Preparation and Characterization of Long-Chain Multimaleimide-Containing Aralkyl Group Linkages

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ABSTRACT: Poly(aralkyl maleimide) (PAMI) and poly(glycidyl ether of aralkyl novolac) resin (PANE) with phenylmethylene units were synthesized from the intermediates poly(aralkyl amino) resin (PAAR) and poly(aralkyl novolac) resin (PANR), which were obtained from the reactions of *p*-xylylene glycol with aniline or phenol, respectively. The oligomers were characterized by ^{1}H-NMR, Fourier transform infrared spectroscopy, gel permeation chromatography, and potentiometry. The corresponding PAMI and PANE were cured with 4,4′-diaminodiphenylmethane (DDM) at an equal equivalent ratio, and their curing behaviors were investigated by differential scanning calorimetry. The reaction involved a major chain extension and a minor homopolymerization. The thermal, mechanical, and electric properties of the PAMI–DDM system were better than that of those PANE–DDM system. Furthermore, the PAMI–DDM has a low internal stress. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 90: 1066–1072, 2003

Key words: poly(aralkyl maleimide); chain extension; internal stress

INTRODUCTION

Aromatic polyimides are well known as highly heat-resistant materials. With high thermal stability and excellent mechanical and electrical properties, as well as radiation resistance, this class of polymer has found broad commercial applications ranging from household and automotive uses to separation membranes and electronic devices.[1–3] However, condensation-type polyimides are often insoluble and infusible in the fully imidized form because of rigid chain characteristics, leading to processing difficulties.[4,5] Epoxy resins are widely used as matrix resins for structural composites because of their well-balanced properties and ease of handling and processing.[6] But the limitation on the upper use temperature and hot/wet performance restricts their application.

A thermosetting resin having excellent heat resistance is obtained by thermally polymerizing an aromatic bismaleimide (BMI) alone, which has typically been used in various applications such as multiplayer printed circuit boards (PCBs) for large-scale computers, advanced composites for aerospace industries, structural adhesives,

and potting resins. BMI bridges the temperature performance gap between epoxies and polyimides, but, inconveniently, it is very brittle and has poor flexibility. To overcome these drawbacks, thermoplastics are increasingly used in the toughening of BMI resins to improve their brittleness.[7,8] In addition, the Michael addition of primary or secondary diamines to BMIs is also usually done to carry out chain extension prior to thermal curing, so as to reduce the crosslink density and brittleness of the cured networks.[9–13]

The object of this study was to develop a process to produce a poly(aralkyl maleimide) (PAMI) that could maintain excellent heat resistance, mechanical strength, and electrical insulating properties and could reduce internal stress by reducing elastic modulus and coefficient of thermal expansion (CTE) to form prepregs and green sheets in order to manufacture laminate plate for integrated circuit (IC) substrates. The synthesized PAMIs were then copolymerized with 4,4′-diaminodiphenylmethane (DDM) through the Michael addition and then crosslinked to produce a network polymer. The properties of this polymer were compared with those of a polyaralkyl-based epoxy resin and of a conventional BMI.

EXPERIMENTAL

Materials

Aniline, phenol, and epichlorohydrin were purchased from Aldrich (Milwaukee, WI). They were all vacuum-

distilled before use. *p*-Toluenesulfonic acid monohydrate (PTSA) was purchased from Ferak (Berlin, Germany) and vacuum-dried before use. *p*-Xylylene glycol was obtained from TCI (Tokyo, Japan), and 4,4′-bismaleimidophenylmethane (BMI), nickel acetate tetrahydrate, and triethylamine were purchased from Aldrich (USA). All reaction chemicals were used as received, then stored in a reduced-pressure dry box, and solvents were purified by standard methods before use.

Synthesis of poly(aralkyl amino) resin

To a 500-mL reactor equipped with a stirrer, a reflux condenser, a thermometer, a Dean–Stark water separator, and a nitrogen inlet were charged 69.75 g (0.75 mol) of aniline and 41.4 g (0.3 mol) of *p*-xylylene glycol in 120 mL of xylene. After purging with N_2 for 10 min, 7.8 g of PTSA was added to the above solution at room temperature. The mixture was heated with stirring under reduced pressure (ca. 200 mmHg) until the internal temperature reached 130°C. Then the mixture was further heated while removing the aqueous distillate by the Dean–Stark trap until the internal temperature reached 185°C and was maintained at this temperature for 8 h. The reaction mixture was cooled to 60°C, and a dilute aqueous solution of potassium hydroxide was added to the resulting solution. The solution was washed several times with deionized water until the wash solution was neutral. The organic phase was placed on a rotary evaporator under a full vacuum at 205°C for 3 h to remove the excess aniline. IR (KBr) cm^{-1}: 3430, 1625 (NH. Str.), 2880–3100 (aromatic and alkane groups); mass spectrometry of the product detected $m/e = 290$ ($n = 0$), 485 ($n = 1$), 680 ($n = 2$), 875 ($n = 3$), 1070 ($n = 4$) as the main peaks, corresponding to the formula for poly(aralkyl amino) resin (PAAR). The reaction is shown in Scheme 1.

## Preparation of poly(aralkyl maleimide)

A 500-mL four-necked flask equipped with an addition funnel and a nitrogen inlet was charged with a solution of maleic anhydride (1 mol) in 100 mL of methyl isobutyl ketone (MIBK). Under a nitrogen atmosphere, poly(aralkyl amino) resin (0.1 equivalent) in 50 mL of MIBK was added dropwise to the above mixture. The materials were dissolved with stirring at room temperature, and the resulting mixture was heated to 45°C and maintained at this temperature for 6 h. The white precipitate was collected, washed with fresh MIBK to remove excess maleic anhydride, and then dried in a vacuum oven to obtain poly(aralkyl maleamic acid) (PAMI).

To a 500-mL round-bottomed flask was added the 0.1 equivalent of the above-synthesized poly(aralkyl maleamic acid) resin (PAMA) and 100 mL of dimeth-

ylformamide. The solution was heated to 40°C with stirring; then 15.8 g of acetic anhydride, 0.18 g of nickel acetate tetrahydrate, and 2 g of triethylamine were added, and the mixture was heated to 70°C and maintained at this temperature for 8 h. After completion of the reaction, the mixture was cooled to room temperature with constant stirring and then poured into ice water with stirring for 30 min. The precipitate was filtered and washed with a dilute aqueous solution of sodium hydrogen carbonate and then with water. A white solid was obtained and then dried in a vacuum. IR (KBr) cm^{-1}: 1763, 1721 (imide ring), 1413 (C—N—C), 697 (maleimide ring), 2880–3100 (aromatic and alkane groups); mass spectrometry of the product detected $m/e = 450$ ($n = 0$), 725 ($n = 1$), 1000 ($n = 2$), 1275 ($n = 3$) as main peaks, corresponding to the formula for PAMI. The reaction is shown in Scheme 1.

Synthesis of poly(aralkyl novolac) resin

Into a 1-L three-necked, flat-bottomed flask equipped with a heating mantle, a thermocouple, a temperature controller, a magnetic stirrer, and a Dean–Stark trap with a reflux condenser was added a solution of 69 g (0.5 mol) of *p*-xylylene glycol in 200 mL of 1-butanol and 141 g (1.5 mol) of phenol. After purging with
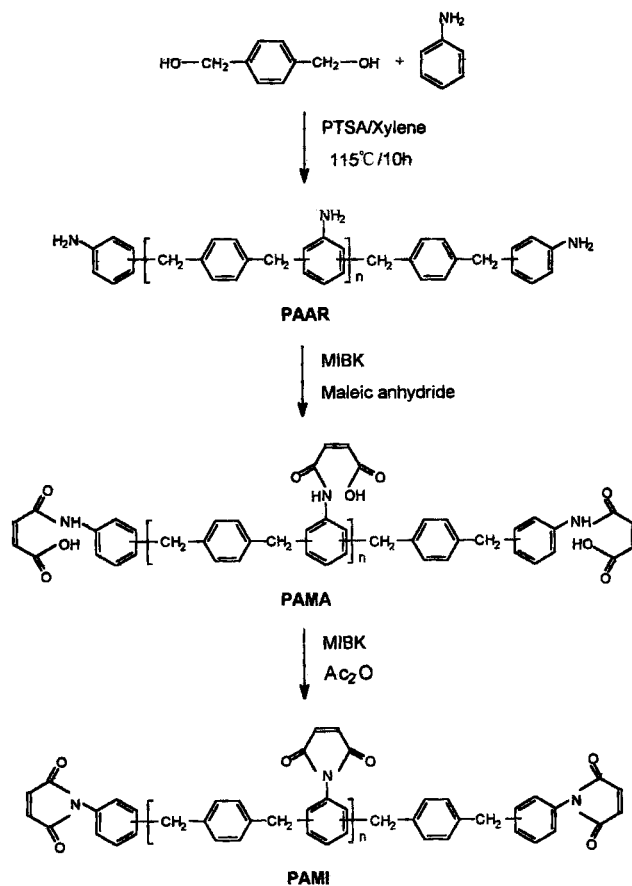
Scheme 1

nitrogen for 10 min, PTSA (1.0 g) was added to the above solution at room temperature. The mixture was allowed to react with stirring at 115°C–120°C for 10 h under reduced pressure (ca. 200 mmHg), while simultaneously removing water by azeotroping with 1-butanol and returning the distilled 1-butanol to the system. The resulting mixture was neutralized with a dilute aqueous potassium hydroxide solution and washed with deionized water until the wash solution was neutral. The organic phase was finally "rotavapped" at 195°C under full vacuum for 3 h to remove the excess phenol until it was less than 0.3% (as analyzed by liquid chromatography). Measurements by gel permeation chromatography (GPC) and infrared (IR) absorption spectroscopy were made on the resins. IR (KBr) cm^{-1}: 3540, 1206 (OH. Str.), 2900-3050 (aromatic and alkane groups); ^{1}H-NMR (200 MHz, CDCl$_3$) δ 3.95 (s, CH$_2$), δ 4.38 (s, NH$_2$), δ 6.71–7.25 (m, phen. H), mass spectrometry of the product detected m/e = 292 ($n = 0$), 488 ($n = 1$), 684 ($n = 2$), 880 ($n = 3$), 1076 ($n = 4$) as main peaks, corresponding to the formula for poly(aralkyl novolac) resin (PANR). The reaction is shown in Scheme 2.

Preparation of poly(glycidyl ether of aralkyl novolac) resin

The epoxy resin was synthesized by the modified procedure, which was previously reported.[14] To a 1-L four-necked, round-bottomed flask, equipped with a stirrer, a heating mantle, a thermocouple, a temperature controller, and a Dean–Stark trap with a reflux condenser was added the 0.5 equivalent of the above-synthesized aralkyl novolac resin, 5 mol of epichlorohydrin, and 80 g of methyl ether of propylene glycol (1-methoxy-2-hydroxy propane) as a solvent. 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 42 g of a 48 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 epichlorohydrin and solvent; in the meantime, distilled epichlorohydrin 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 neutralized with a dilute aqueous potassium hydroxide solution and washed with deionized water. The organic phase was finally rotavapped at 190°C under full vacuum for 3 h to remove the excess epichlorohydrin and solvent. The residue was extracted with methyl isobutyl ketone (MIBK), the extract was washed four times with deionized water, and the MIBK was distilled off at 190°C to recover 103 g of an epoxy resin. IR (KBr) cm^{-1}: 914 (oxirane ring); epoxy equivalent weight: 215. The general schematic drawing preparation of poly(glycidyl ether of aralkyl novolac) resin (PANR) is shown in Scheme 2.

Measurements

The ^{1}H-NMR spectra were registered using a Bruker Analytik WP-200 spectrometer (Germany) using CDCl$_3$ as a solvent. The ^{1}H chemical shifts were calibrated by using tetramethylsilane (TMS) as an internal standard. The gel permeation chromatography (GPC) measurements were performed on a Waters (USA) RI system equipped with a UV detector, a differential refractometer detector, and an Ultrastyagel column at 30°C, using tetrahydrofuran as an elute and calibrated with polystyrene standards. Fourier transform infrared (FTIR) spectra were recorded on a Perkin–Elmer (USA) 16PC spectrometer with KBr pellets. Spectra in the optical range of 400–4000 cm^{-1} were obtained by averaging 32 scans at a resolution of 4 cm^{-1}. Electron Impact source mass spectrometric analyses (England) were performed on a VG 70-250S GC/MS spectrometer with a solid inlet. The epoxy equivalent weight) of the epoxy resin was determined by the HClO$_4$/potentiometric titration method.[15] 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 (Du Pont 910; USA) measurements were used in this study. Samples weighing approximately 5–6 mg were sealed in hermetic aluminum pans and scanned in the calorimeter, with a heating rate of 20°C/min in the range of 40°C–400°C under an N$_2$ atmosphere, and the glass-transition temperature (T_g) was taken as the change in the specific heat in the heat flow curves. For dynamic scanning calibration of the calorimeter was conducted for the heating rate using an indium standard. Dy-

Scheme 2
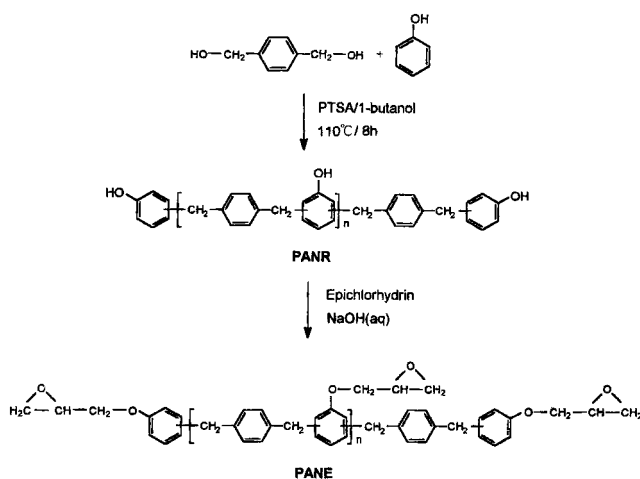
namic viscoelastic properties were studied on a Perkin–Elmer 7 series thermal analysis (USA) with a DMA mode between 50°C and 300°C with a heating rate of 5°C/min at a frequency of 1 Hz. A temperature/time scan in a three-point bending mode was chosen, and the dimensions of the specimen were 15 × 5.5 × 1.5 mm, according to ASTM Standard D 790-95a: 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 thermomechanical analysis mode of a Perkin–Elmer 7 instrument in accordance with ASTM Standard E831-86: a specimen 2 mm in thickness was used at a heating rate of 5°C/min. The CTE values were calculated from the slope. Thermogravimetric analyses (TGA, USA) were performed on a Perkin-Elmer TGA-7 thermal analyzer using a heating rate of 10°C/min in N_2 at a purge pressure of 25 psi within the temperature range of 30°C–800°C. The decomposition temperature (T_d) was measured as the onset temperature from thermogravimetric analyzer curves. The dielectric constants were measured with a Du Pont DEA 2970 dielectric analyzer (USA) at a frequency of 1 kHz, 300 N, and a temperature of 30°C in an N_2 atmosphere at a flow rate of 500 cm^3/min by the

TABLE I
Average Molecular Weights and EEWs of PAAR, PANR, and PANE Measured via GPC and Potentiometry

Sample	M_n GPC	M_w GPC	EEW potentiometery
PAAR	712	1153	
PANR	886	1099	
PANE			215

two parallel plates mode. The specimens were subjected to vacuum pretreatment of 1 mmHg at 120°C for 4 h to eliminate absorbed water. The amounts of moisture absorption were measured by immersing specimens [3 mm (T) × 50 mm (D)] of each cured resin disk in boiling water for 100 h, and the weight differences after aging were determined.

RESULTS AND DISCUSSION

Synthesis of oligomers and analysis

Schemes 1 and 2 show the synthetic routes to poly(aralkyl maleimide) (PAMI) and poly(glycidyl ether of aralkyl novolac) resin (PANE), respectively. A two-step method was used to prepare poly(aralkyl maleimide) via the corresponding poly(aralkyl maleamic acid) (PAMA), using acetic anhydride in the presence of a catalytic amount of nickel acetate tetrahydrate/triethylamine at a specific temperature. The typical infrared spectra of PAMI exhibited several distinctive differences with the corresponding PAMA. The PAMI showed strong absorption at 1721 and 697 cm^{-1} and weak absorption at 1763 cm^{-1}, assigned, respectively, to asymmetrical and symmetrical C=O stretching vibration of the imide ring, and it lacked absorption at 3300–3400 cm^{-1}, associated with carboxylic OH stretching vibration, and at 1562 cm^{-1}, for the NH stretching of the amide group, as shown in Figure 1. Consequently, imidization could be monitored by these absorption values. The molecular weights of the PAAR and PANR, which in this case were determined using GPC, are summarized in Table I. GPC measurement of the PANR was performed, and the average molecular weights (M_n and M_w), shown in Table I, were calculated by using of a GPC calibration curve. The preparation of PANE involves reacting the PANR with a halohydrin in the presence of an alkali metal hydroxide, as shown in Scheme 2. The structure of aralkyl novolac resin was confirmed by IR and its mass spectrum. The typical infrared spectra of PANR showed several distinctive differences with the corresponding PANE. The PANR showed a strong absorption peak at 3400–3600 cm^{-1}, representing the —OH functional groups of the synthesized PANR, and a peak at 914 cm^{-1} representing the oxirane ring absorption of PANE. In addition, an excess amount of
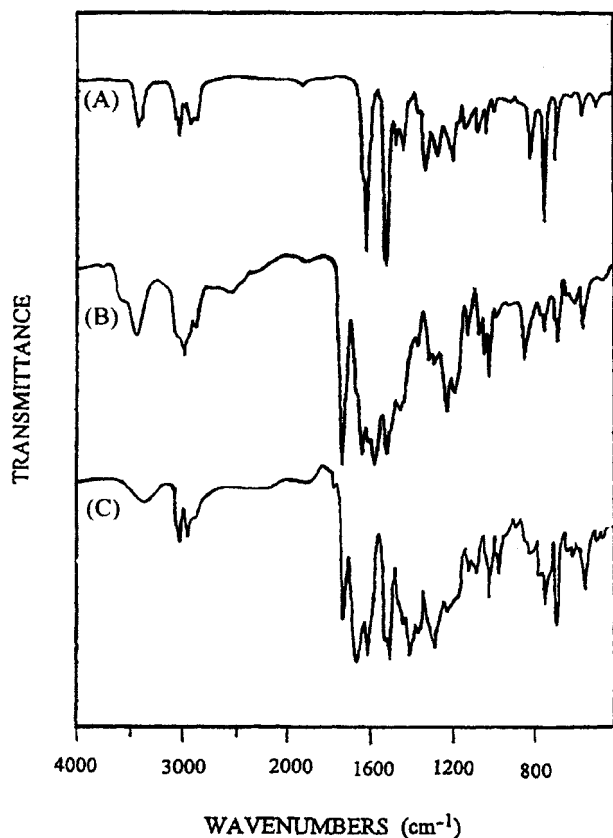

Figure 1 FTIR spectra of (A) poly(aralkyl amino) resin (PAAR); (B) poly(aralkyl maleamic acid) (PAMA); and (C) poly(aralkyl maleimide) (PAMI).
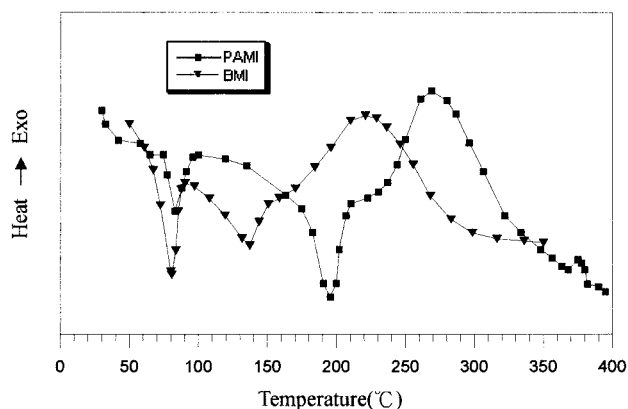
Figure 2 DSC scans for PAMI–DDM and BMI–DDM at a heating rate of 20°C/min.

epichlorohydrin was used to minimize high-molecular-weight species. Hence, the typical product contained about 100 wt % PANE.

Thermal behavior and molecular structure

Figure 2 shows typical DSC scans of the PAMI–DDM and BMI–DDM systems. The composition (equivalent ratio 1:1) exhibited two separate endothermic peaks. The first endotherm indicates the melting of the diamine (ca. 90°C), and the second endothermic peak is broad and shifted to a lower temperature compared to the neat PAMI or BMI, indicating the diamine may have caused an immediate chain extension reaction with PAMI or BMI. The polymerization exothermic peak is retarded and shifted to the lower temperature compared to that of the neat PAMI or BMI, indicating that the nucleophilic addition of amines to the maleimide double bonds occurred readily and at a lower temperature than the neat exothermic temperature of PAMI or BMI (also in comparison with other BMIs reported in the literature[16–18]) to a network structure. The curing characteristics are given in Table II.

TABLE II
DSC Data of Neat PAMI, BMI, PAMI/DDM, and BMI/DDM

Sample	$T_m{}^a$ (°C)	$T_i{}^b$ (°C)	T^c (°C)
PAMI	191	230	289
BMI[d]	163	181	237
PAMI/DDM[e]	175	212	266
BMI/DDM[f]	146	157	231

[a] Melting point from DSC with a heating rate of 20°C/min.
[b] Onset temperature for curing reaction.
[c] Maximum temperature of polymerization exotherm.
[d] Also in compare with other BMI from refs. [16–18].
[e,f] Cure: 200°C (4 h) + 250°C (5 h).

Polymerization of PAMI–DDM or BMI–DDM has been shown to be quite complex. The reaction can yield combinations of chain extensions (secondary amine formation),[19] tertiary amine formation, amidization reaction,[20] and homopolymerization.[21] In general, the rate of tertiary amine formation in the maleimide–amine system is much less than the rate of secondary amine formation, even when substantial amounts of secondary amine are present, clearly indicating that the secondary amine has much less reactivity than the primary amine, so that the primary amine has four times the reactivity of the secondary amine.[22] A side reaction (amidization reaction) has been reported for maleimides and primary amine, resulting in opening of the maleimide ring and formation of an amide group.[20] However, this ring-opening reaction does not appear to have played a significant role in the chemistry of the aromatic maleimide–amine system. In addition, for the maleimide–amine mixture cured over the temperature range studied, homopolymerization of maleimide only played a minor role. From the above discussion, the resulting structure of the maleimide–amine after heat-induced reactions, along with the possible products and/or unreacted species, are depicted in Figure 3.

Thermal stability and physical properties of polymers

Thermogravimetric analyses (TGA) of the cured samples were carried out in a nitrogen atmosphere. The
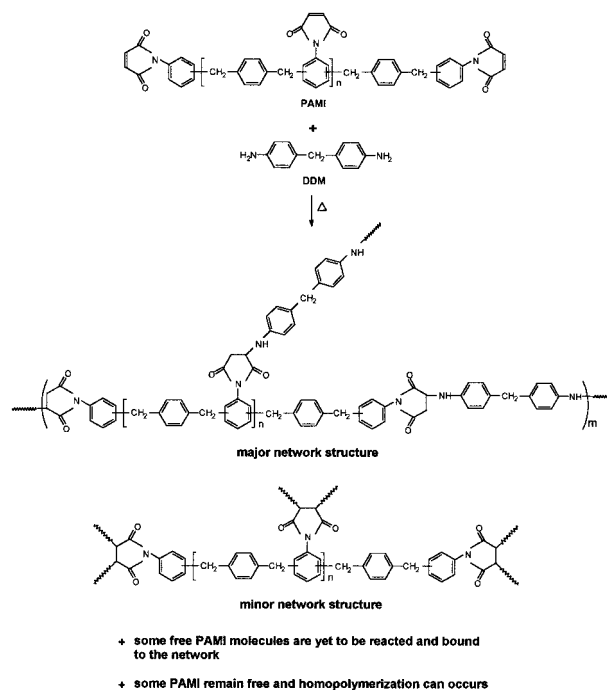

Figure 3 Schematic drawing of the final network structure showing the products of PAMI–DDM system after heating.

TABLE III
Thermal Stability and Physical Properties of Polymers

Polymer code	T_g (°C)		$T_d{}^b$ 10 wt % in N_2	$T_{max}{}^c$ (°C)	Char yield 600°C (%)	MA^d (%)	DC^e
	DMA	TMA[a]					
PAMI/DDM (I)	243	195	416	440	50	1.12	3.05
BMI/DDM (II)	246	197	419	447	56	1.58	3.25
PANE/DDM (III)	176	151	395	410	30	2.43	4.45

[a] T_g: Onset temperature from glassy state transition to rubbery state.
[b] T_d: Onset temperature for decomposition.
[c] T_{max}: Temperature of maximum rate of weight loss.
[d] MA: Moisture absorption.
[e] DC: Dielectric constant.

thermal stabilities of these resins were compared by the temperature of decomposition (T_d), temperature of maximum rate of weight loss (T_{max}), and percent char yield at 600°C. They are presented in Table III. The glass-transition temperature (T_g), thermal stability, and anaerobic char yield of network polymer I was somewhat lower than those of polymer II, indicating that polymer I had a relatively longer (phenyl-methylene, ph—CH₂) distance between the two maleimido end caps than did polymer II. In general, epoxy resins exhibited lower T_g, T_d, and char yield than did maleimide resins, which may be attributed to their lower crosslink density and a lower bond dissociation energy than maleimide resins. Absorbed moisture in a composite was found not only to plasticize the resin, causing a lowering of the T_g and in turn affecting mechanical response, but also to cause package cracking. This cracking (the so-called popcorn phenomenon) is caused by evaporation and expansion of absorbed moisture in the package at the temperature of reflow soldering (215°C–260°C) during mounting semiconductors on printed circuit boards.[23] The moisture absorption results are given in Table III. In general, maleimide resins absorbed less moisture than did the epoxy system because epoxy molecules have some OH groups. The moisture absorption and dielectric constant values of polymer I were lower than those of polymer II. This was attributed to a weak intermolecular dipole–dipole interaction between the imide groups.[24] In other words, the increasing hydrocarbon character, derived from bulkier coupling units (phe-

nyl-methylene), progressively lowered the dielectric constant by diluting dipole concentrations and decreasing the electronegativity of the substituted benzene ring.

Thermomechanical properties

Table IV shows the thermomechanical and dynamic viscoelastic properties. The coefficient of thermal expansion (CTE) in the glassy state below T_g was taken from 60°C to 130°C and the CTE above T_g was taken from 210°C to 260°C. The CTE values of polymer II were slightly lower than those of polymer I. High crosslink density (decreasing distance between the crosslink points in BMI–DDM) increased the elastic modulus in the rubbery state, as shown in Figure 4). Low-molecular-weight maleimides limited the segmental motions of the cured material and reduced the free volume, thus giving lower CTE values.

TABLE IV
Dynamic Viscoelastic and Thermal
Mechanical Properties

Polymer code	E (GPa)			CTE (10^{-5}/K)	
	$E'_{60°C}$	$E'_{120°C}$	$E'_{avg}{}^a$	$<T_g$	$>T_g$
PAMI/DDM (I)	3.19	3.07	3.13	5.8	13.9
BMI/DDM (II)	5.25	4.93	5.09	5.6	14.7
PANE/DDM (III)	1.17	1.16	1.17	8.7	21.3

[a] Average of storage modulus, $E'_{avg} = (E'_{60°C} + E'_{120°C})/2$.
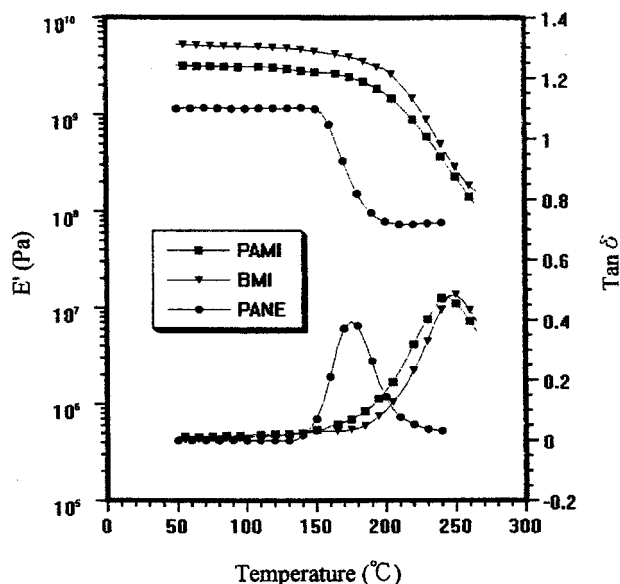

Figure 4 DMA results for the PAMI, BMI, and PANE–DDM adducts at equal equivalent ratio.

TABLE V
Internal Stress Parameter of Polymers

Polymer code	E'_{avg} (GPa)	CTE $(10^{-5}/K)$	$E'_{avg} \times$ CTE (kPa/K)
PAMI/DDM (I)	3.13	5.8	181.5
BMI/DDM (II)	5.09	5.6	285.0
PANE/DDM (III)	1.17	8.7	101.8

Internal stress

Many diamines have been used to modify the properties of various BMI resins to obtain materials meeting specific application requirements. The preparation of maleimide resins, extended with diamine, lowers the crosslinking density and increases the toughness of the cured materials. The sources of internal stress that results from the use of plastic encapsulants are considered to be shrinkage of the plastic upon curing and thermal mismatch between the resin and the device. Reduction of internal stress by lowering of either the thermal expansion coefficient or the flexural modulus of the encapsulant has been reported.[25,26] Internal stresses are considered to be predominantly a result of cooling of the cured material specimen from its curing temperature to room temperature, by which internal stress would accumulate in the resin matrix. The internal stress of cured specimens is closely related to the product of the elastic modulus (E'_{avg}) and the CTE below the T_g of the cured materials. Table V lists the elastic moduli (E'_{avg}), CTEs, and internal stress parameters ($E'_{avg} \times$ CTE) of the materials investigated. The results show that the internal stress of polymer III was lower than that of maleimide resins. With the same ph—CH_2 group but a longer distance between the two maleimido end caps, the corresponding polymer I had lower internal stress.

CONCLUSIONS

Phenyl-methylene-based aralkyl maleimide and aralkyl novolac resin were synthesized by condensing *p*-xylylene glycol with aniline or phenol, respectively. The oligomers were cured at equal equivalent ratio. The combination of the special features of the aralkyl group with those of polymultimaleimide produced higher glass-transition temperature, thermal stability, and elastic modulus and lower coefficient of thermal expansion, moisture absorption, and dielectric constant than those of the phenyl-methylene unit epoxy system.

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