



# Growth methods, enhanced photoluminescence, high hydrophobicity and light scattering of 4,4'-bis(1,2,2-triphenylvinyl)biphenyl nanowires

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## ABSTRACT

4,4'-bis(1,2,2-triphenylvinyl)biphenyl (BTPE) nanowires have been facily grown by either post-annealing its vacuum-sublimed amorphous film or slowly evaporating its chloroform solution. The morphology and density of the nanowires can be easily tuned by changing the growth conditions. For example, isolated single nanowires were readily obtained by thermally depositing the BTPE on a heated substrate, while bundles of nanotubes can be achieved by slowly evaporating a droplet of the BTPE solution. The self-assembled BTPE nanowires show enhanced and blue-shifted photoluminescence compared with that of its amorphous film. The nanowires exhibit a highly hydrophobic surface with a water contact angle of 126°. Also, the BTPE nanowires scatter the light effectively due to the random orientation of the wires. By depositing the BTPE nanowires on the backside of the glass substrate as a scattering media for organic light-emitting diodes, a 31.5% efficiency improvement has been achieved.

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## 1. Introduction

As the quantum size effect of nanostructured materials may induce new optical, electronic, magnetic and mechanical properties compared with those of conventional materials, nanotechnology has become a subject of intensive study in recent years. Among the nanostructured materials, one dimensional (1D) organic nanostructures like nanowires, nanorods, nanoneedles, nanotubes and nanobelts attracted considerable interest recently due to their milder growth conditions such as low-temperature and catalyst-free, compared with those of their inorganic counterparts [1–19]. With their novel structural, optical and electronic properties, the facily grown 1D organic nanostructures have found potential applications in solar cells [1,2], field emitters [3–9], chemical sensors [10], hydropho-

bic surfaces, etc [11,12]. Several small molecular organic materials that were used to grow 1D nanostructures have been reported recently. For example, 1D nanostructures of copper phthalocyanine (CuPc) [1,2,17], 1,5-diaminoanthraquinone (DAAQ) [3,10], tris(8-hydroxyquinolinato)aluminum (Alq<sub>3</sub>) [5,6,13–16], metal-tetracyanoquinodimethane (TCNQ) [7–9], anthracene (AN) and perylene (PY) [18] have been successfully obtained using a wide range of growth methods such as vacuum sublimation [1,3,4], vapor condensation under Ar atmosphere [5,6], physical vapor deposition [2,7,8,13] or solution processing [9,14].

4,4'-bis(1,2,2-triphenylvinyl)biphenyl (BTPE), a lumino-genic molecule that emits efficiently in its solid state with fluorescent efficiency reaching unity, has been employed as a bluish-green emitter and host for organic light-emitting diodes (OLEDs) recently [19,20]. Previously, we also found that the BTPE has a strong tendency to form microfibers in solution. By slowly evaporating its THF/ethanol solution, self-assembled microfibers have been facily obtained [19]. In this work, we show that the BTPE

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nanowires can also be obtained by post-annealing its vacuum-sublimed amorphous film. The growth mechanism is ascribed to the nucleation and molecular migration within the film on annealing. We characterized these 1D nanowires using scanning electron microscopy (SEM) and found that the morphology and density of the nanowires can be easily tuned by changing the growth conditions. For example, isolated single nanowires can be obtained by vacuum subliming the BTPE on a heated substrate, while bundles of nanotubes can be achieved by slowly evaporating a droplet of the BTPE solution. The self-assembled BTPE nanowires exhibit unique characteristics. For example, the nanowires show enhanced and blue-shifted photoluminescence (PL) compared with that of its amorphous film. Also, the nanowires exhibit a highly hydrophobic surface with a water contact angle of 126°. Furthermore, the BTPE nanowires scatter light effectively due to the random orientation of the wires. By depositing the BTPE nanowires on the backside of the glass substrate as a scattering media for the Ir(ppy)<sub>3</sub> based OLEDs, the efficiency at a driving current of 1 mA/cm<sup>2</sup> has been boosted from 78.2 cd/A to 102.8 cd/A, representing a 31.5% enhancement. The facilely grown luminescent, hydrophobic and scattered BTPE nanowires may therefore find potential applications in nano-optoelectronic devices, self-cleaning surfaces or brightness enhancement films.

## 2. Experimental

### 2.1. Preparation and characterization of BTPE nanowires

BTPE was synthesized according to the scheme reported in Ref. [19]. The molecular structure of BTPE is shown in Fig. 6(a) inset. The BTPE thin films were deposited on glass or glass/Au (20 nm) substrates using a thermal evaporator with a base pressure of  $5 \times 10^{-7}$  Torr. The as-deposited BTPE thin films were then annealed using a hot plate with a temperature of 110 °C. For growing isolated single nanowires, the BTPE was deposited on a 150 °C heated glass/Au substrate. Top or cross section views of the morphologies of the BTPE nanowires were investigated using a JEOL-JSM-6490 SEM. The contact angle of the nanowires surface was measured by using a GBX DIGIDROP contact angle meter. The haze of the nanowires was determined by measuring the total transmission and specular transmission using a UV-Vis spectrometer and an integrating sphere.

### 2.2. OLED fabrication and testing

Prior to deposition the OLEDs, amorphous BTPE films were deposited on the backside of the glass substrate using a thermal evaporator with a base pressure of  $5 \times 10^{-7}$  Torr. The BTPE films were then annealed using a hot plate with a temperature of 110 °C for 1 h. The OLEDs were then fabricated in a multi-source vacuum chamber at a base pressure of around  $5 \times 10^{-7}$  Torr with structure glass/ITO/NPB (60 nm)/8% Ir(ppy)<sub>3</sub>:CBP (20 nm)/TPBi (40 nm)/LiF (1 nm)/Al (100 nm), (ITO: indium-tin-oxide; NPB: *N,N'*-bis(naphthalen-1-yl)-*N,N'*-bis(phenyl)-benzidine; Ir(ppy)<sub>3</sub>: Tris(2-phenylpyridine)iridium(III); CBP: 4,4'-bis

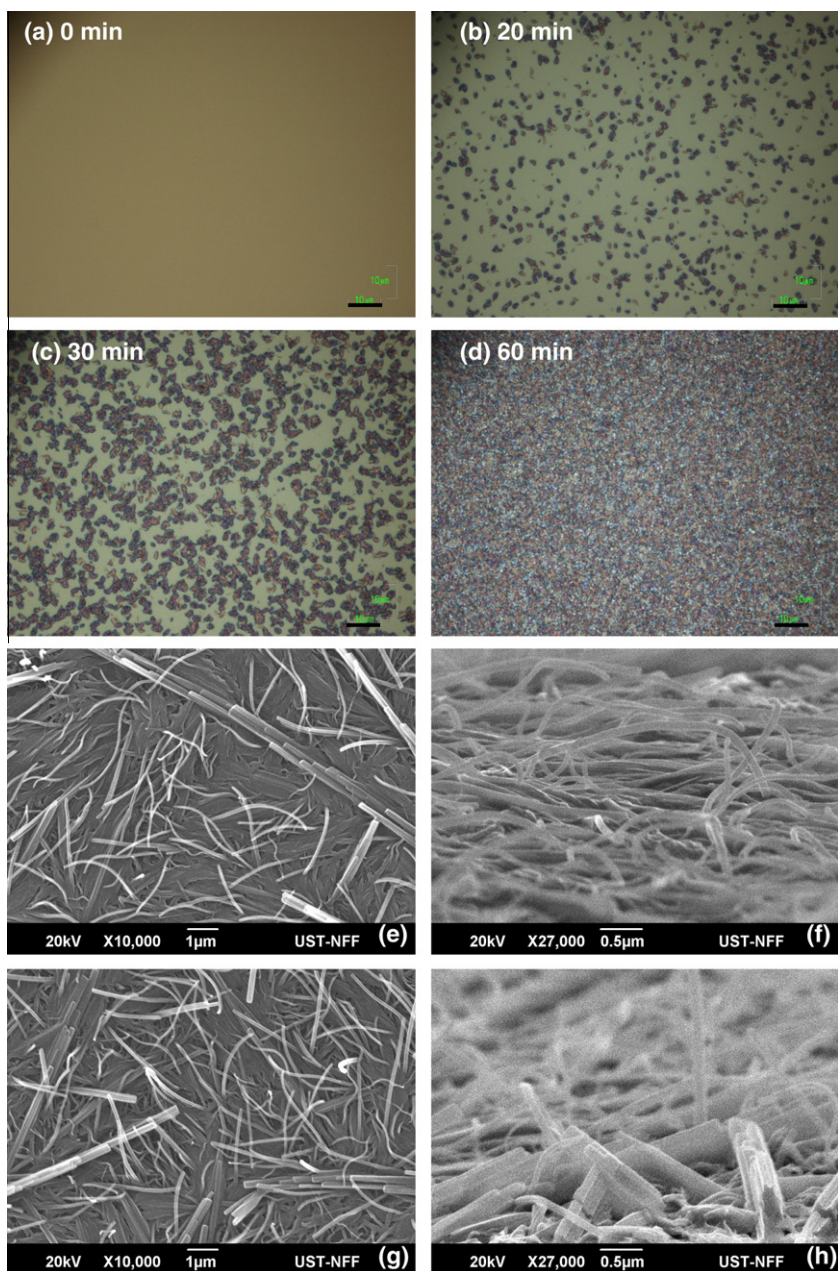
(carbazol-9-yl)biphenyl; TPBi: 2,2',2''-(1,3,5-benzinetriyl)-tris(1-phenyl-1-H-benzimidazole)), The current density-voltage-luminance characteristics of the devices were measured by a HP4145B semiconductor parameter analyzer and a calibrated UDT PIN-25D silicon photodiode. The luminance and external quantum efficiency of the devices were inferred from the photocurrent of the photodiode.

## 3. Results and discussion

### 3.1. Growth methods and mechanism

A 200 nm BTPE film was deposited on a glass substrate with a deposition rate of 2–3 Ångström/s, and then was transferred to a hot plate with a heating temperature of 110 °C. Optical microscopic images as displayed in Fig. 1(a)–(d) shows that the surface of the as-deposited thin film is very smooth. After 20 min annealing, parts of the smooth film become rough due to the generation of microparticles, while the others remain smooth. The arisen particles indicate that the BTPE molecules may self-assemble themselves in a form of microparticles on annealing. These particles may serve as the nucleation seed for further growing the particles/wires. The particle intensity increases as the annealing time is prolonged. After 60 min, the initially smooth film is completely replaced by the micro/nanoparticles, resulting in a rough surface. Closer observation using SEM found that the micro/nanoparticles are actually composed of numerous nanowires, as shown in Fig. 1(e)–(h). Some nanowires agglomerate together, forming the microsheets. The length of the wires can reach tens of μm with a diameter in the range of 100–300 nm. Fig. 1(f) shows the cross section view of the nanowires grown on glass. Some of the wires stand up out-of-plane while most of the wires adhere to the glass surface due to the strong surface free energy of the glass substrate. The surface energy of the glass can be reduced by coating a 20 nm Au film on the glass substrate [3]. With the relatively low surface energy glass/Au (20 nm) substrate, more out-of-plane standing nanowires appear, as shown in Fig. 1(h). Some of the wires can even stand vertically onto the glass/Au substrate, rendering them to be suitable for serving as field emitters [3–9].

The growth mechanism of the self-assembled nanowires may be explained as below. On heating, the molecules may acquire enough thermal energy to adjust their position and assemble with adjacent molecules via  $\pi$ - $\pi$  and/or van der Waals interaction [3,4]. Due to the highly twisted molecular structure of BTPE, the molecules tend to stack in a form of wire rather than plane. Once several molecules are assembled, other molecules could migrate from the nearby film and pile up with the pre-assembled molecules to form nanowires. The initially assembled molecules therefore serve as a nuclei seed for further growth. With a continuous supply of BTPE molecules from the nearby film, the nanowires grow longer and wider. Initially, the formation of nanowires occurs only in some areas; with prolonged heating time, more areas transform to nanowires and the wires become longer and larger. Some wires stick to the glass surface and agglomerate

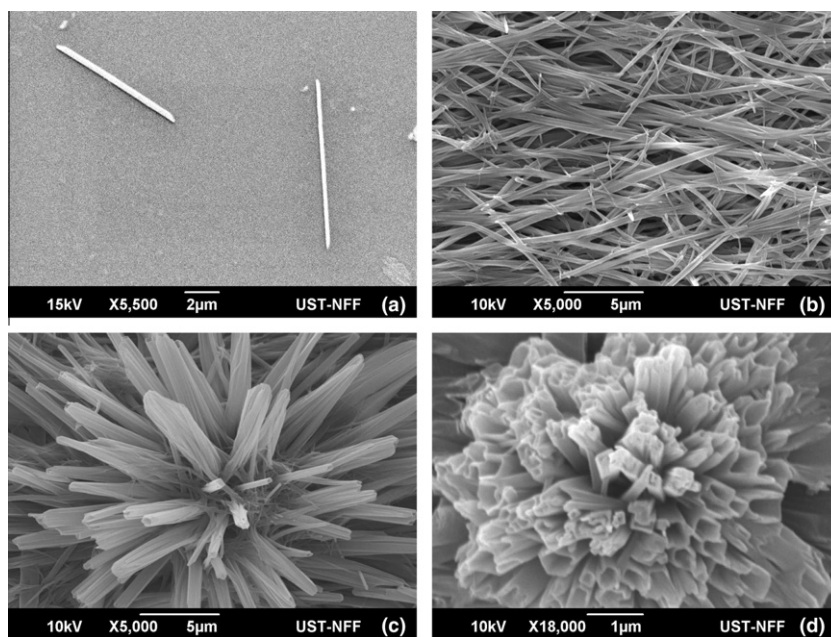


**Fig. 1.** (a)–(d) Optical microscopic images display the evolution of BTPE from a smooth amorphous film to a rough nanosurface. The vacuum-sublimed 200 nm BTPE films on glass substrate were annealed at 110 °C with 0 min (a), 20 min (b), 40 min (c) and 60 min (d) annealing time. Top view (e) and cross section view (f) of 200 nm BTPE on glass substrate. Top view (g) and cross section view (h) of 200 nm BTPE on glass/Au substrate. Due to the reduced surface energy of glass/Au substrate, more nanowires stand up out-of-plane.

forming the microsheets, while others are able to overcome the confinement of the surface and stand up out-of-plane. Finally, the as-deposited film is completely transformed to nanowires, as shown in Fig. 1.

The density and morphology of the nanowires can also be easily tuned by changing the growth conditions/methods. By vacuum subliming 20 nm BTPE on 150 °C heated glass/Au (20 nm) substrate, we have successfully obtained an isolated single nanowire as shown in Fig. 2(a). Thanks to

the thermal energy provided by the heating source and the relatively low surface energy of the glass/Au substrate, the sublimed BTPE molecules may migrate freely on reaching the substrate and subsequently assemble with the pre-arrival molecules, thus self-assembling themselves into a nanowire. The density of the nanowires can be controlled by controlling the amount of the deposited BTPE molecules. The isolated single nanowire may be used to construct novel nanodevices. Also, it offers a means for



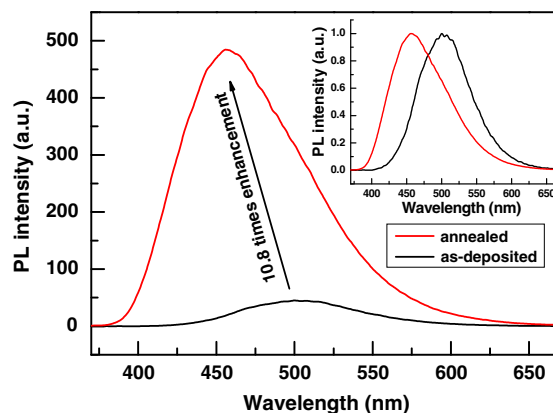
**Fig. 2.** SEM images of the BTPE nanostructures prepared at different conditions. (a) Isolated single nanowire obtained by vacuum subliming 20 nm BTPE on a 150 °C heated glass/Au substrate. (b) Long and entangled BTPE nanowires prepared by immersing a glass substrate into its chloroform solution, followed by slowly pulling the substrate out of the solution for evaporating the solvent. (c) and (d) bundle of standing BTPE nanotubes fabricated by slowly evaporating a droplet of its chloroform solution in air.

testing its properties. For example, the electrical conductivity of the wire can be easily tested by using a high magnification probe station.

The BTPE nanostructures can also be obtained by simply using solution processing. Fig. 2(c) and (d) shows a BTPE flower – bundle of standing BTPE nanotubes obtained by slowly evaporating a droplet of its chloroform solution in air; while long and entangled BTPE nanowires as shown in Fig. 2(b) can be fabricated by immersing the glass substrate into its chloroform solution, followed by slowly pulling the substrate out of the solution for evaporating the solvent. As will be shown in below sections, the self-assembled nanowires exhibit unique characteristics such as brighter and bluer PL emission, lower surface wettability, and stronger light scattering ability compared with those of their smooth amorphous film.

### 3.2. Enhanced and blue-shifted PL emission

Fig. 3 compares the PL emission of the 200 nm as-deposited and 60 min post-annealing BTPE film. Compared with the as-deposited thin film, the post-annealing film exhibits a 10.8-fold enhanced and 50 nm blue-shifted PL emission which may be mainly due to: (1) crystallization-enhanced emission of BTPE. Previously, we found that the self-assembled nanowire is actually composed of crystallized BTPE [19]. When the BTPE molecules are locked in their crystalline lattices, the non-radiative paths like the intramolecular rotation are inhibited effectively, thus resulting in an enhanced PL emission. (2) Light scattering induced by the rough nanowire surface. Due to large index mismatching of BTPE ( $n = 1.8$ ) and air ( $n = 1$ ), most of the



**Fig. 3.** PL intensity of the 200 nm as-deposited amorphous film and 60 min 110 °C annealed film. Inset: normalized PL intensity. Due to crystallization-enhanced emission and strong light scattering ability of the nanowires, the annealed film exhibits stronger and bluer PL emission than that of the as-deposited film.

photoexcited light suffers from the total internal reflection and thus is trapped inside the BTPE film. With the scattering of the anisotropically oriented nanowires, the originally trapped light can be effectively extracted hence leading to an enhanced emission. The strong wavelength dependence ( $\sim \lambda^{-4}$ ) of the Rayleigh scattering indicates that the short wavelength light is scattered more effectively than that of the long wavelength light, thus a bluer PL emission is observed. A similar enhanced and blue-shifted PL emission of the Alq<sub>3</sub> nanostructures has also been reported [15,16].

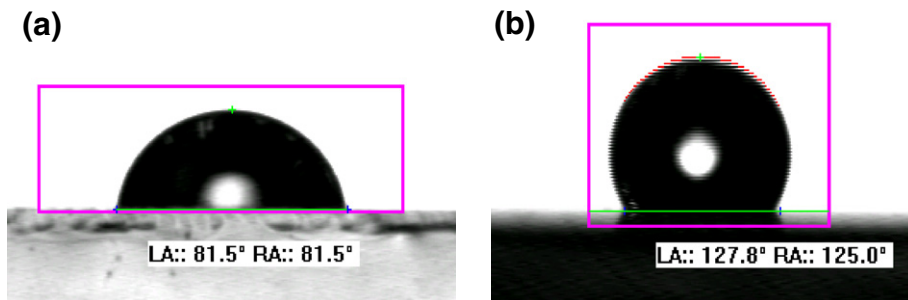


Fig. 4. Contact angle of the 200 nm as-deposited BTPE film (a) and 60 min 110 °C annealed film (b).

### 3.3. High hydrophobicity surface

We further examine the wettability of the BTPE film. As shown in Fig. 4(a), the as-deposited BTPE film already exhibits a moderate wettability with a water contact angle of 81.5°, indicating a highly hydrophobic surface may be obtained if further surface modification measures are taken. Previously, it was shown that a hydrophobic surface can be obtained by carrying out nanoscale surface roughening like plasma etching [21], anodic oxidation [22], colloidal assembly [23] and self-assembly of organic materials via physical vapor deposition [11]. Indeed, by transforming the as-deposited smooth film into nanowires via annealing, a highly hydrophobic surface was obtained with water contact angle remarkably improved from 81.5° to 126.4°, as shown in Fig. 4(b). The reduced wettability is possibly due to the nano-crevices created by the nanowires. The water does not fill the nano-crevices of the rough surface because air is trapped between the liquid and the rough morphology. The durability of the film to long term water exposure is expected to be good, since the water does not damage the BTPE film. However, the mechanical performance of the BTPE film is poor as it is an organic-based material. To protect the BTPE film from mechanical damage, one more inorganic film like SiO<sub>2</sub> can be adopted to package it. The highly hydrophobic BTPE nanowires may find potential applications in self-cleaning surfaces [22], microfluidic devices and other important industrial processes [12].

### 3.4. Strong light scattering ability

The light scattering ability of the films was characterized by measuring the total transmission and specular transmission of BTPE. Then the haze that indicates how many percent of the light is scattered was calculated by  $HAZE = (T_{total} - T_{specular})/T_{total}$ . Fig. 5(a) shows the total transmission of the annealed films as measured by using an integrating sphere combined with a UV–Vis spectrometer. All samples show nearly the same total transmission of around ~90%. However, the case is quite different for the specular transmission. Due to strong light scattering induced by anisotropically oriented nanowires, the specular transmission of the annealed films is remarkably lower than the total transmission. For example, the specular transmission at 520 nm is 68.4%, 55.3% and 58.9% for the

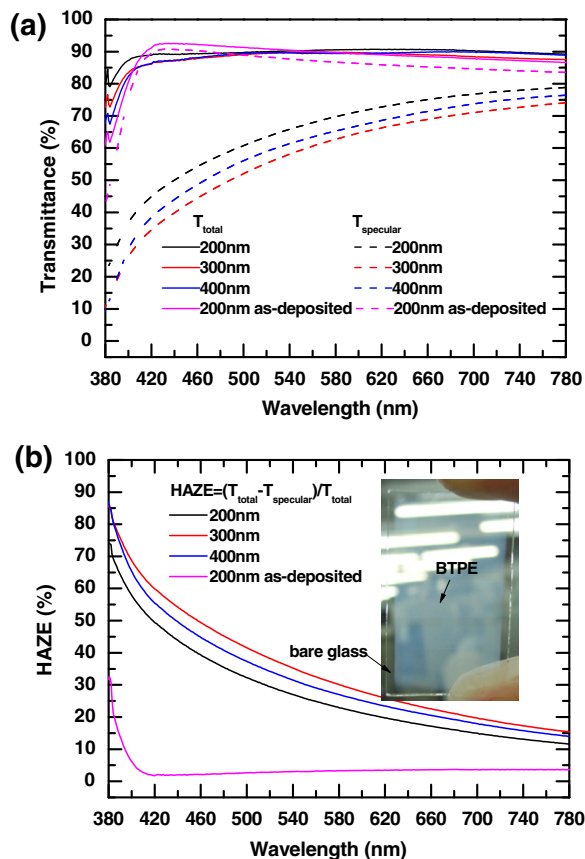
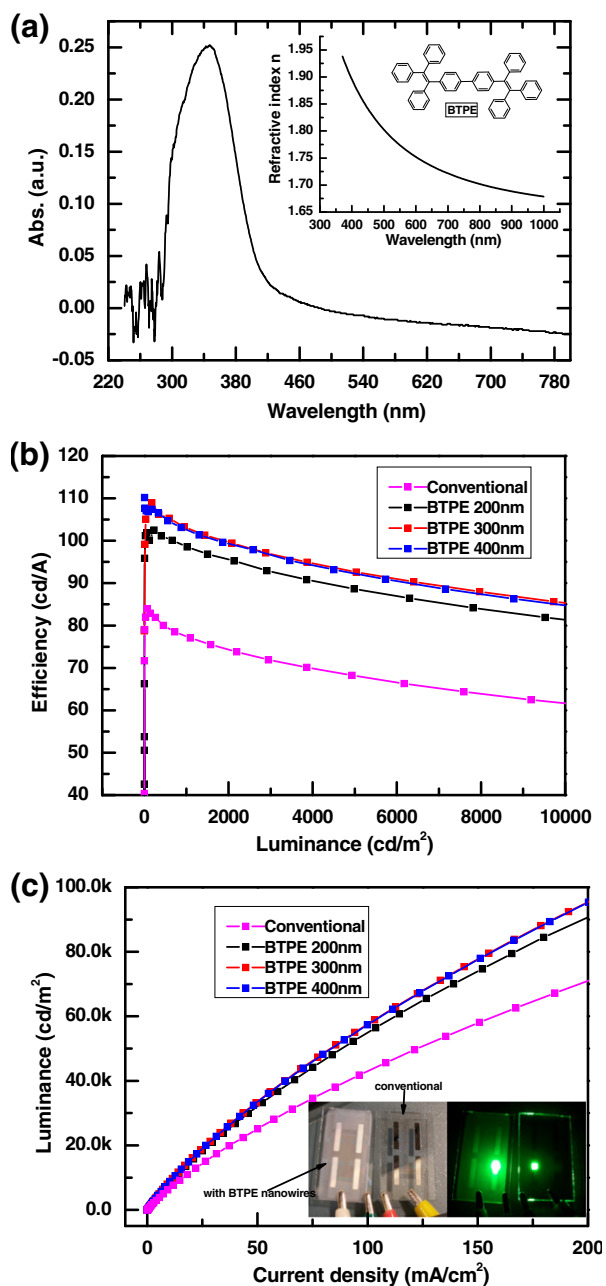


Fig. 5. Total transmission, specular transmission (a) and haze (b) of the 200 nm, 300 nm and 400 nm annealed films. Due to strong light scattering effect, the annealed film exhibits a milky appearance (photo of inset).

200 nm, 300 nm and 400 nm annealed film, respectively, significantly lower than ~90% for the total transmission, resulting in a haze of 29.5%, 38.2% and 34.2%, respectively. However, the haze for the 200 nm as-deposited smooth film is only 2.8%. It is noted that the haze of the annealed films can be tuned by varying the thickness of the BTPE. The relatively low haze of the 200 nm annealed film is ascribed to the low density of the nanowires which cannot scatter the light effectively. The density of the nanowires



**Fig. 6.** (a) Absorption spectrum and refractive index of BTPE. (b) Efficiency–luminance and (c) luminance–current density characteristics of the BTPE nanowire modified OLEDs. A 31.5% efficiency improvement has been achieved due to the effective extraction of the substrate waveguide light by the scattered nanowires. Inset of (c) shows the photos of the OLEDs. The nanowire modified OLEDs emit brighter than the conventional OLEDs.

increases as the thickness increases. Maximum haze is achieved at a thickness of 300 nm. Further increasing the thickness leads to a decrease of the haze, which is mainly due to the reduction of scattering possibility caused by agglomeration of the nanowires at high density. Due to the strong wavelength dependence ( $\sim \lambda^{-4}$ ) of the Rayleigh scattering, the blue light is scattered much more than the

red light; hence the haze in the short wavelength region is higher than that in the long wavelength region, as shown in Fig. 5(b). Like any light diffusing objects, the annealed film exhibits a milky appearance, as shown in Fig. 5(b) inset. Prompted by the strong light scattering ability of the BTPE nanowires, we then explore its possibility of serving as a brightness enhancement layer for OLEDs.

### 3.5. Light enhancement layer for OLEDs

Classical ray optics indicates that about 30% of the OLED photons are waveguided by the glass substrate due to index mismatching of the glass and air [24]. The substrate waveguide light can be extracted by roughening or texturing the substrate [25–27]. Previously, Chen et al. demonstrated that a 20% improvement of efficiency can be achieved by simply sand-blasting the glass substrate [25], while Cheng et al. fabricated a PDMS nanostructure on the glass for extracting the substrate waveguide light [26]. However, the fabrication of PDMS nanostructure is relatively complex including preparation of porous anodic aluminum oxide (AAO) template, molding of PDMS and removing of template. Here we show that by depositing the facily grown BTPE nanowires on the backside of the glass substrate as a scattering media for OLEDs, the substrate waveguide light can also be effectively extracted.

As shown in Fig. 6(a), the BTPE does not absorb of the visible light and exhibits a high total transmission of  $\sim 90\%$ . Also, the refractive index of BTPE ( $n = 1.8$ ) is larger than that of glass; hence all the light can be coupled from the glass to the BTPE. Fig. 6(b) and (c) shows the current efficiency–luminance and luminance–current density characteristics of the Ir(ppy)<sub>3</sub> based OLEDs. The efficiency and luminance are greatly improved due to the strong light scattering ability of the nanowires. For example, at a current density of  $1 \text{ mA/cm}^2$ , the current efficiency is  $98.6 \text{ cd/A}$ ,  $102.8 \text{ cd/A}$  and  $102.3 \text{ cd/A}$  for the devices with 200 nm, 300 nm and 400 nm BTPE nanowires, respectively, remarkably higher than  $78.2 \text{ cd/A}$  for the conventional device, and represent a 28.4%, 31.5% and 30.8% improvement, respectively. The key performance of the devices is compared in Table 1. The highest efficiency improvement by the 300 nm annealed film may be likely due to its strongest light scattering ability as evidenced by its highest haze compared with the other two. Inset of Fig. 6(c) shows the photos of the OLED with and without the BTPE nanowires. Obviously, compared with the conventional devices, the modified OLEDs emit brighter and the edges become darker, signifying an effective conversion of substrate waveguide light into forward emission with the help of BTPE nanowires. The facily grown BTPE nanowires are hence an ideal candidate for serving as a brightness enhancement layer.

## 4. Summary

In summary, we show that various BTPE nanostructures like nanowires, isolated single nanowires and bundles of nanotubes can be facily obtained by post-annealing its vacuum-sublimed thin film, depositing its molecules on

**Table 1**Key performance of the Ir(ppy)<sub>3</sub> based green OLEDs at a driving current density of 1 mA/cm<sup>2</sup>.

Structure	HAZE (%)	L (cd m <sup>-2</sup> )	η <sub>L</sub> (cd/A)	η <sub>P</sub> (lm W <sup>-1</sup> )	η <sub>ext</sub> (%)	L <sub>Enhancement</sub> (%)
Conventional	–	782	78.2	53.5	22.8	–
BTPE 200 nm	29.5	986	98.6	67.7	28.4	26.1
BTPE 300 nm	38.2	1028	102.8	69.8	29.6	31.5
BTPE 400 nm	34.2	1023	102.3	69.0	29.5	30.8

heated glass/Au substrate or evaporating its chloroform solution. The self-assembled BTPE nanowires exhibit unique characteristics such as stronger and bluer PL emission, lower wettability and stronger light scattering ability; thus they may find potential applications in nano-opto-electronic devices, self-cleaning surfaces or brightness enhancement films. Initial attempts show that a 31.5% efficiency improvement can be achieved by employing the nanowires as scattering media for the OLEDs.

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