Synthesis and Properties for Interpenetrating Polymer Network of Modified Epoxy Resins

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ABSTRACT: The control epoxy resin, CE (i.e., without modified), containing naphthalene group was synthesized directly from 1,6-dihydroxynaphthalene with epichlorohydrin and a stoichiometric of NaOH. The MDI (4,4′-diphenylmethane diisocyanate)-modified epoxy resins, ME, were prepared from the direct process, MDI with CE. Subsequently, they were cured from various composition of ME/CE with 4,4′-diaminodiphenylsulfone (DDS) and tetramethyl bisphenol F dicyanate (TBFD) in equal equivalent, respectively. In addition, the cure reaction was studied through a cross-reference between in situ Fourier transform infrared and DSC (differential scanning calorimeter) dynamic scanning. The kinetic model gives a good description of curing paths. The modified epoxy systems had higher activation energy than that of control epoxy resin. The epoxy resins with 10 wt % ME-modified was found to obtain appropriate result in combination with optimum properties and content (e.g., thermal stability, glass-transition temperature, coefficient of thermal expansion, moisture absorption, dielectric constant, and modulus etc.), which may be attributed to the naphthalene, oxazolidinone structure, and crosslink density contributed to optimal state in polymer matrices. Furthermore, the intermolecular interaction hydrogen bonding of interpenetrating polymer networks. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 1872–1879, 2006

Key words: naphthalene group; oxazolidinone structure; modified epoxy resin; interpenetrating polymer networks

INTRODUCTION

Epoxy resins are used for many important applications such as encapsulation of electronic devices, high temperature adhesives, and structural aerospace materials since they have excellent mechanical, thermal, and adhesive properties.[1–9] The diglycidyl ether of bisphenol A, *o*-cresol formaldehyde novolac epoxy, and phenol formaldehyde novolac epoxy resins have been used widely in industry because of their ease of handling and processability, physical strength after curing, and cost advantage. Unfortunately, epoxy resins have relatively poor performance at high temperatures, have high dielectric constants, and exhibit significant water absorption. Moreover, the chemical resistance of epoxy resin is not as good as that of cynate esters. Cyanate ester resins have improved performance relative to conventionally cured epoxy resins. The main reaction pathway of dicyanate ester is identified as the cyclotrimerization of dicyanate groups to form a highly crosslinked polytriazine network. In general, adding cyanate ester resin component into the epoxy resin enhances the effective improvement in glass-transition temperature (T_g) and thermal stability.[10]

Kinetic behavior of thermoset resin is fundamental to understand structure/characterization/processing relationships for manufacturing and utilization of high performance composites. In recent years, some authors[11,12] have proposed a complex reaction mechanism for the polymerization of epoxy associated with cyanate ester resins. In this study, the MDI-modified epoxy resins, which form a higher concentration of oxazolidinone and structure epoxy resin networks, were achieved by coreaction with cyanate ester. The curing mechanism of epoxy resin and cyanate ester was investigated through a cross-reference of the *in situ* Fourier transform infrared (FTIR) and the DSC dynamic scanning results. In addition to curing kinetics, the curing rate, variation of rate at various temperatures, and activation energy are useful for the study of mixed system applications. These kinetic data provide information for the curing cycles of epoxies to ensure that the adequately cured system is able to meet the requirements of its end use. The synthesized naphthalene-containing epoxy resin was characterized by FTIR and potentiometric titration method. The cur-

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ing kinetics, thermal behavior, and physical properties of cured systems were also evaluated by differential scanning calorimeter (DSC), thermogravimetric analyzer (TGA), dynamic mechanical analyzer (DMA), and thermal mechanical analyzer (TMA), respectively.

EXPERIMENTAL

Materials

1,6-Dihydroxynaphthalene (DHN), 4,4′-diphenylmethane diisocyanate (MDI), 4,4′-diaminodiphenyl sulfone (DDS), tetramethyl bisphenol F dicyanate (TBFD), and sodium carbonate were obtained from Aldrich (Milwaukee, WI). Epichlorohydrin (ECH), 1-methoxy-2-hydroxy propane, and methyl isobutyl ketone (MIBK) were obtained from Ferak (Berlin, Germany). 2-Phenylimidazole, triphenylphosphine (Ph_3P), acetylacetone copper (II), and nonyl phenol were obtained from TCI (Tokyo, Japan). All the reaction chemicals and solvents were reagent grade or were purified by standard methods before use.

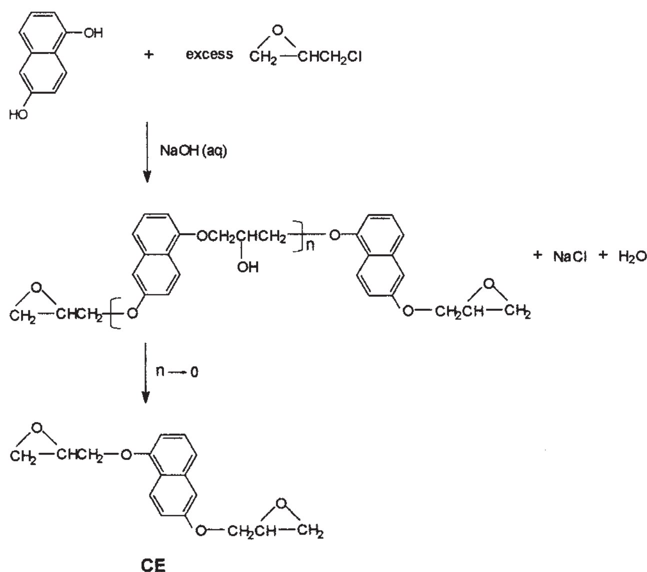
Measurements

Fourier transform infrared (FTIR) spectra were recorded on a Perkin–Elmer (Wellesley, MA) 16PC spectrometer with KBr pellets, spectra in the optical range of 400–4000 cm^{-1} were obtained by averaging six scans at a resolution of 8 cm^{-1} to minimize the effect of dynamic scanning. The epoxy equivalent weight (EEW) of the epoxy resin was determined by the $HClO_4$/potentiometric titration method.[13] The solution was used as a titrant, and the end points were detected by the first derivative of the potential versus the volume of the titrant. Differential scanning calorimeter (Perkin-Elmer DSC7, Wellesley, MA) measurements were used in this study. Samples weighing approximately 5–6 mg were sealed in hermetic aluminum pans and scanned in the calorimeter, with heating rates of 5, 10, 20, and 40°C/min in the range of 40–400°C under a N_2 atmosphere. For dynamic scanning, calibration of the calorimeter was conducted for the heating rate, using an indium standard. Dynamic viscoelastic properties were studied on a Perkin–Elmer 7 series thermal analysis with a DMA mode between 50°C and 350°C and with a heating rate of 5°C/min at a frequency of 1 Hz. A temperature/time scan in a three-point bending mode was chosen, and the dimensions of the specimen were 15 × 5.5 × 1.5 mm^3, according to ASTM Standard D790–95a: the distance of the support span was 10 mm. The elastic modulus, E′ and the tan δ were determined.

The coefficient of thermal expansion (CTE) was measured in the thermal mechanical analysis (TMA) mode of a Perkin–Elmer 7 instrument in accordance with ASTM Standard E831–86: a specimen of 2 mm in thickness was used at a heating rate of 5°C/min, and the CTE values were calculated from the slope in front of T_g. Thermogravimetric analyses (TGA) were performed on a Perkin–Elmer TGA-7 thermal analyzer (7–10 mg samples), using a heating rate of 10°C/min in N_2 at a purge pressure of 25 psi within the temperature range of 30–800°C. The decomposition temperature (T_d) was measured as the onset temperature from TGA curves. Moisture uptake was obtained with a difference in mass. Samples (D: 50 mm and T: 3 mm) of each cured resin disk were immersed in hot water (about 90°C) for 5 h (until equilibrium saturation). The quantity of water absorbed as a result of the value was determined by weight differences before and after immersion. Before each weighing measurement, the samples were wiped to remove surface water and weighed immediately. A new value of the water absorbed is checked with the one obtained earlier, and the same iterative procedure can be repeated until the value to within 1% deviation is achieved. The dielectric constants were determined by the bridge method with Du pont-2970 dielectric analyzer at a frequency of 1 KHz, 300*N*, and 30°C in a N_2 atmosphere at a flow rate of 500 cm^3/min by the two parallel plate mode. The specimens were subjected to vacuum pretreatment of 1–3 mmHg at 100°C for 2 h to eliminate absorbed water. The bulk density ρ was evaluated from a buoyant principle of mixture, $\rho = \rho_1 \upsilon_1 + \rho_2 \upsilon_2$ (where, the subscript 1 represents the water, 2 stands for the sodium carbonate 30 wt % solution, and υ_i refers to the volume fraction of component *i*).

Syntheses of diglycidyl ether of DHN (DGEDN or CE)

The monomer epoxy was synthesized by the most common method. To a 250 mL four-necked, round-bottomed flask, equipped with a heating mantle, stirrer, thermometer, and a Dean-Stark trap with reflux condenser, were charged 8.00 g (0.05 mol) of DHN, 46.25 g (0.5 mol) of ECH, and 20 mL of 1-methoxy-2-hydroxy propane. After stirring at a temperature and atmospheric pressure to thoroughly mix the contents, the temperature was raised to 65°C, and the pressure was reduced to 160 mmHg absolute. To the resulting solution was continuously added 25 mL of a 40 wt % sodium hydroxide solution at a constant rate via a metering pump over a period of 3 h while maintaining the reaction temperature at 65°C under a reduced pressure. During the addition of the NaOH, the water was removed from the system by codistilling with ECH and solvent; in the meantime, distilled ECH, and solvent were returned to the system. On completion of the addition, the reaction mixture was maintained at 65°C for an additional 30 min. The resulting mixture was washed with deionized water. The organic phase was finally rotary evaporated at 170°C under full vac-



Scheme 1 Synthesis of naphthalene-containing control epoxy resin.

uum for 3 h to remove the excess ECH and solvent completely. The residue was extracted with MIBK, the extract was washed five times with deionized water, and the MIBK was distilled off at 170°C to recover 12.24 g of an epoxy resin (DGEDN), and sample code is CE. Epoxy equivalent weight 139 (calcd: 136); Elem Anal. Calcd for $C_{16}H_{16}O_4$: C, 70.59%; H, 5.88%. Found: C, 70.25%; H, 5.96%. IR (KBr) cm^{-1}: 912 (oxirane ring). The reaction is shown in Scheme 1.

Preparation of MDI-modified epoxy resins

To a 250 mL four-necked, round bottomed flask, equipped with a $CaCl_2$ drying tube, heating mantle, N_2 inlet, a 100 mL addition funnel, mechanical stirrer, thermocouple, and temperature controller was added 40.8 g (0.144 mol) of DGEDN. The epoxy resin was heated to 170°C and 350 ppm (based on DGEDN) of 2-phenylimidazole was added. Melted MDI of 7.2 g (0.029 mol) was added dropwise to the stirring DGEDN under nitrogen atmosphere over a period of 30 min. After the completion of addition, the reaction mixture was maintained at 190°C for another 3 h, and using rotary, evaporated at 180 °C under high vacuum for 4 h to remove the excess DGEDN. Completion of the epoxide-isocysnate reaction was confirmed by infra-red spectroscopy for the disappearance of the —NCO group absorption, and sample code is ME. Epoxy equivalent weight 402 (calcd: 397); Elem Anal. Calcd for $C_{47}H_{42}N_2O_{10}$: C, 71.03%; H, 5.29%; N, 3.53%. Found: C, 70.91%; H, 5.46% N, 3.43%. IR (KBr) cm^{-1}: 912 (oxirane ring); 1754 (oxazolidinone ring). The reaction is shown in Scheme 2.

Cure reaction of CE/ME with DDS or TBFD

The control epoxy resin CE and various content ME epoxy resin (i.e., ME5, ME10, ME15; weight percentage: 5, 10, and 15%) were mixed with a stoichiometric (i.e., equal equivalent) amount of curing agent (DDS) and Ph_3P in a mill at moderate temperature to give a thermosettable epoxy resin powder. The resin powder was cured in a mold at a temperature of 160°C and 50 kg/cm^2 for a period of 1 h and then postcured at 180°C for 2 h and 200°C for 3 h to obtain a cured specimen. Another system was used as curing agent (TBFD) for epoxy resins, with 1% acetylacetone copper (II) and 4% nonyl phenol as accelerators. The resin powder was cured in a mold at a temperature of 160°C and 50 kg/cm^2 for a period of 1 h and then postcured at 200°C for 2 h and 220°C for 3 h to obtain a cured specimen.

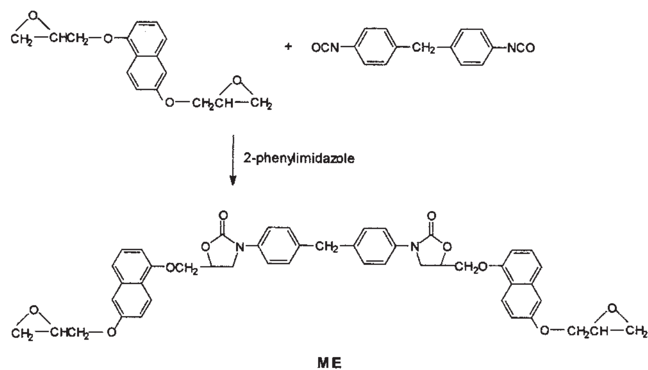
Sample codes

The sample codes for the control epoxy/curing agent systems are designated CES and CED, respectively, as shown in Table I. CE stands for control epoxy and S (or D) stands for curing agent of DDS (or TBFD). However, for the modified-epoxy/curing agent, systems are designated as MEXS and MEXD, respectively. ME stands for modified-epoxy, S (or D) stands for curing agent of DDS (or TBFD), and the number X after the ME is the weight percentage of the modified-epoxy.

RESULTS AND DISCUSSION

Synthesis of epoxy resin and analysis

In the direct process, a calculated excess of ECH governs the degree of polymerization. However, preparation of the higher molecular weight species is subject to practical limitation. In the preparation DGEDN (or CE), an excess amount of ECH (mole ratio: 10/1) was used to minimize high molecular weight species.



Scheme 2 The preparation of modified epoxy resin.

TABLE I
Apparent Activation Energy of the Reaction of Epoxy Resin with Curing Agent Obtained from the Kissinger Method

Sample code	Epoxy resin	Curing agent	E_1 (KJ/mol)	E_2 (KJ/mol)
CES	Control[a]	DDS	51.64	
ME5S	5 wt % ME[b]	DDS	55.42	
ME10S	10 wt % ME	DDS	61.35	
ME15S	15 wt % ME	DDS	63.28	
CED	Control	TBFD	78.43	59.72
ME5D	5 wt % ME	TBFD	85.92	63.25
ME10D	10 wt % ME	TBFD	96.34	68.17
ME15D	15 wt % ME	TBFD	102.26	71.84

[a] CE is without modified epoxy monomer, i.e. DGEDN.
[b] ME (5 wt %) is modified epoxy (ME) with control epoxy (CE) to yield a mixture and 5 wt % represent the weight fraction of ME is 5%, and the residual weight fraction of CE is 95%.

Hence, the typical product contained ~98% by weight of the monomer DGEDN as shown in Scheme 1, where the degree of polymerization was nearly zero ($n \rightarrow 0$).

Figure 1 shows typical infrared spectra of control, unmodified epoxy resin, and the corresponding modified epoxy resin exhibited several distinctive differences. The oxazolidinone structure showed a strong absorption at 1754 cm^{-1}, and the completion of this reaction was confirmed by the disappearance of the —NCO group absorption peak at 2250 and 2270 cm^{-1}. The epoxide absorption (912 cm^{-1}) still emerged in the two conditions.

Coreactive cure

Amine (DDS) is the most widely used curing agent for epoxy resins. Reaction of primary, secondary, and

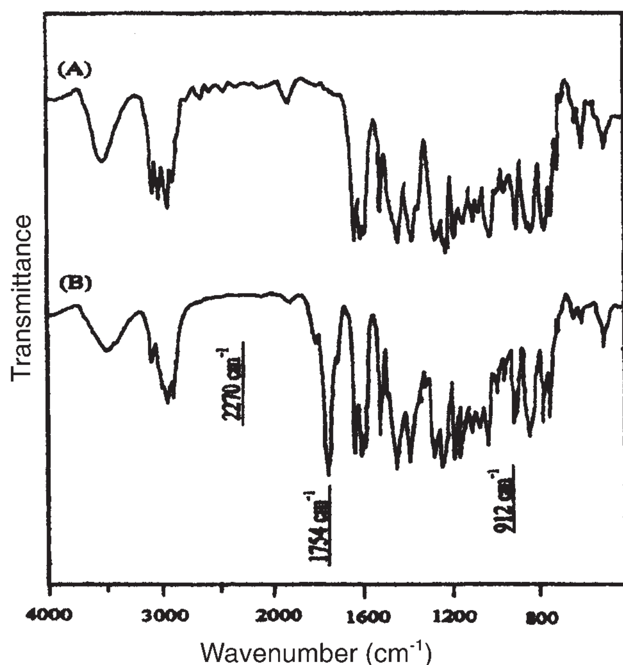


Figure 1 IR spectra of (A) control epoxy resin (CE) and (B) modified epoxy resin (ME).

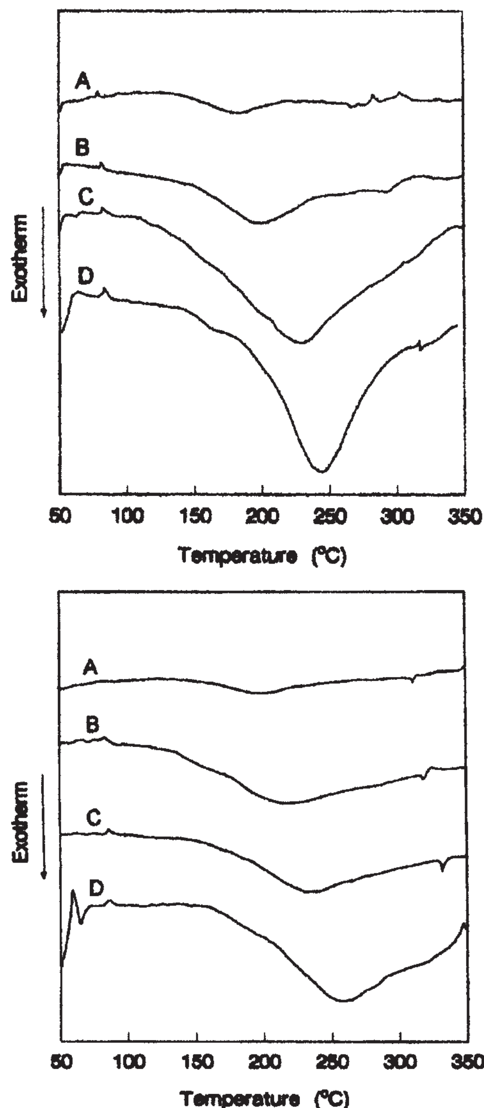


Figure 2 DSC curves of polymers: top, CES; bottom: ME10S at various heating rates (A: 5, B: 10, C: 20, and D: 40°C/min).

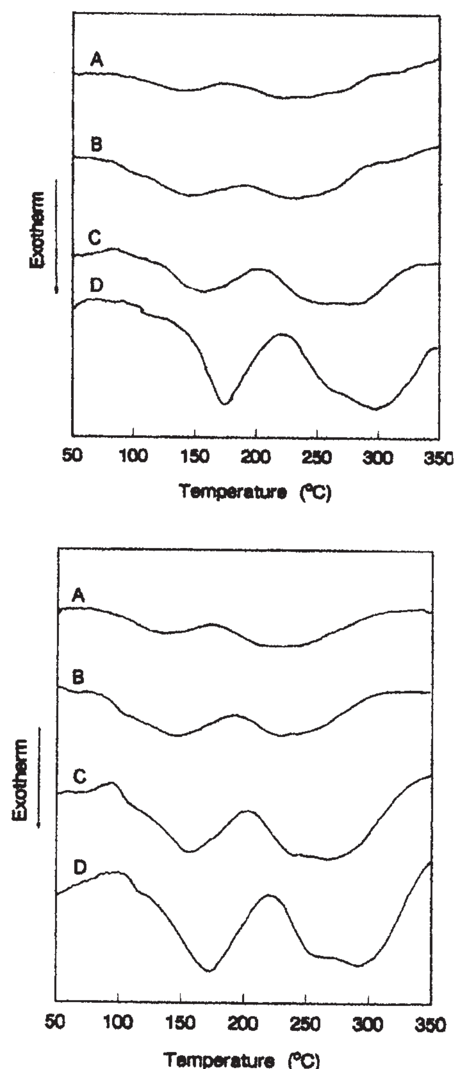


Figure 3 DSC curves of polymers: top, CED; bottom: ME10D at various heating rates (A: 5, B: 10, C: 20, and D: 40°C/min).

tertiary amine with epoxy groups that can take place simultaneously with secondary hydroxyl and ether group formation reactions are shown in the following

Therefore, the DSC scans of the epoxy/DDS systems exhibited only one exothermic peak, as shown in Figure 2. However, the DSC scans of the epoxy/TBFD systems showed two separate exothermic peaks,[14,15] and the occurrence of the phenomena are expected to explain the course of this curing reaction as well as to propose a reaction mechanism via cross-reference of dynamic DSC thermograms and FTIR spectra, as shown in Figure 3 and 4. To make the infrared spectroscopy comparable to the DSC thermogram, we performed the infrared study for dynamic cure of this system at the same heating rate (10°C/min) from room temperature to 270°C.

The FTIR scans were performed at each 20°C increment of the progressively ascending temperature during the heating process, and the dynamic heating from room temperature to about 220°C (just passing the borderline of two exothermic peaks of DSC trace) shows the consumption of most cyanate groups. Simultaneously, concomitant reactions include the formation of triazine rings, isocyanurates, and oxazoline linkages. The occurrence of these reactions can be verified by observing the following phenomena, disappearing absorptions of cyanate group (2250 and 2270 cm^{-1}) and epoxide group (912 cm^{-1}); however, emerging absorption of triazine (1582 cm^{-1}), isocyanurate structure (1690 cm^{-1}), and from the absorption intensity of the cyanurate ring and isocyanurate structure, we know that the total rate of formation of the aryl and alkyl cyanurate (path 1 and 2) is faster than the total rate of its disappearance (path 3). The early stage (the lower temperature peak in DSC scan) of curing process are shown as follows

The substituted isocyanurate at high temperature can further react with additional glycidyl ether to generate oxazolidinone structure (path 4). However, the reaction mixture of a cyanate ester with an epoxide does not produce the oxazolinyl linkages alone during polymerization, and oxazoline structure (1300 cm^{-1}) via rearrangement produce oxazolidinone structure (path 5 and 6), and the peak showed a absorption at 1748 cm^{-1}. But the mark showed at same position (1748 cm^{-1}) in the original stage is due to the epoxide-isocyanate reaction of MDI-modified epoxy resin. The reaction path is shown as follows

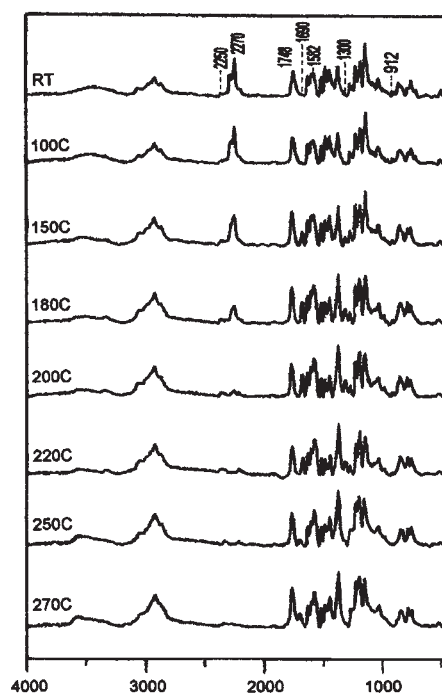


Figure 4 FTIR spectra of ME10D system during the dynamic curing stages from room temperature to 270°C at a heating rate of 10°C/min.

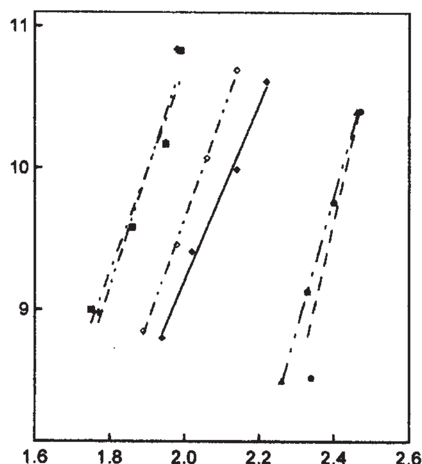


Figure 5 Plot of $-\ln\Phi/T_p^2$ versus $10^3/T_p$ for epoxy/curing agent (CED, •: first peak and ■: second peak; ME10D, ▲: first peak and +: second peak; ◆:CES, ◇: ME10S).

In summary, the mechanism proposed here for the curing system of epoxy/cyanate essentially follows the Bauer pathway.[16,17]

Curing kinetics

The kinetic parameters from dynamic DSC measurements[18–21] are analyzed by Kissinger's method

$$-\ln\frac{\Phi}{T_p^2} = \ln\frac{E/R}{An(1-x)_p^{n-1}} + \frac{E}{RT_p}$$

where Φ is the heating rate, T_p is the maximum rate temperature, and E the apparent activation energy.

We can get the data for the plot of $-\ln \Phi/T_p^2$ against $1/T_p$ as shown in Figure 5 and Table I. The apparent activation energy is clearly decreased with control epoxy resin, while the short-chain epoxy CE generally possesses a higher probability of molecular collision than the long-chain epoxy ME. The E_1 values of polymers ME5D, ME10D, and ME15D were higher than those of the corresponding polymers ME5S, ME10S, and ME15S. The reason can be explained due to the oxazolidinone structure which hinders the molecular chain movement, and hence has lower motion effect.

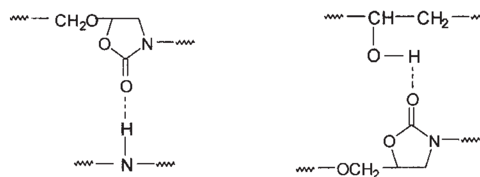


Figure 6 Schematic drawing of hydrogen bonding between carbonyl group (in oxazolidinone) and hydroxyl/amine (using DDS as curing agent) group (in epoxy).

The properties of polymer

Thermal gravimetric analysis of the cured samples was carried out in a nitrogen atmosphere. Thermal stabilities of these resins were compared by the temperature of decomposition (T_d) and percent char yield at 700°C, these are presented in Table II. Glass-transition temperature (T_g), thermal stability, and anaerobic char yield of polymers ME5–15S were higher than that of polymers CES. This may be attributed to the increased hydrogen bonding (see Fig. 6), and hence the intermolecular interaction hydrogen bonding of interpenetrating polymer networks (IPNs) as shown in Figure 7. In addition, the oxazolidinone structure also greatly increased the thermal properties of the resultant polymers.

The other properties of cured products are shown in Table III. The CTE was taken from the glassy state below T_g, and the CTE values of polymers ME5D, ME10D, and ME15D were lower than those of the corresponding polymers ME5S, ME10S, and ME15S. Furthermore, the epoxy resins, which contained planar naphthalene and oxazolidinone structure, have a lower CTE than that of common electronic material of epoxy resins, about 65 ppm. Absorbed moisture in a composite was found not only to plasticize the resin, causing a lowering of the T_g and in turn affecting

TABLE II
Thermal Properties of Control and Modified Epoxy Resins with the Various Curing Agent

Sample code	Epoxy resin	Curing agent	T_g (DSC) (°C)	T_d (°C)	Char yield 700°C (%)
CES	Control	DDS	194	396	26.28
ME5S	5 wt % ME	DDS	201	410	30.20
ME10S	10 wt % ME	DDS	218	426	36.84
ME15S	15 wt % ME	DDS	223	430	38.27
CED	Control	TBFD	204	412	28.69
ME5D	5 wt % ME	TBFD	215	421	32.89
ME10D	10 wt % ME	TBFD	232	438	38.46
ME15D	15 wt % ME	TBFD	237	447	40.19

T_d, onset temperature for decomposition.

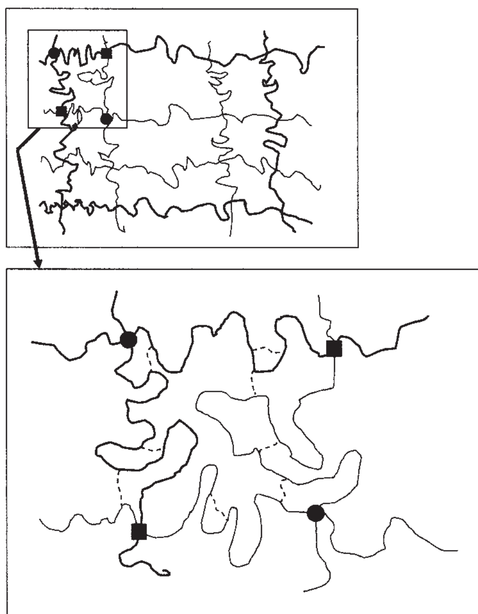


Figure 7 Idealized structure of a crosslink polymer (IPNs, constituted by a mutual combination of two crosslinked polymers; ●: same molecular crosslinks, ■: different molecular crosslinks, and dot line: hydrogen bonding).

mechanical response, but also to cause package cracking. This crack is caused by evaporation and expansion of absorbed moisture in the package at the temperature of reflow soldering during mounting semiconductors on printed circuit boards,[22] while the naphthalene, oxazolidinone structure and high crosslink density also greatly reduced the CTE and moisture absorption of the resultant polymers (common bisphenol A epoxy system absorbs ~3% moisture). Although, the molecular structure of CE and ME was rather similar, but the molecular volumes (evaluated from density, see Table III) were different, ME5–15S and ME5–15D packed denser than those of corresponding polymers CES and CED (the influences of crosslink, hydrogen bonding, naphthalene structure, and molecular space), which led to a lower dielectric constant values (for typical epoxy resins, ~4.0). The modulus values of all the polymers, except control epoxy (such as CES and CED) had moderate values. The thermomechanical properties were determined via DMA. The modulus values observed in these epoxy resins were comparable to those with very stiff (naphthalene and oxazolidinone) structures, and because of this high stiffness, they provide polymers with high in-plane orientation.

CONCLUSIONS

The structure of these products (CE and ME) were confirmed by the epoxy equivalent weight (EEW, by potentiometery analysis) and elemental analysis (elemental composition), so that the proposed network behavior is in good agreement with the IPNs. The DSC scan of epoxy/DDS showed only one exothermic peak, however, for the epoxy/TBFD system exhibited two separate exothermic peaks. The first reaction is always the trimerization of the cyanates to build up aryloxy-substituted triazines. Subsequently, the glycidyl ether is inserted into the cyanurate and forms the corresponding alkyl cyanurate, which isomerizes to the isocyanurate structure, and while the isocyanurate at high temperature, and can further react with additional glycidyl ether to generate oxazolidinone structure.[23] These good properties are presumably the reason for the contribution to optimal state, and which IPNs formed by a cure reaction, depending on two different epoxy monomer (i.e., epoxy CE and epoxy ME) with curing agent. In addition, hydrogen bonding is also formation from the intermolecular interaction between carbonyl group (in oxazolidinone) and hydroxyl/amine group (in epoxy) in polymer matrices.[24]

TABLE III
Other Properties of Control and Modified Epoxy Resin with the Various Curing Agent

Sample code	Epoxy resin	Curing agent	MU (%)	CTE (10^{-5}/K)	E'_{avg} GPa	ρ (g/cm^3)	DC
CES	Control	DDS	1.92	5.9	0.85	1.2436	3.8
ME5S	5 wt % ME	DDS	1.76	5.5	1.24	1.2381	3.6
ME10S	10 wt % ME	DDS	1.69	5.1	1.72	1.2215	3.3
ME15S	15 wt % ME	DDS	1.57	4.6	1.91	1.2197	3.2
CED	Control	TBFD	1.80	5.8	0.89	1.2862	3.6
ME5D	5 wt % ME	TBFD	1.71	5.3	1.27	1.2678	3.4
ME10D	10 wt % ME	TBFD	1.63	4.8	1.82	1.2583	3.1
ME15D	15 wt % ME	TBFD	1.54	4.2	2.06	1.2394	2.9

The average of modulus, $E_{avg}' = (E_{120°C}' + E_{60°C}')/2$.
MU, moisture uptake; ρ, bulk density; DC, dielectric constant.

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