Low-Temperature-Curable and Photo-Patternable Benzocyclobutene-Derived Aggregation-Induced Emission (AIE)-Active Polymer Dielectrics

Ziwei Yuan, Meng Xie, Jianlei Qian, Wenjie Fan, Menglu Li, Liao Guo, Yan Sun*, and Wenxin Fu*

1Institute of Chemistry Chinese Academy of Sciences
2Henan University

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Abstract

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1. INTRODUCTION

With the rapid evolution of fifth-generation mobile communication technology (5G),[1] the fields of Internet of Things (IoT) and Augmented Reality (AR) technologies have witnessed significant progress. This surge in technological advancement has given rise to an array of electronic devices such as smartphones, smart bracelets, and smart glasses, along with other flexible wearable products, which have become ubiquitous in our daily lives. These products are continuously evolving towards achieving higher performance, miniaturization, and increased integration.[2-4] The feature size of chips is constantly shrinking. Consequently, chip feature sizes continue to shrink, placing greater demands on packaging technology and materials. Among the commonly employed packaging materials, such as epoxy,[5-6] polyimide,[7-8] polybenzoxazole,[9-11] BCB thermosets have emerged as superior choices for the multilayer wiring processes in integrated circuit manufacturing.[12-16] For example, commercial DVSBCB resins, known for their exceptional dielectric properties, high thermal stability, low water absorption and dimensional stability, serve as ideal interlayer dielectric (ILD) materials in multi-chip assemblies.[17-19] However, there remains considerable room for advancement within the realm of traditional BCB thermosets.

In the context of flexible wearable electronic products, the imperative development of packaging materials featuring low curing temperatures ([2] 200 °C) becomes apparent, as these substrates are unable to withstand high curing temperatures.[20-21] BCB, composed of a thermostable benzene ring and a kinetically unstable four-membered ring, typically undergoes ring-opening reaction to yield o-quinodimethane at elevated temperatures ([2] 250 degC).[22-27] It is well-established that the introduction of substituents onto the four-membered ring of BCB can substantially reduce the activation energy barrier for ring-opening reactions, consequently lowering the ring-opening temperature (as indicated by the peak temperature of DSC curves). Previously, Chino et al. have reported several BCB monomers with the ring-opening temperatures [2] 230 degC. However, their investigations primarily focused on thermal or radical polymerization processes, with limited exploration into the packaging properties as polymer materials.[21, 28-29] Gies et al. described an ether bond functionalized BCB monomer and degradation pathways of thermal cured polymers.[30] Dobish et al. reported the successful reduction of the cross-linking temperature by 100 degC through the incorporation of 1-alkoxy-substituted BCB as pendant side chains in a polyacrylate matrix.[31] Nonetheless, the majority of studies have predominantly revolved around complex BCB monomer syntheses, with unstable polymer or polymerization relying on extra crosslinkers, no subsequent research on the packaging properties of BCB...
resins.

In addition, increasing the demand for enhanced positioning accuracy during the wafer bonding processes in three-dimensional (3D) stacked packages has become a prominent concern. Current positioning tools for multilayer wafer bonding predominantly rely on optical microscopes. In response to this challenge, we are exploring the replacement of existing interlayer positioning packaging materials with luminescent materials to achieve superior positioning accuracy through fluorescence microscopy. Luminescent materials have garnered widespread attention across various fields, including luminescent sensing, information storage, bioimaging, and crack detection in recent decades. Tetraphenylethylene (TPE) stands as one of the most extensively studied aggregation-induced emission (AIE) molecules within the realm of luminescent materials, owing to its ease of synthesis, versatility in modifications, and intriguing photostability characteristics. Numerous studies have documented the incorporation of TPE moieties into polymers to craft temperature-responsive luminescent materials with reversible cross-linking properties. As we have seen, there are no relevant reports on the use of TPE materials for interlayer positioning in 3D packaging.

Building upon these backgrounds, we designed a novel BCB-based monomer, TPE-BCB, featuring a substituent on the four-membered ring, with the aim of producing BCB resins that exhibit low curing temperatures. TPE-BCB was synthesized through Williamson reaction with an impressive 74.3% yield (Scheme 1). Notably, this monomer exhibits the ability to undergo full curing at low temperatures (190 degC) and demonstrates an aggregation-induced emission effect. Our approach involved the utilization of hydroxy-modified tetraphenylethylene (1,2-bis(4-hydroxyphenyl)-1,2-diphenylethylene) as a precursor for Williamson reaction. This dual-purpose strategy aimed to achieve two critical objectives: firstly, the introduction of phenolic hydroxyl groups onto the four-membered ring of BCB to reduce the ring-opening temperature; secondly, the incorporation of TPE moiety inducing an AIE effect in this monomer. The fully cured resins resulting from this strategic synthesis displayed outstanding mechanical and dielectric properties, low water absorption and photo-patternable capabilities, promising to deliver enhanced positioning accuracy within the realm of microelectronics.

SCHEME 1. Functional low-temperature cured BCB monomer reported previously and TPE-BCB monomer with AIE effect in this work.
2. RESULTS AND DISCUSSION

2.1. Synthesis and characterization of TPE-BCB and BCB-diphenol monomers

The Williamson reaction’s mild reaction temperature makes it an ideal choice for the preparation of BCB monomers capable of low-temperature crosslinking. However, the synthesis routes typically reported for functional BCB monomers tend to be intricate, often yielding less-than-optimal results. In our approach, we employed compounds containing phenol hydroxyl groups as raw materials. These compounds readily undergo activation by inorganic alkalis, such as cesium carbonate, resulting in significantly improved reaction yields of 59% and 74.9%. The synthetic pathway was outlined in Scheme 2. Furthermore, Figures S1 to S4 displayed the $^1$H NMR and $^{13}$C NMR spectra of both monomers for comprehensive characterization. The successful incorporation of BCB units was verified by $^1$H NMR. As shown in Figure S1, the emergence of proton signals at 5.63-3.24 ppm, corresponding to the CH$_2$ and hypomethyl CH groups on the four-membered BCB ring, unequivocally confirmed the successful synthesis of the desired product, TPE-BCB. A parallel observation was made in the case of BCB-diphenol.

SCHEME 2. Synthesis routes for TPE-BCB and BCB-diphenol monomers.

2.2. Curing Behavior of TPE-BCB and BCB-diphenol monomers

The ring-opening temperature serves as a critical parameter for defining curing conditions. Differential scanning calorimetry (DSC) is the conventional method for determining the curing temperature of polymer materials. For BCB derivatives substituted on the benzene ring, such as DVSBCB, the typical ring-opening temperature hovers around 250 °C. Whereas, in the case of TPE-BCB and BCB-diphenol, a noticeable reduction in the ring-opening temperature to 188 °C and 190 °C, respectively, was observed, as depicted in Figure 1a. This reduction can be attributed to the incorporation of ether bonds onto the BCB four-membered rings, resulting in an approximate 25% decrease on the ring-opening temperature of the traditional BCB ones.

The thermal crosslinking mechanism was illustrated in Figure 1b and several studies have previously reported the potential breakage of the ether bond between the eight-membered ring formed after BCB cross-linking and the benzene ring.$^{[30]}$ To investigate this phenomenon, Fourier transform infrared (FTIR) analyses were conducted to identify phenolic hydroxyl groups formed as a consequence of ether bond rupture. In Figure 1c, a distinct stretching vibration peak of the hydroxyl group, arising from the breakage of the ether bond, was observed within the range of 4000 – 3000 cm$^{-1}$. Notably, BCB-diphenol exhibited incomplete curing due to its susceptibility to degradation. In contrast, during the curing process of TPE-BCB, no stretching vibration peak within the 4000 – 3000 cm$^{-1}$ range was observed (Figure 1d), signifying the stability of the ether bond throughout curing (where full curing was achieved at 170 °C for 3 h).
FIGURE 1. (a) DSC curves of TPE-BCB and BCB-diphenol monomer. Heat flow: 10 °C/min; (b) The thermal crosslinking mechanism of TPE-BCB polymer; (c–d) FTIR spectras of polymers with different thermal curing durations.

To further elucidate the thermal crosslinking mechanism, Raman spectroscopy was employed to determine the involvement of double bonds in crosslinking. The Raman spectra of TPE-BCB monomer and resin were shown in Figure S5. The presence of a band at 1564 cm$^{-1}$, attributed to the stretching vibration of olefinic C=C bonds, exhibited distinct signals before and after crosslinking.$^{[45]}$ It had been confirmed that the double bonds in tetraphene did not engage in the crosslinking process.

2.3. Dielectric properties and Hydrophobicity of TPE-BCB resin

The dielectric constant ($D_k$) and dielectric loss ($D_f$) are pivotal parameters that serve as indicators of the insulating properties of polymer materials, significantly influencing packaging performance. The parallel-plate capacitor method was employed to measure the $D_k$ and $D_f$ of TPE-BCB and DVSBCB resins in the frequency range of 1 kHz to 1 MHz, as presented in Figure 2c and 2d. It is evident that the average $D_k$ of TPE-BCB was lower than that of DVSBCB, while the average $D_f$ was a little bit higher. Importantly, we also evaluated the dielectric performance of TPE-BCB at 10 GHz, utilizing the split post dielectric resonator method as shown in Figure 2b. Notably, TPE-BCB demonstrated exceptional dielectric properties, with $D_k$ below 2.8 and $D_f$ below 2.0×10$^{-3}$.

In the context of serving as low-dielectric packaging materials for diverse microelectronic devices, it is imperative for BCB resins to exhibit excellent hydrophobicity. This attribute ensures the stability of the material’s mechanical and dielectric properties. Our assessment of hydrophobicity employed static contact angle analysis and water adsorption experiments on TPE-BCB resin. The static water contact angle image was presented in Figure 2a, showcasing a static water contact angle of 109° on the surface of TPE-BCB resin, which closely matched that of commercial DVSBCB resin (108°). Furthermore, TPE-BCB resin displayed low water uptake values of 0.23% at room temperature for 72 h (Table S2). These findings, characterized by a substantial static contact angle and minimal water uptake, affirmed the outstanding hydrophobicity exhibited by cured TPE-BCB resin.
2.4. Mechanical properties and thermostability of TPE-BCB resin

The mechanical properties of the materials were assessed by nanoindentation measurements (Figure 3). The upper sections of Figure 3a and 3b illustrated the load-displacement curves of two resins. The prominent step at the peak load represents the phase where the material sustains the maximum load, maintaining a constant force. Concurrently, the indenter continued to exert pressure on the TPE-BCB material, leading to a discernible increase in displacement. Conversely, the step observed at the end of the unloading curve arises from the sample’s elastic characteristics. During unloading, the indenter was displaced due to the elastic recovery of the substrates.

In the hardness-depth curves of TPE-BCB (middle section of Figure 3a), a rapid decline in hardness was evident as the indentation depth increased from 0 to 130 nm. This phenomenon, known as the scale effect, arose from the heterogeneous mechanical properties within resins and the presence of surface hardening. A similar observation was made in the case of DVSBCB with the indentation depth increased from 0 to 190 nm. The higher content of benzene rings within the TPE-BCB resin contributed to a more pronounced scale effect and a greater degree of surface hardening.

The hardness of TPE-BCB resin measured approximately 0.33 GPa, surpassing that of DVSBCB (0.27 GPa). Additionally, the Young’s modulus of TPE-BCB was determined to be 4.5 GPa, once again surpassing DVSBCB (3.7 GPa). The improvement in mechanical properties of TPE-BCB could be attributed to the increased abundance of benzene rings within the resin’s structure.
The thermostability of TPE-BCB resin was evaluated by thermogravimetric analysis (TGA) under a N$_2$ atmosphere, and the results were presented in Figure S6. The resin exhibited a 5% weight loss temperature ($T_{d5}$) of 248.2 °C, indicating its stability within the working environment of flexible wearable devices (≤130 degC).

2.5. The photophysical properties of TPE-BCB and BCB-diphenol monomers

The photophysical properties of TPE-BCB and its analogues BCB-diphenol molecules were further investigated. UV-visible absorption spectra of both monomers in THF solution were presented in Figure 4a. TPE-BCB displayed a maximum absorption peak at 256 nm, while BCB-diphenol exhibited its maximum absorption peak at 238 nm. This spectral distinction arose from TPE-BCB’s replacement of two ethyl groups with phenyl rings, resulting in an extended conjugation length and consequently inducing a redshift in the absorption spectrum.

Figure 4c–d illustrated the fluorescence spectra of BCB-diphenol and TPE-BCB monomers in tetrahydrofuran (THF)/water mixed solutions with varying water volume fractions. As observed in Figure 4c, BCB-diphenol exhibited its maximum emission peak at 315 nm. However, at water volume fractions of 30%, 50%, and 70%, the fluorescence intensity gradually decreased with increasing water content, with no AIE phenomenon. Conversely, in Figure 4d, TPE-BCB exhibited the maximum emission peak at 332 nm. Interestingly, TPE-BCB displayed a different behavior, transitioning from a dissolved state to an aggregated state as water content increased at water volume fractions of 10%, 20%, and 30%. This transition led to a progressive enhancement in fluorescence intensity, clearly indicating the presence of the distinct AIE phenomenon, as visually represented in Figure 4b under the exposure of 365 nm UV light. This phenomenon underscored that in the aggregation state, the rigid double bond impeded the rotation of the benzene ring, leading to the dissipation of energy in the form of light. Conversely, when flexible alkyl groups replace the
benzene rings, luminescence did not occur.

FIGURE 4. (a) The UV-vis absorption spectra of TPE-BCB and BCB-diphenol monomers in THF solution. (concentration: 10⁻⁴ mol/L, λ_{exc} = 350 nm); (b) Fluorescence photos of TPE-BCB monomer dispersed in water/THF mixture with varied water fractions; (c) Fluorescence spectra of BCB-diphenol monomer dispersed in water/THF mixture with varied water fractions; (d) Fluorescence spectra of TPE-BCB monomer dispersed in water/THF mixture with varied water fractions.

2.6. Lithographic ability for photosensitive fabrications

Lithography plays a pivotal role in semiconductor processing and integrated circuit chip fabrication. It enables structured processing of photosensitive polymer materials through mask exposure. By manipulating the mask’s shape, photosensitive BCB resins can be directly patterned, greatly simplifying the encapsulation process. The negative lithography process involves spin-coating photosensitive polymer onto substrates, such as silicon wafers, followed by mask placement. Subsequently, the polymer film, bearing the mask pattern, is exposed to ultraviolet (UV) light. In this process, exposed regions undergo cross-linking under UV exposure, while unexposed regions dissolve upon exposure to a developing solution, forming a pattern that mirrors the mask pattern.
For lithography, we employed the azide-based photosensitive agent BAC-E. BAC-E generates highly reactive nitrene structures under 365 nm UV light irradiation, capable of reacting with unsaturated C=C bonds in prepolymer. The lithography mechanism was outlined in Figure 5a. The optical microscope images and fluorescence microscope images of the lithographic pattern were displayed in Figure 5b–e. Additionally, probe profiler results (Figure S7) and SEM images (Figure S9) indicated good film-forming ability, uniform thickness (230 nm), and a clear lithography edge, affirming the convenient photolithographic capability of TPE-BCB resin employing an azide crosslinker in this work.

3. CONCLUSION

In conclusion, we successfully synthesized a novel low-temperature-cured BCB-type monomer, TPE-BCB, through Williamson reaction. The modification of the BCB four-membered ring imparts remarkable low-temperature curing characteristics, with a remarkably low ring-opening temperature of 190 °C. Upon complete cross-linking, the resulting resins demonstrate outstanding dielectric properties, both at low and high frequencies. Over the range of 1 kHz to 1 MHz, its dielectric constant remains lower than that of the commercial DVSBCB. Even at 10 GHz, its dielectric constant remains below 2.8. Furthermore, these resins exhibit excellent hydrophobicity, mechanical properties, and photo-lithography ability (achieving a negative lithographic resolution of up to 10 μm). Notably, TPE-BCB resin also displays distinctive fluorescence characteristics, rendering it suitable for application as wafer bonding materials with superior positioning accuracy. This study serves as an exemplar in the development of functional low-temperature-cured BCB monomers, thereby expanding the application of BCB dielectric materials in flexible wearable devices.

EXPERIMENTAL SECTION

Materials: Titanium tetrachloride (99.9%), 4-hydroxybenzophenone (99%), zinc (99%) were purchased from Shanghai Aladdin Biochemical Technology Co., Ltd. Tetrahydrofuran (99.9%, water [?] 50 ppm) was obtained from Beijing Innochem Science Technology Co., Ltd. Caesium carbonate (98%) and dimethyl sulfoxide (99.7%, water [?] 50 ppm) were purchased from Shanghai Energy Chemicals Co., Ltd. 1,3,5-trimethylbenzene was purchased from Shanghai Macklin Biochemical Co., Ltd.

Synthesis: TPE-BCB: 1 g (2.746 mmol) 4,4’-(1,2-Diphenylethene-1,2-diyl)diphenol and 4.47 g (13.73 mmol) Cs₂CO₃ were placed in a flame-dried three-neck round-bottom flask. Then 20 mL dimethyl sulfoxide (99.7%, water [?] 50 ppm) was added. The mixture was bubbled with nitrogen for 30 min to remove oxygen at room
temperature. 1.25 g (6.87 mmol) 1-bromobenzocyclobutene (1-Br-BCB) was added at last. The reaction mixture was stirred for 12 h at 50 °C. After the reaction, the mixture was filtered through a short silicone pad, extracted with brine and ethyl acetate, and then purified by column chromatography (petroleum ether: ethyl acetate = 300:1), and 1.159 g yellow solid was obtained with 74.3% yield.

**1H NMR** (300 MHz, CDCl₃, δ) 7.32 (t, J = 6.0 Hz, 2H, ArH), 7.24–7.20 (m, 3H, ArH), 7.19–7.14 (m, 4H, ArH), 7.13–7.04 (m, 9H, ArH), 6.98 (t, J = 9.0 Hz, 4H, ArH), 6.76 (dd, J = 12.0, 9.0 Hz, 4H, ArH), 5.61 (t, J = 6.0 Hz, 2H, CH), 3.66 (m, 2H, CH₂), 3.27 (dd, J = 15.0 Hz, 2H, CH₂).

**13C NMR** (75 MHz, CDCl₃, δ) 156.52, 156.48, 144.78, 144.22, 144.11, 142.63, 139.85, 136.88, 136.79, 132.66, 131.67, 129.87, 127.73, 127.62, 127.38, 127.34, 126.27, 126.24, 123.45, 123.10, 114.31, 114.25, 74.21 (C-O-Ar), 39.49 (CH₂).

BCB-diphenol: 176 mg (0.66 mmol) Diethylstilbestrol and 1.075 g (3.3 mmol) Cs₂CO₃ were placed in a flame-dried three-neck round-bottom flask. Then 6 mL dimethyl sulfoxide (99.7%, water [?] 50 ppm) was added. The mixture was bubbled with nitrogen for 30 min to remove oxygen at room temperature. 300 mg (1.65 mmol) 1-Br-BCB was added at last. The reaction mixture was stirred for 12 h at 50 °C. After the reaction, the mixture was filtered through a short silicone pad, extracted with brine and ethyl acetate, and then purified by column chromatography (petroleum ether: ethyl acetate = 300:1), and 184 mg yellow solid was obtained with 59% yield.

**1H NMR** (300 MHz, CDCl₃, δ) 7.40–7.27 (m, 6H, ArH), 7.24–7.13 (m, 7H, ArH), 7.03 (d, J = 9.0 Hz, 4H, ArH), 5.72 (s, 2H, CH), 3.76 (dd, J = 15.0, 6.0 Hz, 2H, CH₂), 3.36 (d, J = 15.0 Hz, 2H, CH₂), 2.15-2.22 (m, 6H, CH₂), 0.82 (t, J = 9.0 Hz, 6H, CH₃).

**13C NMR** (75 MHz, CDCl₃, δ) 156.50, 144.89, 142.68, 138.80, 135.52, 130.94, 130.08, 129.88, 129.86, 127.41, 123.49, 123.08, 114.99, 114.53, 74.33 (C-O-Ar), 39.65 (CH₂), 28.63 (CH₂), 13.49 (CH₃).

**Characterization:** The **1H NMR** and **13C NMR** spectra were recorded on Fourier 300 using CDCl₃ as solvent. Thermogravimetric analysis (TGA) was performed with a TA Q600 instrument using 3–5 mg of samples. The experiments were performed at a heating rate of 10 °C[?]min⁻¹ from 40 to 800 degC under a nitrogen atmosphere at a flow rate of 50.0 mL[?]min⁻¹. Differential scanning calorimetry (DSC) was conducted using a TA Q2000 calorimeter at a heating rate of 10 degC[?]min⁻¹ from -10 to 250 degC under a nitrogen atmosphere. Fourier transform infrared (FTIR) spectroscopy was recorded using a VERTEX 70 vacuum microfourier infrared spectrometer. The dielectric constant and dielectric loss were tested using a Novocontrol Concept 40 broad frequency dielectric spectrometer with frequencies ranging from 1 kHz to 1 MHz and split post dielectric resonator (SPDR) at a frequency of 10 GHz (Poland QWED) equipped with microwave Q-Meter. The static contact angles of the polymer discs were characterized using a KRUSS DSA 100 Instrument at room temperature. The water absorption of polymer discs was characterized by the average of the mass changes before and after immersion in deionized water for 72 h at room temperature. The nanoindentation test was characterized by Keysight G200 technology, and the maximum penetration depth was about 2000 nm and the maximum pressure was about 23 mN. UV-vis spectra experiments were carried by a Lambda 1050+ ultraviolet-visible near-infrared spectrophotometer. Fluorescence spectra were measured using a FluoroMax+ fluorescence spectrometer. The profile of the lithography pattern was obtained using a probe profiler (DEKTAK-XT, Bruker). The morphologies of the photolithographic patterns were observed by IX83olympus inverted fluorescence microscope and high and low temperature crystal structure analysis system Lecia DM4P equipped with microscope DMC5400. Scanning electron microscopy (SEM) photos were obtained by Tungsten filament scanning electron microscope (KYKY-EM6900LV, China).

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**CONFLICT OF INTERESTS**

The authors declare no conflict of interests.

**DATA AVAILABILITY STATEMENT**
Research data are not shared.

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REFERENCES

A novel tetraphenylethylene (TPE)-functionalized benzocyclobutene (BCB) monomer is prepared. TPE-BCB, with ether bonds on the BCB’s four-membered ring, demonstrates remarkable low-temperature curing properties. Upon complete cross-linking, resins exhibit exceptional dielectric and mechanical properties, coupled with minimal water absorption. Additionally, the incorporation of TPE enhances the resins’ luminescence and photolithographic capabilities. It serves as an exemplar for functional low-temperature-cured BCB materials.

Keywords: benzocyclobutene, low-temperature curing, aggregation-induced emission, Williamson reactions, wafer Bonding
Low ring-opening temperature
Exceptional dielectric properties
Good photolithographic capabilities