

铈配合物共掺杂电致发光器件效率滚降的延缓

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摘要: 将 $\text{Alq}_3[\text{tris}(8\text{-hydroxyquinoline}) \text{ aluminium}]$ 和 $\text{Eu}(\text{TTA})_3\text{phen}$ (TTA=thenoyltrifluoroacetone, phen=1,10-phenanthroline) 共掺杂进入主体材料 CBP(4,4'-N,N'-dicarbazole-biphenyl) 中, 我们制作并研究了一系列电致发光器件。经过优化 Alq_3 的掺杂浓度, 在不改变色纯度的情况下, 器件的效率滚降被大幅降低并获得了近乎加倍的最大亮度。发光层中的 Alq_3 分子不仅促进了电子的注入和传输, 还延缓了空穴的传输。借助电致发光光谱, 我们证实 Alq_3 分子作为阶梯加速空穴从 CBP 分子到 $\text{Eu}(\text{TTA})_3\text{phen}$ 分子的迁移, 从而促进了电子和空穴在 $\text{Eu}(\text{TTA})_3\text{phen}$ 分子上的平衡。因此, 我们认为器件的效率滚降受到抑制的原因有两点: 一是复合区间的加宽, 二是 $\text{Eu}(\text{TTA})_3\text{phen}$ 分子上空穴和电子的分布更加平衡。

关键词: 铈(III)配合物; 荧光性质; 电致发光; 效率滚降

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Suppression of Efficiency Roll-off in Co-doped Electroluminescent Devices Based on Europium Complex

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Abstract: By co-doping $\text{Alq}_3[\text{tris}(8\text{-hydroxyquinoline}) \text{ aluminum}]$ and $\text{Eu}(\text{TTA})_3\text{phen}$ (TTA=thenoyltrifluoroacetone, phen=1,10-phenanthroline) into CBP (4,4'-N,N'-dicarbazole -biphenyl), a series of electroluminescent (EL) devices were fabricated and investigated. After optimizing the co-doping concentration of Alq_3 , significant suppression of EL efficiency roll-off and almost doubled maximum brightness were realized without deteriorating the color purity. The presence of Alq_3 molecules in light-emitting layer (EML) not only facilitates the injection and transport of electrons but also retards the transport of holes. Alq_3 molecules function as ladders of hole transfer from CBP to $\text{Eu}(\text{TTA})_3\text{phen}$ molecules as supported by the EL spectroscopy results, thus facilitating the balance of holes and electrons on $\text{Eu}(\text{TTA})_3\text{phen}$ molecules. Therefore, two possible reasons are suggested to be responsible for the suppression of EL efficiency roll-off: one is the broadening of recombination zone, the other is the improved balance of holes and electrons on $\text{Eu}(\text{TTA})_3\text{phen}$ molecules.

Key words: Eu^{3+} complexes; luminescent properties; electroluminescence; efficiency roll-off

Trivalent europium (Eu^{3+}) complexes have attracted considerable attention in the application of organic light-emitting devices (OLEDs) because of their monochromatic red light at around 612 nm

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corresponding to the $^5D_0 \rightarrow ^7F_2$ transition of Eu^{3+} ions, and theoretically 100% internal quantum efficiency^[1,2]. In the past years, several groups have devoted to the synthesis of novel europium complexes and the optimization of device structures^[3-7]. Due to the poor charge-carrier transporting ability and film-forming ability, doping europium complexes into host materials has been proven to be an efficient strategy to enhance the electroluminescent (EL) performance^[7-10]. However, the low-lying HOMO levels of europium complexes make it difficult to choose proper host materials for device fabrication^[3,9]. As a result, most europium complexes trap only one type carrier (either holes or electrons) and the other one resides mainly on host materials^[4]. For example, $\text{Eu}(\text{TTA})_3\text{phen}$ (TTA = thenoyltrifluoroacetone, phen = 1,10-phenanthroline) has been demonstrated to trap only electrons when it is doped into CBP (4,4'-N,N'-dicarbazole-biphenyl)^[11]. Consequently, the asymmetric carrier trapping causes the unbalanced carrier distribution on both CBP and $\text{Eu}(\text{TTA})_3\text{phen}$ molecules, thus limiting the improvement of EL efficiency because carrier trapping is the dominant EL mechanism of these doped devices.

In a recent paper, we have reported the gradual decrease of electron injection by controlling the thicknesses of LiF and Al layers. With decreasing electron injection, electrons situated on $\text{Eu}(\text{TTA})_3\text{phen}$ molecules decrease gradually; as a result, holes and electrons on $\text{Eu}(\text{TTA})_3\text{phen}$ molecules tend to become more balanced. Therefore, maximum EL current efficiency and power efficiency as high as $9.53 \text{ cd} \cdot \text{A}^{-1}$ and $5.35 \text{ lm} \cdot \text{W}^{-1}$, respectively, are obtained with serious efficiency roll-off^[7,12-13]. To suppress the rapid roll-off of EL efficiency in the devices based on europium complexes, various methods have been designed and attempted by different groups^[8,12,14,15]. Among those methods, co-doping fluorescence or phosphor dye into light-emitting layer (EML) as assistant dopant has been demonstrated to be the most efficient one^[8,15]. For example, by selecting the well known red dye DCJTB (4-(dicyanomethylene)-2-*t*-butyl-6 (1,1,7,7-tetramethyljulolidyl-9-enyl)-4*H*-pyran)

as assistant dopant, Ma et al.^[8] obtained the $9.0 \text{ cd} \cdot \text{A}^{-1}$ maximum EL current efficiency and the decreased efficiency roll-off; as a result, maximum brightness of $2\,450 \text{ cd} \cdot \text{m}^{-2}$ was realized. While significant improvements have been proven, co-doping fluorescence or phosphor dye into EML is usually accompanied by two troublesome problems: one is the emission of dye, which deteriorates the colour purity of these devices; the other is the relatively high price of most dyes, which increases the fabrication cost of these devices.

In this work, the excellent electron transport material tris(8-hydroxyquinoline) aluminum (Alq_3) with relatively low price was selected as the assistant dopant. By co-doping Alq_3 and $\text{Eu}(\text{TTA})_3\text{phen}$ into CBP, a series of EL devices with slower efficiency roll-off and higher brightness were fabricated. Excitingly, no Alq_3 emission was observed in these co-doped devices. In addition, the EL mechanisms of these co-doped devices were investigated in detail by analyzing the energy levels, the transport and distribution of holes and electrons, and the EL spectra. Firstly, increased electron transport and decreased hole transport in EML after Alq_3 co-doping were confirmed by comparing the electron only devices with and without Alq_3 co-doping, which indicates the broadening of recombination zone in the co-doping devices. Then, with the help of EL spectra, Alq_3 molecules co-doped in EML were demonstrated to accelerate the transfer of holes from CBP to $\text{Eu}(\text{TTA})_3\text{phen}$, thus facilitating the balance of holes and electrons on $\text{Eu}(\text{TTA})_3\text{phen}$ molecules. Therefore, wider recombination zone and better carriers balance were suggested to be responsible for the suppression of EL efficiency roll-off.

1 Experimental

Most of the organic materials used in this study were obtained commercially and used as received, while $\text{Eu}(\text{TTA})_3\text{phen}$ was synthesized and purified in our laboratory. The devices used in this investigation were fabricated by thermal evaporation in vacuum onto ITO (indium-tin-oxide) coated glass substrates

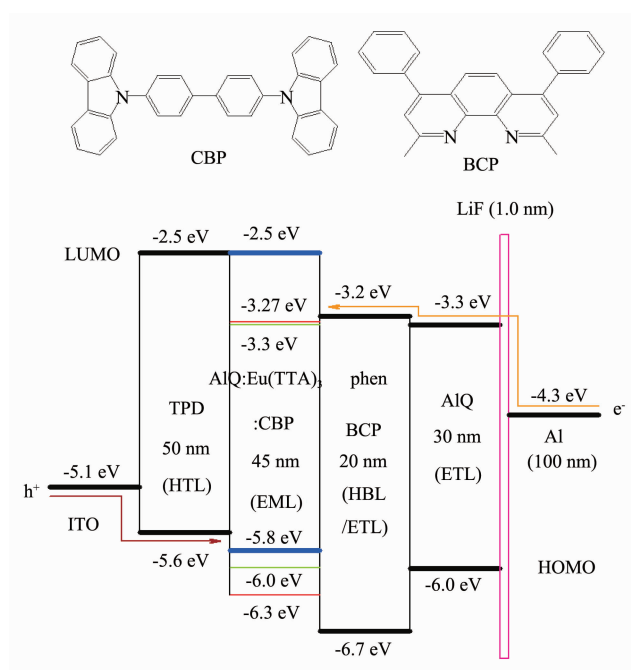
with the sheet resistance of $15 \Omega \cdot \text{sq}^{-1}$. The low-pressure oxygen plasma treatment of ITO substrates was performed in a special chamber with the pressure of 10 Pa. All the organics except Alq_3 and $\text{Eu}(\text{TTA})_3\text{phen}$ were evaporated with the rate of $0.05 \text{ nm} \cdot \text{s}^{-1}$ under high vacuum ($\leq 3 \times 10^{-5}$ Pa). The $\text{Eu}(\text{TTA})_3\text{phen}$ doped CBP layers were prepared by co-evaporating $\text{Eu}(\text{TTA})_3\text{phen}$ and CBP from two individual sources, while the Alq_3 and $\text{Eu}(\text{TTA})_3\text{phen}$ co-doped CBP layers were prepared by co-evaporating Alq_3 , $\text{Eu}(\text{TTA})_3\text{phen}$ and CBP from three individual sources. During these processes, the doping concentrations were determined by controlling the evaporation rate of Alq_3 and $\text{Eu}(\text{TTA})_3\text{phen}$, respectively. LiF and Al were evaporated in another vacuum chamber ($\leq 8.0 \times 10^{-5}$ Pa) with the rates of 0.01 and $1 \text{ nm} \cdot \text{s}^{-1}$, respectively, without being exposed to the atmosphere. The thicknesses of these deposited layers and the evaporation rate of individual materials were monitored in vacuo with quartz crystal monitors. The shape of the cathode was defined by using a shadow mask during the deposition of Al. The active area of these devices as defined by the overlapping area of the cathode and the anode is 9 mm^2 . After fabrication,

all the devices were measured immediately in air at room temperature without encapsulation. Current density-brightness-voltage characteristics were measured by using a Keithley source measurement unit (Keithley 2400 and Keithley 2000) with a calibrated silicon photodiode. The EL and photoluminescence (PL) spectra were measured with a Hitachi F-7000 fluorescence spectrophotometer.

2 Results and discussion

2.1 Suppression of EL efficiency roll-off after co-doping Alq_3 into EML

In this study, Alq_3 were selected as the assistant dopant to suppress the rapid efficiency roll-off in the doped EL devices based on $\text{Eu}(\text{TTA})_3\text{phen}$. Fig.1 shows the proposed energy level diagram of the device structure used in this investigation and also the thickness of each layer [7,11,16-17]. ITO substrates treated with low-pressure oxygen plasma were selected as the anodes, TPD (*N*, *N'*-Diphenyl-*N*, *N'*-bis (3-methylphenyl)-1,1'-diphenyl-4,4'-diamine) was selected as the hole transport layer (HTL), Alq_3 and $\text{Eu}(\text{TTA})_3\text{phen}$ co-doped into CBP as the EML, BCP is 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline as the hole



The thickness of each layer and the chemical structures of CBP and BCP are also shown

Fig.1 Proposed energy level diagram of the devices used in this paper

Table 1 Performance of the devices with different co-doping concentrations of Alq₃

Device (wt% of Alq ₃)	B_{\max}^a / (cd·m ⁻²)	EL efficiency (cd·A ⁻¹) at different current density (mA·cm ⁻²)				
		0.1	1	10	100	500
0%	1372.4	4.58	3.33	2.11	0.90	0.26
0.1%	1693.3	4.64	3.33	2.11	0.99	0.35
0.2%	2035.3	4.73	3.34	2.13	1.01	0.41
0.3%	2394.6	4.98	3.44	2.14	1.09	0.48
0.4%	2199.1	4.58	3.10	1.85	0.95	0.42
0.6%	2094.8	4.07	2.77	1.73	0.93	0.38

^a Maximum brightness

block/electron transport layer (HBL/ETL), Alq₃ was selected as the electron transport layer (ETL), and LiF/Al is the cathode. The chemical structures of CBP and BCP are shown in Fig.1.

Keep the concentration of Eu (TTA)₃phen constant at 3%, five co-doped devices were fabricated by controlling the concentrations of Alq₃ to be 0.1%, 0.2%, 0.3%, 0.4%, and 0.6%, respectively. For comparison, a device without Alq₃ co-doping was fabricated as the reference device. The maximum brightness and the EL efficiencies of these devices operating at different current densities are summarized in Table 1. At relatively low current density, EL efficiency increases slightly and then decreases gradually with increasing co-doping concentration of Alq₃. Compared with this result, the more striking phenomenon is the significant enhancement of EL efficiency at relatively high current density after co-doping Alq₃ into EML. Among these devices, the 0.3% co-doped device shows the highest EL efficiency of 5.60 cd·A⁻¹. For convenience, the EL

efficiency-current density (η - J) characteristics of the 0.3% co-doped device and the reference device are compared in Fig.2. With increasing current density, EL efficiency of the 0.3% co-doped device decreases slower than that of the reference device. At relatively high current density of 500 mA·cm⁻², the 0.3% co-doped device shows the current efficiency of 0.48 cd·A⁻¹, which is about 85% higher than the corresponding value of 0.26 cd·A⁻¹ obtained by the reference device. Eventually, the 0.3% co-doped device obtains the 2 394.6 cd·m⁻² maximum brightness, while the corresponding value of the reference device is only 1 372.4 cd·m⁻².

Simultaneously, EL spectra of the 0.3% co-doped device operating at different current densities are shown in Fig.3. Besides the emission of Eu³⁺, two other emission peaks centered at 390 ~490 nm, respectively. The emission peaked at 390 nm originates from CBP due to the incomplete Frster

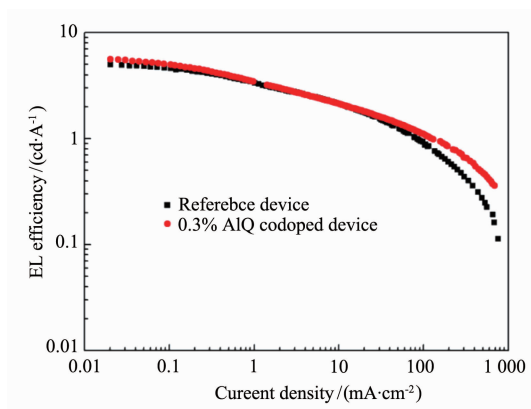
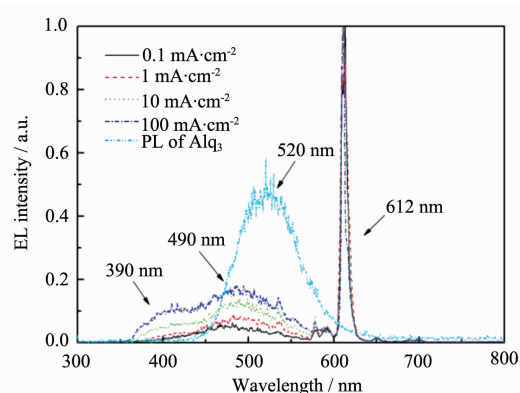


Fig.2 EL efficiency-current density (η - J) curves of the 0.3% co-doped device and the reference device



The PL spectrum of Alq₃ is also shown

Fig.3 Normalized EL spectra of the 0.3% co-doped device operated at different current densities

energy transfer from CBP to $\text{Eu}(\text{TTA})_3\text{phen}$ ^[7,13]. In a previous paper, we have demonstrated that the emission peaked at 490 nm originates from BCP due to the accumulation of electrons in HBL and the penetration of holes from EML into HBL^[18]. With increasing current density, the relative intensities of CBP and BCP emissions increase gradually versus that of Eu^{3+} ; however, no Alq_3 emission (at 520 nm) is observed from beginning to end. On the basis of these results, we conclude that co-doping Alq_3 and $\text{Eu}(\text{TTA})_3\text{phen}$ into CBP can significantly slow down the efficiency roll-off without deteriorating the color purity, thus causing the significant enhancement of maximum brightness. Furthermore, the relatively low price of Alq_3 is helpful to decrease the fabrication cost of these co-doped devices.

2.2 Wider recombination zone in the co-doped devices

To make clear the EL mechanisms of these co-

doped devices, it is necessary to investigate the carriers transport processes firstly. As shown in Fig.4, we schematically describe the injection and transport processes of electrons and holes. Considering the near lowest unoccupied molecular orbital (LUMO) levels of BCP (-3.2 eV) and Alq_3 (-3.3 eV)^[7,11], electron injection from BCP to Alq_3 molecules in EML (process 1) is believed to be possible, namely, the co-doping of Alq_3 molecules into EML provides another path for electron injection from HBL into EML. And, the only 0.03 eV difference between the LUMO levels of Alq_3 and $\text{Eu}(\text{TTA})_3\text{phen}$ makes it easy for electrons to transfer between them (processes 2 and 3), thus accelerating the transport of electron in EML^[7,11,19-20]. On the other hand, the co-doping of Alq_3 is believed to retard the transport of holes in EML due to its poor hole transport ability and lower highest occupied molecular orbital (HOMO) level than that of CBP^[11,19-20].

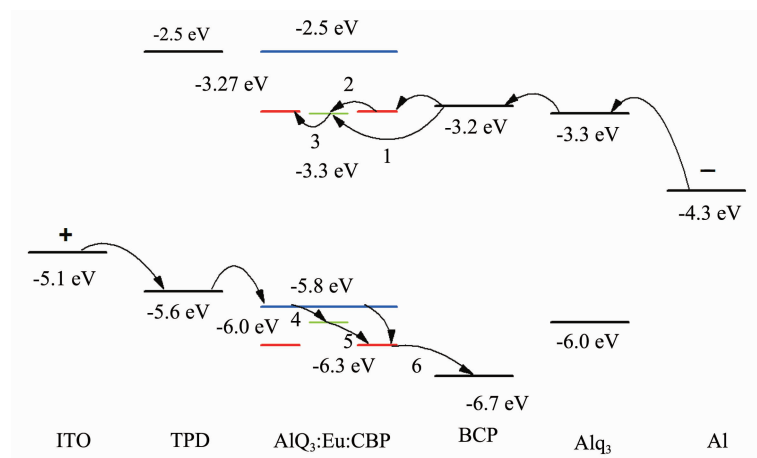


Fig.4 Schematic representation of carriers injection and transport processes in the co-doped devices

To verify our above inference, we have designed and fabricated two three-layer devices with and without Alq_3 co-doping to reveal the injection and transport mechanisms of electrons.

Device 1: anode/ Alq_3 (20 nm)/ $\text{Eu}(\text{TTA})_3\text{phen}$:CBP (3%, 60 nm)/ Alq_3 (20 nm)/LiF (1 nm)/Al (100 nm);

Device 2: anode/ Alq_3 (20 nm)/ Alq_3 :Eu(TTA)₃phen:CBP (0.3%, 3%, 60 nm)/ Alq_3 (20 nm)/LiF (1 nm)/Al (100 nm).

Only electrons are expected to transport in

devices 1 and 2, i.e. electron only devices^[19-20]. Current density-voltage (J - V) characteristics of devices 1 and 2 are shown in Fig.5. At certain driving voltage, the current density of device 2 is higher than that of device 1, which validates our inference that the co-doping of Alq_3 molecules into EML accelerates the injection and transport of electrons. On the other hand, as shown in Fig.6, current density of the 0.3% co-doped device is lower than that of the reference device. In this case, the decreased current density after Alq_3 co-doping confirms the decreased hole

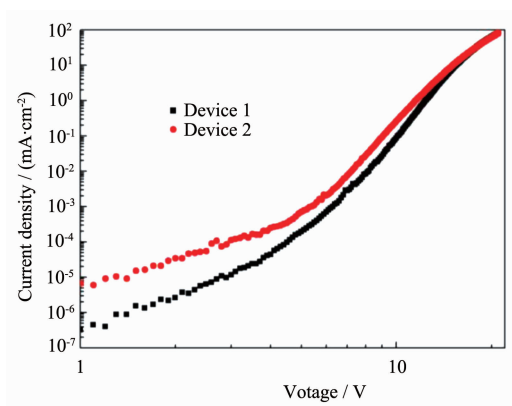


Fig.5 Current density-Voltage characteristics (J - V) of the electron only devices with and without Alq_3 doping

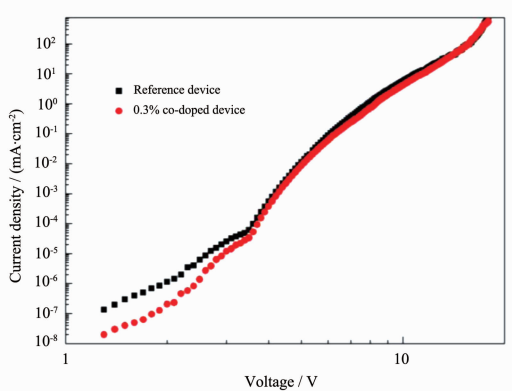


Fig.6 Current density-Voltage characteristics (J - V) of the reference device and the 0.3% co-doped device

transport in EML because Alq_3 molecules in EML have been demonstrated to accelerate the injection and transport of electrons.

Based on these results, we conclude that the recombination center would shift towards anode after co-doping Alq_3 molecules into EML due to the increased electron transport and the decreased hole transport. In previous reports^[11,18], the recombination centers of these devices have been demonstrated to be near the interface between EML and HBL. Then, the shift of recombination center towards anode will cause the broadening of recombination zone. Here, the width of recombination zone is defined as the average distance at which all injected carriers undergo recombination^[21-22]. In this case, energy back transfer and triplet-triplet (T-T) annihilation are the main mechanisms of EL efficiency roll-off^[7,23]. Theoretically speaking, wider recombination zone is favorable to

decrease the density of excitons, thus suppressing the notorious T-T annihilation^[7]. Therefore, the slowdown of efficiency roll-off after co-doping can be partially interpreted as the wider recombination zone.

2.3 Better balance of holes and electrons on Eu(TTA)₃phen molecules in the co-doped devices

According to the theory of Kalinowski^[22,24], the broadening of recombination zone would result in the decrease of recombination probability. At relatively low current density, no marked T-T annihilation take place; therefore, EL efficiency of the co-doped devices is expected to be lower than that of the reference device^[7,11,13]. Surprisingly, our experiments show the contrary result that the 0.3% co-doped device obtains higher efficiency at relatively low current density compared with the reference device. Take this fact into consideration, another reason is believed to be responsible for the suppressed efficiency roll-off and the enhancement of maximum EL efficiency, which can compensate for the decrease of recombination probability caused by the broadening of recombination zone.

To further understand the EL mechanisms of these co-doped devices, it is necessary to investigate the distributions of holes and electrons in EML and HBL. Here, the holes and electrons mean the charge carriers injected from anode and cathode, respectively, which would contribute to electroluminescence^[20]. At relatively low current density, EL efficiencies of these devices have been demonstrated to be determined by the densities of holes and electrons on Eu(TTA)₃phen molecules because carrier trapping is the dominant EL mechanism^[7,11-13]. In previous report^[12], we have demonstrated that the better balance of holes and electrons on Eu(TTA)₃phen molecules helps to enhance the recombination probability, thus enhancing the EL efficiency. As shown in Fig.4, the HOMO of Alq_3 locates between the HOMOs of CBP and Eu(TTA)₃phen. Theoretically speaking, holes can transfer firstly from CBP to Alq_3 molecules (process 4) and then transfer to Eu(TTA)₃phen molecules (process 5); namely, the presence of Alq_3 molecules in EML provides a ladder for hole transfer from CBP to

Eu(TTA)₃phen molecules^[18,20]. As a result, faster hole transfer from CBP to Eu(TTA)₃phen molecules is expected, which would facilitate the balance of holes and electrons on Eu(TTA)₃phen molecules because holes have been demonstrated to be the minority carriers^[11-12].

Fig.7 shows the EL spectra of these co-doped devices operated at 0.1 mA·cm⁻². Interestingly, BCP emission increases gradually with increasing co-doping concentration of Alq₃, which indicates the increasing hole penetration from EML into HBL. Previously, we have demonstrated that Eu(TTA)₃phen molecules in EML function as the ladders between CBP and BCP molecules (process 6)^[18]. So the increasing hole penetration from EML into HBL implies the increasing hole intensity on Eu(TTA)₃phen molecules, thus the increasing hole transfer from CBP to Eu(TTA)₃phen molecules. As shown in Fig.8a and b, the increasing hole transfer from CBP to Eu(TTA)₃phen molecules will facilitate the balance of holes and electrons on Eu(TTA)₃phen molecules. Therefore, the slight increase of EL efficiency at relatively low current density can be attributed to the increased hole transfer from CBP to Eu(TTA)₃phen in the co-doped devices. Based on these discussions, it may be concluded that the suppression of efficiency roll-off after co-doping Alq₃ into EML stems from the broadening of recombination zone and the better balance of holes and electrons on Eu(TTA)₃phen molecules.

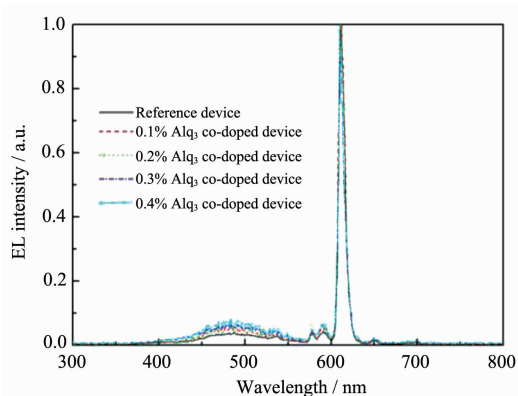


Fig.7 Normalized EL spectra of reference device and these co-doped devices with different concentrations of Alq₃ operated at 0.1 100 mA·cm⁻²

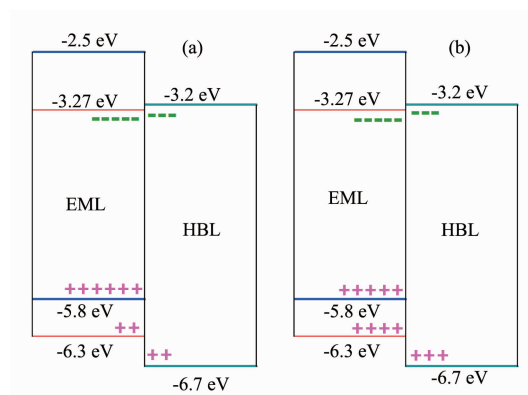


Fig.8 Distribution of holes and electrons in EMLs and HBLs of the reference device (a) and the 0.3% co-doped device (b)

3 Conclusions

By selecting Alq₃ as the assistant dopant, we have demonstrated an efficient method to suppress the efficiency roll-off of the EL devices based on Eu(TTA)₃phen. After optimizing the co-doping concentration of Alq₃, significant enhanced EL efficiency at relatively high current density and nearly doubled maximum brightness are realized without deteriorating the color purity. The co-doping of Alq₃ into EML not only accelerates the injection and transport of electrons into EML but also facilitates the transfer of holes from CBP to Eu(TTA)₃phen molecules, thus causing the broadening of recombination zone and the better balance of holes and electrons on Eu(TTA)₃phen molecules. This method provides a general guide for the optimization of EL devices based on europium complexes. Moreover, the relatively lower price of Alq₃ compared with fluorescence or phosphor dye is helpful to decrease the fabrication cost, which is very important for the commercial application of OLEDs.

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