Synthesis and Properties of Fluorinated Biphenyl-Type Epoxy Resin

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ABSTRACT: A novel fluorinated biphenyl-type epoxy resin (FBE) was synthesized by epoxidation of a fluorinated biphenyl-type phenolic resin, which was prepared by the condensation of 3-trifluoromethylphenol and 4,4′-bismethoxymethylbiphenyl catalyzed in the presence of strong Lewis acid. Resin blends mixed by FBE with phenolic resin as curing agent showed low melt viscosity (1.3–2.5 Pa s) at 120–122°C. Experimental results indicated that the cured fluorinated epoxy resins possess good thermal stability with 5% weight loss under 409–415°C, high glass-transition temperature of 139–151°C (determined by dynamic mechanical analysis), and outstanding mechanical properties with flexural strength of 117–121 MPa as well as tensile strength of 71–72 MPa. The thermally cured fluorinated biphenyl-type epoxy resin also showed good electrical insulation properties with volume resistivity of 0.5–0.8 $\times$ 10^{17} Ω cm and surface resistivity of 0.8–4.6 $\times$ 10^{16} Ω. The measured dielectric constants at 1 MHz were in the range of 3.8–4.1 and the measured dielectric dissipation factors (tan δ) were in the range of 3.6–3.8 $\times$ 10^{-3}. It was found that the fluorinated epoxy resins have improved dielectric properties, lower moisture adsorption, as well as better flame-retardant properties compared with the corresponding commercial biphenyl-type epoxy resins. © 2009 Wiley Periodicals, Inc. J Appl Polym Sci 113: 1429–1437, 2009

Key words: epoxy; dielectric properties; water absorption; flame retardancy

INTRODUCTION

The versatility in chemical formulations have resulted in advanced epoxy resins as microelectronic packaging materials to be used extensively, including epoxy molding compounds (EMC), printed circuit boards, adhesives, and encapsulants.[1–6] With the rapid development of microelectronic packaging technology, epoxy resins should be improved accordingly in both performance and functionality to satisfy the higher requirements, such as higher thermal stability, lower dielectric constant and moisture absorption, lower coefficient of thermal expansion, higher flame-retardant properties, higher mechanical strength, and lower modulus.[7,8] Thus, many efforts were made in recent years to develop novel epoxy resin materials with specially designed chemical structures. For instance, multiaromatic (such as biphenyl, naphthalene, anthracene, diphenylether, or pyrene) structures, containing epoxy resins, have been reported as heat-resistant thermosets.[9–12] Iji and Iwasaki[13,14] reported that novel EMC derived from biphenyl-contained epoxy resins exhibited very good flame-retardant properties without any added flame retardants.

It is known that the insertion of some fluorinated groups into epoxy resins have many positive benefits such as reducing moisture absorption and dielectric constant due to the small dipole and the low polarizability of the C—F bond as well as its big free volume.[15] Hence, many efforts were made in recent years to prepare novel fluorinated epoxy resins. Maier reported many fluorinated epoxy resins with low dielectric constant for potential microelectronic applications.[16] Sasaki and Nakamura[17,18] reported that perfluorobutenyloxyphthalic anhydride-cured bisphenol A type epoxy could reduce the water absorption by 75% and the dielectric constants to 2.7–2.8. However, thermal stabilities and anaerobic char yields were also improved in the materials incorporated with trifluoromethyl groups.[19–21]

In this article, a novel fluorinated biphenyl-type epoxy resin (FBE) was synthesized and characterized. It was cured with fluorinated biphenyl-type phenolic resin (FBP) and commercial biphenyl-type phenolic resin (BP). The synthesis, characterization, and relationship between the chemical structure and the properties of cured resin were systematically investigated. Furthermore, the epoxy resins based on commercial biphenyl-type epoxy (BE) cured with the same hardeners were also prepared for comparison, respectively.

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MATERIALS AND METHODS

Materials

Methylisobuthyl ketone (MIBK), and *p*-tolunesulfonic acid (pTS) were purchased from Beijing Beihua Fine Chemicals (China) and used without further purification. Epicholorohydrin (ECH) was reagent grade from Beijing Beihua Fine Chemicals (Beijing, China) and distilled before use. 3-Trifluoromethylphenol was purchased from Fuxin Sanbao Chemical (Fuxin, China), and it was purified by distillation before use. Triphenylphosphine (TPP) and 4,4′-bismethoxymethylbiphenyl (BMMB) were commercially obtained and used without further purification. Phenol 4-4′-dimethylbiphenylene phenolic resin (BP) (MEH-7851ss, hydroxyl equivalent 200 g/mol, softening point 67°C) was purchased from Meiwa Kasei (Ube City, Yamaguchi, Japan). Phenol 4-4′-dimethylbiphenylene novolac epoxy resin (BE) (NC-3000, epoxy equivalent 270 g/mol, softening point 57°C) was purchased from Nippon Kayaku (Tokyo, Japan).

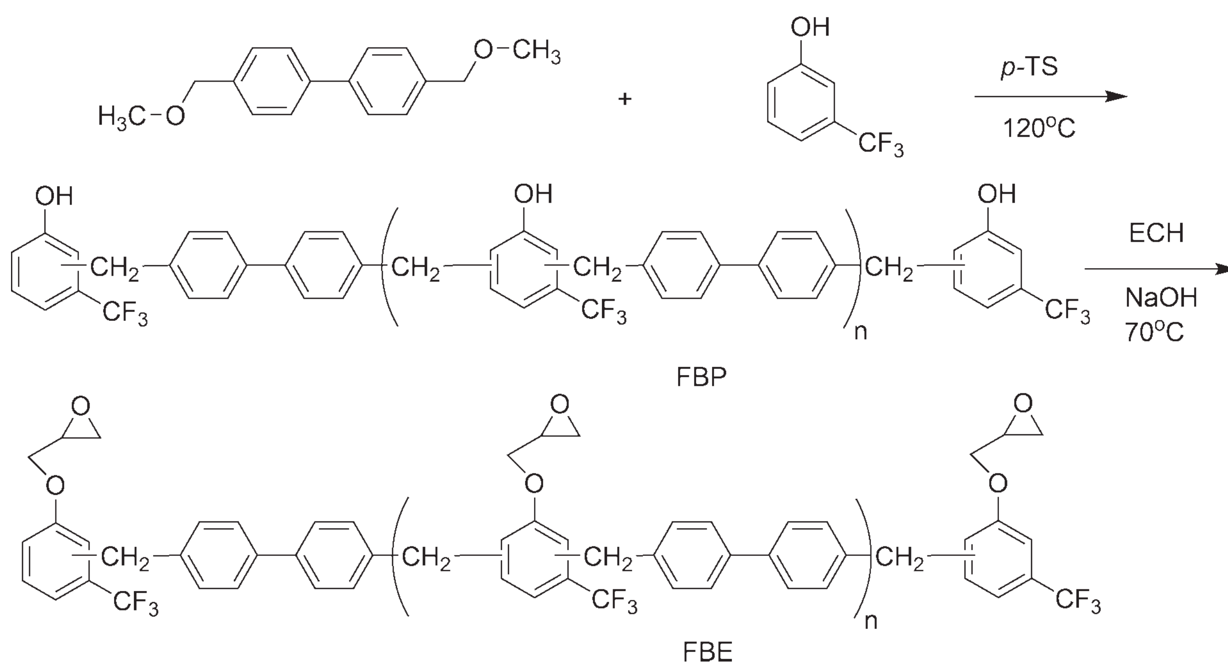
Characterization

^{1}H-NMR spectra were performed on a Bruker AVANCE 400 Spectrometer at 400 MHz by using $CDCl_3$ as solvents. Fourier transform infrared (FTIR) spectra were recorded on a Perkin–Elmer 782 FTIR spectrophotometer. Matrix-assisted laser desorption/ionization time-of-flight (MALDI-TOF) mass spectra were obtained by using a Biflex III MALDI-TOF mass spectrometer (Bruker, Billerica, Germany). Differential scanning calorimetry (DSC), thermal gravimetric analysis (TGA), and dynamic mechanical analysis (DMA) were recorded on a Perkin–Elmer 7 series thermal analysis system under nitrogen. Water uptake was calculated through difference between weights of cured epoxy specimen (Φ 50 × 3 mm) before and after immersion in water at 100°C for 6 h. The dielectric constants and dissipation factors were determined by the parallel plate method with an AS2851Q capacitance meter at a frequency of 1 MHz and 25°C. Viscosity (η^*) was measured on a TA AR2000 rheometer in Oscillation mode. Gel permeation chromatography (GPC) was carried on Waters 1515 equipped with HR1, HR0.5 Waters Styragel columns, using tetrahydrofuran as eluent. Flame retardant properties of the cured resins were determined by the limited oxygen index (LOI) method, UL94 test, and cone calorimeter test (Fire Testing Technology, heat flux of 50 kW/m^2).

Resin synthesis

3-Trifluoromethylphenol-4,4′-biphenyl phenolic resin

Into a 1000-mL four-neck round-bottom flask equipped with Dean-Stark trap was added 3-trifluoromethylphenol (486.0 g, 3.0 mol), BMMB (121.0 g, 0.5 mol), and pTS (0.5 g, 2.5 mmol). The mixture was heated at 120°C with stirring under nitrogen atmosphere and then the reaction was continued for 5 h. The methanol generated from the polycondensation process was removed simultaneously by distillation. After cooling down to room temperature, the reaction mixture was washed with deionized water five times to give a neutralized solution. Subsequently,



Scheme 1 Synthesis passageway of fluorinated biphenyl-type epoxy (FBE).

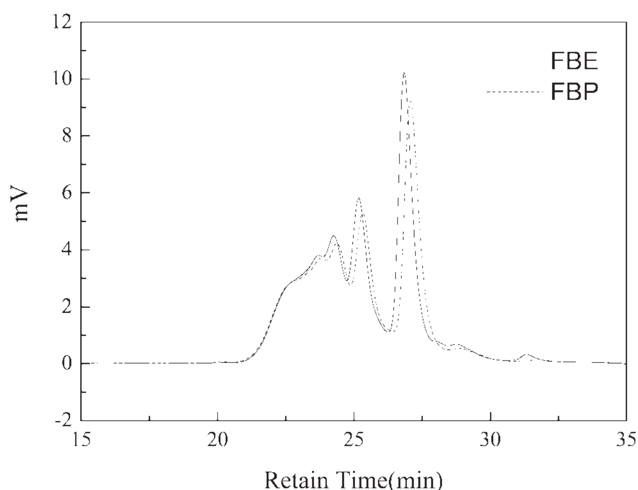


Figure 1 GPC curves of fluorinated biphenyl-type epoxy (FBE) and fluorinated biphenyl-type phenolic resin (FBP).

the solution was dried by $MgSO_4$ and vacuum distilled at 130°C to remove the unreacted 3-trifluoromethylphenol. As a result, a light red solid resin was obtained (224.7 g). Softening point: 66°C, the hydroxy equivalent weight: 271 g/equiv, GPC: M_n = 1589 g/mol. IR (cm^{-1}): 3401, 3027, 2923, 1914, 1701, 1619, 1499, 1443, 1323, 1242, 1119, 1048, 907, 810, 725. ^{1}H-NMR ($CDCl_3$, δ, ppm): 6.7–7.6 (broad peak, aromatic proton), 4.9 (—OH), 3.7–4.3 (multiplet, CH_2 linkage between phenol ring and biphenyl). Elem. Anal. Calcd. for $C_{49}H_{35}F_9O_3$ (n = 1): C, 69.83%; H,4.19%. Found: C, 69.99%; H, 4.29%.

3-Trifluoromethylphenol-4,4′-biphenyl epoxy resin

A mixture of FBP resin (135.5 g, 0.5 mol eq) and epichlorohydrin (925.3 g, 10.0 mol) were placed into a 2000-mL four-necked flask equipped with stirrer and a modified Dean-Stark trap. To the solution was added dropwise 50.0 g of 48 wt % sodium hydroxide solution over a period of 3 h while keeping the temperature at 70°C. The additive epichlorohydrin and the water generated in the reaction were removed continuously from the reaction system by azeotropic distillation. The distilled epichlorohydrin was separated and returned to the reaction system. After stirring for an additional hour, the reaction

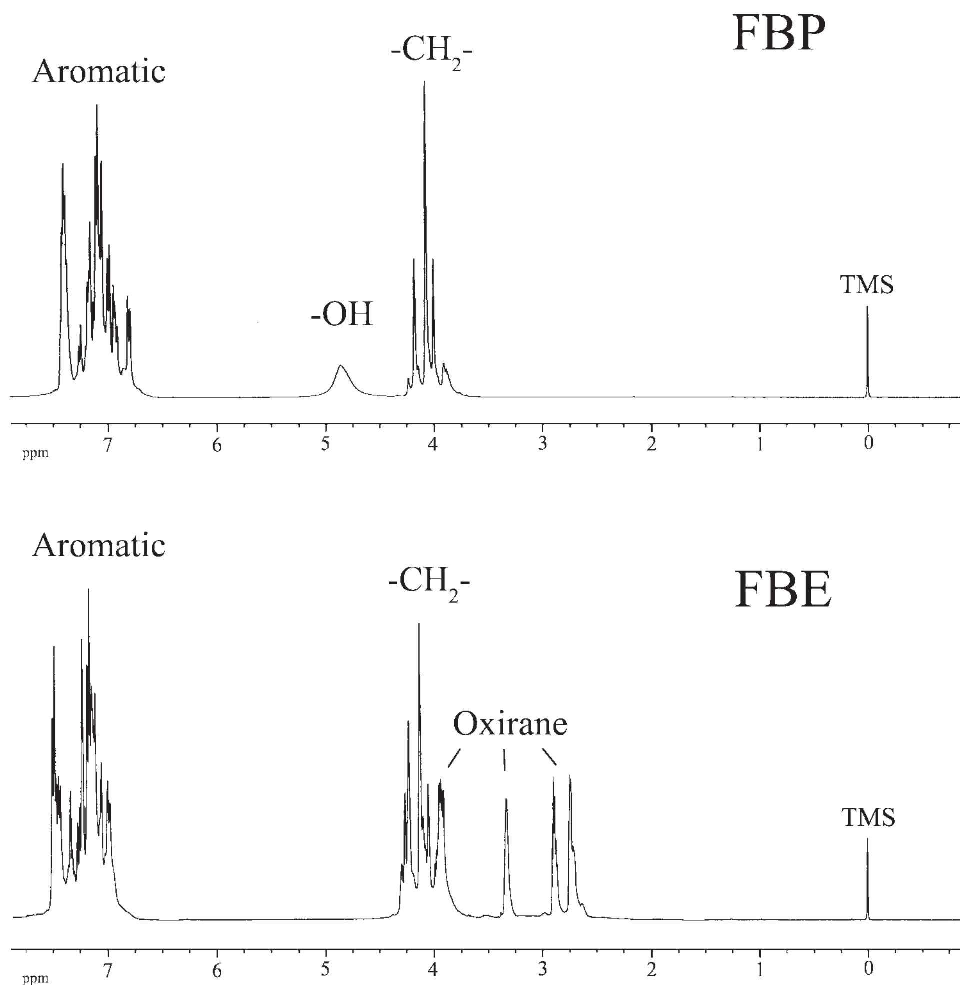


Figure 2 ^{1}H-NMR spectra of fluorinated biphenyl-type epoxy (FBE) and fluorinated biphenyl-type phenolic resin (FBP).

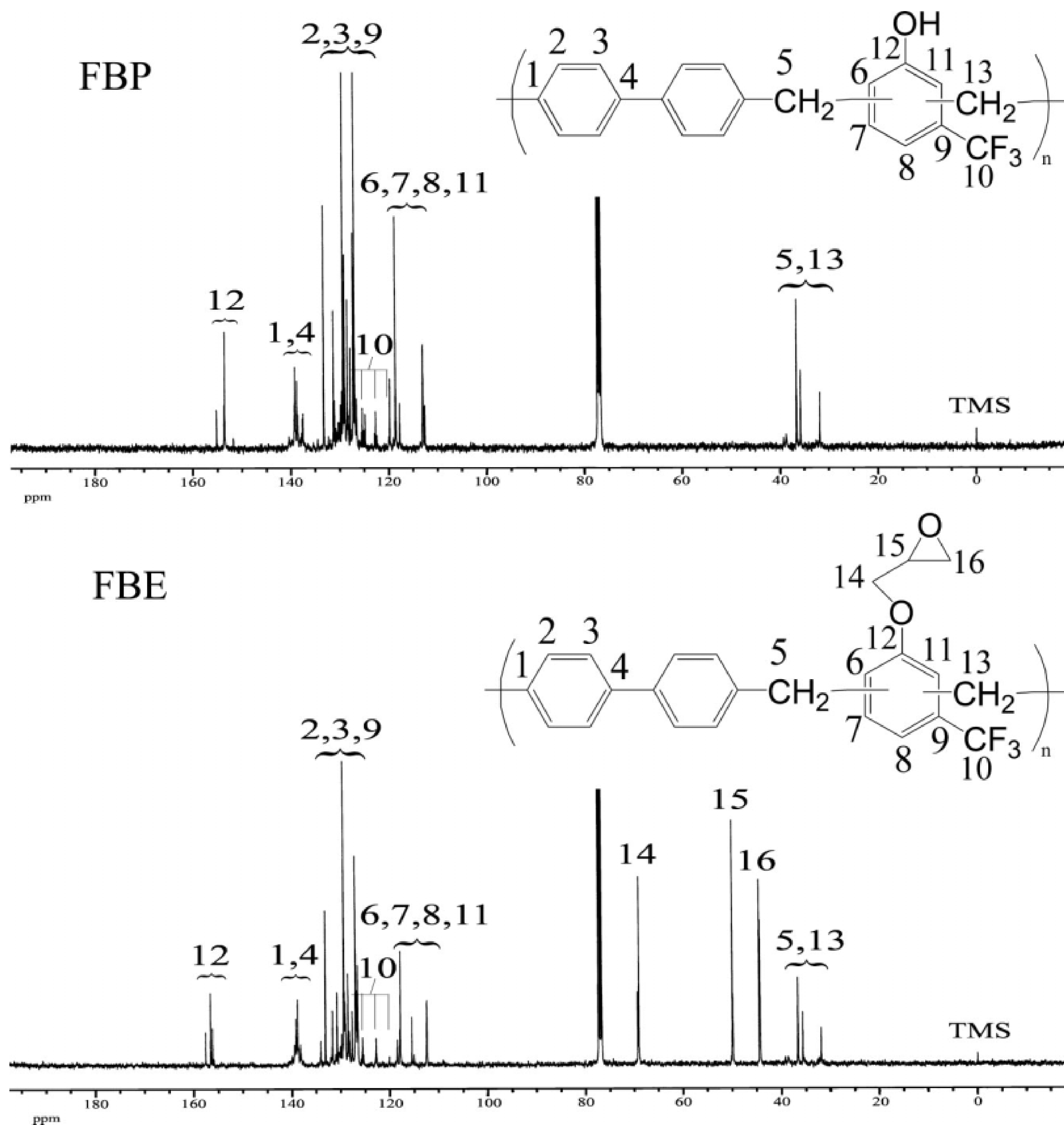


Figure 3 ^{13}C-NMR spectra of fluorinated biphenyl-type epoxy (FBE) and fluorinated biphenyl-type phenolic resin (FBP).

solution was cooled down to room temperature. The solution was filtered to remove the salt generated in the reaction and the obtained organic phase was washed with deionized water four times to give a neutralized solution. The solution was subsequently distilled to remove the unreacted epichlorohydrin and 138.6 g of FBE as a light yellow solid was obtained. Softening point: 58°C; The epoxy equivalent weight: 355 g/equiv. GPC: $M_n = 1623$ g/mol. IR (cm^{-1}): 3025, 2927, 1720, 1618, 1587, 1500, 1426, 1314, 1119, 1042, 915, 860, 810, 670. ^{1}H-NMR (CDCl$_3$, δ, ppm): 6.9–7.6 (broad peak, aromatic proton), 3.9–4.3 (multiplet, CH$_2$ linkage between phenol ring and biphenyl), 2.7–3.9 (multiplet, —CH$_2$, —CH on the epoxy group). Elem. Anal. Calcd. for $C_{58}H_{47}F_9O_6$ (n = 1): C, 68.91%; H,4.69%. Found: C, 68.60%; H, 4.72%.

Thermal curing of the fluorinated epoxy resins

Two phenolic resins (FBP and BP) were used as curing agents to thermally cure FBE resin. The epoxy resin was blended with phenolic resin in a molar

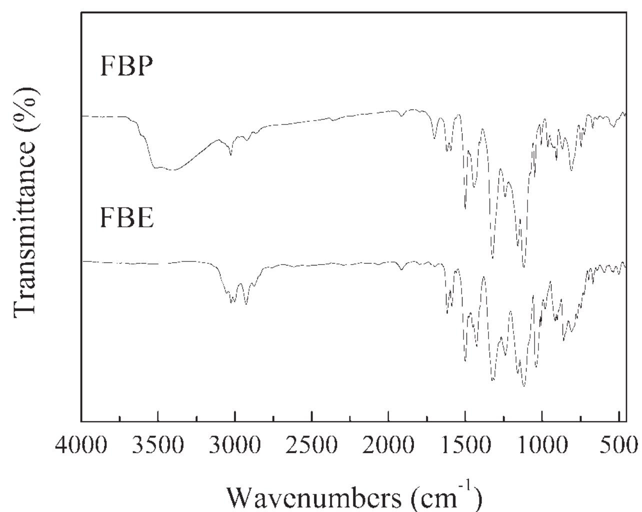


Figure 4 Comparison on FTIR spectra of fluorinated biphenyl-type epoxy (FBE) and fluorinated biphenyl-type phenolic resin (FBP).

stoichiometric ratio of 1 : 1 at 100°C to obtain a homogeneous solution and then TPP (1.0 wt % of the epoxy resin) was added. The mixture was then heated at 150°C for 2 h and postcured at 180°C for 3 h to give fully cured epoxy resins EP-1 (FBE-FBP) and EP-2 (FBE-BP).

In comparison, the commercial biphenyl-type epoxy resin BE (NC-3000) was also cured with FBP or BP at the same condition described above to give cured resins EP-3 (BE-FBP) and EP-4 (BE-BP).

RESULTS AND DISCUSSION

Synthesis and characterization

The fluorinated biphenyl-type epoxy resin (FBE) was synthesized by a two-step process as shown in Scheme 1. Figure 1 shows the GPC chromatograms of FBE and FBP, suggesting that both FBE and FBP are mixed with oligomeric compounds having similar molecular weight distribution. Figure 2 is ^{1}H-NMR spectra of FBE and FBP. The singlet signal at 4.9 ppm was attributed to hydroxyl proton in FBP, while the glycidyl protons in FBE were observed at 2.5–3.9 ppm. Figure 3 depicts ^{13}C-NMR spectra of FBE and FBP. The quartet absorption peaks at 119.4–127.8 ppm were attributed to the C_{10} in FBE and FBP, which are assigned to the $^{1}J_{C-F}$ coupling of the carbons linked with fluorine atoms. Further the glycidyl carbons in FBE were detected at 69, 49, and 44 ppm. Figure 4 exhibits the FTIR spectra of FBE and FBP, in which the C—F multiple stretching absorptions in the range of 1300–1100 cm^{-1} are observed. Additionally, the absorption in the range of 3300–3500 cm^{-1} was assigned to the hydroxyl groups in FBP and the characteristic band of oxirane rings in FBE was detected at 915 cm^{-1}. Figure 5 shows MALDI-TOF spectrum of FBE, which contains at least six chemical species with different molecular weights in the range of 721.6–2702.6. It is confirmed that the six chemical species could be assigned to FBE oligomers with n value of 0–5.

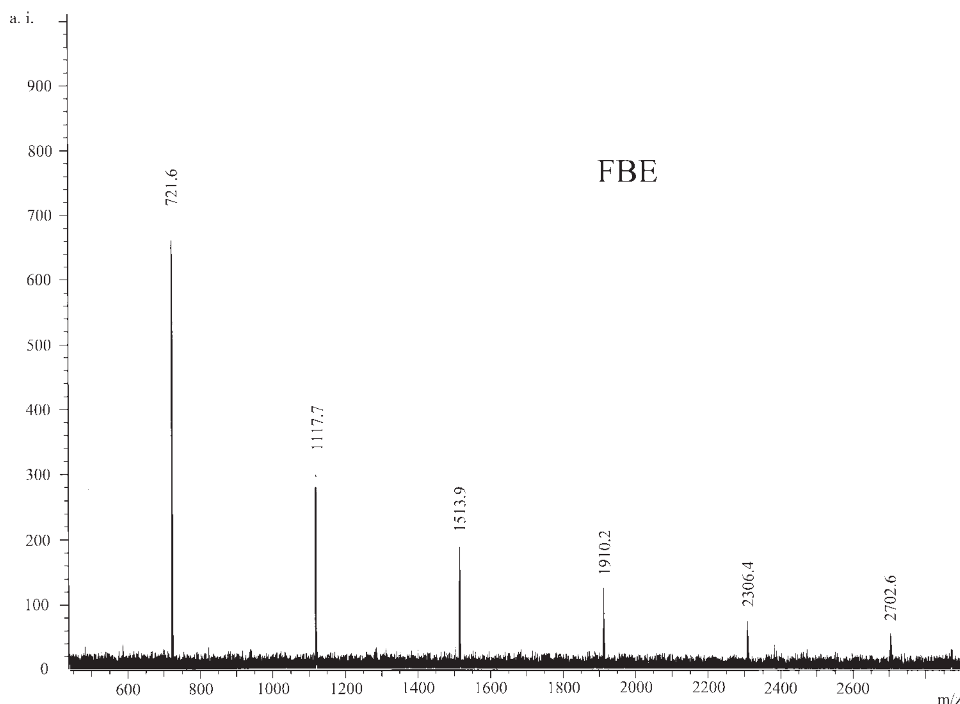


Figure 5 MALDI-TOF mass spectrum of fluorinated biphenyl-type epoxy (FBE).

TABLE I
Rheological and Curing Parameters of Thermal Curing Process

Samples	Minimum complex viscosity (Pa s)	E_a (kJ/mol)	n
EP-1	1.3 at 122°C	76.8	0.914
EP-2	2.5 at 120°C	73.4	0.911
EP-3	1.3 at 127°C	72.1	0.908
EP-4	5.1 at 115°C	74.7	0.914

Thermal curing

To understand the thermal curing behaviors of the epoxy resins, a plate rheology test was performed in oscillation mode. The resulting data are shown in Table I. As can be seen from the results, the complex viscosity of epoxy resin samples decreased in the beginning, reached lowest value (10^0) at 120–127°C before rapidly increasing by several orders of magnitude (10^4) with gelation (Fig. 6). The fluorinated biphenyl-type epoxy resin samples (EP-1, EP-2) showed relatively lower viscosity than the commercial biphenyl-type epoxy (EP-4), where lower viscosity is good for advanced microelectronic packaging materials.

The dynamic cure reaction parameters of epoxy resin samples were estimated by using DSC at different heating rates. The DSC traces of these epoxy resin samples at a heat rate of 10°C/min were collected in Figure 7. The activation energy can be obtained from the maximum curing reaction rate at the peak exotherm temperature according to Kissinger's method,[22] where the relation could be expressed as:

$$\frac{d\left[\ln\left(q/T_p^2\right)\right]}{d\left(1/T_p\right)} = -\frac{E_a}{R} \qquad (1)$$

where T_p is the peak exotherm temperature, q is the heating rate, E_a is the activation energy, and R is the gas constant. Figure 8 shows the linear fit process of $\ln(q/T_p^2)$ versus $1/T_p$ of the fluorinated epoxy resins. E_a could be calculated from slope of the plot of $\ln(q/T_p^2)$ versus $1/T_p$ without any assumption about the conversion-dependent equation. The activation energy of the fluorinated epoxy resins calculated from the slope of eq. (1) was in the range of 72.1–76.8 kJ/mol, which is similar to other epoxy/phenolic systems (69–78 kJ/mol).[23,24]

The cure reaction order can be calculated by using the Crane method as shown in eq. (2):

$$\frac{d(\ln q)}{d\left(1/T_p\right)} = -\frac{E_a}{nR} \qquad (2)$$

where n, the reaction order, can be calculated from the slope of the plot of $\ln(q)$ versus $1/T_p$. The reaction orders for all of the resin blends were measured in the range of 0.908–0.914.

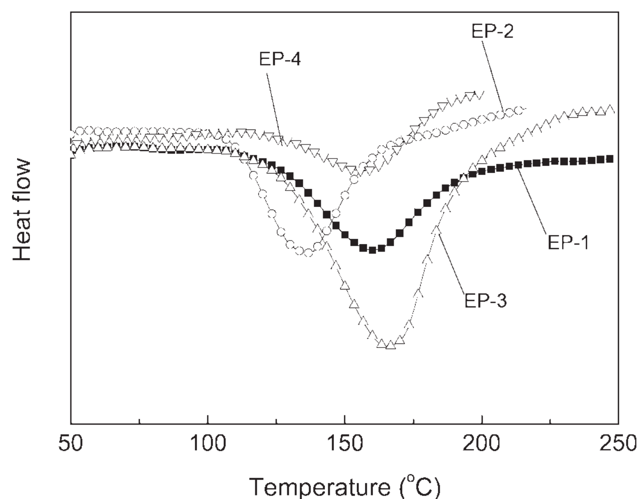


Figure 7 DSC curves of FBE and BE resins curing with different hardeners.

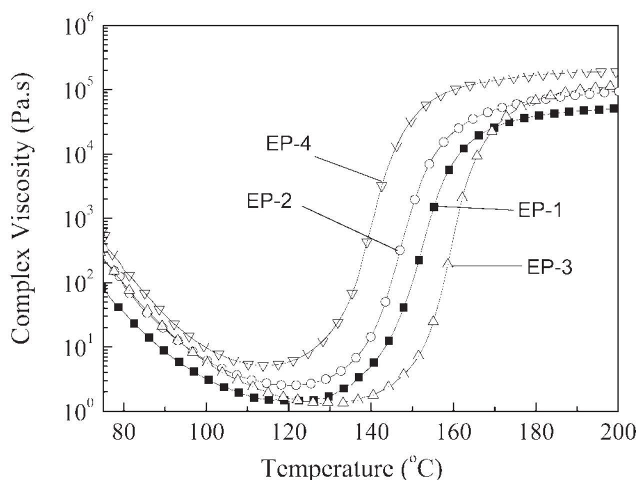


Figure 6 Rheological curves of FBE and BE resins curing with different hardeners.

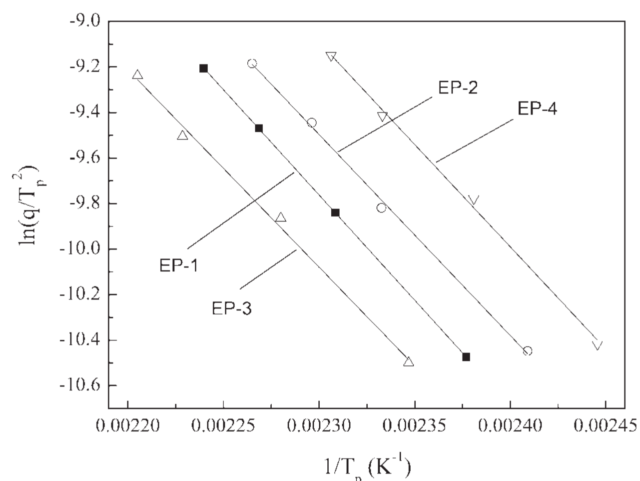


Figure 8 Linear fit of curing parameters using Kissinger's method.

TABLE II
Thermal Properties of the FBE and BE Resins Cured with Different Curing Agents

Samples	$T_{g,DMA}$ (°C)	T_d (°C)[a]	T_5 (°C)[b]	Char (%)[c]
EP-1	151	424	415	55.8
EP-2	139	429	409	46.9
EP-3	149	438	426	44.9
EP-4	129	431	424	32.7

[a] Initial decomposition temperature in nitrogen.
[b] Temperature at 5 wt % loss.
[c] Char yield at 650°C in nitrogen.

These results indicated that the curing mechanism of epoxy resins was not significantly affected by the introduction of fluorinated substituent.

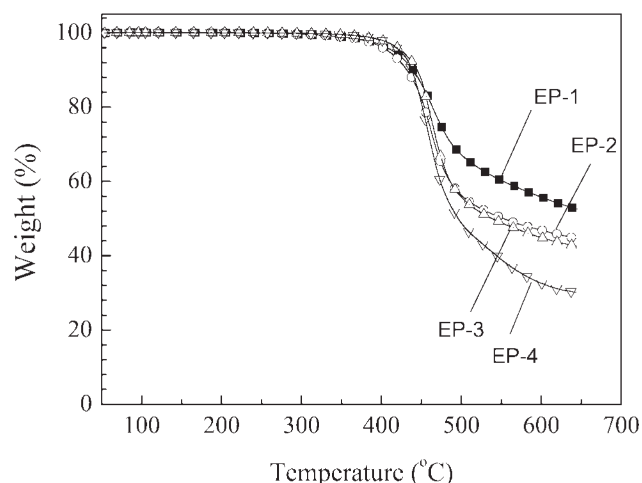


Figure 10 TGA curves of FBE and BE resins cured with different curing agents.

Thermal properties

Thermal properties of the cured resins were investigated by DMA and TGA, and the results are tabulated in Table II. Figure 9 shows storage modulus and tan δ curves of the cured epoxy resins. The onset temperatures of the storage modulus curves are in the order: EP-1 > EP-3 > EP-2 > EP-4, demonstrating that the fluorinated epoxy resins exhibited higher glass transition temperature (T_g) and better thermal-mechanical properties. The presence of bulky CF_3 groups in the resins hindered the motion of segments of the macromolecules, which resulted in increased T_g.[25] Figure 10 is a comparison of TGA curves of the cured epoxy resins. As can be seen from the results, there was no weight loss in all of the cured resins up to 300°C, and their initial decomposition temperatures (T_d) were determined in the range of 424–437°C. Meanwhile, the anaerobic char yields at 650°C were measured in the order of EP-1 > EP-2 > EP-3 > EP-4. The fluorinated epoxy resin exhibited better thermal stability than commercial epoxy cured with the same hardener. This might be attributed to the effect of the CF_3 groups in the resin backbone which resulted in improved thermal stability of cured resins.[25]

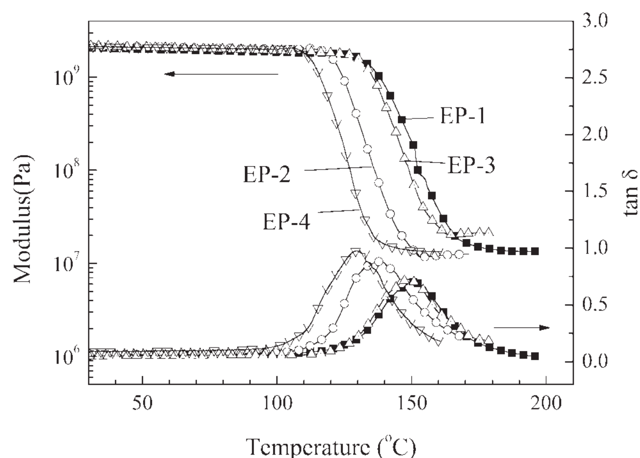


Figure 9 DMA curves of FBE and BE resins cured with different curing agents.

Mechanical, water absorption, electrical insulation, and dielectric properties

The mechanical properties of the cured epoxy resins were investigated and the results are summarized in Table III. EP-1 and EP-3 have similar flexural strengths of 121 and 135 MPa, respectively, which are 4 and 10 MPa higher than the corresponding epoxy cured with BP. The tensile strengths were measured in the range of 71–80 MPa and the elongation at breakage was in the range of 3.1–5.3%. FBE and BE cured with FBP showed the elongation of 4.3 and 5.3%, also superior to those cured with BP. The epoxy resin cured with fluorinated phenolic hardener thus exhibited improved mechanical properties than those cured with commercial hardener. Moreover, these results also indicated that all the cured resins have as good mechanical properties as compared with DGEBA epoxy cured with aromatic

TABLE III
Mechanical Properties of the FBE and BE Resins Cured with Different Curing Agents

Samples	Tensile Strength (MPa)	Tensile Modulus (GPa)	Elongation at Breakage (%)	Flexural Strength (MPa)	Flexural Modulus (GPa)
EP-1	72	2.2	4.3	121	2.4
EP-2	71	2.7	3.1	117	2.5
EP-3	79	2.5	5.3	135	2.6
EP-4	80	2.5	4.8	125	2.6

TABLE IV
Electrical, Dielectric Properties, and Water Absorption of the FBE and BE Resins Cured with Different Curing Agents

Samples	Water absorption (wt %)	$\rho_v \times 10^{-17}$ (Ω·cm)[a]	$\rho_s \times 10^{-16}$ (Ω)[b]	ε_r (1 MHz)[c]	Tan $\delta \times 10^3$ [d]
EP-1	0.27	0.8	4.6	3.8	3.6
EP-2	0.33	0.5	0.8	4.1	3.8
EP-3	0.29	1.3	1.5	4.1	4.5
EP-4	0.37	1.6	3.1	4.2	4.0

[a] ρ_v: volume resistivity.
[b] ρ_s: surface resistivity.
[c] ε_r: dielectric constant at 1 MHz at 25°C.
[d] Tan δ: dissipation factor.

amines as well as biphenyl epoxy resin cured with phenol novolac.[11,19]

Table IV shows the water absorption, electrical insulation, and dielectric properties of the cured epoxy resins. Low water absorption is a very important factor for the advanced microelectronic packaging applications because the absorbed H_2O molecules have obviously adverse impact on materials electrical insulation and dielectric properties. The cured fluorinated biphenyl-type epoxies (EP-1 and EP-2) have water absorption of 0.27 and 0.33%, lower than that of commercial biphenyl-type epoxies (EP-3 and EP-4), which is probably due to the strong hydrophobic effect of fluorinated groups in the resin backbone.[26]

The volume resistivity (ρ_v) and surface resistivity (ρ_s) were measured in the range of 0.5–1.6 $\times 10^{17}$ Ω cm and 0.8–4.6 $\times 10^{16}$ Ω, respectively. All the cured epoxy resins exhibited as good electrical insulation properties as commercial DGEBA epoxy cured with aromatic amines.[19] The dielectric constants (ε_r) and dielectric dissipation factors (tan δ) at 1 MHz were determined in the range of 3.8–4.2 and 3.6–4.5 $\times 10^{-3}$. The dielectric constants of highly aromatic epoxy resins measured were slightly higher than aliphatic epoxy resins.[16] However, the fluorinated epoxy resins (EP-1 and EP-2) exhibited better dielectric properties than that of commercial epoxy resins (EP-3 and EP-4), which could be interpreted by the low polarizability of the C—F bond and the large free volume of CF_3 groups.[27,28]

Flame-retardant properties

Flame retardancy is required for industrial application of epoxy resins to insure safety. The LOI, UL94 test, and cone calorimeter test have been widely used to evaluate the flame-retardant properties of polymer materials. Commonly used DGEBA epoxy cured with aromatic amine and *o*-cresol novolac epoxy cured by phenol novolac usually have a LOI value of 21–23 and cannot even pass UL94 V-2 classification.[29]

The LOI test results of EP 1–4 are in the range of 35.9–37.6 (Table V). It is obvious that the highly aromatic epoxy resins possess much higher LOI values than commercial DGEBA epoxy resins as well as *o*-cresol novolac epoxy resins. It also can be seen that the fluorinated biphenyl-type epoxy resins have higher LOI values than the commercial biphenyl-type epoxy resins cured with the same hardener. For instance, EP-1 shows a LOI value of 37.6, higher than that of EP-3 (36.4); EP-2 shows a LOI value of 36.4, higher than that of EP-4 (35.9). This might be attributed to higher thermal stability determined by TGA from the fluorine containing substituent.

UL94 test results indicated that all of the epoxy resins did not drop melt drips and ignite cotton. The fluorinated biphenyl-type epoxy resin exhibits shorter combustion time than commercial biphenyl-type epoxy resin. Total combustion times of EP-1 and EP-2 are 17.8 and 36.4 s, which are 0.3 and 51.1 s shorter than that of EP-3 and EP-4, respectively.

Figure 11 compares the heat release rates of the cured epoxy resins determined by cone calorimeter test, which is considered one of the most bench-scale methods for evaluating the polymer flammability. The heat release rate, especially the peak of heat release rate (pHRR), is the most important parameter to evaluate fire safety. It can be induced that fluorinated epoxy substituent in the chemical structure could reduce HRR and pHRR values. The total heat release value of EP-1 and EP-2 are 73 and 93 kW/m^2, which are 3 and 71 MJ/m^2 lower than that of EP-3 and EP-4, respectively. The fluorinated biphenyl-type epoxy resin exhibited lower heat release than commercial biphenyl-type epoxy resin cured with the same hardener. The cone calorimeter test data are summarized in Table V. The fluorinated biphenyl-type epoxy resins exhibited better flame retardancy because of longer ignition time, lower heat release, and smoke release.

TABLE V
Flame-Retardant Properties of the FBE and BE Resins Cured with Different Curing Agents

Sample	LOI	Burning Time (s)	UL94 Grades	TTI[a] (s)	pHRR[b] (kW/m^2)	THR[c] (MJ/m^2)	TSR[d]
EP-1	37.6	17.8	V-0	134	693	73	2514
EP-2	36.4	36.4	V-1	133	795	93	3194
EP-3	36.4	18.1	V-0	133	619	76	2122
EP-4	35.9	87.5	V-1	80	860	164	4948

[a] TTI: time to ignition.
[b] pHRR: peak of heat release rate.
[c] THR: total heat release.
[d] TSR: total smoke release.

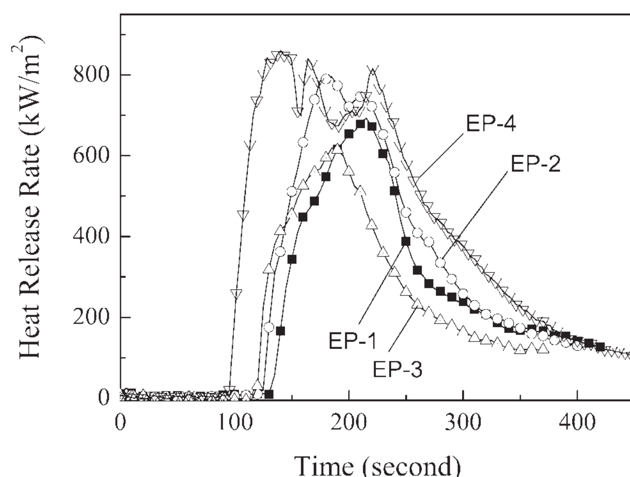


Figure 11 HRR curves of FBE and BE resins cured with different curing agents.

CONCLUSIONS

A fluorinated biphenyl-type epoxy resin (FBE) and a fluorinated biphenyl-type phenolic resins (FBP) were synthesized and characterized. Experimental results indicated that the fluorinated epoxy resins cured with phenolic hardeners exhibited good thermal stability, high thermal mechanical properties, superior dielectric properties, and low moisture absorption. Excellent flame-retardant properties were found for the fluorinated biphenyl-type epoxy resins with high LOI value and UL94 V-0 ratings.

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