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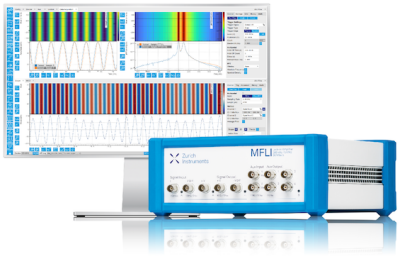

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Emission mechanism in organic light-emitting devices comprising a europium complex as emitter and an electron transporting material as host

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The emission mechanism in organic light-emitting devices, where the emission layer is composed of $Eu(DBM)_3$pyzphen (DBM=Dibenzoylmethane, pyzphen=pyrazino-[2,3-f][1,10]-phenanthroline) doped into electron transporting/hole blocking material BPhen (4,7-diphenyl-1,10-phenanthroline), is investigated. Energy transfer and carrier trapping simultaneously exist in the luminescence process, and carrier trapping is a main process. Direct carrier trapping by $Eu(DBM)_3$pyzphen molecules is confirmed by the difference of electroluminescence and photoluminescence spectra as well as *J-V* characteristics. Efficient Föster and Dexter energy transfer from BPhen to $Eu(DBM)_3$pyzphen molecules were speculated in terms of analysis of photoluminescence spectra of fixed solutions, triplet energies, and phosphorescent lifetimes. Based on these mechanisms, the overall performances of these devices were improved. High efficiencies were obtained under carrier trapping by $Eu(DBM)_3$pyzphen molecules, and the emission of BPhen was eliminated by efficient energy transfer from the BPhen to $Eu(DBM)_3$pyzphen molecules. © *2007 American Institute of Physics*. [DOI: 10.1063/1.2655225]

I. INTRODUCTION

Great progress has been made on device structures and materials since Tang developed efficient bilayered organic light-emitting devices (OLEDs) based on Alq_3.[1–3] Despite such tremendous achievements, efficient pure red OLEDs with narrow emission line is still a great challenge compared to blue and green OLEDs. Using organic Eu complexes as emitter is an attractive way to solve the problem. Eu complexes are characterized by extremely sharp red emission bands due to inner *f* orbits of the central Eu^{3+} ion. Furthermore, since the emission is due to electronic transitions of the central Eu^{3+} ion, the emission efficiency is not restricted by the spin inhibition rule,[4] which lowers the efficiency of normal π-conjugated organic compounds. For these reasons, Eu complexes are anticipated to exhibit high efficiencies as well as good purity of color in OLEDs.

In previous reports, several groups have produced Eu-complex OLEDs and achieved certain progresses in electroluminescence (EL) performances.[5–12] However, the properties of Eu-complex OLEDs are still not satisfying; for example, the EL efficiencies of Eu-complex devices were relatively lower than that of normal phosphorescent devices, significant decreases of efficiencies were observed at high current densities owing to triplet-triplet annihilation, and the red emission of Eu^{3+} ion is usually accompanied by the broad-band luminescence from the host or electron transporting material. Up to now, only a few materials have been used as the hosts of Eu-complex OLEDs such as 4,4-N,N-dicarbazole-biphenyl (CBP), N,N′-diphenyl-N,N′-bis(3-methylphenyl)-1,1′-biphenyl-4,4′-diamine (TPD), and 2-(4′-biphenyl)-5-(4″-tert-butylphenyl)-1,3,4-oxadiazole (PBD). Moreover, the emission mechanisms were not clear in more detail,[7,10,13] especially that no attempt has been made to make clear the Dexter energy transfer from host to Eu complex. In order to improve the performances of Eu-complex OLEDs, a doping system with an efficient Eu complex and an appropriate host should be designed, and the emission mechanisms need to be investigated further.

In recent research, we successfully fabricated an efficient red emitting OLED by doping $Eu(DBM)_3$pyzphen (DBM =Dibenzoylmethane, pyzphen=pyrazino-[2,3-f][1,10]-phenanthroline) into electron transporting/hole blocking material BPhen (4,7-diphenyl-1,10-phenanthroline). A reduced efficiency roll off, improved efficiency, and extended operating lifetime were obtained.[14] However, the detailed emission mechanism remains unclear and the device structure needs further optimization. In this paper, the emission mechanism in BPhen doped $Eu(DBM)_3$pyzphen system is investigated in terms of energy transfer and carrier trapping on $Eu(DBM)_3$pyzphen. Carrier trapping is identified by the difference of photoluminescence (PL) and electroluminescence (EL) spectra as well as current versus voltage characteristics (*J-V*). Energy transfer might simultaneously exist in the EL process, which was inferred by analyzing the PL spectra of fixed solutions, triplet energies, and phosphorescent lifetimes.

Energy transfer and carrier trapping are two main EL mechanisms in doping system.[15] The two mechanisms may simultaneously exist in the EL process, or one of them is dominant, which depends on the device structure and the used dopant and host. Uchida *et al.* have shown that charge

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carrier trapping by dopant molecules was beneficial to enhance EL efficiency.[15] But in such a trapping device, the emission of Eu^{3+} is generally accompanied by broad-band luminescence from the host due to the poor energy transfer between the host and the dopant.[7,11] On the other hand, the emission of the host could be eliminated by using efficient energy transfer from the host to the dopant, but high efficiency is hard to be obtained from such a device. In our device based on $Eu(DBM)_3$pyzphen doped into BPhen, a main EL mechanism of carrier trapping in company with efficient energy transfer simultaneously exists in the EL process. As a result, not only high efficiency but also pure red emission were obtained.

II. EXPERIMENT

The structure of the OLEDs used in this paper is indium tin oxide (ITO)/TPD (50 nm)/$Eu(DBM)_3$pyzphen:BPhen (30 nm)/BPhen (35 nm)/LiF (1 nm)/Al (100 nm). $Eu(DBM)_3$pyzphen was synthesized in our lab; other materials were commercially available without further purification. All layers were evaporated onto a pretreated ITO coated glass substrate (25 Ω/sq) at a chamber pressure of about 5×10^{-4} Pa. The doping process is carried out by coevaporation method. The emitting area is 2.5×4 mm^2. The PL and EL spectra were detected by a Hitach-4000 fluorescence spectrophotometer. Absorption spectra were measured with a Shimaduz UV-3000 spectrophotometer, and the luminance-current-voltage characteristics were measured using a calibrated multifunctional system for EL measurement. The relative alignment of the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) levels of $Eu(DBM)_3$pyzphen and BPhen was estimated from the results of cyclic voltammetry (CV) and absorption spectra; others are cited from literature.[16,17] For the optical measurements, thin films of $Eu(DBM)_3$pyzphen and BPhen were prepared by spin coating from a chloroform solution on a quartz substrate, respectively. The PL decay times were measured by a spectrometer (Spex 1403), a photomultiplier, a box car averager, and the sample was excited by yttrium aluminum garnet (YAG):Nd laser at a wavelength of $\lambda=355$ nm. All the measurements were carried out in atmosphere at room temperature.

III. RESULTS AND DISCUSSION

We first optimized the concentrations of $Eu(DBM)_3$pyzphen in the emission layer (EML) to obtain a optimal EL performances. Figure 1(a) shows the efficiency-current (*L-J*) characteristics for the devices with different $Eu(DBM)_3$pyzphen concentrations (15, 25, 30, 40, and 100 wt %). It can be seen that the efficiency-current curve is very sensitive to the doping concentration and the doped devices show much higher efficiencies than the neat (100 wt %) device. With increasing $Eu(DBM)_3$pyzphen concentration, the efficiency of the devices increase first, reach a maximum at the concentration of 25–30 wt %, and then decrease. High efficiencies of 2.4% and 2.5% were obtained from the 25 and 30 wt % devices, respectively. The optimal device (30 wt % device) exhibited a maximum luminance of

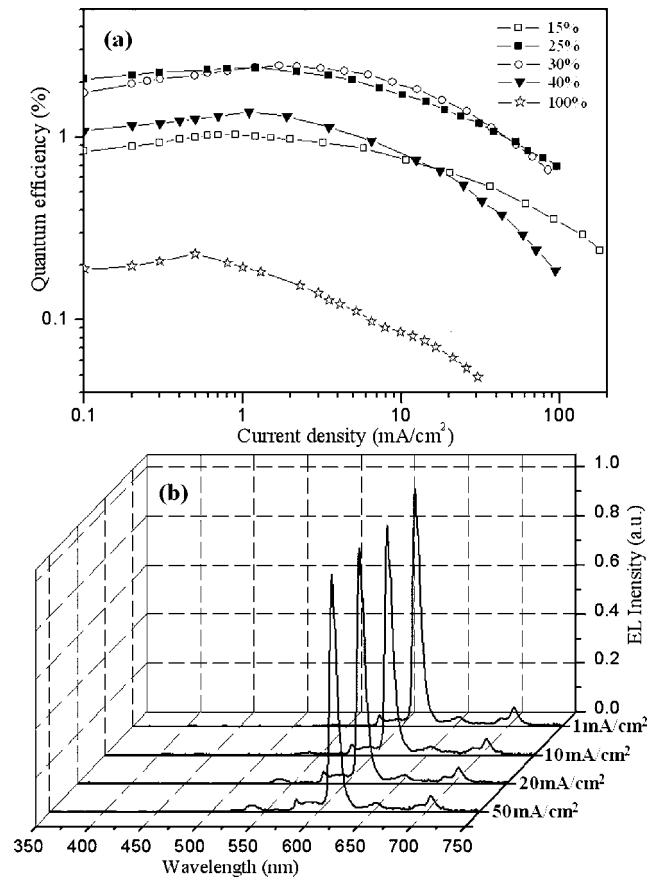

FIG. 1. (a) Efficiency-current characteristics for ITO/TPD/$Eu(DBM)_3$pyzphen:BPhen/BPhen/LiF/Al with different $Eu(DBM)_3$pyzphen concentrations: 15, 25, 30, 40, and 100 wt %. (b) EL spectra of the optimal device at the current densities of 1, 10, 20, and 50 mA/cm^2.

1320 cd/m^2. An external quantum efficiency of 2.5% and a current efficiency of 5.3 cd/A were achieved at the current density of 1.7 mA/cm^2. When the current density increased, high efficiencies of 1.9% and 1.5% were maintained at the current density of 10 and 20 mA/cm^2, respectively. Figure 1(b) shows the EL spectra of the optimal device at different current densities. Pure red emission was obtained at a large range of current densities, and the color coordinates were (0.66, 0.33) and (0.65, 0.34) at the brightness of 200 and 1000 cd/m^2, respectively.

We now discuss the emission mechanism in $Eu(DBM)_3$pyzphen:BPhen doping system in terms of energy transfer and direct carrier trapping.

Figure 2 shows the PL, excitation, and absorption spectra of $Eu(DBM)_3$pyzphen and BPhen films; also shown is their molecular structure. It can be seen that there is a large overlap between the PL spectrum of BPhen and absorption spectrum of $Eu(DBM)_3$pyzphen. According to the Föster energy transfer theory, the transfer rate parameters are proportional to the overlap area. So we can speculate that there is an efficient Föster energy transfer between $Eu(DBM)_3$pyzphen and BPhen. An experiment was set up to investigate the existence of energy transfer simply by measuring the PL emission. We prepared chloroform solutions of BPhen (1×10^{-2} g/l) mixed with 0, 1×10^{-4}, 3×10^{-4}, 6

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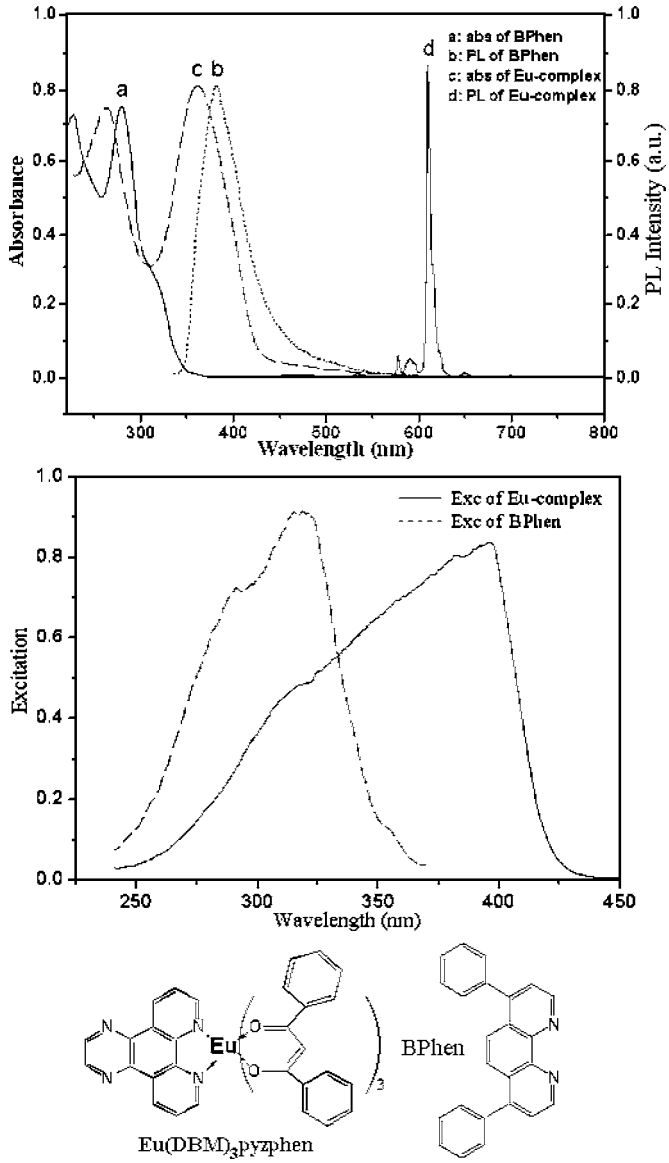

FIG. 2. PL, absorption, and excitation spectra of $Eu(DBM)_3$pyzphen and BPhen films and their chemical structures.

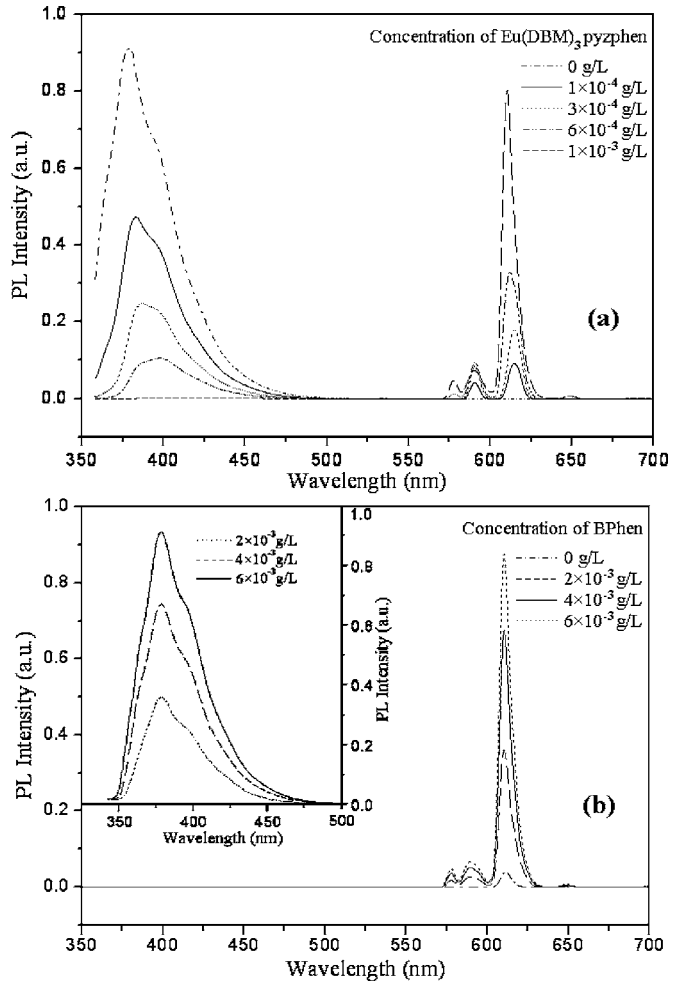

FIG. 3. PL spectra of (a) BPhen chloroform solutions (1×10^{-2} g/L) mixed with 0, 1×10^{-4}, 3×10^{-4}, 6×10^{-4}, and 1×10^{-3} g/l $Eu(DBM)_3$pyzphen, respectively; (b) $Eu(DBM)_3$pyzphen chloroform solutions (1×10^{-3} g/L) mixed with 0, 2×10^{-3}, 4×10^{-3}, and 6×10^{-3} g/l BPhen, respectively; and [(b) (inset)] 2×10^{-3}, 4×10^{-3}, and 6×10^{-3} g/l BPhen chloroform solutions. The spectra were photopumped at $\lambda=280$ nm.

$\times10^{-4}$, and 1×10^{-3} g/l (0, 1, 3, 6, and 10 wt %) $Eu(DBM)_3$pyzphen, respectively, and collected emission spectra. The samples were photopumped at 280 nm, which is close to the maximum absorption of BPhen. At the excitation wavelength of 280 nm, the Eu complex has a weak excitation and might influence the results of energy transfer. In order to exclude the effect of Eu complex direct excitation, we designed a contrastive experiment and subtracted the corresponding direct excitation PL intensity from mixed spectra. Figure 3(a) shows the results which having been excluded direct excitation effect. The broad band of 380 nm arises from BPhen emission, while the sharp peak at 612 nm corresponds to the $^5D_0\rightarrow{}^7F_2$ transition of Eu^{3+}. The intensity of the 612 nm peak clearly grows when the Eu complex concentration increases, while the intensity of the BPhen emission decreases dramatically. When the Eu complex concentration increases to 1×10^{-3} g/l (10 wt %), the emission from BPhen is already being quenched completely. These results proved that the excited BPhen molecules have transferred its energy to $Eu(DBM)_3$pyzphen molecules. In the case of 1×10^{-3} g/l $Eu(DBM)_3$pyzphen chloroform solutions mixed with 0, 2×10^{-3}, 4×10^{-3}, and 6×10^{-3} g/l (0, 200, 400, and 600 wt %) BPhen, respectively, similar results are observed [Fig. 3(b)]. These solutions were excited at 280 nm. With the same Eu-complex concentration, the PL intensity of Eu complex grows obviously when BPhen concentration increases, indicating that the pump energy is absorbed by the BPhen and then transferred to the Eu-complex ligand. It is observed that the intensity of the 612 nm peak is almost ten times larger when 2×10^{-3} g/l BPhen is present. For 6×10^{-3} g/l BPhen, the intensity increases to twenty-five times larger. The PL spectra do not show any peak associated with the emission of BPhen even at large BPhen concentration. Since no obvious concentration quenching was observed in the range of 2×10^{-3}–6×10^{-3} g/l BPhen solutions [Fig. 3(b) (inset)], the disappearance of BPhen emission should be attributed to the efficient Föster energy transfer from BPhen to $Eu(DBM)_3$pyzphen.

For rare earth complex, it is generally recognized that the first ligand plays a main role in energy transfer and the

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second ligand acts to saturate the coordination number of central ion and also to improve the fluorescence intensity, volatility, and stability of the rare earth complex.[18] Thus in the energy transfer process of our doping system, host was excited at the first and then transferred its energy to the first ligand site of Eu complex. If this energy transfer is Föster energy transfer, the energy is transfer from the singlet state of BPhen to that of DBM and then transferred to triplet state of DBM by intersystem crossing. Finally, the excited triplet state transferred its energy to central ion ($S_{\mathrm{BPhen}} \rightarrow S_{\mathrm{DBM}} \rightarrow T_{\mathrm{DBM}} \rightarrow \mathrm{Eu}^{3+}$). Besides Föster energy transfer, there may exist Dexter energy transfer, which has a different energy transfer route: the energy is directly transferred from the triplet state of BPhen to that of DBM and then to central ion ($T_{\mathrm{BPhen}} \rightarrow T_{\mathrm{DBM}} \rightarrow \mathrm{Eu}^{3+}$).

Because there is a large doping concentration in the optimized device (30 wt %, 9.50 mol %), the molecule distance between BPhen and $\mathrm{Eu(DBM)_3pyzphen}$ molecule would be relatively small. It is possible that the energy is transferred from BPhen to $\mathrm{Eu(DBM)_3pyzphen}$ through Dexter process. In Dexter mechanism, the energy transfer is based on exchange interaction, where an electron and a hole simultaneously transferred from one molecule to the next with no change in spin.[19] Different from the Föster process which could only transfer singlet energy, Dexter process could transfer triplet energy from one molecule to another.[20] It means that Dexter process might make the most of the host's energy and get better EL performances.

Four processes determine the overall efficiency of triplet energy transfer in Dexter process: the forward and reverse triplet transfer rates between the dopant and host, k_F and k_R, respectively, and the rates of exciton relaxation on the dopant and host, k_G and k_H, respectively. To maximize the dopant EL efficiency, we require that either $k_G \gg k_H$ or $k_F > k_R$.[21] If the phosphorescence lifetime of the host is much larger than that of the dopant, the requirement of $k_G \gg k_H$ will be fulfilled, and $k_F > k_R$ needs that the triplet energy level of the host is higher than that of the dopant. Since the Dexter energy transfer route of our doping system is $T_{\mathrm{BPhen}} \rightarrow T_{\mathrm{DBM}} \rightarrow \mathrm{Eu}^{3+}$, to have an efficient Dexter energy transfer, we required that $\tau_{\mathrm{BPhen}} \gg \tau_{\mathrm{Eu(DBM)3pyzphen}}$ and $T_{\mathrm{BPhen}} > T_{\mathrm{DBM}}$. Here τ_{BPhen} and $\tau_{\mathrm{Eu(DBM)3pyzphen}}$ are the phosphorescent lifetime of BPhen and $\mathrm{Eu(DBM)_3pyzphen}$, T_{BPhen} and T_{DBM} are the triplet energy levels of BPhen and DBM.

In order to estimate the efficiency of triplet energy transfer from BPhen to $\mathrm{Eu(DBM)_3pyzphen}$, we measured the phosphorescence lifetime and relaxed triplet state energies of both the donor and acceptor molecules. The phosphorescence lifetimes were estimated using their decay curves of PL emission, and the triplet energy levels were calculated from phosphorescent PL spectra.

Figure 4 shows the decay curves at the phosphorescence PL peak wavelength of BPhen (550 nm, $T=77$ K) and $\mathrm{Eu(DBM)_3pyzphen}$ (612 nm, $T=298$ K). The decay curves of the PL intensity, $I_{\mathrm{PL}}(t)$, are fitted according to

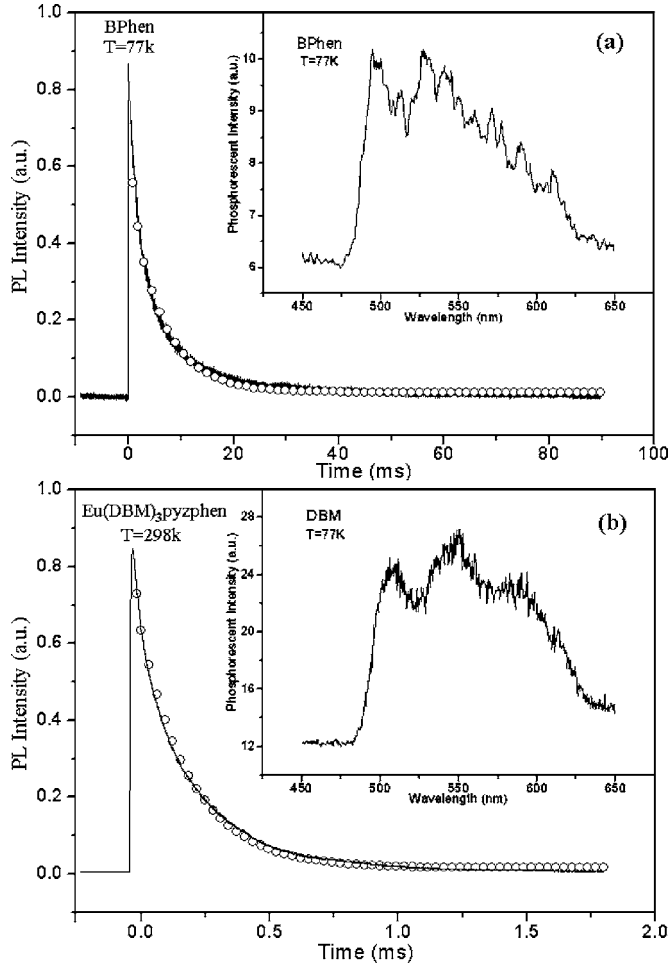

FIG. 4. PL emission decay curves of BPhen (550 nm, $T=77$ K) and $\mathrm{Eu(DBM)_3pyzphen}$ (612 nm, $T=298$ K) and corresponding monoexponential curve (○). Inset: Phosphorescence spectra of BPhen and DBM. The spectra were recorded at $T=77$ K due to their low phosphorescence efficiency at room temperature.

$$I_{\mathrm{PL}}(t) = \sum_{i=1}^{n} a_i \exp\left[\frac{-(t-t_0)}{\tau_i}\right],$$

where a_i are the preexponential factors, and τ_i is the decay time. The decay curves in BPhen and $\mathrm{Eu(DBM)_3pyzphen}$ film are both fitted to monoexponential function. The fitted parameters of BPhen and $\mathrm{Eu(DBM)_3pyzphen}$ are ($\tau_i=5$ ms, $a_i=100\%$) and ($\tau_i=0.28$ ms, $a_i=100\%$), respectively. The calculated phosphorescence lifetime of BPhen was significantly longer than that of $\mathrm{Eu(DBM)_3pyzphen}$, thus in $\mathrm{Eu(DBM)_3pyzphen}$:BPhen doping system, $k_G \gg k_H$. From the phosphorescent spectra in Fig. 4 (inset), we deduced that the triplet energy levels of BPhen and DBM are 2.5 and 2.4 eV, respectively; the demand of $k_F > k_R$ is also fulfilled. Furthermore, the gap between BPhen and DBM triplet energy levels is 0.1 eV, which is close to resonance, thus a large numbers of both guest and host triplets could exist. From the analysis mentioned above, we can infer that the energy could be efficiently transferred from host to dopant through Dexter process in our doping system.

Back Dexter energy transfer from an Eu-complex ligand to host was reported to be a main factor that significantly

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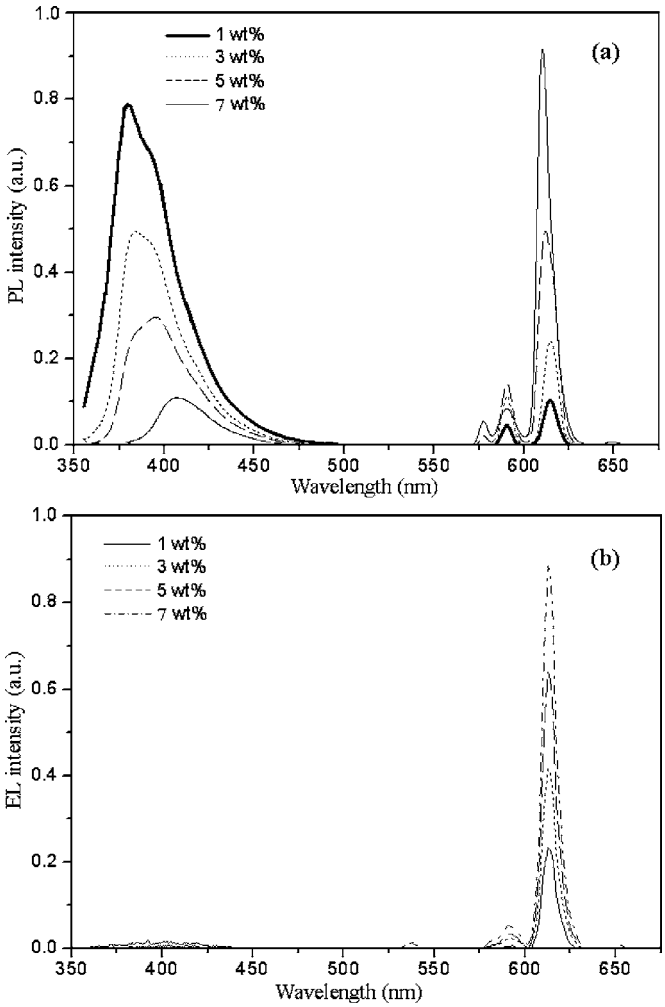

FIG. 5. PL (a) and EL (b) spectra of the devices doped with 1, 3, 5, and 7 wt % $Eu(DBM)_3$pyzphen, respectively.

decreased the efficiency even at low currents. At high currents, *T-T* annihilation on the host molecules which is caused by back energy transfer would lead a serious efficiency roll off.[7] In our doping system, the Dexter energy transfer route is from host to Eu complex, where $T_{BPhen} > T_{DBM}$ and $\tau_{BPhen} \gg \tau_{Eu(DBM)3pyzphen}$ can efficiently prevent back energy transfer. This is consistent with the facts that the optimal device exhibited high efficiencies and an alleviated efficiency roll off.

Next we discuss the probability of carrier trapping by $Eu(DBM)_3$pyzphen by comparing its optical and electrical excitation spectra. Figure 5 shows the PL and EL spectra of devices doped with 1, 3, 5, and 7 wt % $Eu(DBM)_3$pyzphen. It can be seen that there is a large difference between the PL and EL spectra. Emissions from the host BPhen can be observed even at the high $Eu(DBM)_3$pyzphen concentration of 7 wt % in PL spectra, whereas in EL spectra, the emissions of BPhen were much smaller than that in PL, and when the concentration is larger than 3 wt %, the EL spectra were only composed of $Eu(DBM)_3$pyzphen emission. This huge difference between the EL and PL spectra should be ascribed to selective carrier trapping by $Eu(DBM)_3$pyzphen molecules. If energy transfer from BPhen to $Eu(DBM)_3$pyzphen is a main route, then the shape of the EL spectra should coincide

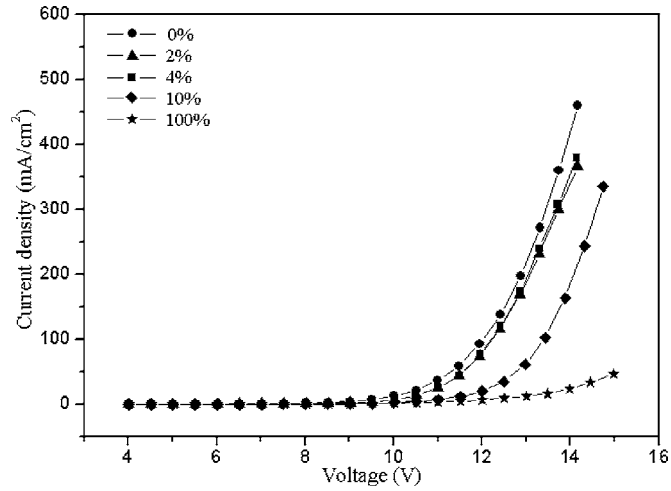

FIG. 6. Forward bias *J-V* characteristics in the devices with various concentrations of $Eu(DBM)_3$pyzphen: 2, 4, 10, and 100 wt %.

with that of PL spectra. Since the EL spectra were not the same as the PL spectra and the EL spectra were mainly composed of $Eu(DBM)_3$pyzphen emission, it is probable that the $Eu(DBM)_3$pyzphen molecules trap charge carriers.

Further evidence for carrier trapping by $Eu(DBM)_3$pyzphen can be inferred from the forward biased *J-V* characteristics. Figure 6 summarize the *J-V* characteristics of the devices with various concentrations of $Eu(DBM)_3$pyzphen. The device with neat BPhen as EML exhibited the largest current density. When $Eu(DBM)_3$pyzphen was doped into BPhen (2 wt %), a decrease of current density was observed, which would be attributed to the carriers captured by $Eu(DBM)_3$pyzphen molecules. As the concentration of $Eu(DBM)_3$pyzphen was increased (4 wt %), the distance between the $Eu(DBM)_3$pyzphen molecules becomes shorter, and the carrier can hop via $Eu(DBM)_3$pyzphen molecules;[22] accordingly the current density slightly increased. When the concentration of $Eu(DBM)_3$pyzphen was increased further, the current density decreased rapidly due to the poor film-forming ability of $Eu(DBM)_3$pyzphen.

Since both carrier trapping by $Eu(DBM)_3$pyzphen and efficient energy transfer from BPhen to $Eu(DBM)_3$pyzphen were confirmed, we now discuss which of them is the main mechanism in the EL process. Figure 7 shows the energy level diagram of the device, with the relative alignment of the HOMO and LUMO levels of each layer. The LUMO of $Eu(DBM)_3$pyzphen is 3.3 eV, which is much lower than that of BPhen, thus electrons injected into EML can be easily

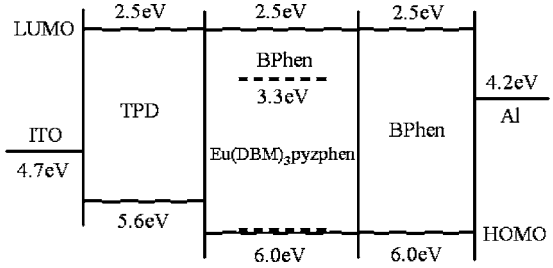

FIG. 7. Energy level diagram of the device used in this paper.

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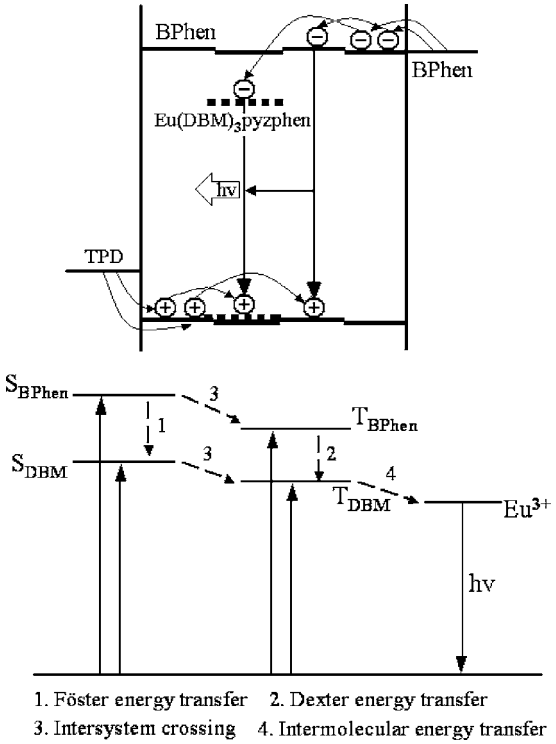

FIG. 8. Schematic emission mechanism diagram of $Eu(DBM)_3$pyzphen:BPhen doping system.

trapped by $Eu(DBM)_3$pyzphen. Since the electrons are trapped in the energetically favored LUMO of the $Eu(DBM)_3$pyzphen, the holes have a higher probability to also recombine on the $Eu(DBM)_3$pyzphen.[10] Therefore we can confirm that carrier trapping by $Eu(DBM)_3$pyzphen is the main EL mechanism. Considering BPhen has the similar HOMO level with $Eu(DBM)_3$pyzphen, carrier trapping by $Eu(DBM)_3$pyzphen could not complete and a part of excitons might form on BPhen. And since the energy transfer from BPhen to $Eu(DBM)_3$pyzphen was demonstrated to be quite efficient, the excited BPhen molecules would then transfer their energy to $Eu(DBM)_3$pyzphen molecules. Based on the discussion above, we reckon that in $Eu(DBM)_3$pyzphen:BPhen devices, carrier trapping is a main process and energy transfer from the host to guest is a subprocess.

As shown in Fig. 8, in the EL process, excitons that came from direct recombination of holes and electrons are mainly formed on the Eu-complex ligand, which created excited DBM singlets and triplets. Meanwhile, a part of excitons formed on BPhen and created excited BPhen singlets and triplets. The singlets of BPhen may convert to DBM singlets or BPhen triplets through Föster process and intersystem crossing, respectively. The DBM singlets and BPhen triplets then converted to DBM triplets through Dexter process and intersystem crossing, respectively. Finally, the DBM triplets that came from these routes undergo a nonradiative transition and converted to the excited state of the central Eu^{3+}. The Eu^{3+} exhibit sharp spectral bands corresponding to ^5D_x-^7F_x transitions.

IV. CONCLUSION

In conclusion, high efficiency, pure red emission was obtained from the optimized $Eu(DBM)_3$pyzphen:BPhen OLED. The emission mechanisms in the device are discussed. Energy transfer and carrier trapping simultaneously exist in the luminescence process, and carrier trapping is a main process. High efficiencies were obtained under carrier trapping by $Eu(DBM)_3$pyzphen molecules, and the emissions of BPhen were eliminated by efficient energy transfer from the BPhen to $Eu(DBM)_3$pyzphen molecules. With the back energy transfer been prevented by efficient Dexter energy transfer from host to Eu complex, the efficiency roll off at high current densities also alleviated.

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