Exciplex elimination in an organic light-emitting diode based on a fluorene derivative by inserting 4,4'-N,N'-dicarbazole-biphenyl into donor/acceptor interface*

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Organic light-emitting diodes (OLEDs) composed of a novel fluorene derivative of 2,3-bis(9,9-dihexyl-9H-fluoren-2-yl)-6,7-difluoroquinoxaline (F2Py) were fabricated, and exciplex emission was observed in the device. To depress the exciplex in an OLED for pure colour light emission, 4, 4'-N,N'-dicarbazole-biphenyl (CBP) was inserted as a separator at the donor/acceptor interface. It was found that the device without the CBP layer emitted a green light peaking at 542 nm from the exciplex and a shoulder peak about 430 nm from F2Py. In contrast, the OLED with CBP layer emitted only a blue light peak at about 432 nm from F2Py. Device efficiencies were calculated by a simulative mode in an injection controlled type mechanism, and the results showed that exciplexes yield much lower quantum efficiency than excitons. The device with CBP has a higher power efficiency as no exciplex was present.

Keywords: exciplex elimination, fluorene derivative, organic light-emitting diode, 4,4'-N,N'-dicarbazole-biphenyl

PACC: 7860F, 7855K, 7135, 7360R

1. Introduction

Since the milestone work of organic light-emitting diodes (OLEDs) with high brightness using tris(8-hydroxyquinolinato)aluminum (Alq_3) as an emitting layer at low bias voltage was reported by Tang and Van Slyke in 1987,[1] OLEDs with heterojunction structure have attracted more and more attention. Progress in developing novel organic materials, optimising device configuration and fabrication process has been made continuously.[2−5] However, the performances of blue emitting materials are not sufficient enough for practical applications, so that blue light-emitting materials with high luminescence and efficiency are still needed. To attain this goal, many kinds of blue emitting organics have been employed in OLEDs.[6−9] Among them, molecular fluorene derivative is a promising kind of blue material because it has a high thermal stability, an energy band gap above 2.9 eV, and a photoluminescent (PL) quantum efficiency between 60% and 80%.[10−12] In some multilayer OLEDs, including OLEDs based on fluorene derivatives, a bimolecular recombination state called an exciplex will be realized at the organic/organic interface.[13−16] The existence of the exciplex in OLEDs usually leads to a redshifted and broadened emission spectrum, and a significant reduction of device efficiency and an unstable electroluminescent (EL) spectrum at different bias voltages accordingly. Therefore, several methods have been adopted to eliminate the exciplex to obtain electroluminescence only from the monomer, or manipulate the exciplex emission in OLEDs to tune light-emitting colour,[17−19] and to obtain green, yellow,

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or red emissive components for white OLEDs.[20−22] To avoid exciplex emission in donor/acceptor system OLEDs, solutions have been reported which include the insertion of a hole transport material with high highest-occupied molecular orbital (HOMO) between the donor and acceptor layers,[14,23] and to employ doping or blending techniques to reduce the probability of exciplex formation.[24,25] However, the latter method does not seem to be a promising solution for the interface exciplex.[23]

In this work, multilayer devices are fabricated using N,N'-Di-[(1-naphthalenyl)-N,N'-diphenyl]-(1,1'-biphenyl)-4,4'-diamine (NPB) as a hole transporter and Alq_3 as an electron transporter, and exciplex emission is studied in the EL process. To depress this exciplex to gain a pure chromatic emission from F2Py, a thin layer of CBP is employed as a separator between the hole transporting material NPB and F2Py. To investigate the exciplex effect on device performance, the PL spectra of materials, the EL spectra and current–voltage-luminescence properties are studied. Assuming devices operated under the injection controlled mode of the charge transporting mechanism, the quantum efficiencies of OLEDs are computed by simulation to scrutinize the effect of the exciplex in OLEDs on the operation mechanism and the impact of inserted CBP layer on the exciplex.

2. Experimental

Indium tin-oxide (ITO)-coated glass with a sheet resistance of 12 Ω/sq was employed as anode substrate. Prior to the deposition of organic layers, ITO-coated glasses were pretreated according to a conventional cleaning procedure, using detergent, deionized water, acetone, and ethanol respectively, and then dried with nitrogen gas. Afterwards, the substrate was treated in an oxygen plasma atmosphere for 15 min to enhance the work function of ITO and thus improve its hole injecting capability.[26] The thermal evaporation of organic materials embracing NPB, CBP, F2Py and Alq_3 was carried out at a typical pressure of 2×10^{-4} Pa at a typical deposition rate 1–2 Å/s (1 Å=0.1 nm). NPB and CBP were used as hole transporting layers (HTL). F2Py and Alq_3 were used as the emitting layer (EML) and electron transporting layer (ETL), respectively. Then, Mg:Ag cathode (with ratio 10:1 by weight) deposited at a pressure of 3×10^{-3} Pa and at an evaporation rate 10–15 Å/s. The emissive area of the device is 5×5 mm^2, and unencapsulated devices were tested at room temperature under atmospheric circumstances.

Current density (J)–bias voltage (V)–luminance (L) characteristics were measured with a Keithley-4200 and ST-86LA semiconductor characterisation system. Meanwhile, the EL spectra of devices A and B were obtained by a PR650 system. The normalised absorption spectra of F2Py, NPB, CBP, NPB:F2Py and CBP:F2Py blend in film state were obtained by using a Shimadzu-UV1700 system. Moreover, the PL spectra of materials and their blend films were measured at room temperature, excited by a 365 nm wavelength light source. Figure 1 shows the chemical structures of materials in this work.

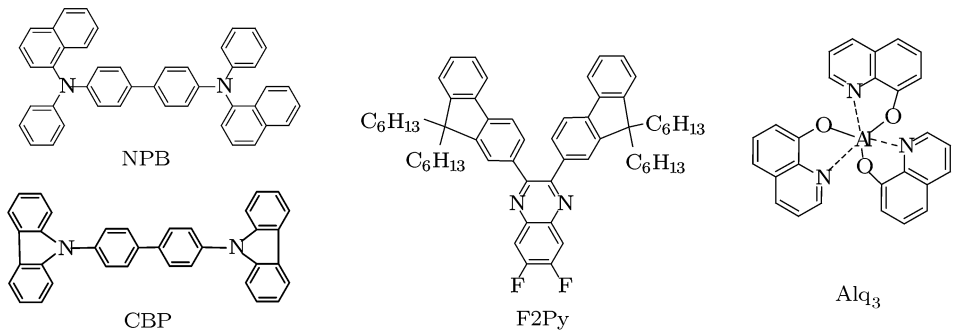


Fig. 1. Molecular structures of F2Py, NPB, CBP and Alq_3.

3. Results and discussions

3.1. Absorption and photoluminescence

Figure 2 reveals the UV–vis absorption spectra of spin-coated films of F2Py, NPB, CBP, and blends of F2Py:NPB and F2Py:CBP on quartz substrates. There are multiple bands with maxima at 280, 312 and 383

nm for F2Py; 221, 277 and 348 nm for NPB, and 296, 331 and 345 nm for CBP. The absorption spectra of F2Py:NPB and F2Py:CBP blends are nearly a linear combination of the absorption of individual components, indicating that the components in the composite films do not have any interaction.

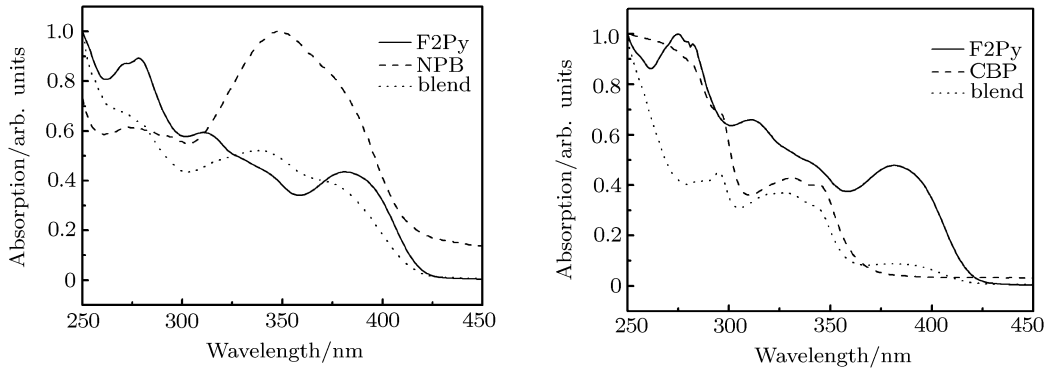


Fig. 2. Normalised UV–vis absorption spectra of organic materials in film as functions of λ.

The normalised PL spectra of F2Py, NPB, CBP, F2Py:NPB and F2Py:CBP blends in film state are shown in Fig. 3. The PL spectra of F2Py, NPB and CBP show PL bands peaking at 447, 440, and 409 nm, respectively. In contrast, the PL spectrum of NPB:F2Py spin-coating blend film with a ratio 1:1 by weight has a new band peaking at 545 nm, which is very different from that of individual NPB or F2Py. While no new features can be observed at the absorption spectrum of the NPB and F2Py mixture, it indicates that the new light-emitting band does not directly come from the ground state of the charge-transfer complex. From the previous work,[27,28] new emitting portions come from the exciplex formed at the interface of NPB and F2Py. In contrast, the PL of CBP:F2Py mixture with a ratio 1:1 by weight shows nearly identical emission spectra relative to F2Py, which means no exciplex formed at the CBP/F2Py interface.

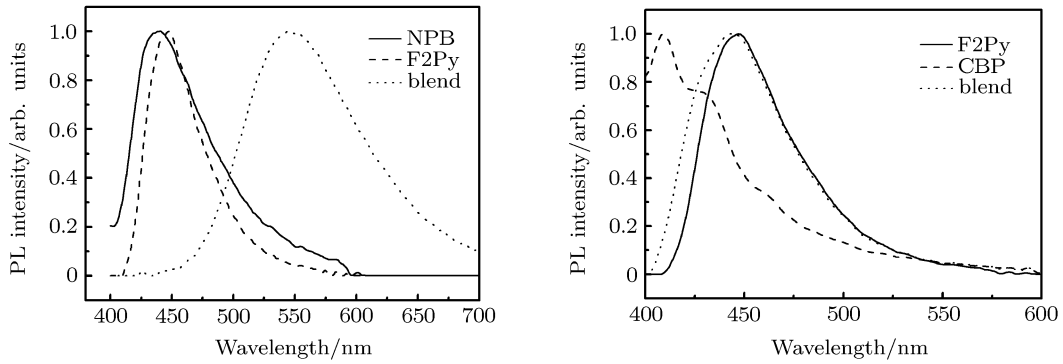


Fig. 3. Normalised PL spectra of materials in film as functions of λ.

3.2. Electroluminescence

To further investigate the electroluminescence (EL) of OLEDs, two devices with configuration as follows were fabricated: Device A: ITO/NPB (50 nm)/F2Py (20 nm)/Alq_3 (10 nm)/Ag:Mg (200 nm), Device B: ITO/NPB (50 nm)/CBP (6 nm)/F2Py (20 nm)/Alq_3 (10 nm)/Ag:Mg (200 nm).

EL spectra at a bias voltage of 9 V of the two devices are shown in Fig. 4. Device A has a low energy emission band peak at 542 nm with a blue shoulder peak around 430–450 nm, and the blue emission is enhanced with increasing bias voltage (not shown here). To confirm the source of the low energy emission in device A, we also fabricated a double layer (DL) device with a structure of ITO/NPB (50 nm)/F2Py (20 nm)/Ag:Mg (200 nm), the EL spectra of which is also shown in Fig. 4.

Although no thin layer of Alq_3 is employed as ETL, a low energy emission band peak at 529 nm can still be observed in this DL device, implying that the low energy emission band does not originate from Alq_3. Besides, a shoulder peak around 430 nm also appears in this DL device. The DL device has an EL spectrum which is very similar to device A, the shift of low energy emission peak may result from the difference between the intensities of blue shoulder peaks. However, as no Alq_3 layer works as ETL to enhance the amount of electrons injected from cathode, the DL device has a very low luminescence. Therefore, the low energy emission of 542 nm in device A is not from NPB, F2Py, or Alq_3 excitons, but from a new species named the exciplex. The blue shoulder peak around 430–450 nm is ascribed to light emission from F2Py excitons. Device B has a blue emission peak at about 432 nm, which keeps constant when the voltage increases. The full width at half maximum (FWHM) of NPB, F2Py film state PL and device B EL are 71, 51 and 52 nm, respectively. The proximity of FWHM between device B EL spectrum and F2Py PL spectrum indicates that the blue emission peak at 432 nm comes from the F2Py exciton instead of NPB. We postulate that the difference between PL and EL of F2Py spectra peaks probably results from the different excited mechanisms.

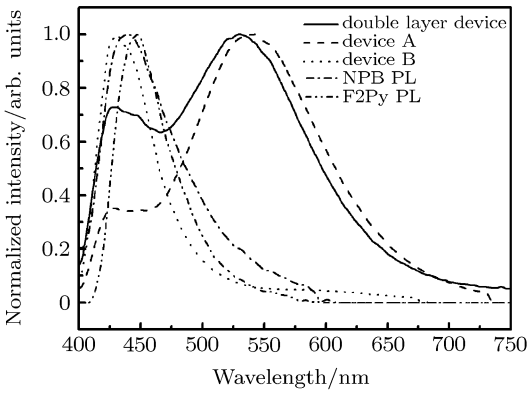


Fig. 4. Normalised EL spectra of double-layer device ITO/NPB/F2Py/Mg:Ag, devices A and B at 9 V and PL spectra of NPB and F2Py (as functions of λ).

An exciplex is a common bimolecular-involved excited state complex in OLEDs, formed at the interface of the donor/acceptor system, with one in the excited state and the other in the ground state. In this study, the exciplex emission peak at 542 nm is only realised in device A. The energy levels of organic materials are significantly important in the origin of exciplex formation. The lowest unoccupied molecular orbital (LUMO) and the highest occupied molecular orbital (HOMO) of F2Py are respectively −2.92 eV and −6.19 eV, obtained with the cyclic voltammetry method.[29] The LUMO and HOMO of NPB are −2.1 eV and −5.2 eV, respectively.[30] The exciplex is formed by a donor molecule of NPB and an acceptor molecule of F2Py at the interface in OLEDs. The exciplex emission energy can be expressed as[31−33]

$$h\nu_{\text{exciplex}} = E_{\text{LUMO}}(\text{acceptor}) - E_{\text{HOMO}}(\text{donor}),$$

where

$$E_{\text{LUMO}}(\text{acceptor}) = -2.92 \text{ eV},$$

the LUMO of F2Py; E_{HOMO}(donor)=–5.2 eV, the HOMO of NPB, and ν_{exciplex} is the frequency of exciplex emission. According to $\lambda_{\text{exciplex}} = c/\nu_{\text{exciplex}}$, where $\lambda_{\text{exciplex}}$ is the wavelength calculated to be 544 nm. This value fits well the experimental result, implying that the emission at 542 nm in EL of device A does result from the exciplex at the NPB/F2Py interface.

CBP is conventionally employed as a bipolar charge-transport material or hole-transporting host for phosphorescent material with PL peaking at about 380 nm and 409 nm,[34] HOMO and LUMO levels are respectively −6.3 and −3.0 eV.[35] As shown in Fig. 3, the PL spectrum of F2Py:CBP blend in film state shows the characteristics of F2Py without low energy emission band. Hence, no new exciplex formed at CBP/F2Py interface, and pure blue light emission of F2Py in OLEDs could be expected using CBP as HTL and separator between NPB and F2Py. The device B fabricated verifies this assumption, i.e., there is no low energy emission band observed in the EL spectrum of the device, which implies that the exciplex of the NPB/F2Py system is depressed and no new exciplex forms at the CBP/F2Py interface.

3.3. *L–J–V* properties and charge transporting characteristics

The turn-on voltages (defined as the operating voltage when luminescence is equal to 1 cd/m^2) of devices A and B are 7 V and 8 V, respectively. The *V–J* and *V–L* curves are shown in Fig. 5. In the low electric field range, device A has higher luminance and current density than device B at the same operating voltage. Emission of device A dominates with the exciplex, while device B emits only blue light from F2Py.

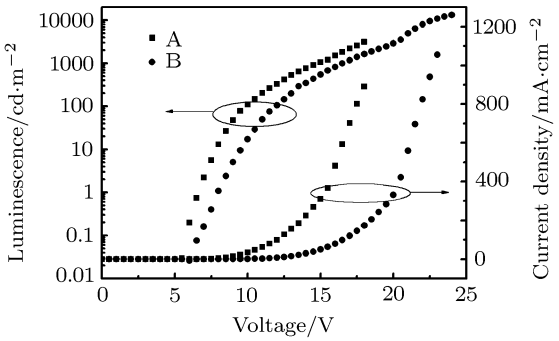


Fig. 5. L–V–J characteristics of devices A and B.

The current–voltage properties are primarily determined by charge injection from electrodes and space charge effects but not by p-i-n heterojunctions in OLEDs; herein, we assume that the leakage of holes and electrons toward the cathode may be neglected in high current regime, that is, a very small percentage of holes and electrons crosses through organic layers without recombination. This small fraction of charge carriers can be ignored in the overall device current. Hence, devices may be considered as injection-controlled EL devices (ICEL). The injected holes from the anode remain within the NPB layer, bound to its imaginary charge by Coulombic attraction. Only those holes which in the diffusion process overcome this Coulombic barrier contribute to the current. Since an external electric field lowers the Coulombic barrier, the current becomes field-dependent.[36] Device current–field curves could be approximated by the field-assisted thermionic injection of charge carriers, described by its one-dimensional Onsager plots[37] $\log(j/F^{3/4})$ versus $F^{1/2}$, as shown in Fig. 6. This simulation model was presented by Kalinowski.[38] Therefore, the relationship between current density j and electric field F can be expressed as

$$j \sim F^{3/4} \exp(aF^{1/2}), \tag{1}$$

where

$$a = 2(\beta e/kT)^{1/2} = (e/kT)(e/4\pi\varepsilon_0\varepsilon)^{1/2}, \tag{2}$$

From Fig. 6, the currents of both OLEDs are not likely to be space charge limited since $\log(j/F^{3/4})$ against $F^{1/2}$ plots deviate from straight lines at lower electric fields ($< 10^6$ V/cm).[36] The experimental value of coefficient a at high electric fields ($1\times10^6-4\times10^6$) can be obtained from the slopes of straight-line plots $\log(j/F^{3/4})$ against $F^{1/2}$: $a_1 = 0.79\times10^{-2}$, $a_2 = 0.55\times10^{-2}$ $(\mathrm{cm/V})^{1/2}$. Moreover, $a_{\mathrm{theor}} = 0.77\times10^{-2}$ $(\mathrm{cm/V})^{1/2}$ is obtained from Eq. (2) with dielectric constant of NPB $\varepsilon = 3.6$.[39] This a_1 fits well the theoretical value. However, the constant a_2 of device B only constitutes a small fraction of a_{theor}. The main reason is that CBP layers inserted between NPB and F2Py layer decreases the charge carrier mobility of OLED since hole transport mobility in CBP is much lower than in NPB. Meanwhile, we realise the difference slopes of low and high field ranges from $\log(j/F^{3/4})$ against $F^{1/2}$ curves. It is mainly attributed to different carrier injection processes as at organic/electrode interface, which is found to obey Schottky-type carrier injection via impurity or structural disordered levels with thermal assistance.[40] Moreover, the slopes also alter again distinctly at higher fields ($> 4\times10^6$ V/cm). This is closely related to the reverse internal electric field formed by accumulated charges, when operating voltage becomes high enough to inject the amount of carriers exceeding the capacity in organic materials.

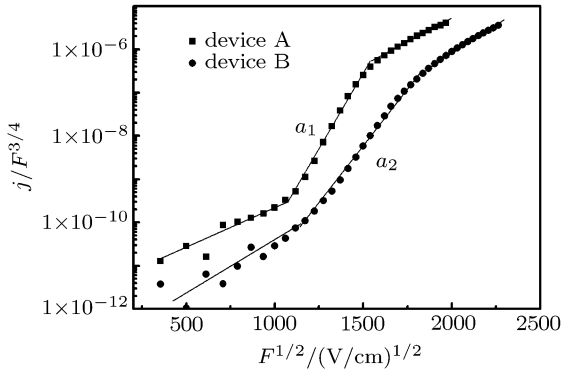


Fig. 6. Current–electric field characteristics (in $\log(j/F^{3/4})$ versus $F^{1/2}$ representation) of devices A and B.

Since insertion of CBP alters the mobility of barriers in materials, at low operating voltages, device A has a lower turn-on voltage and higher brightness. Device A thus gains a higher luminance and current density as the carrier transport mobility of CBP is lower than NPB, and more charges can be injected in device A than in device B and the former has higher luminance than the latter at the same operating voltage. Also, the current density in device A is higher than in device B at the same bias voltage. Hence, in order to provide evidence of how the exciplex affects device performance, calculation of the efficiencies of the two kinds of devices is needed.

3.4. Device efficiencies

The equation of power efficiency of devices can be expressed as

$$\eta_{\mathrm{p}} = \frac{\pi BS}{IV}, \tag{3}$$

where B is emissive brightness, S is emissive area, V is operating voltage, and I is operating current. After calculation, power efficiency as a function of device operating voltage is shown in Fig. 7. Power efficiencies of

both devices A and B increase with bias voltage, and after reaching their maxima, reduced with increasing voltage. The maximum efficiencies of 0.12 lm/W at 6.5 V for device A and 0.52 lm/W at 8.5 V for device B are obtained. That device B has a higher efficiency than device A indicates that the existence of exciplexes decreases the device efficiency. The relationship between power efficiency and external quantum efficiency can be expressed as $\gamma\eta_{\rm ext} = \eta_{\rm p}$, where $\eta_{\rm ext}$ stands for external quantum efficiency, $\eta_{\rm p}$ is power efficiency, and γ represents the function relation between external quantum efficiency and power efficiency,[38] expressed as

$$\gamma = \frac{683\hbar c}{Ve}\frac{\int_{380}^{780} g(\lambda)K(\lambda)\mathrm{d}\lambda}{\int_{380}^{780} g(\lambda)\lambda\mathrm{d}\lambda}. \tag{4}$$

Here $g(\lambda)$ is relative EL intensity at a certain wavelength, and $K(\lambda)$ is the Commission International de l'Eclairage chromaticity (CIE) standard photopic luminous efficiency function. Using this simulation model, the external quantum efficiencies of devices A and B are calculated to be 0.031 % and 0.522 % at 9 V, respectively. Apparently, the device with the CBP layer has a significantly higher external quantum efficiency.

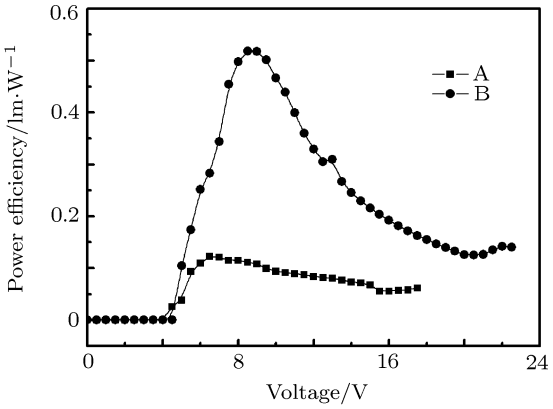


Fig. 7. Relationship between power efficiency of devices A and B and bias voltage.

In injection-controlled EL devices, considering the thickness of the device, we can assume that no holes and electrons can penetrate through organic layers without recombination with oppositely charged carriers, and hence device current is mainly determined by holes from the anode and electrons from the cathode instead of the heterojunction. Furthermore, we suppose that charge carriers injected into organic layers all undergo some kind of recombination,[41] which can be divided into four groups:

(i) $R_{\rm et}$, radiative recombination of excitons in F2Py molecules;

(ii) $N_{\rm et}$, nonradiative recombination of excitons in F2Py molecules;

(iii) $R_{\rm ex}$, radiative recombination of exciplexes at NPB/F2Py interface;

(iv) $N_{\rm ex}$, nonradiative recombination of exciplexes at NPB/F2Py interface.

Therefore, the total current I is the sum of contributions of these four channels of recombination

$$I = I_{R_{\rm et}} + I_{N_{\rm et}} + I_{R_{\rm ex}} + I_{N_{\rm ex}}. \tag{5}$$

In device A, both excitons in F2Py and exciplexes at the NPB/F2Py interface contribute to EL performance. In contrast, only excitons in F2Py emit light in device B. As only the radiative recombination process, which produces photons that actually radiate from the OLED, can contribute to the external quantum efficiency, this quantum efficiency in devices A and B can be expressed as

$$\eta_{\rm ext}(A) = \frac{I_{R_{\rm et}} + I_{R_{\rm ex}}}{I}, \tag{6}$$

$$\eta_{\rm ext}(B) = \frac{I_{R_{\rm et}}}{I}. \tag{7}$$

By analysing devices A and B at 9 V, the ratio of radiative contribution of excitons to that of exciplexes in device A is obtained from the EL spectrum, i.e. from the ratio of integrals of the two spectral components — excitons around 430–440 nm and exciplexes around 545 nm. For device A, $I_{R_{\rm et}}/I_{R_{\rm ex}} = 0.19$, while for device B, since no exciplex is formed, $I = I_{R_{\rm et}} + I_{N_{\rm ex}}$, the ratio of $I_{R_{\rm et}}/I = 0.52\%$ is gained by $\eta_{\rm ext}$ at 9 V. The emission quantum efficiency of F2Py excitons should be identical in devices A and B, since the introduction of CBP does not affect the device operating mechanism on a significant level. Thus, the quantum efficiency of excitons or exciplexes, defined as the ratio of radiative excitons or exciplexes to the amount of excitons or exciplexes formed in devices, which can be obtained from Eqs. (6) and (7), $\eta_{\rm exciton} = I_{R_{\rm et}}/I = 0.52\%$, $\eta_{\rm exciplex} = I_{R_{\rm ex}}/(I_{R_{\rm ex}} + I_{N_{\rm ex}}) = 0.028\%$. The quantum efficiency of the exciplex is significantly lower than that of F2Py excitons. Hence, the exciplex in OLEDs indeed reduces the device efficiency. Insertion of a thin layer of CBP as a separator will eliminate the exciplex in device B, and thus greatly improve the EL efficiency of the device. However, the low carrier transporting mobility in CBP creates a remarkable mobility barrier in HTL, and reduces the amount of injected holes from the anode. Therefore, device B has a lower luminescence and current density corresponding to device A at low electric field.

4. Conclusion

Exciplex emission has been observed in the PL spectrum of NPB:F2Py blend and the EL spectrum of device A by using a novel blue light-emitting fluorene molecular derivative of F2Py as a probe. By inserting a thin CBP layer (6 nm) between NPB and F2Py, the exciplex at the NPB/F2Py interface can be completely depressed. Devices without CBP layer have lower turn-on voltage, higher brightness and current density at low electric field, but lower power efficiency than those with CBP. By calculation of the external quantum efficiency of devices with a simulation mode in injection-controlled EL device, the quantum efficiencies of F2Py excitons and the NPB/F2Py exciplex have been obtained. F2Py excitons have a higher quantum efficiency than exciplexes, indicating that the existence of the exciplex can greatly reduce the device efficiency. Since fluorene derivatives are relatively easy to form exciplexes with donor materials at interfaces, exciplex elimination in blue OLEDs based on fluorene derivative is vitally important for high device efficiency. Optimising a proper separator to be inserted in the donor/acceptor interface can eliminate the exciplex efficiently. A high efficient OLED based on fluorene derivatives can be expected by using this method.

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