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Mechanism of Liq's "N-Type Doping" in Alq Based Organic Light-Emitting Diodes

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摘要: Electron-only devices with different structures were prepared by doping Liq(8-hydroxyquinolino-lithium) into the electron transport layer Alq(tris(8-hydroxyquinolino) aluminum). The experimental results show that the electrical properties of doped devices are inferior to those of non-doped devices with Liq/Al composite cathodes and superior to those of non-doped devices with Al only cathodes. This indicates that Liq's doped with Alq does not show any significant "n-doping" effect. The effect exhibits dual roles: Liq molecules dispersed at the Alq/Al cathode interfaces after doping display as electron injection layers, which enhances the device currents by enhancing electron injection; those in the bulk of Alq after doping have a detrimental effect on electron transport due to their own poor conductivity, consequently decrease the device currents. In the tests of electroluminescent devices, the doping of Liq shows a similar behavior. The performance of Liq-doped device is between the non-doped devices with Liq/Al cathode and Al cathode structures, with maximum current efficiencies of 3.96, 4.27 and 2.27 cd/A for the three devices, respectively, and no additional changes caused by charge transfer are observed in the absorption and photoluminescent spectra.

关键词: [organic semiconductor](#) [Liq](#) [Alq](#) [interface modification](#) [charge transfer](#)

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0 Introduction

Since the efficient organic light-emitting diode (OLED) was first reported in 1987 ^[1], OLED has become a research hotspot in flat panel display technology due to its advantages of light weight, high contrast and fast response. Although good progress has been made in improving the performance of OLEDs, low driving voltage and high luminous efficiency OLEDs are still the main research targets ^[2]. However, due to the low intrinsic carrier concentration of organic semiconductors and the mismatched energy level alignment ^[3] between the organic material and the electrode interface, the driving voltage of OLED devices is usually higher than that of inorganic devices. As a dual charge carrier injection device, improving the ability of electron injection and transmission is the key to improve the above problems. This is because the electron mobility in organic semiconductors is usually in the range of $10^{-4} \sim 10^{-6} \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$. The hole mobility is generally in the range of 10^{-2}

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$\sim 10^{-4} \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$. The hole mobility is 2 orders of magnitude higher than the electron mobility, which leads to the imbalance of the number of holes and electrons in the light-emitting layer. The excess holes will pass through the device and enter the cathode directly, which limits the improvement of OLED luminous efficiency. Similar to the doping of inorganic semiconductors, electrical doping in organic semiconductors has also been used as a method to enhance carrier transport and enhance device performance^[4-8].

When n-type doping is performed on the electron transport layer, the n-type dopant is required to have a low work function and a high conduction band or the highest occupied molecular orbital energy level, which makes the n-type dopant more unstable and less eligible. N-type doping is technically difficult to achieve^[9-10]. In earlier studies, alkali metals such as lithium (Li)^[11] and cesium (Cs)^[12] were used for n-type doping in the electron transport layer due to the lower work function meeting the requirements of n-type dopants. However, there are still some problems: on the one hand, alkali metals are chemically active and extremely unstable when oxygen and water are present in the environment, which will lead to difficulties in device manufacturing; on the other hand, alkali metal cations can easily diffuse into the adjacent luminescent layer, causing exciton quenching. Alkali metal compounds are used as effective n-type dopants due to their easy processing and chemical stability, such as lithium fluoride (LiF), lithium carbonate (Li_2CO_3), cesium fluoride (CsF) and cesium azide (CsN_3), etc.^[13-18], but inorganic dopants such as alkali metal compounds have a higher evaporation temperature, which will destroy the organic layer below and cause instability of the device. Therefore, organic n-type dopants with lower evaporation temperature have entered the field of vision of researchers. Among them, the most typical is Liq (8-hydroxyquinolinato-lithium), the evaporation temperature is about 200 °C^[19], and it is often used as an OLED electron injection layer. The study of^[20] indicates that near-ohmic contact can be achieved at the Alq / Liq / Al interface. However, Liq has good thermal stability, and it is still worth looking forward to whether it can give electrons as easily as alkali metals and act as n-type dopants. In addition, the mechanism of Liq doping leading to device performance changes is more worthy of study.

In this paper, electron-only single-carrier devices with different cathode structures (Al, Liq / Al) were designed with different proportions of Alq : Liq as electron transport layers. The experimental results show that the electrical properties of Alq : Liq doped devices are improved when Al cathode structure is used, while the electrical properties of Alq : Liq doped devices are reduced when Liq / Al cathode structure is used. In the electroluminescent (EL) device test, the performance of the Liq-doped device is between that of the Liq / Al and Al cathode non-doped devices, and the maximum current efficiency of the three structural devices is 3.96, 4.27 and 2.27 cd / A, respectively.

1 Experiment

In the experiment, the OLED device was fabricated on an ITO (Indium Tin Oxides) glass substrate with a sheet resistance of about $10 \Omega / \square$, and the light-emitting area of the device was 1 mm × 5 mm. Before using the ITO substrate, it is necessary to use the detergent solution, acetone and deionized water to ultrasonically clean it in turn. After drying with an infrared lamp, it is quickly placed in a vacuum chamber. Under the condition that the vacuum degree is about 6×10^{-4} Pa, all functional

layers are evaporated to the ITO substrate in a specific order by conventional resistance heating. The thickness of the organic layer and the cathode layer is monitored by a quartz crystal oscillator thickness monitor. The Alq : Liq thin film was obtained by co-evaporation in two independent sources, and the doping concentration was controlled by changing the evaporation rate of different evaporation sources. Firstly, a single-carrier device with only electron transport was prepared, and the structure was ITO / Al (2 nm) / Alq : Liq (60 nm, x wt %) / cathode. x = 0, 5, 15, 25, 35, the cathode is Al or Liq (0.5 nm) / Al. Al (2 nm) close to the anode ITO is used to reduce the work function of ITO to prevent holes from being injected from the anode side. Subsequently, an electroluminescent device with the structure of ITO / MoO₃ (0.5 nm) / TPD (50 nm) / Alq (30 nm) / Alq : Liq (25 wt %, 30 nm) / Al was prepared. For comparison, non-doped devices with similar structures were also prepared : ITO / MoO₃ (0.5 nm) / TPD (50 nm) / Alq (60 nm) / cathode (Liq / Al, Al). The chemical structure of the organic materials used in the experiment is shown in Fig.1. Among them, MoO₃, TPD is used as hole injection layer and hole transport layer respectively, Alq is used as electron transport layer and light emitting layer, and Liq is doped into Alq as dopant. The absorption spectra of the films were measured by an ultraviolet-visible-near infrared spectrophotometer Lambda35. The electrical properties of the devices were measured by a Keithley 2400 digital source meter. The optical properties of the devices were measured by a Libero PR650 spectral scanning colorimeter. All measurements were performed at room temperature without any protective treatment.

Fig. 1 Chemical structure of organic materials

2 Results and discussion

When Al and Liq / Al are used as cathode structures, the current density-voltage (J-V) curves of electron-only single-carrier devices with different Liq doping ratios are shown in Figure 2 (a) and (b), respectively. As shown in figure 2 (a), for the device with Al cathode, when the doping concentration is in the range of (0 ~ 25) wt %, the current density at the same voltage increases monotonously with the increase of the doping concentration of Liq in Alq. When the doping concentration exceeds 25 wt %, the current density of the device begins to decrease, indicating that 25 wt % is the optimal doping concentration. For example, at a driving voltage of 8 V, the current densities corresponding to doping concentrations of 0 wt %, 5 wt %, 15 wt %, 25 wt %, and 35 wt % are 0.14, 1.77, 10.3, 39.2, and 21.2 mA / cm², respectively. Compared with undoped devices, the current density of devices with Liq doping layer is increased by 1 ~ 2 orders of magnitude. In single-carrier devices, the increase in current density is due to the increase in the number of electrons. Whether the increase in the number of electrons is due to the introduction of Liq brings additional free electrons or the decrease in the electron injection barrier leads to an increase in electron injection needs further discussion. However, in the case of Liq / Al composite cathode, in the doping range of 0 wt % ~ 35 wt %, the current density of the device is lower than that of the undoped device at the same voltage, and the electrical performance of the device gradually deteriorates with the increase of doping concentration. The above results show that the effect of Liq doping on electron transport in Alq depends on the modification of cathode electron injection. The above concentration of Liq doping has a positive effect on the improvement of the electrical properties of Al-cathode devices, but it has a

negative effect on the performance improvement of Liq / Al-cathode devices. In fact, the purpose of Liq incorporation into Alq is to increase the concentration of carriers or electrons through certain charge transfer (CT) mechanisms, which has been observed and demonstrated in p-type doping systems [21-22]. If the doping of Liq does increase the number of electrons, that is, the concentration of carriers, the electrical properties of the device should be improved after Liq doping, whether the cathode structure is Al or Liq / Al. However, the experimental results give the opposite conclusion. Based on the above experimental facts, it is believed that the n-type doping induced by CT does not occur in the Liq-Alq system.

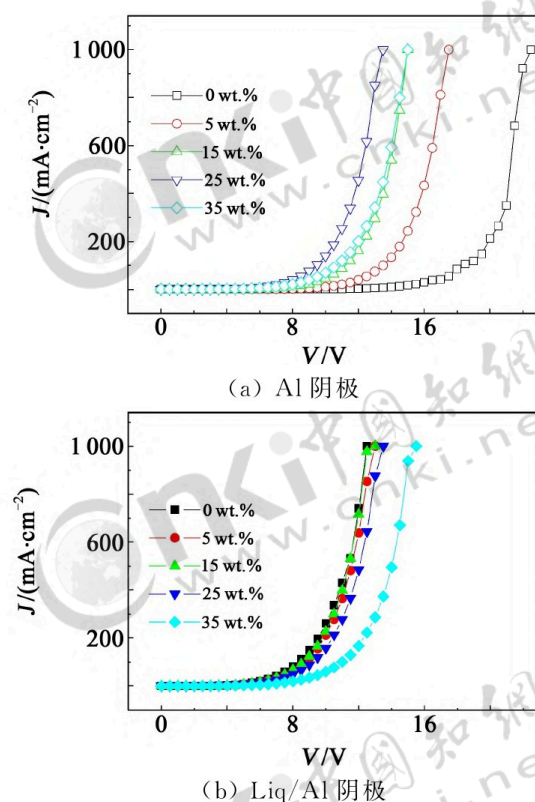


Fig. 2 J-V curves of single-carrier devices with different doping ratios of Alq : Liq under Al and Liq (0.5 nm) / Al cathodes

The study of n-type doping of Liq in Alq shows that there are two reasons for the increase of device current density caused by Liq doping in Alq : one is that Liq decomposes to give electrons [23] ; secondly, compared with Alq, Liq is a more ordered organic semiconductor. After adding Liq into Alq, the disorder degree of the system decreases. Since the carrier mobility increases with the decrease of energy disorder, the doping of Liq makes the carrier mobility of the device increase [24] . In the first argument, Juang et al. [23] also prepared a single-carrier device with only electron transport. In the case of Al cathode, the increase of current density caused by Liq doping Alq at fixed voltage is attributed to the low binding energy of Li and hydroxyquinoline ligand in Liq, and then Li will be released during evaporation, transferring electrons to Alq, thereby increasing the concentration of free electrons and making the E_F of Alq move to the LUMO level. When p-type doping of hole transport materials, such as MoO_3 doping NPB, the structure of single-carrier devices with only hole transport

near the anode is usually ITO / MoO₃ / NPB : MoO₃, and the improvement of device electrical performance is attributed to the formation of CT complex between NPB and dopant MoO₃. In the n-type doping experiment on the electron transport layer, such as Liq, LiF, Na₂CO₃ and other n-type dopants doped with Alq, the structure of the single-carrier device with only electron transport near the cathode is usually Alq : n dopant / Al, and the improvement of the electrical performance of the device is also attributed to the charge transfer between Alq and the so-called ' n-type dopant '. Compared with the p-type doped structure, it is not difficult to find that the n-type doped structure will lose the electron injection layer after doping. However, if the n-type dopant really gives electrons, the process should not be stopped by adding an electron injection layer at the interface between the electron transport layer and the Al cathode. On the contrary, by adding an electron injection layer to modify the cathode, the electron injection barrier will be reduced, so that the electrical performance of the device is further improved on the basis of the doped layer. However, no such design has been reported in the literature. Combined with the data in Figure 2 (b), Liq does not really give electrons. In the second argument, Liq doping leads to a decrease in the disorder of the Alq film, which leads to an increase in carrier mobility. There is also no selectivity to the presence or absence of the electron injection layer, but this is not the case in the experimental results. In Fig.2, the improvement of the electrical properties observed after doping Liq in Alq is actually due to the interface effect : Liq near the Al cathode forms a special interface structure, thereby improving the electron injection of the cathode. Therefore, for Al cathode devices, Liq close to the cathode interface acts as an electron injection layer, which reduces the electron injection barrier and improves the device performance. For Liq / Al cathode devices, the cathode electron injection has been well modified, and the electron injection barrier is very low. The conductivity of Liq doped into the bulk Alq is worse than that of Alq [25], thus reducing the device performance. This is confirmed by the experimental results shown in Fig.2 (b). In Fig.2 (b), when the Liq doping concentration is 15 wt % and 5 wt %, the current density of the device is close to that of the non-doped but Liq / Al cathode device. This is because Liq is an organic semiconductor material, and the LUMO energy level (3.1 eV) [26] is close to the LUMO (3.0 eV) [27] energy level of Alq, and both are quinoline organic compounds. Therefore, Liq doped into Alq films will not have a significant effect on electron transport at low concentrations. And when Liq is used as the electron injection layer, the device performance shows a low sensitivity to the thickness of the injection layer [20], so the device will show obvious performance degradation at higher Liq doping concentration.

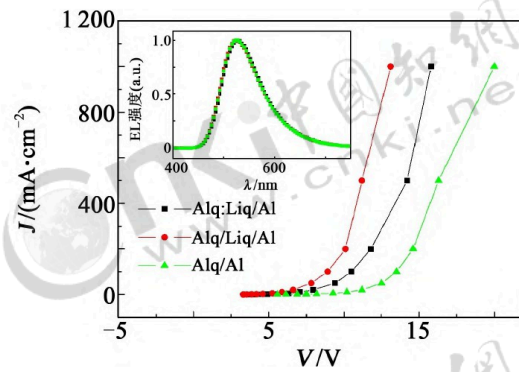
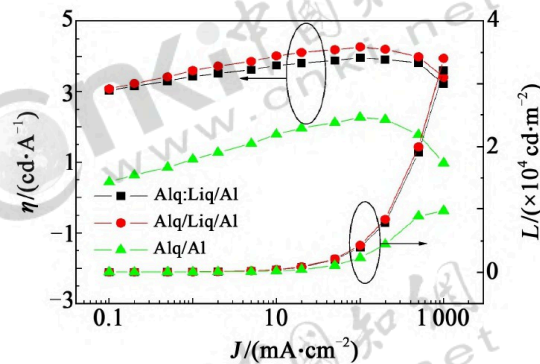
Combined with the data in Figure 2 (a) and (b), Figure 3 is obtained. It can be clearly seen that when Liq is doped in Alq, at the same voltage, the current density of doped devices with different cathode structures (Al, Liq / Al) is between the undoped Al cathode device and the Liq / Al composite cathode device. The hollow and solid symbols in the figure correspond to the devices with Al cathode and Liq / Al cathode, respectively. When the cathode structure is Al, the current density of the 35 wt % doped device begins to decrease compared to the current density of the 25 wt % doped device at the same voltage, but it is still much better than the undoped device. Although the conductivity of Liq is not as good as that of Alq, when there is no electron injection layer on the cathode, Liq doping will still lead to the improvement of device performance, which is enough to illustrate the importance of cathode interface modification for electron injection of OLED devices.

Fig.3 J-V curve of single carrier device

Fig. 4 Absorption and Photoluminescence Spectra of Alq : Liq Films with Different Ratios

In the NPB : MoO₃ doping system of the hole transport material, a very good p-type doping effect can be observed. The p-type doping effect originates from the CT process and is accompanied by an additional absorption peak corresponding to the CT process. In order to determine whether this process also occurs in the Alq : Liq doped system, the absorption test of Liq doped and undoped Alq films was carried out, and the photoluminescence (PL) test was also carried out. The results are shown in Figure 4. From Figure 4 (a), it can be found that the absorption spectrum of the pure Alq film reaches a peak^[28] at about 390 nm, while the absorption spectrum of the doped Liq film slightly shifts to the short-wave region. With the increase of Liq doping concentration, the blue shift of the absorption spectrum becomes more obvious. However, no additional absorption peak was observed in the Alq : Liq system, such as in the NPB : MoO₃ system. Combined with the results of figure 2, this means that the possibility of CT process is very small. In fact, the blue shift can be attributed to the solid-state solvation effect^[29] caused by Liq separating Alq molecules to some extent. In the PL spectra of Alq : Liq samples with different proportions, the spectra are almost unchanged, as shown in figure 4 (b). No direct evidence of CT process can be obtained from these experimental results.

Finally, the photoelectric properties of the EL structure devices in the experimental part are compared. The devices are recorded as Alq : Liq / Al and Alq / Liq / Al and Alq / Al, respectively. The EL spectra (@ 20 mA / cm²), J-V characteristics and current efficiency-luminance-current density (η -L-J) characteristics of the device are shown in Figure 5 (a) and (b), respectively. As can be seen from the inset of Fig.5 (a), the EL spectra of the three devices are almost identical, with the emission peak at about 520 nm, which is a typical Alq emission^[30]. In the J-V curve, the performance of the EL device after Liq doping also depends on the electron injection modification on the cathode. It can be seen from Fig.5 (a) that at a fixed voltage, the current density of the device with Alq : Liq structure is significantly higher than that of the device with Alq / Al structure, but lower than that of the device with Alq / Liq / Al structure, which is basically consistent with the effect of Liq doping in single-carrier devices. This is because in the EL device, the number of holes is more than that of electrons, resulting in an imbalance in the ratio of the two. The performance of the device still depends on the minority carrier, that is, the injection and transmission of electrons. In addition, the current efficiency and brightness of the devices under the three structures also show similar trends. As shown in Figure 5 (b), at a current density of 100 mA / cm², the maximum current efficiency (and corresponding brightness) of the three devices are 3.96 (3 959 cd / m²), 4.27 (4 267 cd / m²) and 2.27 cd / A (2 271 cd / m²), respectively. The device with Alq / Liq / Al structure has the maximum current efficiency of 4.27 cd / A. This indicates that the incorporation of Liq into Alq will not have a positive impact on device performance.

(a) J - V (插图为 EL 光谱 (@ 20 mA/cm²))

(b) 电流效率-亮度-电流密度特性曲线

Fig. 5 J-V characteristics and current efficiency-brightness-current density characteristic curve

In the test results of single-carrier and EL devices which only transport electrons, it can be found that the introduction of Liq into Alq does not form n-type doping, and Liq dispersed near the Alq / Al cathode interface after doping acts as an electron injection layer. The specific role of Liq as the electron injection layer is similar to [20,31] reported in the previous literature. We also believe that it is the formation of the dipole layer, that is, because Liq is a highly polar molecule, the Liq present on the Alq / Al cathode interface will produce a certain degree of polar ordered arrangement, so that a dipole layer is formed between Alq and Al, and the effective barrier of electron injection from Al to Alq is reduced.

3 Conclusion

In this paper, it is found that the doping of Liq in Alq cannot cause the 'n-type doping' effect. The Liq near the Al cathode will act as an electron injection layer. By forming a special interface structure, the electron injection barrier is reduced and the device current is increased. Although the chemical properties of Liq and Alq dispersed in the Alq bulk phase are similar, the conductivity of Liq is inferior to that of Alq, which reduces the device current at high concentration. In the conventional double-layer EL devices, the maximum current efficiency (3.96 cd / A) of the Liq-doped device is higher than that (2.27 cd / A) of the non-doped device with Al cathode, but lower than that (4.27 cd / A) of the non-doped device with Liq / Al cathode. These findings provide a different perspective for

understanding the improvement of electron injection in organic devices by using doped or thin-layer Li_q.

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