder" reaction between the maleimide group of BMI and the allyl group of DBA, and self-polymerization of BMI via the double bond addition of maleimide groups. Since a large molar ratio of BMI and DBA is used in this work, it is possible that more of the BMI component remained even after "Ene" and "Diels–Alder" reactions between BMI and DBA at lower temperature. This could result in more BMI self-polymerization occurring at higher temperature and thus higher portion of the corresponding network formed in the cured resin structure. Hence, the first damping peak of the cured BMI/DBA at the lower temperature around 288°C could be probably due to the T_g of the addition product resulting from "Ene" and "Diels–Alder" reactions between maleimide groups and allyl groups. And the second damping peak at about 367°C might be attributed to the homopolymerization of BMI and further crosslinking structure between BMI and DBA. The storage modulus of BMI/DBA is obviously higher than that of pure b10 and other IPN resin systems within the whole testing temperature range, and the intensity of damping peak shows the lowest value among all resin systems. This also implies very high crosslinking density and compact structure of the cured BMI/DBA resin.

The relatively low crosslinking density of b10 with the rotational freedom oxygen —O— linkage is responsible for its lower storage modulus in the initial testing temperature range. CE b10 undergoes glass
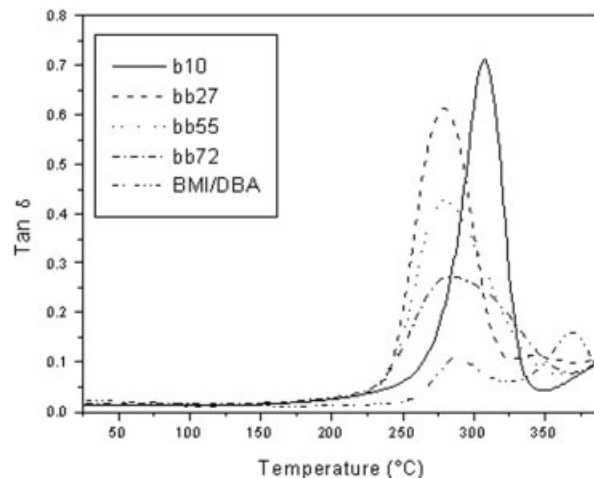

Figure 7 Overlay curves of tan δ vs. temperature of BMI/DBA–CE IPN resin systems.

TABLE II
Summary of Damping Peak Values of BMI/DBA–CE IPN Resin Systems

Resin systems	b10	bb27	bb55	bb72	BMI/DBA
tanδ peak intensity value	0.71	0.61	0.43	0.27	0.11
					0.16
tanδ peak half width (°C)	32.5	43.3	55.7	74.7	29.7
					30.8
T_g (°C)[a]	307.1	278.7	279.0	282.0	288.1
					367.5
Loss modulus Peak value (MPa)	235.5	225.4	202.2	171.1	139.5
					95.89
Loss modulus Peak temperature (°C)	286.9	258.8	259.3	261.5	286.0
					355.1

[a] T_g: glass transition temperature obtained using temperature of loss tangent peak.

transition at around 304°C, resulting in a much larger change in storage modulus E' as the material softens. It exhibits a very sharp and clear damping peak, which can be identified by a very narrow half width of tan δ peak around 32.5°C (see Table II). This is consistent with the tan δ peak shape observed in another report about CE (b10),[29] though it reported a much lower T_g around 289°C, which may be attributed to the different curing schedule used.

Single damping peaks are generally detected for all the BMI/DBA–CE IPN resin systems. The corresponding damping peak temperatures, which are taken as glass transition temperatures (T_g), are listed in Table II. IPN resin systems bb27, bb55, and bb72 exhibited T_g at 278, 279, and 282°C, respectively, with very little significant variation. It is known that in totally incompatible polymer blends or copolymers, each phase maintains the properties, e.g., T_g, of the pure homopolymer. As some molecular mixing occurs, whether because of altered thermodynamic equilibrium conditions or because of mechanical entrapment, the glass transitions shift inward between its individual T_g's and broaden.[25,27] It is unexpected that the observed glass transition temperatures of the cured BMI/DBA–CE IPN resin systems are lower than both two neat resins. This phenomenon could be attributed to a higher degree of networks interlocking and greater diffusion control effect resulting from two networks' interaction during the curing process, especially at the end stage of curing. Network interlock not only provides a sterically hindered environment for IPN curing reactions, but also may restrain chain mobility, especially in the network formed by rigid aromatic rings. Hence, the introduction of the b10 network into the BMI/DBA resin may hinder further stages of curing between BMI and DBA. This could explain the lower glass transition temperatures observed in these BMI/DBA–CE IPN resin systems. A similar network interlock phenomenon has been reported on IPNs based on unsaturated polyester and epoxy.[30] And the relatively lower glass transition temperatures of IPNs were observed in a recent work on

the sequential IPN of BMI and CE in polycyanate-rich blends,[31] though no explanation was given.

Only one damping peak observed in BMI/DBA–CE IPN resin systems can be interpreted by assuming that BMI/DBA and CE (b10) networks formed a substantial degree of interpenetration during curing. We recall that, before the curing process, the BMI/DBA–CE mixture is molecularly homogeneous. During curing, the molecules grow in size and the entropy contribution to the free energy of mixing decreases. Consequently, phase separation in these BMI/DBA–CE IPN resin systems tends to occur. However, the formation of interpenetration and entanglement between two discrete networks formed by BMI/DBA and CE (b10) respectively, particularly in a densely crosslinked network, may counter this tendency and essentially enhance phase homogeneity. The observed predominant single damping peaks in DMA spectrum indicate that these BMI/DBA–CE IPN resin systems are molecularly homogeneous at least up to the scale of the dynamic mechanical test. In other words, the fact that the T_g of the investigated samples is well defined and only slightly broadened indicates that the phase domains, if exist, are smaller than the size of the segments that are responsible for the primary molecular relaxation.

The shape of tan δ peak may be used as convenient indicator of the morphological state of the phases within IPNs. The broadness of a relaxation indicates the complexity of the IPN structure. Although only one damping peak was observed for these BMI/DBA–CE IPN resin systems, their tan δ peaks do tend to become broader with increasing BMI/DBA content, which is indicated by the increase in half width of tan δ damping peaks (see Table II). Such a phenomenon suggests an increasing possibility of the existence of more than one phase with increasing BMI/DBA concentration.

From the view of molecular structure, the intensity of the damping peak reflects the rigidity of materials. Figure 7 shows the overlay plot of the tan δ damping peak of BMI/DBA–CE IPN resin systems, in which

pure b10 and BMI/DBA resins are also included for comparison. It can be seen that the intensity of damping peaks of BMI/DBA–CE IPN resin systems decrease with increasing BMI/DBA concentration. This means that the greater the BMI/DBA content, the more rigid the IPN structure. The lowest damping peak intensity of BMI/DBA suggests that the BMI/DBA component in the cured resin may bring a strong restraining effect on IPN polymer network segments mobility. This restraint is enhanced with increasing the BMI/DBA weight fraction, which is also in accordance with the trend of the increment of storage modulus in Figure 6.

CONCLUSIONS

IPNs based on different ratios of modified BMI resin (BMI/DBA) and CE (b10) have been prepared via prepolymerization followed by thermal curing. A systematic study of both static and dynamic mechanical properties of the cured BMI/DBA–CE IPN resin systems was carried out through flexural testing, impact testing, and DMA. The flexural strength, flexural strain at break, and impact strength of the cured BMI/DBA–CE IPN resin systems is lower than that calculated by rule of mixture of two individuals: BMI/DBA and b10. It could be understood as IPN resin systems may not attain the same level of curing as the pure resins due to a higher degree of network interlocking and greater diffusion control effect during curing process, especially at the end stage of curing. However, the flexural moduli of BMI/DBA–CE IPN resin systems show more consistent features compared with that calculated by rule of mixture. This is because the elastic modulus measured in the glassy state relies more on the packing density or concentration of the chain segments in the cured resin structure.

The glass transition temperatures of the cured BMI/DBA–CE IPN resin systems show lower values as compared to two individuals, i.e., BMI/DBA and b10. This could be once again attributed to the influence of a higher degree of network interlocking and greater diffusion control effect resulting from two networks' interaction during the curing process, especially at the end stage of curing. Single damping peaks are generally observed in DMA spectrums of all BMI/DBA–CE IPN resin systems. This could be interpreted by assuming two networks in an IPN gain a substantial degree of interpenetration during curing. The shapes of tan δ damping peaks of BMI/DBA–CE IPN resin systems do tend to become broader with increasing the content of BMI/DBA, whereas the intensity of

damping peaks decreases. Such a phenomenon suggests an increasing trend of presence of more than one phase and more rigid IPN structure with increasing BMI/DBA concentration.

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