Electric field-induced hole injection-enhanced photoluminescence in a N,N'-bis(3-methylphenyl)-N,N'-bis(phenyl)-benzidine-based emitter*

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Non-monotonic, asymmetrical electric field dependence of photoluminescence (PL) intensity is observed in a monolayer sample of tris-(8-hydroxyquinoline) aluminum (AlQ) doped N,N'-bis(3-methylphenyl)-N,N'-bis(phenyl)-benzidine (TPD). A possible model is proposed: the charge separation from the dissociated photoexcited excitons causes energy band bending in the organic films and improves the hole injection from the electrode, which brings about the extra fluorescence. This mechanism is further verified by a series of experiments using a series of samples, variously featuring symmetrical electrodes, block layers, and hosts with lower hole mobilities.

Keywords: photoluminescence, doped structure, external electric field, energy level

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1. Introduction

The properties of charge injection/transport and exciton diffusion/recombination are considered to be among the most useful parameters in improving the performance of organic light-emitting diodes (OLEDs). However, it was previously reported[1,2] that the EL quantum efficiency of OLEDs might be attenuated when driven by a high current density (electric field), which implies a complicated effect of external electric field on these key properties. So, in order to make clear the origin of this effect, the electric field-induced photoluminescence (PL) spectrum, which is a method showing directly the effect of electric field on the exciton after its excitation by light, is widely applied. Since it was observed in 1972,[3] electric field-induced PL quenching has been reported for various samples consisting of polymer,[4–6] small-molecule organic materials[7] or doping materials.[8] The phenomenon was mostly attributed to mechanisms such as electric field-assisted exciton nonradiative decay,[9] current-induced exciton quenching,[10] thermal exciton quenching,[11] and quenching for some other reason, such as the Stark effect.[12]

However, PL enhancement by electric field has been much less reported. One report was presented by Popvic *et al.*,[4] who observed the electric field-induced PL enhancement as well as a spectral shift in the yellow pigment DFC, and explained them as the strong coupling between the electric field and the dipole moment of the excited state. Ohta *et al.*[13] also found the fluorescence enhancement in the donor–acceptor complexes dissolved in the PMMA matrix, and they ascribed it to the increased back-electron transfer by electric field. More recently, Gulbinas *et al.*[14] discovered both the quenching and enhancement of PL by electric fields in DMABI single-layer samples. They suggested that the PL intensity is dependent on the trapped carriers, while the density of trapped carriers varies in different electric fields. However, systematic research directly comparing the PL changes under positive and negative fields has not been conducted, and the mechanism of the phenomenon is not yet quite clear. Thus, further work is needed to reveal a common mechanism responsible for electric field-induced PL intensity change.

In this paper, we present specific electric field-induced PL intensity changes in AlQ-doped TPD samples (indium tin oxide (ITO)/TPD:AlQ/Al). It turns out that in a negative electric field, the PL intensity first decreases with the electric field, and then quickly increases when the intensity of electric field is further increased; while in a positive electric field, the PL intensity first increases steeply with the electric field but soon starts to decrease at a higher electric field. Consequently, a possible underlying mechanism has been newly proposed: the photo-caused space charge promotes the injection of carriers under electric field, which in turn results in an enhancement of the fluorescence intensity. This mechanism can well explain the observed electric field-induced non-monotonic and the asymmetrical fluorescence intensity change, and it has been further confirmed by other experiments.

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2. Experiment

The samples were all fabricated on glass substrates coated by conducting transparent indium tin oxide (ITO) with a sheet resistance of about 17 Ω/sq. The substrates were pretreated by ultrasonic cleaning and UV zone treatment[15,16] before being loaded into a high-vacuum chamber with a base pressure of 1×10^{-5} Pa. Both the organic and metallic films were then sequentially deposited onto the substrate without a vacuum break, while the thickness and concentration of the films were determined *in situ* by a quartz crystal sensor. The Al_2O_3 was grown "film by film" by oxidation of a thin Al film (about 0.5 nm) each time. Oxidation was accomplished by the simple exposure of the Al film in O_2 atmosphere for about 10 min.[17]

The electric field-induced PL spectra measurements were carried out in an inert ($O_2 \sim 2$ ppm, $H_2O \sim 1$ ppm) atmosphere at room temperature. The constant driving voltage and current density–voltage (J–V) characteristics were determined using a Keithley 236 unit, with ITO connecting to the positive electrode and Al to the negative electrode. The excitation source of the PL was provided by a continual wave laser at 375 nm, incident through ITO. The PL spectra were recorded by the Spectra Scan 705 (from Photo-Research Inc.) on the same side, avoiding the reflected light. Note that EL emission may occur and overlap the PL emission when the positive electric field exceeds a threshold (usually 0.5 MV/cm ∼ 1.5 MV/cm[18]). It was verified that no EL emission was detected under the testing electric field without illumination.

The solar illumination was conducted by the 91192 Full Spectrum Solar Simulator (from the Newport Corp.) with a lamp wattage of 1000 W.

3. Results and discussion

3.1. Electric field-induced PL spectra in monolayer-doped samples

Figure 1(a) shows the PL spectra of pure AlQ, TPD, and AlQ-doped TPD. The doped material exhibits a stronger emission from the AlQ (at around 520 nm) than TPD (at around 410 nm), which implies the influence of the Föster energy transfer from TPD to AlQ.

While no shift of wavelength is observed in the doped structure under external electric field, an obvious change of emission intensity from AlQ is detected (as shown in Fig. S1). Figure 1(b) depicts how the AlQ's luminescence depends on the external electric field for the monolayer-doped sample (ITO/TPD:AlQ(2.3 wt%)(100 nm)/Al(100 nm) (sample 1)). The field dependence of PL intensity is defined as:

$$K_{\mathrm{PL}} = \frac{I_{\mathrm{PL}}(x)}{I_{\mathrm{PL}}(0)}, \tag{1}$$

where $I_{\mathrm{PL}}(0)$ and $I_{\mathrm{PL}}(x)$ denote the PL intensity at zero field and at external field of x MV/cm, respectively.

It can be seen that in a negative electric field, the PL intensity first decreases slightly with increasing electric field. After achieving a local minimum of 99% at −0.05 MV/cm, it starts increasing abruptly. When the electric field reaches −1.2 MV/cm, the PL intensity is observed to be higher than 110%. In contrast, in positive electric field, the PL enhances sharply once the electric field is applied and it soon reaches its local maximum at about 0.2 MV/cm with a value of 115%, which is even larger than the strongest fluorescence in a negative electric field. When the electric field is further increased, PL begins to quench and it ultimately reaches around 110%. Consequently, the electric field dependence of the PL intensity from AlQ in sample 1 is both non-monotonic and asymmetrical. A similar dependence is obtained with different weight rates of AlQ varying from 2% to 5% (as is shown in the supporting information Fig. S2). However, no obvious change in PL intensity of TPD with the electric field is observed in the doped sample (as shown in the supporting information Fig. S3). This may be due to the strong Förster energy transfer and the weak signal to noise ratio (SNR) of this emitting species, but future verification is required.

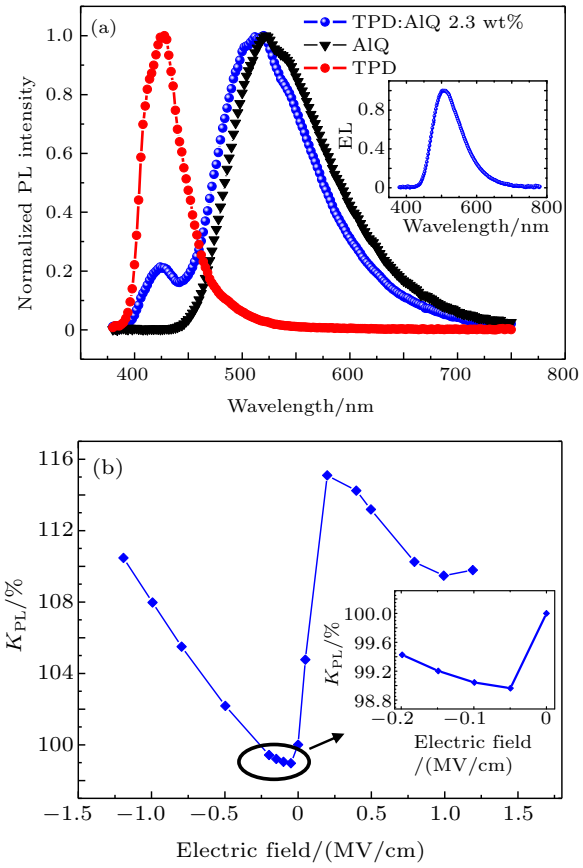


Fig. 1. (color online) (a) PL spectra of 2.3 wt% AlQ-doped TPD, along with pure TPD and AlQ. The inset shows the EL of the structure of ITO/TPD:AlQ(2.3 wt%)(100 nm)/Al(100 nm) with driving voltage of 20 V and current density of ∼ 1 mA/cm^2. (b) The electric field dependence of AlQ's PL intensity in ITO/TPD:AlQ(2.3 wt%)(100 nm)/Al(100 nm). The inset shows a blowup of the field dependence of PL intensity of the sample around 0 MV/cm.

3.2. Comparison with the known results and mechanism

In comparison with the previously reported photoluminescence quenching and enhancement, the results in sample 1 stand out with the following characteristics. First, while both ITO/AlQ/Al and ITO/TPD/Al show only electric field-induced PL quenching,[9] the doped sample 1 shows more complex results, with both quenching and enhancement. So the doping structure may be a key factor in these results. Secondly, while almost all of the previous reports depict similar dependencies of PL intensity upon the positive and negative electric fields,[13,14,19] the PL intensity in sample 1 is absolutely asymmetric with the electric field. Consequently, mechanisms like back-electron transfer[13] cannot account for this phenomenon. In addition, other articles report a local minimum (maximum) at zero electric field or in a positive electric field, which should be easy to understand in terms of the built-in electric field. But in sample 1, the local minimum is observed in negative electric field. So the phenomenon also cannot agree with the mechanisms offered by Gulbinas.[14]

3.3. A mechanism for electric field-induced hole injection-enhanced PL

A possible mechanism was proposed to explain the unique non-monotonic, asymmetrical electric field dependence of PL intensity in sample 1. First of all, both TPD and AlQ in the doped film are excited by the incident laser and form Frenkel excitons. The Frenkel excitons then diffuse to the interfaces of TPD/AlQ and dissociate into electrons and holes. Confined by the high energy barriers of about 0.7 eV $\sim$ 0.9 eV (as shown in Fig. 2(a)),[20–22] electrons are trapped[23] in the guest (AlQ), while holes are mostly located in the host (TPD). When an external electric field is applied, the electrons remain trapped in the original AlQ, due both to a long average distance between the nearest AlQ molecules and a lack of transport pathways. On the other hand, owing to its excellent mobility in the host (TPD), the holes are well transported between TPDs and ultimately collected by the electrode. Therefore, the local electrons develop a space-charge-effect in the doped film, causing the energy band bending. Compared with the energy level without this bending (as shown in Fig. 2(a)), the barrier becomes much thinner and holes can be injected more easily. Upon injection, the recombination of the injected holes and the electrons trapped there occurs, and energy is released as light emission.[24] In a negative electric field, the holes must be injected across the barrier between the Fermi energy of Al and the HOMO of the organic layer, which is rather hard (as shown in Fig. 2(b)), so the hole-injection still remains weak in a low electric field (as shown in Fig. 2(c)). In this case, the fluorescence enhancement is suppressed by electric field-induced quenching,[8] resulting in decreased the PL intensity. When the electric field is increased, the hole injection barrier is further reduced (as shown in Fig. 2(d)). Finally, the injection-caused fluorescence enhancement suppresses the quenching, and the PL intensity increases. On the other hand, with a positive electric field, the hole barrier is located between the Fermi energy of ITO and the HOMO of the organic film, which is much easier to overcome (as shown in Fig. 2(e)). Consequently, the enhancement of fluorescence covers the quenching, even in a low electric field (as shown in Fig. 2(f)). PL increases once the electric field is applied. However, this is not an endless increase because the hole-injection becomes so strong with the field that the recombination is over-saturated. Further injection of holes cannot bring about enhancement but quenches fluorescence.[25] Hence, PL intensity starts to decrease.

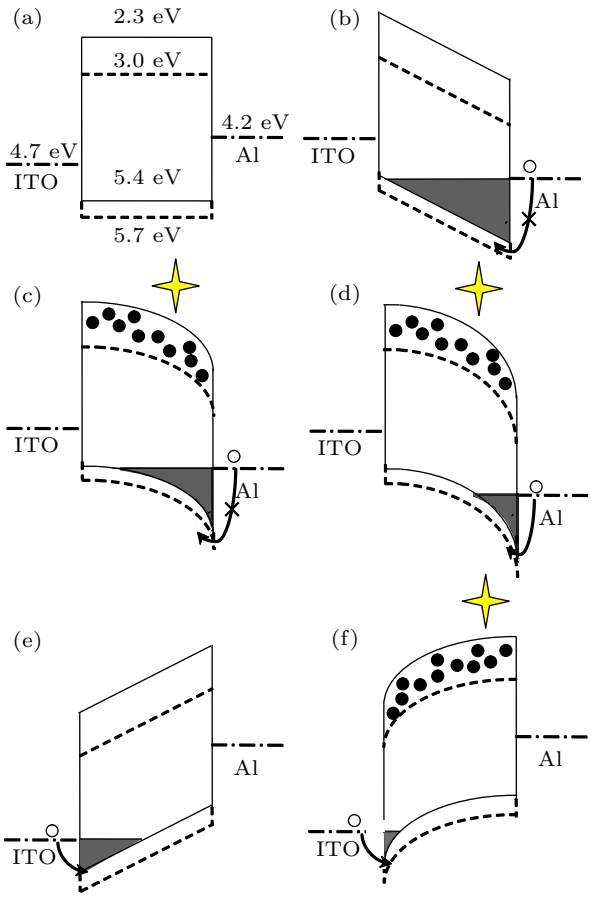


Fig. 2. (color online) Energy level diagram of samples (a) without neither field nor illumination, (b) with negative field and without illumination, (c) with low negative field and illumination, (d) with high negative field and illumination, (e) with positive field and without illumination, (f) with positive field and illumination. The solid line represents the energy level of the host and the dash line is the energy level of the guest. • represents the electron, ○ represents the hole. The gray area represents the hole injection barrier.

In conclusion, the dissociation of the photoexcited excitons causes energy band bending, which promotes the injection of holes and brings about extra fluorescence. Consequently, the asymmetry in sample 1's behavior may be due to the difference in hole-injection barriers under a positive or negative field.

3.4. Further verifications

(i) Dissociation and recombination

Figure 3 exhibits current density–voltage (J–V) characteristics for two samples of ITO/LiF(2 nm)/TPD:AlQ(100 nm)/LiF(2 nm)/Al(100 nm) with differing concentrations of AlQ. The layers of LiF are introduced to inhibit[22] dissociation at the electrode/organic interface. While no significant short-circuit current density (J_{sc}) is obtained in the dark, the J_{sc} under solar illumination increases as the AlQ's concentration increases from 2.3 wt% to 33 wt%. This suggests that dissociation of a photo-generated exciton may happen in the bulk heterojunction. The larger J_{sc} in Fig. 3(b) may imply a larger density of the dissociated exciton.

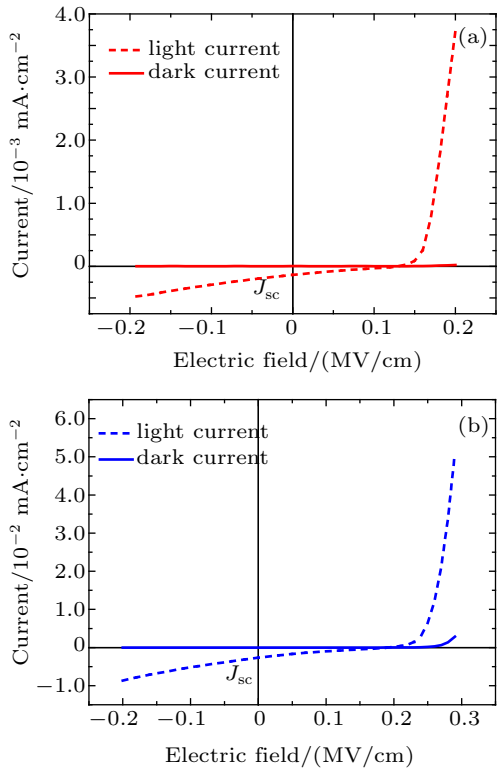


Fig. 3. (color online) J–V (J–E) characteristics of the bulk heterojunction cells (ITO/LiF(2 nm)/TPD:AlQ(100 nm)/LiF(2 nm)/Al(100 nm)) with AlQ's concentration of (a) 2.3 wt% and (b) 33 wt%. The dashed line represents the density of light current and the solid line represents the density of dark current.

The inset of Fig. 1(a) exhibits the PL of sample 1, and only the emission from the AlQ around 520 nm is observed. This may be good evidence that the injection finally contributes to the emission of AlQ.

(ii) Injection and symmetry

According to the mechanism, a sample with symmetrical electrodes may obtain a more symmetrical photoluminescence intensity. Figure 4 depicts the electric field dependence of AlQ's PL intensity in the symmetrical sample, ITO/Al(5 nm)/TPD:AlQ(3.1 wt%)(103 nm)/Al(100 nm) (sample 2). In a negative electric field, just as for sample 1, PL intensity initially decreases with electric field slightly and reaches its minimum of 99% at −0.05 MV/cm. When the electric field is further increased, it starts to increase and obtains 106% by the electric field of −1.2 MV/cm. However, in a positive electric field the PL intensity increases monotonically and reaches 106% at 1.2 MV/cm, which implies approximately the same enhancement as in the negative electric field. Consequently, by introducing symmetrical electrodes, PL intensity in the positive electric field increases more slowly in the low electric field and is without a decrease in the high electric field. The electric field dependence of the PL intensity in sample 2 becomes more symmetrical. This is easy to understand in terms of our mechanism. In the negative electric field, the holes are injected from Al to the organic film, which is just the same as in sample 1. Since the electrodes hardly affect the dissociation of the excitons and bending of the band, the electric field dependence of injection is also unchanged. Consequently, PL remains slightly quenched initially and quickly enhanced later on by the electric field. However, in a positive electric field, the hole injection electrode changes from original ITO to Al. Injection increases so slightly[26] that the recombination cannot be saturated even in the high electric field. Consequently, the PL intensity of sample 2 increases monotonically and gently with the positive electric field.

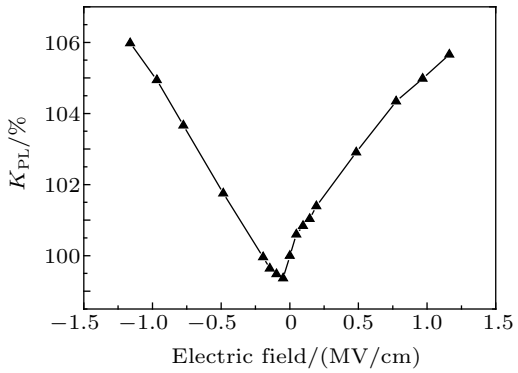


Fig. 4. The electric field dependence of AlQ's photoluminescence intensity in the symmetrical sample of ITO/Al(5 nm)/TPD:AlQ(3.1 wt%)(103 nm)/Al(100 nm).

However, it should be noted that in Fig. 4 the PL intensity is not completely symmetrical to the electric field, which may be caused by the penetration of the top metal atoms into the organic film[27] or the asymmetrical thickness of the electrodes. Taking into account the above-mentioned factor, block layers of insulating Al_2O_3 are introduced into the symmetrical sample. Figure 5 shows the electric field dependence of AlQ's PL intensity in ITO/Al(5 nm)/Al_2O_3(4.5 nm)/TPD:AlQ(3.0 wt%)(100 nm)/Al_2O_3(4.5 nm)/Al(100 nm) (sample 3). It is interesting to find

that the PL intensity in sample 3 drops initially and then improves in both positive and negative electric fields. Moreover, the local minimum of 98% is reached at about −1 MV/cm (recall that the value of 99% appeared at −0.05 MV/cm in sample 2); that is, both the PL quenching and the threshold electric field for enhancement ($V_{\rm th}$) are increased by the block layers. According to our mechanism, when the block layers are inserted, the injections are reduced in both a negative and positive electric field, and the injection-related fluorescence enhancement turns out to be much weaker than before. Assuming that the quenching is independent of the injection, quenching effect suppresses the enhancement more vigorously for sample 2 in a low electric field. So the PL quenching becomes more pronounced. Meanwhile, in order to suppress the quenching, a larger electric field is required to get enough injection. The $V_{\rm th}$ shifts to a larger electric field. The mechanism we proposed can therefore be well verified by this experiment.

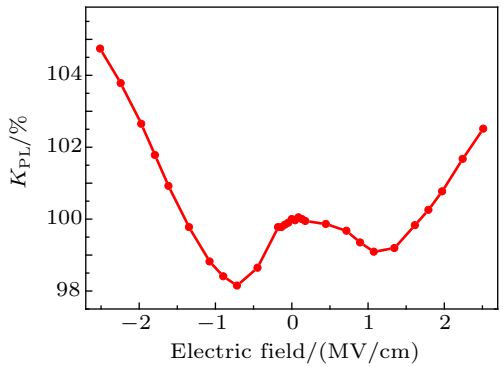


Fig. 5. (color online) The electric field dependence of AlQ's PL intensity in the sample of ITO/Al(5 nm)/Al_2O_3(4.5 nm)/TPD:AlQ(3.0 wt%)(100 nm)/Al_2O_3(4.5 nm)/Al(100 nm).

(iii) Hole mobility

In addition, our mechanism also indicates that good hole mobility of the host can lead to an effective separation of holes and trapped electrons. Then, the energy band bends notably and holes are injected. Thus, if AlQ, a low hole mobility material, is chosen as the host, with electron-trapping material 4-(Dicyanomethylene)-2-methyl-6-(4- dimethylaminostyryl)-4H-pyran (DCM) as the guest, holes cannot be effectively transported in the organic film to get rid of the electrons in DCM. Consequently, the band bending caused by the space charges is not vigorous and the holes are barely injected by the electric field. It should be inferred that there is no PL enhancement by the electric field in sample of AlQ:DCM. Figure 6(a) exhibits the experimental results of DCM's PL intensity as a function of the external electric field in ITO/AlQ:DCM (1.2 wt%)(100 nm)/ Al(100 nm). The PL of DCM is quenched monotonously in both positive and negative electric fields, as predicted by our mechanism. Moreover, when the host is replaced by TPD, the non-monotonic, asymmetrical electric field dependence of PL intensity is recovered, as shown in Fig. 6(b). Therefore, the experimental results above clearly confirm the effect of the hole mobility on PL intensity enhancement by electric field and the mechanism is further verified.

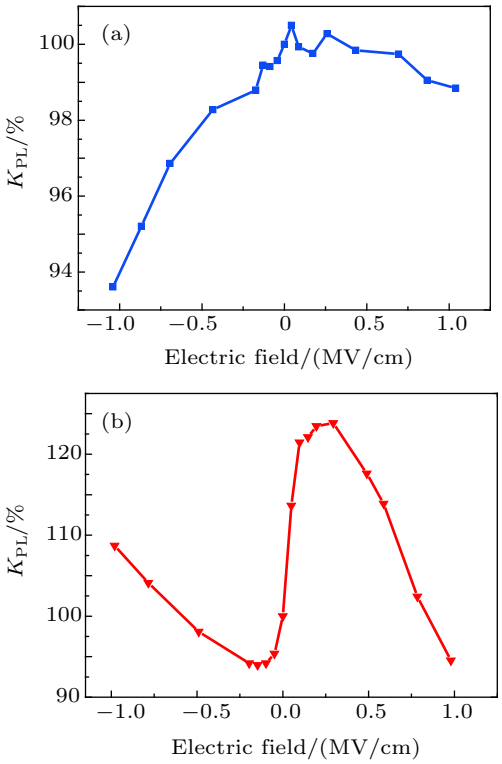


Fig. 6. (color online) The electric field dependence of the DCM's photoluminescence intensity in the sample of (a) ITO/AlQ:DCM(1.2 wt%)(100 nm)/Al(100 nm) and (b) ITO/TPD:DCM(1.2 wt%)(100 nm)/Al(100 nm).

4. Conclusions

An abnormal non-monotonic, asymmetrical electric field dependence of AlQ's PL intensity is observed in the TPD-based structure of ITO/TPD:AlQ/Al. Since this dependence is not symmetrical to the built-in electric field and also does not apply to the pure TPD/AlQ structure, the known mechanisms cannot explain it any more. Consequently, a new possible mechanism is proposed: the dissociation of photoexcited excitons under electric field causes energy band bending in the organic film and improves hole injection from the electrode. Then, the injected holes recombine with electrons in the organic film to generate further fluorescence. While the asymmetrical dependence is attributed to the different hole injection electrode in the cases of a positive or a negative field, the non-monotonicity arises because the competition between PL quenching and enhancement varies with field. Furthermore, experimental results measured from the samples with symmetrical electrodes, block layers, and the host with a low hole mobility are also well explained, and the mechanism presented is explicitly validated.

Acknowledgment

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Appendix A

A1. Electric field dependence of wavelength

In order to show how the wavelength of a doped structure varies with the external electric field, the PL versus wavelength under electric field in the structure of ITO/TPD:AlQ(2.3 wt%)(100 nm)/Al(100 nm) is measured. The electric fields of −1 MV/cm, −0.05 MV/cm, 0 MV/cm, 0.05 MV/cm, 1 MV/cm are adopted, and PL intensity in the y axis is depicted relative to a constant number (shown in Fig. S1). No clear wavelength shift or change of TPD PL intensity is observed, while an obvious change of AlQ's emission is detected.

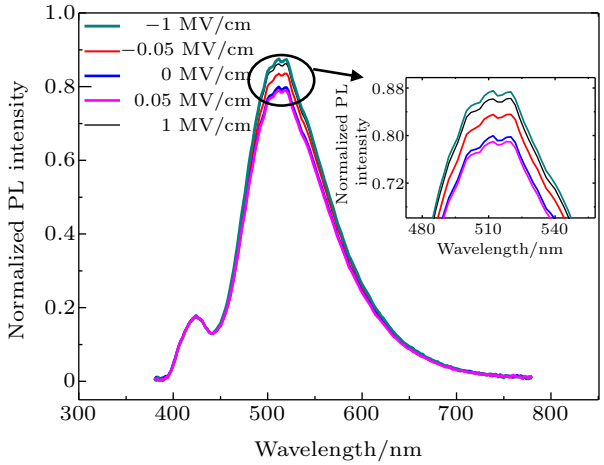


Fig. S1. (color online) PL versus wavelength in the structure of ITO/TPD:AlQ(2.3 wt%)(100 nm)/Al(100 nm) at different external fields.

A2. Influence of the concentration of AlQ

Figure S2 shows how AlQ's PL intensity depends on the electric field with various concentrations of AlQ in the doped structure: ITO/TPD:AlQ (100 nm)/Al(100 nm). When the weight rate of AlQ is between 2% ∼ 5% (i.e. 2 wt %, 3.7 wt%), the curve shows a similar trend. However, when the weight rate is over 10% (i.e. 10.4 wt%), PL quenches at high negative field, which is approximately symmetrical to the positive field. This may be caused by the concentration-induced-over-injection of holes or the concentration quenching effect. Further work is required to detect its variation.

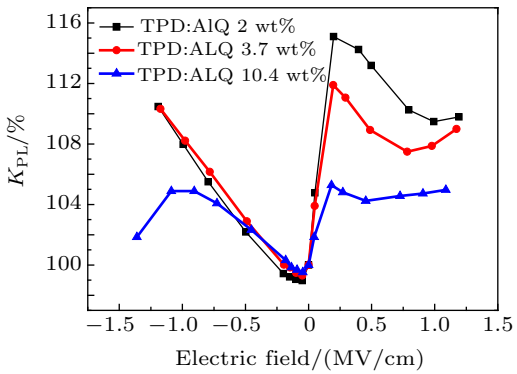


Fig. S2. (color online) The electric field dependence of AlQ's PL intensity in ITO/TPD:AlQ (100 nm)/Al(100 nm) with AlQ's concentration varies from 2 wt% to 11 wt%.

A3. Electric field dependence of TPD's PL intensity

The TPD's photoluminescence intensity dependence on electric field is illustrated in Fig. S3 for sample 1 (ITO/TPD:AlQ (2.3 wt%)(100 nm)/Al(100 nm)). It is indicated that no obvious trend is observed, which may be due to the strong Förster energy transfer and the weak signal to noise ratio (SNR) of this emitting species; however, future verification is required.

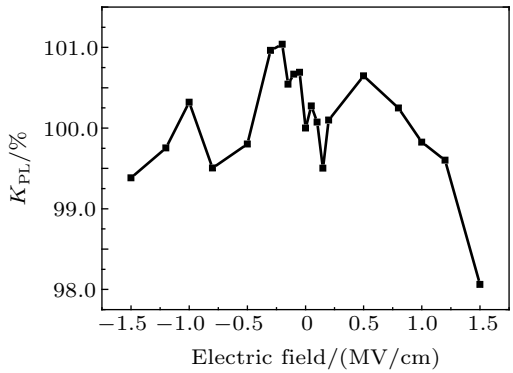


Fig. S3. Electric field dependence of TPD's PL intensity in ITO/TPD:AlQ (2.3 wt%)(100 nm)/Al(100 nm).

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