European Polymer Journal 109 (2018) 110–116

Contents lists available at ScienceDirect

European Polymer Journal

journal homepage: www.elsevier.com/locate/europolj

Preparation and characterization of ultralow dielectric and fibrous epoxy thermoset cured with poly(arylene ether ketone) containing phenolic hydroxyl groups

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ARTICLE INFO

Keywords:
Poly(arylene ether ketone)
Electrospinning
Ultralow dielectric constant
Epoxy resins

ABSTRACT

Poly(arylene ether ketone) containing naphthalene and phenolic hydroxyl groups (HPAEK) was prepared by polycondensation and demethylation reaction. Polymer cured epoxy resin with high-*Tg* was obtained through reactions between oxirane ring of epoxy and phenolic hydroxyl groups of HPAEK. Electrospinning technology was used to fabricate porous film. The porous film shows a high *Tg* value (262 °C), a low dielectric constant (1.9 at 1 MHz), and a low coefficient of thermal expansion (52 ppm °C^{-1}). The thermal stability and water contact angle were also measured. The naphthalene and phenolic hydroxyl containing poly(arylene ether ketone) provides us with a new strategy to achieve ultralow dielectric constant materials via electrospinning.

1. Introduction

Epoxy resins have been used as insulation materials for dielectric devices due to their good balance of properties such as superior electrical and mechanical properties, excellent solvent and chemical resistance and good adhesion to many substrates [1,2]. However, unmodified epoxy thermosets are relatively brittle, thus displaying poor resistance to crack propagation [3]. One of the widely used approaches for improving the toughness of epoxy resin is to modify it by liquid rubbers [4–7]. The dispersed rubber particles could enhance the toughness of epoxy resin significantly. Nevertheless, a decrease in thermal stability and tensile modulus was observed for the resulted thermosets. Another approach is to incorporate engineering polymers into epoxy matrix [8,9]. The thermal stability of thermosets was also greatly improved. However, poor interfacial adhesion between different phases leads to a decrease in the improvement effect of the fracture toughness. To improve interfacial adhesion, functionalized polymers containing epoxy [10,11], amine [12,13] and phenolic hydroxyl groups [14,15] are used as the modifier or curing agent to form covalent linkage. Low molecular weight poly(phenylene oxide) (PPO) with terminal phenolic hydroxyl groups was utilized to modify epoxy resin, and the thermosets showed good thermal stability and improved dielectric property (2.6–3.1 at 1 GHz) [16]. Lin et al. prepared poly(aryl ether ketone) with phenol pendent group in every repeating unit. Flexible and transparent films were obtained, which showed high *Tg* and thermal stability [17].

To meet the requirements of modern electronic industry, numerous investigations have been carried out to prepare kinds of modified epoxy resins, such as the incorporation of fluorine [18,19], nanoparticles [20,21] and porous structures [22]. Since the design of molecule structure is complicated and time-cost, the strategy of incorporation of voids ($k \approx 1$) into epoxy resins has been an attractive approach to decrease the dielectric constant.

Over the years, electrospinning has been considered as an efficient technique to fabricate fibrous films, due to its potential for industrial-scale processing and repeatability in control of fiber dimension [23,24]. It has been demonstrated that electrospun polymer films exhibit ultralow dielectric constant (usually below 2.2) compared with as-cast films [25–28]. However, it is more difficult to fabricate high quality fibrous films using epoxy solution with low concentration. Thus, beads and defects are inevitable. Adding polymers into solution is an efficient way to increase viscosity and obtain electrospun epoxy film at a low concentration.

In this work, we synthesized fluorinated poly (arylene ether ketone) containing naphthalene and phenolic hydroxyl groups (HPAEK) and presented a simple and effective approach for the fabrication of epoxy films with ultralow dielectric constant through electrospinning technique. 4,4′-Diglycidyl (3,3′,5,5′-tetramethylbiphenyl) epoxy resin (TMBPER) is a kind of liquid crystal epoxy resin possessing high thermal and mechanical properties.[29–31] Fluorinated HPAEK was

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https://doi.org/10.1016/j.eurpolymj.2018.08.059
Received 7 July 2018; Received in revised form 17 August 2018; Accepted 31 August 2018

used as a suitable macromolecule curing and toughness agent due to its linear structure and phenolic hydroxyl groups in the repeating unit. Flexible and tough films were prepared through cast and electrospinning. The detailed characterization including thermal stability, dielectric constant and water contact angle was also provided.

2. Experimental

2.1. Materials

1,5-Bis(4-fluorobenzoyl)-2,6-dimethoxynaphthalene (DMNF) and 4,4′-diglycidyl (3,3′,5,5′-tetramethylbiphenyl) epoxy (TMBPE) were synthesized according our previous work [32,33]. 2-Methylimidazole, 4,4′-(hexafluoroisopropylidene)diphenol and boron tribromide (BBr_3) were purchased from Alddin chemistry Co. Ltd and used as received. Toluene, sulfolane, N-methyl-2-pyrrolidinone (NMP), dichloromethane, potassium carbonate were purchased from Beijing chemical company and used as received.

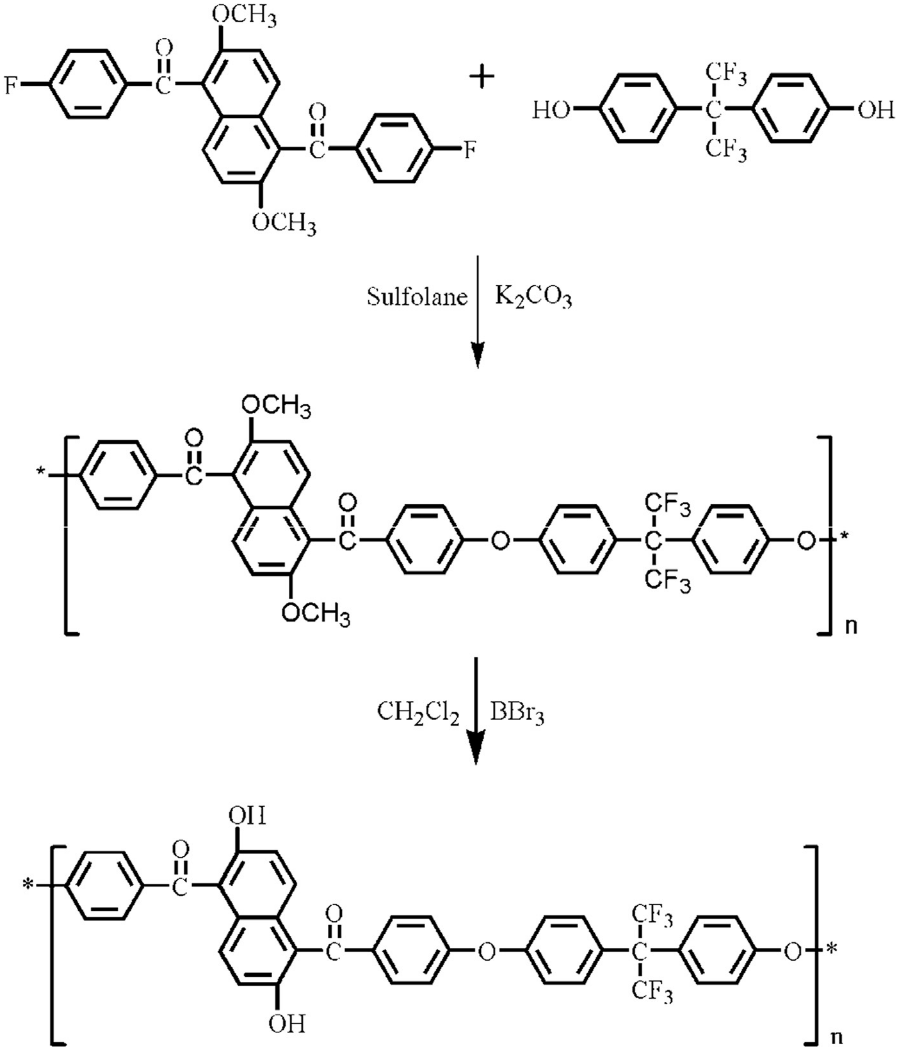
2.2. Synthesis of poly(arylene ether ketone) containing naphthalene and phenolic hydroxyl groups (HPAEK)

HPAEK was synthesized through polycondensation of DMNF and 4,4′-(hexafluoroisopropylidene)diphenol, and demethylation reaction, as shown in Scheme 1. A mixture containing DMNF (6.48 g, 0.015 mol), 4′-(hexafluoroisopropylidene) diphenol (5.04 g, 0.015 mol) and K_2CO_3 (2.277 g, 0.0165 mol), sulfolane (24 mL) and toluene (8 mL) was added to a three-necked flask equipped with a nitrogen inlet, a mechanical stirrer and a Dean-Stark trap. The mixture was heated at 140 °C for 3 h to remove the water by azeotropic distillation with toluene. Then the reaction was heated to 200 °C and stirred for 2 h. The high viscosity mixture was coagulated into a large excess of deionized water. The precipitation was washed with deionized water several times and dried under vacuum at 80 °C for 24 h.

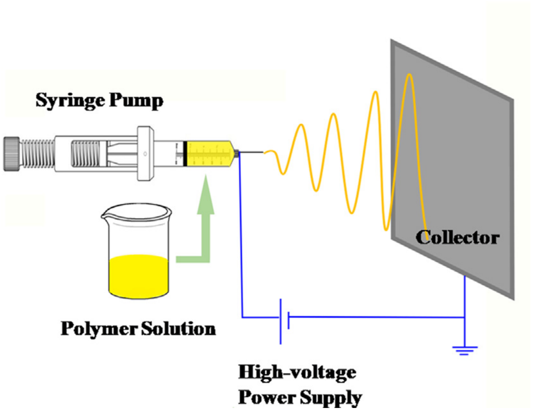
The demethylation of obtained polymer was performed as follows: 1.0 g of polymer was dissolved into 40 mL anhydrous dichloromethane. The solution was cooled to 0–3 °C, and 5 mL solution of BBr_3 in dichloromethane ($1\ \text{mol L}^{-1}$) was added dropwise. The mixture was stirred at room temperature under nitrogen atmosphere for 24 h. The mixture was poured into ice water to hydrolyze the BBr_3 and the boron complexes. The HPAEK was washed with deionized water. The resulting polymer was dried at 80 °C for 24 h.

2.3. Preparation of macromolecule cured TMBPER resin

HPAEK (2.62 g) and TMBPE (1.34 g) were dissolved in 15 mL DMF. After all contents were dissolved, 2-methylimidazole (0.04 g) was added into the solution. The solution was poured onto glass plate and heated at 60 °C for 24 h to dry the film. And then the film was cured at 100 °C for 2 h, 150 °C for 2 h, at 200 °C for 2 h in a convection oven.



Scheme 1. The synthesis of HPAEK.



Scheme 2. The electrospinning setup.

2.4. Fabrication of electrospun film

HPAEK (1.96 g) and TMBPE (1.01 g) were dissolved in 10 mL DMF. And then 2-methylimidazole (0.03 g) was added into the solution, followed by magnetic stirring for 10 min to obtain the precursor solution. The fiber mat of HPAEK-TMBPER was electrospun using a typical electrospinning machine, as shown in Scheme 2. The polymer solution was loaded into plastic syringe equipped with 19 gauge stainless needle. A high voltage of 12 kV was applied between the needle tip of the spinneret and tinfoil on the collection screen. The distance between the needle tip and collector plate was set at 20 cm. The electrospinning process was carried out at room temperature in air. The as-spun film was cured at 100 °C for 4 h, at 150 °C for 2 h, and at 200 °C for 2 h in a convection oven.

2.5. Characterization

^{1}H-NMR spectra were measured at 500 MHz on a Bruker AVANC-EIII500 spectrometer using deuterated dimethyl sulfoxide (DMSO-d_6) and deuterochloroform (CDCl3) as the solvent and tetramethylsilane (TMS) as the standard. Fourier transform infrared (FT-IR) was performed using a Nicolet Impact 410 spectrometer. Dielectric constant of polymer film was measured at room temperature by a 4292 precision impedance analyser in a range of frequencies from 1 kHz to 1 MHz. And the thickness of all films is about 150 μm. The cure kinetics of the cast film was measured by using differential scanning calorimeter (TA instrument DSC Q20) under a nitrogen flow of 50 mL min^{-1}. Morphology of the fiber mats was characterized by a scanning electron microscopy (SEM, FEI Nova NanoSEM 450). Thermogravimetry analysis (TGA) was performed on Perkin-Elmer Pyris1 thermogravimetric analyser from 80 to 700 °C at a heating rate of 10 °C min^{-1} under a nitrogen atmosphere. Dynamic mechanical analysis (DMA) was carried out with a TA instrument DMA Q800 at a heating rate of 3 °C min^{-1} and a load frequency of 1 Hz in film tension geometry. Thermo-dimensional characteristics of the films were examined using a Mettler Toledo TMA/SDTA 841e thermomechanical analyser at a heating rate of 10 °C min^{-1} under nitrogen atmosphere. The mechanical property of samples was evaluated at room temperature on SHIMADZU AG-I 1 KN at a speed of 5 mm min^{-1}. The Young' modulus and hardness of the cast films were measured with an Agilent Nano Indenter G200 with an XP-style actuator and continuous stiffness measurement (CSM) method.[34] The water contact angle was measured using contact angle goniometer (Drop shape Analysis DSA 30, Kruss, Germany) at room temperature.

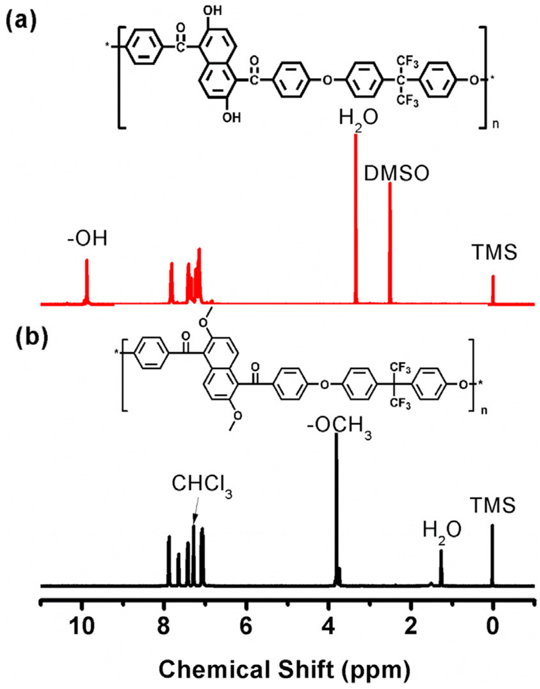


Fig. 1. The ^{1}H NMR spectra of (a) HPAEK and (b) PAEK.

3. Results and discussion

3.1. Characterization of HPAEK and TMBPER based epoxy thermosets

The HPAEK was synthesized by a demethylation reaction, as depicted in Scheme 1. The ^{1}H-NMR spectra of PAEK and HPAEK are shown in Fig. 1. The demethylation of PAEK was conducted in dichloromethane using BBr_3 and the HPAEK precipitated from dichloromethane due to the polar nature of phenolic hydroxyl group. Peaks between 7 and 8 ppm are attributed to the protons on the aromatic ring. The peak at 9.9 ppm corresponds to phenolic hydroxyl group. And the peak at 3.8 ppm which attributed to the $-OCH_3$ group disappeared. The obtained HPAEK can be used as curing and a toughness agent for epoxy resins.

The curing kinetics study of epoxy resins is critical for optimization of processing conditions and production of high performance thermosets.[35]Thermo-curing process of HPAEK-TMBPER was evaluated by differential scanning calorimetry (DSC) at different heating rate of 5, 7.5, 10, 12.5 and 15 °C min^{-1}, and the results are shown in Fig. S1. 2-Methylimidazole was used as a catalyst for the ring-open reaction of epoxy groups. There is one exothermic peak at approximately 150–180 °C, which could be attributed to the reaction between epoxy groups and phenolic hydroxyl groups. Table S1 lists the onset curing temperature (T_o), the peak curing temperature (T_p) and the end set curing temperature (T_e) of HPAEK-TMBPER system. The T_p of HPAEK-TMBPER during the curing process rose with increasing the heating rate. In this work, the curing kinetics of HPAEK-TMBPER were evaluated by Kissinger method to obtain the overall activation energy (Ea).[36] The relation can be expressed as Eq. (1).

$$\frac{d[\ln(\alpha/Tp2)]}{d(1/Tp)} = -\frac{\mathrm{Ea}}{\mathrm{R}} \qquad (1)$$

where α is the heating rate, R is the gas constant (8.314 J mol^{-1} K^{-1}). If the plot of $\ln(\alpha/T_p^2)$ versus $1/T_p$ is linear, the overall activation energy can be obtained from the slope of the corresponding straight line. Fig. S2 shows the curve of $\ln(\alpha/T_p^2)$ versus $1/T_p$ for the HPAEK-TMBPER system. The linear correlation coefficient R-square is 0.9945. The Ea of HPAEK-TMBPER system is 65.2 kJ mol^{-1}.

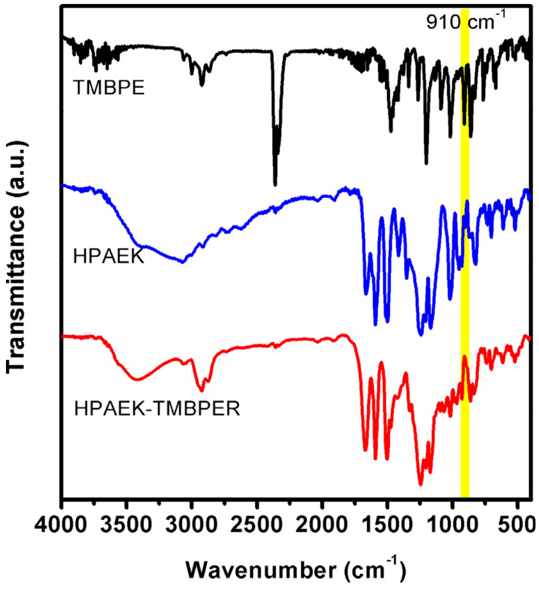


Fig.2. Normalized FT-IR spectra of HPAEK and epoxy resin.

After curing, the films were insoluble in organic solvents, which indicating the network formation. The films show excellent flexibility. Their FT-IR spectra before and after curing are shown in Fig. 2. The complete disappearance of epoxy peak at 910 cm^{-1} in epoxy resins confirmed the success of entirely curing.

3.2. Morphology of ES-HPAEK-TMBPER

The fiber morphology and dimensions of the ES-HPAEK-TMBPER were investigated by SEM (shown in Fig. 3). As the content of HPAEK and TMBPER is 30%, we can fabricate fibrous films by electrospinning. The film shows continuous and crossed fibers similar to nonwoven texture. As seen in Fig. 3, the fibers show smooth surface and no significant defects or beads exist in their structure. The diameter of the fibers varies between 700 and 1000 nm. The electrospinning film is tough and flexible after curing at 200 °C for 2 h.

3.3. Thermal properties of all thermosets

Fig. 4 shows the temperature dependence of storage modulus (E’) and tan δ for HPAEK and all thermosets. The glass transition temperature (*Tg*) is determined by the maximum value of tan δ curves. From the DMA spectra, the *Tg* values of HPAEK-TMBPER and ES-HPAEK-TMBPER are 234 °C and 262 °C, respectively. The HPAEK cured epoxy resins show high Tg, which may be attributed to the aromatic structure of HPAEK. The advantage of HPAEK curing agent could be prominent compared with the Tg values of small molecule cured TMBPER. The *Tg* of methyl hexahydrophthalic anhydride (MeHHPA) cured TMBPER is 156 °C.[37] HPAEK is an epoxy curing agent with high Tg. In addition, it is observed that both storage modulus and tan δ of all HPAEK cured epoxy resins exhibit a single transition curve, suggesting a good compatibility of the system and the network formation.

The dimensional stability is regarded as one of the most important properties for low-*k* materials. Therefore, the coefficient of thermal expansion (CTE) of HPAEK and HPAEK cured films was evaluated by thermomechanical analyzer. As shown in Fig. 5, the CTE values between 50 ~ 150 °C for HPAEK, HPAEK-TMBPER and ES-HPAEK-TMBPER are 74 ppm °C^{-1}, 72 ppm °C^{-1} and 52 ppm °C^{-1}, respectively. After crosslinking, HPAEK-TMBPER film showed lower CTE compared with HPAEK film, which may be attributed to the crosslinking structure. The significant reduction of CTE value for ES-HPAEK-TMBPER film is attributed to the existence of holes in the fibrous structure. When the fibers undergo expansion at high temperature, the increased dimension may occupy the holes in the fibrous structure, resulting in lower thermal expansion.

The thermal stability of HPAEK and epoxy resin thermosets was evaluated by TGA in nitrogen atmosphere, and the results are presented in Fig. 6. And the derivative thermogravimetry (DTG) curves are given in Fig. S3. The relative thermal stability of cured resins was compared by the temperature of 5% and 10% weight loss (T_{d5} and T_{d10}), together with the char yield at 700 °C. The T_{d5} values of HPAEK-TMBPER and ES-HPAEK-TMBPER are 354 °C and 347 °C, respectively. And the T_{d10} of HPAEK-TMBPER and ES-HPAEK-TMBPER are 400 °C and 375 °C, respectively. The percent char yield at 700 °C of HPAEK-TMBPER and ES-HPAEK-TMBPER is 49% and 40%, respectively. The ES-HPAEK-TMBPER showed lower thermal stability than HPAEK-EMBPER, which may be due to the holes in the porous structure. When the temperature increased, the fibers were much easier to degrade compared with dense film. The HPAEK cured epoxy resins showed higher thermal stability compared with MeHHPA cured TMBPER (T_{d5} = 316 °C)[37], which may be attributed to high thermal stability of HPAEK (T_{d5} = 421 °C) and high cross-linking.

3.4. Mechanical property

The mechanical properties of HPAEK and all thermosets were measured by universal mechanical test and nanoindentation measurement, and the results are shown in Table 1. The tensile strength of

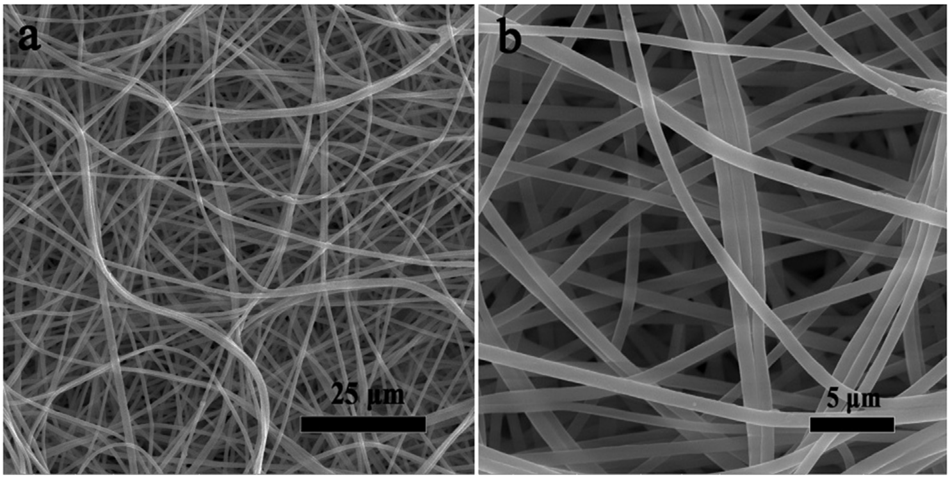


Fig. 3. (a) Magnified × 3000, (b) magnified × 10000 SEM images of ES-HPAEK-TMBPER thermoset.

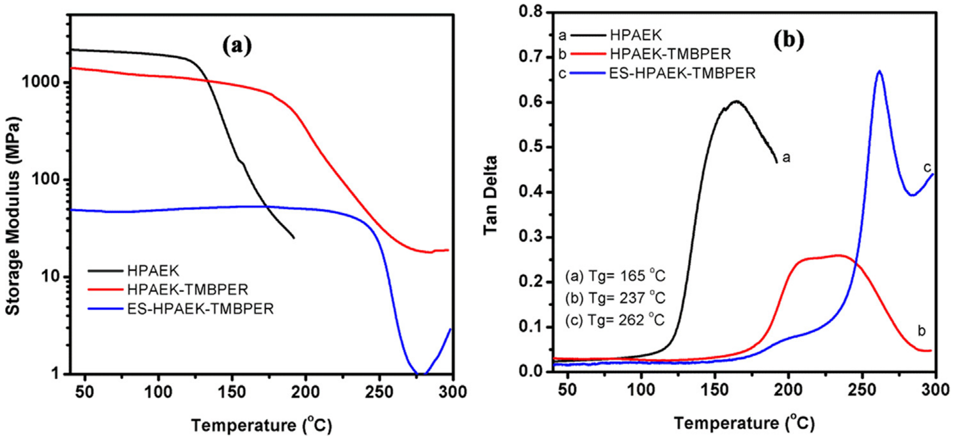


Fig. 4. (a) Storage modulus (E'), (b) tan δ curves of HPAEK and all epoxy resin thermosets.

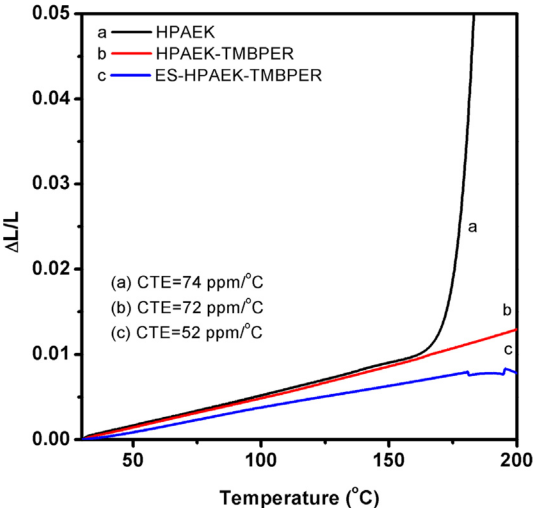


Fig. 5. Thermal expansion curves of HPAEK, HPAEK-TMBPER and ES-HPAEK-TMBPER thermosets.

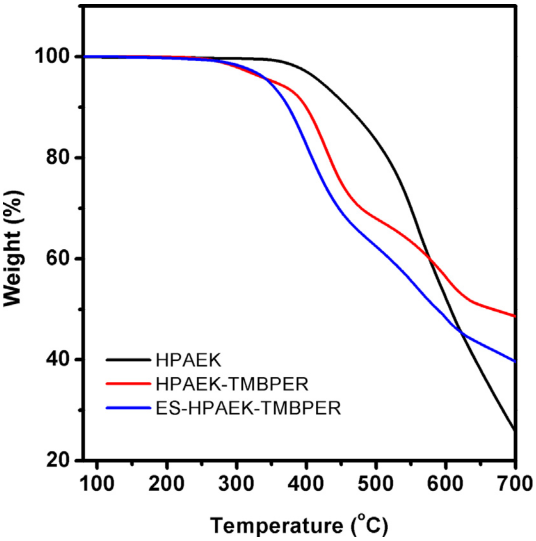


Fig. 6. TGA curves of HPAEK and all epoxy resin thermosets.

HPAEK-TMBPER and ES-HPAEK-TMBPER are 51.39 MPa and 3.46 MPa, respectively. And the elongation at break of HPAEK-TMBPER and ES-HPAEK-TMBPER are 7.79% and 15.89%, respectively. The HPAEK cured epoxy film is flexible and tough. Nanoindentation was used to examine mechanical properties of cured epoxy by analyzing Young's modulus and hardness. HPAEK-TMBPER shows higher Young's modulus and hardness than HPAEK, which may be attributed to the crosslinking structure of HPAEK-TMBPER. The different values generated from the two test methods could be attributed to the different loading rate applied. A displacement rate of 5 mm min^{-1}, which is about 0.0017 s^{-1} in the initial loading stage, was used for the universal mechanical test; while for the nanoindentation measurement, a constant strain rate of 0.05 s^{-1} was applied throughout the test.[38]

3.5. Contact angle

The water absorption is also very crucial for low-k materials to be applied in microelectronics. Materials with low water absorption usually imply that the corresponding electronic devices possess good operating conditions. Water contact angle was measured to investigate the nature of water absorption, as displayed in Fig. 7. It is found that HPAEK-TMBPER (88°) showed higher contact angle than MeHHPA cured TMBPER (83°), suggesting the incorporation of HPAEK could improve the hydrophobicity without influencing the surface wettability significantly. The highest contact angle achieved for ES-HPAEK-TMBPER is 145°, which indicates that porous rough polymer film fabricated by electrospinning possesses enhanced hydrophobicity.

3.6. Dielectric properties

Fig. 8a shows the relationship of dielectric constant of HPAEK and all thermosets with frequency ranging from 1 kHz to 1 MHz at room temperature. The dielectric constant of all samples decreased slightly over the measured frequency. Contrasted with dense film, the ES-HPAEK-TMBPER film exhibits much lower dielectric constant. The dielectric constant of ES-HPAEK-TMBPER is 1.9 at 1 MHz, which is lower than that of HPAEK-TMBPER (3.4), and is considerably lower than other low dielectric epoxy resins (2.0–4.0) [39–44]. The comparison of low dielectric constant epoxy resins is shown in Table S2 in detail. The dielectric constant can be reduced by increasing the free volume, decreasing the molecule's polarization, and introducing voids into the film. In contrast with MeHHPA cured TMBPER thermoset (3.77 at 1 MHz) [37], the lower dielectric constant of HPAEK-TMBPER may be attributed to the increased free volume derived from bulky naphthalene groups and –CF_3 groups with lower polarizability. As to ES-HPAEK-TMBPER, the lower dielectric constant is owing to the fibrous structure and the low dielectric constant of air (≈1). The dielectric loss of all thermosets is displayed in Fig. 8b. It is important to maintain low dielectric loss for achieving lower conversion of electric energy into heat. The dielectric loss of all thermosets is between 0 and 0.04. The

Table 1

Mechanical properties of HPAEK and epoxy resin thermosets.

Sample	Tensile strength (MPa)	Elongation at break (%)	Young's modulus[a] (MPa)	Young's modulus[b] (GPa)	Hardness[b] (GPa)
HPAEK	64.93 ± 1.73	7.70 ± 0.62	1169.80 ± 75.21	3.8 ± 0.4	0.29 ± 0.05
HPAEK-TMBPER	51.39 ± 6.93	7.79 ± 1.24	960.34 ± 55.50	4.2 ± 0.1	0.32 ± 0.01
ES-HPAEK-TMBPER	3.46 ± 0.43	15.89 ± 3.22	56.17 ± 4.72	0.50 ± 0.08	0.17 ± 0.03

[a] Young's modulus was measured by universal mechanical test.

[b] Young's modulus and hardness were determined from the nanoindentation measurements under different loadings.

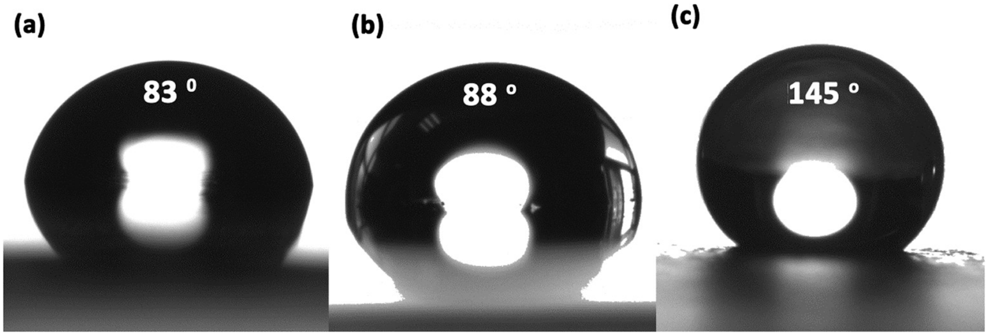


Fig. 7. Water contact angle images of (a) HPAEK, (b) HPAEK-TMBPER and (c) ES-HPAEK-TMBPER.

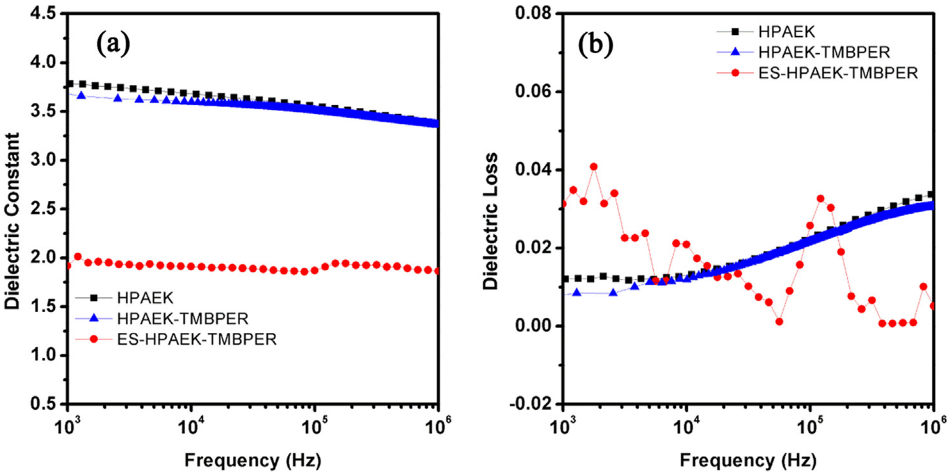


Fig. 8. Frequency dependence of (a) dielectric constant and (b) dielectric loss of HPAEK and all epoxy resin thermosets.

dielectric loss of HPAEK-TMBPER and ES-HPAEK-TMBPER at 1 MHz is 0.031 and 0.005, respectively. These results suggest that initial incorporation of pores into materials plays a significant role in preparing an ultralow dielectric constant material.

4. Conclusions

Poly(arylene ether ketone) containing naphthalene and phenolic hydroxyl groups was prepared and used as the curing and toughness agent for TMBPER thermosets. Flexible films were obtained via the reaction of oxirane ring and phenolic hydroxyl groups. Porous film was fabricated by electrospinning technology. Because of the bulky naphthalene and trifluotomethyl groups in HPAEK, the as-cast film showed high *Tg*, good thermal stability and low dielectric constant (3.4 at 1 MHz). After electrospinning, the fibrous film showed higher *Tg*, lower dielectric constant (1.9) and coefficient of thermal expansion (52 ppm °C^{-1}). Therefore, HPAEK is a good curing and toughness agent for epoxy resins. Furthermore, the fibrous film shows a great potential for low dielectric application.

Acknowledgments

We acknowledge the financial support from the Natural Science Foundation of China (No. 21374034 and 21474036).

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.eurpolymj.2018.08.059.

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