

Role of Fe₃O₄ as a *p*-Dopant in Improving the Hole Injection and Transport of Organic Light-Emitting Devices

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Abstract—Fe₃O₄ has been demonstrated to be an efficient *p*-dopant in improving the performance of organic light-emitting devices. This paper investigates in detail the role of Fe₃O₄ in improving the hole injection and the hole transport by the ultraviolet/visible/near-infrared absorption, x-ray, and ultraviolet photoelectron spectroscopy. The results demonstrated that Fe₃O₄ as a *p*-dopant has different effectiveness when it is doped into different host materials. The improved properties of the OLEDs with the *p*-doped N, N'-diphenyl-N,N'-bis (1,1'-biphenyl)-4,4'-diamine layer is mainly due to the enhanced hole injection through the lowering of the hole injection barrier, while the enhanced hole transport plays a more important role for the OLEDs with the *p*-doped 4,4',4''-tris (3-methylphenylphenylamino) triphenylamine due to their higher ability in the formation of charge transfer complex.

Index Terms—Fe₃O₄, organic light-emitting devices, *p*-dopant.

I. INTRODUCTION

ORGANIC semiconductors are generally in an amorphous state when used as a carrier transport layer through thermal evaporation in organic light-emitting devices (OLEDs). However, the hopping transport in disordered organic thin films leads to low charge mobility in organic semiconductors, which is an obstacle in further improvements of driving voltages and power efficiency [1]. To increase the conductivity of organic semiconductors, doping concepts have been adopted from their inorganic counterparts. The basic concept of doping in organic semiconductor is to add a strong electron-donor or electron-acceptor, which either transfers an electron to the lowest unoccupied molecular orbital (LUMO) of a host molecule to produce a free electron (*n*-type doping) or removes an electron from the highest occupied molecular orbital (HOMO)

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of a host molecule to generate a free hole (*p*-type doping) [2], [3]. The charge transport of the doped transport layers could be enhanced by a few orders through the generation of the free charges. In addition, the doped layers also exhibit their role in enhancing charge injection by the surface dipole formation [4], [5]. Both the dopant and the host material in the doped layer have an impact on the charge transport and the injection of the devices. Although various kinds of dopants have been reported and some of them have been compared by doping into same host material [6], there are very few reports on the relative effectiveness of the host molecules on the free charge generation and surface dipole formation. It is very important to understand this mechanism to select suitable hosts for the dopants and optimize the structure of the doped OLEDs.

Recently, Fe₃O₄ has been reported as an efficient *p*-dopant in OLEDs [7]. This paper studied the role of Fe₃O₄ as a *p*-dopant in different hosts in improving the hole transport and the injection. 4,4',4''-Tris (3-methylphenylphenylamino) triphenylamine (m-MTDATA) and N,N'-diphenyl-N,N'-bis (1,1'-biphenyl)-4,4'-diamine (NPB) were selected as the hosts of Fe₃O₄, which are widely used as hole injection and transport materials. The electroluminescent (EL) properties of the devices with either *p*-doped m-MTDATA or *p*-doped NPB are greatly improved, exhibiting lower driving voltage and higher power efficiency when compared with undoped devices, which can be attributed to both enhanced hole transport and improved hole injection. However, the *p*-doped m-MTDATA and NPB have different effectiveness in improving hole transport and injection, which has been clarified by the investigation of ultraviolet/visible/near-infrared (NIR) absorption, x-ray, and ultraviolet photoelectron spectroscopy (XPS, UPS).

II. EXPERIMENTAL

The structures of tris-(8-hydroxyquinoline) aluminum (Alq₃)-based *p*-doped OLEDs with different hosts were as follows: ITO/Fe₃O₄:m-MTDATA (1:2 by volume, 25 nm)/m-MTDATA (10 nm)/NPB(5 nm)/Alq₃ (50 nm)/LiF (1 nm)/Al (100 nm) and ITO/Fe₃O₄:NPB (1:2 by volume, 25 nm)/NPB(15 nm)/Alq₃ (50 nm)/LiF (1 nm)/Al (100 nm). A ratio of 1:2 was found to be the optimum doping ratio demonstrated in a previous work [7]. For comparison, the undoped devices were fabricated as controls: ITO/m-MTDATA (35 nm)/NPB (5 nm)/Alq₃ (50 nm)/LiF (1 nm)/Al

(100 nm) and ITO/NPB (40 nm)/Alq₃ (50 nm)/LiF (1 nm)/Al (100 nm). Here, both the Fe₃O₄ and the organic layers were sequentially evaporated by vacuum deposition onto an ITO-coated glass substrate. The thermally evaporated Fe₃O₄ under high vacuum from the Fe₃O₄ powder has been confirmed stoichiometrically consist of Fe₃O₄ by XPS measurement, and the oxygen vacancy is negligible (data not shown here). This is somehow unexpected since vacuum deposited oxide materials such as MoO_x and V₂O_x tend to contain large density of oxygen vacancy. However, there are exceptions similar to the current case, for example, thermally evaporated WO₃ [8]. The active area of the devices was 2 × 2 mm². The current density–voltage–luminance (J-V-L) characteristics of the devices were measured by a Keithley 2400 programmable voltage–current source and a Photo Research PR-655 spectrophotometer. The UPS data were measured with a Thermo ESCALAB 250, and during measurement, the samples were biased at −4.0 V to observe the low-energy secondary cutoff. The XPS measurements were performed with Mg Ka x-ray source (1253.6 eV, Specs XR50). The absorption spectra were measured by means of an ultraviolet/visible/NIR absorption spectrometer (UV 3600, Shimadzu).

III. RESULTS AND DISCUSSION

A. Effects of Fe₃O₄ Doped m-MTDATA and NPB on EL Performance of OLEDs

The J-V-L characteristics of the doped and the undoped devices are shown in Fig. 1. Both the current density and the luminance of the devices with Fe₃O₄-doped layer strikingly increased when compared with the undoped devices. For example, the operating voltages required for 100 mA/cm² for the devices with doped m-MTDATA and doped NPB were 5.4 and 6.7 V, respectively, and 7.2 and 10.5 V for the devices with the undoped m-MTDATA and NPB, respectively. The turn-on voltage obtained the luminance of 1 cd/m² was found to be greatly decreased from 3 to 2.4 V and 5 to 2.5 V, respectively, for the devices with doped m-MTDATA and NPB. A high luminance was achieved at low voltages due to the doping of Fe₃O₄, which resulted in an improved power efficiency, as shown in Fig. 2. The power efficiencies at a current density of 20 mA/cm² for devices with the doped m-MTDATA and NPB were 2.6 and 2.0 lm/W, respectively, when compared with 1.5 and 1.2 lm/W of the undoped devices.

MoO₃ has been demonstrated as one of the effective p-dopants to generate the free charges [9]. To study the relative p-doping effectiveness of Fe₃O₄, the authors fabricated the devices with MoO₃-doped m-MTDATA and NPB with an optimized doping ratio for comparison. The structures are ITO/MoO₃:m-MTDATA (1:3 by volume, 25 nm)/m-MTDATA (10 nm)/NPB (5 nm)/Alq₃ (50 nm)/LiF (1 nm)/Al (100 nm) and ITO/MoO₃:NPB (1:3 by volume, 25 nm)/NPB (15 nm)/Alq₃ (50 nm)/LiF (1 nm)/Al (100 nm), and their J-V-L characteristics are compared in Fig. 1. It can be observed that both the current density and the luminance of the Fe₃O₄-doped devices are higher than those of the MoO₃-doped devices for OLEDs with m-MTDATA as the host of the p-dopant, while

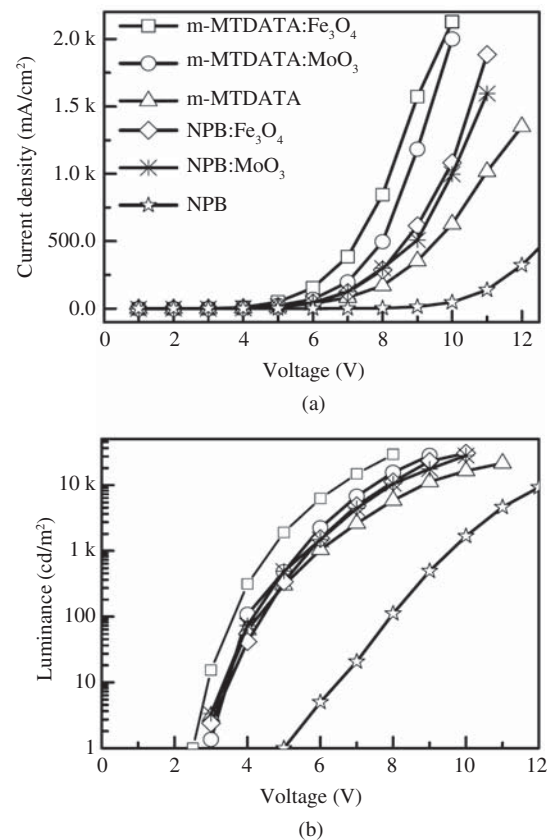


Fig. 1. (a) Current density–voltage and (b) luminance–voltage characteristics of the devices with Fe₃O₄ doped m-MTDATA (1:2), Fe₃O₄ doped NPB (1:2), MoO₃ doped m-MTDATA (1:3), MoO₃ doped NPB (1:3), undoped m-MTDATA and NPB.

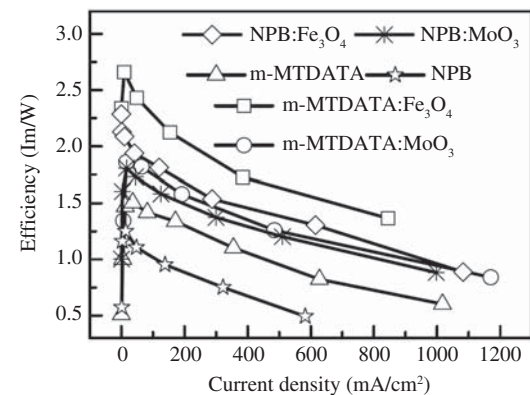


Fig. 2. Power efficiency–current density of the devices with Fe₃O₄ doped m-MTDATA (1:2), Fe₃O₄ doped NPB (1:2), MoO₃ doped m-MTDATA (1:3), MoO₃ doped NPB (1:3), undoped m-MTDATA and NPB.

they are comparatively lower when the NPB is employed as the host. Fig. 2 shows that the power efficiency for both the Fe₃O₄-doped devices is higher than that of the MoO₃-doped devices. Therefore, Fe₃O₄ is a more effective p-dopant than MoO₃ in improving the EL performance of the OLEDs.

B. Effects of Fe₃O₄ as p-Dopant on Hole Transport and Injection of the Doped m-MTDATA and NPB Layers

The doping effect could be confirmed by the emergence of the additional absorption peak in NIR absorption spectroscopy

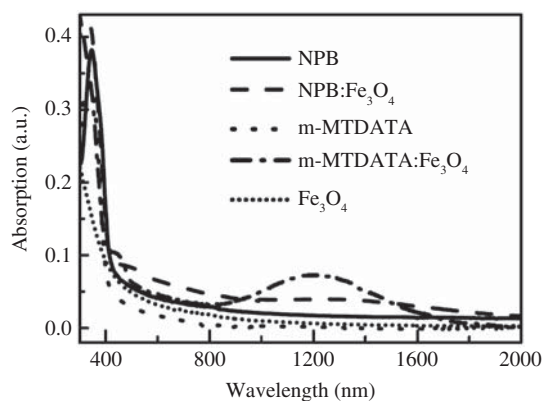


Fig. 3. Ultraviolet/visible/NIR absorption spectra of the neat films of Fe₃O₄ (50 nm), NPB (50 nm) and m-MTDATA (50 nm), the Fe₃O₄ doped m-MTDATA film (50 nm) and Fe₃O₄ doped NPB film (50 nm) deposited on quartz substrate.

due to the induced formation of the charge transfer (CT) complex between the dopant and the host materials [6], [10]–[12]. The absorption spectra of the Fe₃O₄-doped m-MTDATA (1:2, 50 nm), Fe₃O₄ doped NPB (1:2, 50 nm), and the neat m-MTDATA (50 nm), NPB (50 nm) and Fe₃O₄ (50 nm) films deposited on quartz substrates are shown in Fig. 3. The pronounced CT band can be detected by the appearance of the additional absorption peaks at around 440 nm and 1200 nm in the Fe₃O₄ doped m-MTDATA film while the strong absorptions of the neat m-MTDATA and Fe₃O₄ are all located at the wavelength less than 400 nm, which indicates an efficient electron transfer from m-MTDATA to Fe₃O₄ to form the CT complex of Fe₃O₄⁻/m-MTDATA⁺. In case of the Fe₃O₄ doped NPB film, the additional absorption peak (when compared with the neat NPB and Fe₃O₄ films) of about 500 nm and the broad absorption band expanding between 1000 nm and 1600 nm are lower than those of the Fe₃O₄-doped m-MTDATA. These phenomena indicate that the host materials of m-MTDATA and NPB have different effectiveness in the formation of the CT complex. The Fe₃O₄-doped m-MTDATA shows a much higher ability in the CT formation, namely, higher ability of the free hole generation. This easier CT formation can be further confirmed by the energy diagram. The m-MTDATA has a lower HOMO level (5.0 eV) than that of NPB (5.3 eV), and the HOMO level of m-MTDATA is lying below that of Fe₃O₄ (5.2 eV), which results in an easier electron transfer from m-MTDATA to Fe₃O₄ [10]. The higher ability of the free hole generation in the doped m-MTDATA indicates that the hole-transport improvement in the p-doped m-MTDATA layer is higher than that of the p-doped NPB layer. However, the EL properties of the OLEDs with p-doped NPB exhibit larger enhancement than that of its control devices when compared with the OLEDs with doped m-MTDATA, as shown in Fig. 1. There must be some other effects that contribute to the improvements of the EL performance for the OLEDs with p-doped NPB, besides hole transport.

It has been demonstrated that the p-doped transport layers not only enhance the hole transport, but also exhibit their role in enhancing hole injection by surface dipole formation at the anode/p-doped HTL interface due to the appearance of the

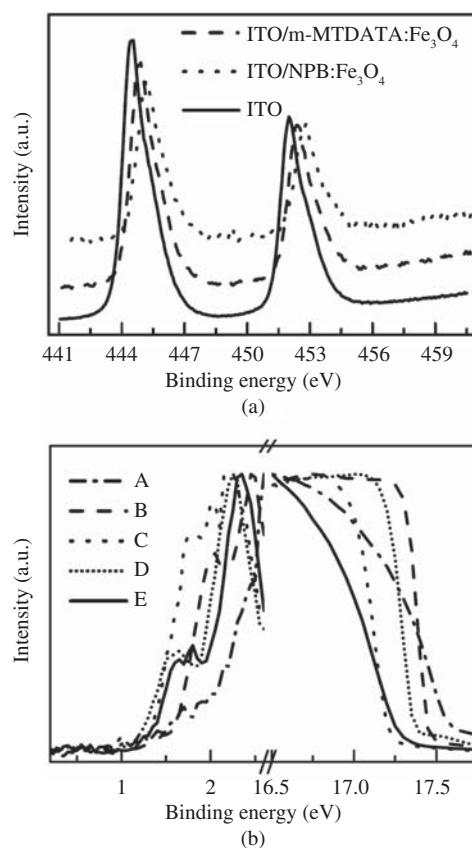


Fig. 4. (a) XPS In 3d core-level spectrum of the ITO, NPB:Fe₃O₄ (2 nm), m-MTDATA:Fe₃O₄ (2 nm) on ITO. (b) UPS spectra of ITO (A), ITO/NPB (15 nm) (B), ITO/NPB:Fe₃O₄ (25 nm)/NPB (15 nm) (C), ITO/m-MTDATA (15 nm) (D), ITO/m-MTDATA:Fe₃O₄(25 nm)/m-MTDATA (15 nm) (E).

p-dopant at the interface. The XPS was performed to analyze the interface of the ITO and the p-doped layer. The XPS spectra of the Indium (In) 3d peaks were measured from the bare ITO, ITO/m-MTDATA: Fe₃O₄ (2 nm) and ITO/NPB: Fe₃O₄ (2 nm), as shown in Fig. 4(a). It can be seen that the In 3d peaks shift toward higher binding energy by approximately 0.4 and 0.7 eV, respectively, after depositing p-doped m-MTDATA and NPB, which implies charge transfer from ITO to the p-doped layers at the interface [4], [5], [13], [14]. As a result, the dipole layer is formed at the interface, which would lead to an abrupt shift of the potential across the dipole layer, and therefore an up-shift in the vacuum level. The up-shift of the vacuum level for the p-doped NPB layer on ITO is greater than that of the p-doped m-MTDATA layer from the XPS data, resulting in an easier hole injection from ITO to the p-doped NPB. Probably, this is the main origin for the EL enhancement of the OLEDs with the p-doped NPB.

To further clarify the effects of the Fe₃O₄-doped layers on charge injection, the interfacial energy level was determined by employing the UPS measurement [15]–[20]. Figure 4(b) shows the UPS spectra of the m-MTDATA, NPB, m-MTDATA: Fe₃O₄/m-MTDATA, and NPB: Fe₃O₄/NPB films deposited on the ITO as well as the bare ITO. A neat layer was deposited on the p-doped NPB and m-MTDATA layer, so that the structure of the samples for the UPS measurement coincide with that of the p-doped OLED, and the extracted energy diagram is

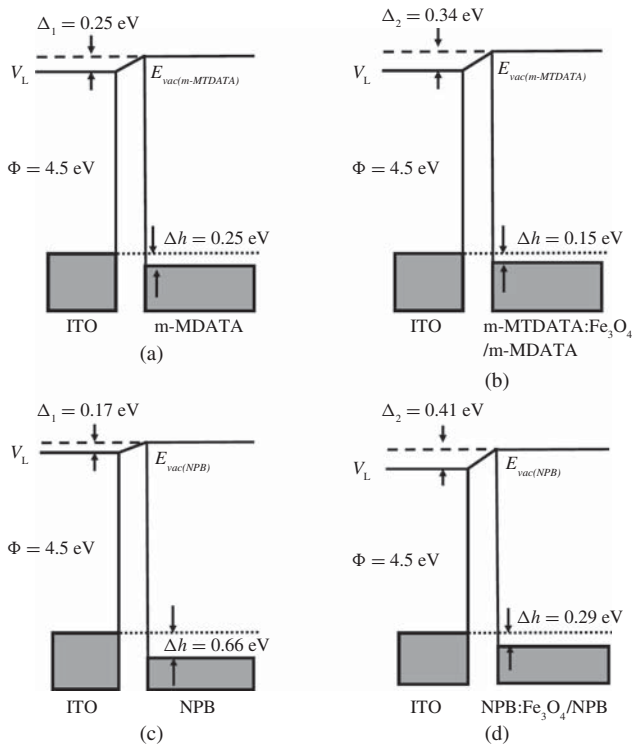


Fig. 5. Energy diagram of (a) ITO/m-MTDATA, (b) ITO/m-MTDATA:Fe₃O₄/m-MTDATA, (c) ITO/NPB, and (d) ITO/NPB:Fe₃O₄/NPB.

applicable for the interface in the OLEDs. There are clear spectral changes after the deposition of the p-doped m-MTDATA and NPB when compared with those of neat films, and both the HOMO onset position and the cutoff position shift toward the lower binding energy, respectively. Noticeably, the shift of the secondary electron cutoff position is due to the formation of the interface dipoles (Δ) at the interface of ITO/m-MTDATA:Fe₃O₄ and ITO/NPB:Fe₃O₄, which reaches 0.34 and 0.41 eV, respectively, while it is 0.25 eV and 0.17 eV for the ITO/m-MTDATA and ITO/NPB interfaces, respectively. The difference in the interface dipoles between ITO/NPB and ITO/NPB:Fe₃O₄/NPB is greater than that between ITO/m-MTDATA and ITO/m-MTDATA:Fe₃O₄/m-MTDATA, which is coincident with the XPS results. We should note that the amounts of the XPS peak shifts do not agree with those of vacuum level shifts extracted from the UPS spectra, which is due to the different structure of the samples between the XPS and UPS measurements. The energy diagrams of the ITO/m-MTDATA, ITO/m-MTDATA:Fe₃O₄/m-MTDATA, ITO/NPB, and ITO/NPB:Fe₃O₄/NPB extracted from the UPS spectra are shown in Fig. 5. The work function of ITO is calculated to be 4.5 eV from the UPS spectra. The shift of the HOMO level by introducing the Fe₃O₄-doped layer will result in a reduced hole-injection barrier, which will greatly enhance the hole-injection efficiency and decrease the turn-on voltage. The hole-injection barrier from ITO to NPB is reduced by 0.37 eV from 0.66 to 0.29 eV, after inserting the p-doped NPB. However, in case of the p-doped m-MTDATA, the injection barrier is reduced by 0.1 eV from 0.25 to 0.15 eV. Obviously, the p-doped NPB is more effective in the

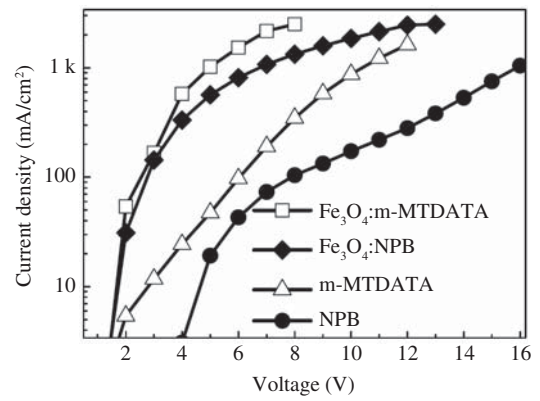


Fig. 6. Current density-voltage characteristics of the hole-only devices with Fe₃O₄ doped m-MTDATA, Fe₃O₄ doped NPB, undoped m-MTDATA and NPB.

reduction of the hole-injection barrier when Fe₃O₄ appears at the ITO/NPB interface. Therefore, hole injection from ITO to the p-doped NPB is much more efficient than that from ITO to the p-doped m-MTDATA, which contributes to the higher EL improvements of the OLEDs with the p-doped NPB.

To further examine the hole injection and the transport characteristics of the Fe₃O₄-doped NPB and m-MTDATA layers, the authors fabricated the hole-only devices [21]–[24] with the following structure: ITO/m-MTDATA:Fe₃O₄ (2:1, 25 nm)/m-MTDATA (10 nm)/NPB (5 nm)/Al (100 nm), ITO/NPB:Fe₃O₄ (2:1, 25 nm)/NPB (15 nm)/Al (100 nm). The control devices of ITO/m-MTDATA (35 nm)/NPB (5 nm)/Al (100 nm), and ITO/NPB (40 nm)/Al (100 nm) were fabricated for comparison. Both the devices with the p-doped layers exhibited a much-improved current density, when compared with that of their control devices, as shown in Fig. 6. However, their effectiveness in improving the current density was different, as concluded from the aforementioned NIR absorption, XPS, and UPS results. The Fe₃O₄-doped m-MTDATA layer has a higher ability in the CT formation, which results in much improved hole transport, while the Fe₃O₄-doped NPB layer has a higher ability in the surface dipole formation, which results in the much-enhanced hole injection.

IV. CONCLUSION

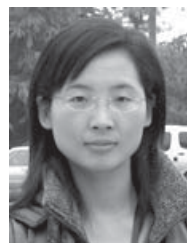
The authors have demonstrated that the OLEDs with Fe₃O₄ doped in two different hosts of m-MTDATA and NPB remarkably enhance the luminance, the current density, and the power efficiency compared to that of the undoped devices. The effectiveness of Fe₃O₄ in the two different hosts has been examined in detail by ultraviolet/visible/NIR absorption, XPS and UPS spectra, and exhibits different roles in improving the device performance. The Fe₃O₄-doped m-MTDATA layer in the OLEDs is more efficient in improving the hole transport, while the Fe₃O₄-doped NPB layer is more efficient in lowering the hole-injection barrier. The results clarify the underlying mechanism of Fe₃O₄ as a p-dopant in different hosts, which could be helpful in optimizing the doped OLEDs through selection of the favorite hosts for the p-dopants.

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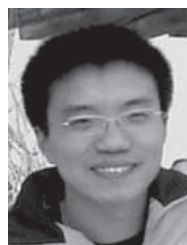


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