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ARTICLE

## Ultra-Thin Gold Nanoparticles Layer Modified Cathode for the Enhanced Performance of Polymer Light Emitting Diodes

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The enhanced performance of the polymer light emitting diodes has been achieved by embedding ultra-thin gold nanoparticles layer on the cathode. The performance of devices could be optimized simply by adjusting the thickness of the gold, which exhibited the improved brightness from 17k cd·m<sup>-2</sup> to 20k cd·m<sup>-2</sup> and the enhanced luminous efficiency from 15.4 cd·A<sup>-1</sup> to 18.3 cd·A<sup>-1</sup>, when greenish emissive polymer P-PPV was applied as emissive layer. The experimental results show that it is mainly attributed to the decreased holes and the increased electrons, resulting in the balanced electron-hole recombination and the shifted light emitting profile to the anode. The results demonstrate that gold nanoparticles constitute a feasible and effective route for achieving high-performance polymer optoelectronic devices through its electric properties.

### Introduction

Organic light-emitting diodes (OLEDs) have attracted much attention for illumination and display applications due to the advantages of low-cost fabrication, lightweight, and flexibility.<sup>1-4</sup> However, electrode quenching is one of the main problems in single carrier transport materials based OLEDs, since most conjugated electroluminescence (EL) materials are *p*-type semiconductors, in which the hole-mobility is usually much higher than the electron-mobility. The unbalanced carrier transportation creates the excess of holes and the formation of excitons take place near the cathode.<sup>5-8</sup> Significant efforts have been made to improve the efficiency of optoelectronic devices using a variety of processing strategies for transportation balance. The cathode modification is effective for the improvement of device performance, including cesium carbonate (Cs<sub>2</sub>CO<sub>3</sub>),<sup>9</sup> metal fluoride,<sup>10</sup> and self-assembled dipole molecules<sup>11</sup> to improve the electrons injection.

Recently metal nanostructures are widely employed in the optoelectronic devices for performance improvement, due to their optical and electrical effects such as metal enhanced fluorescence,<sup>12-18</sup> energy transfer,<sup>19-20</sup> interface effect,<sup>21-22</sup> and so on.<sup>23-24</sup> The modification of the electrode and transporting layer by the metal nanostructures is regarded as one of the

most important and effective methods.<sup>12-17,21,22</sup> The noble metals such as gold (Au), silver (Ag), are rarely chosen for the cathode modification, because of their high work functions. Yang et al, have reported that the Ag cluster at cathode can be used for plasmonic effect and electron injection in OLEDs.<sup>25</sup> Our group also reported Au nanoparticles (NPs) modified ITO cathode used in the inverted polymer light emitting diodes (iPLEDs) for the plasmonic effect induced electrode quenching control.<sup>22</sup> In this work we have used an evaporated ultra-thin gold nanoparticles layer to modify CsF/Al cathode in a conventional device structure and achieved obvious enhancement of brightness and luminous efficiency (LE). The device physics analysis showed that the origin of enhanced performance in a conventional device is different from the inverted devices in previous literatures we reported before.

### Experimental section

**Materials:** Emissive layer polymer material poly(2-(4-(3',7'-dimethyloctyloxyphenyl)-1,4-phenylene-vinylene) (P-PPV) is purchased from Canton OLEDKING Optoelectric Materials Co. Ltd., Guangzhou, China. ITO glass substrates (size 15×15 mm ITO) are purchased from China Southern Glass Holding Corp. The thermally evaporated Au, Ag and Al are purchased from ZhongNuo Advanced Material Technology Co., Limited. Poly[(9,9-bis(3'-(N,N-dimethylamino)propyl)-2,7-fluorene)-alt-2,7-(9,9-dioctylfluorene)] (PFN) is synthesized in our laboratory.<sup>1,5</sup> N-doped silicon is purchased from HeFei KeJing Material Technology Co., Limited.

#### Device fabrication and characterization

**PLEDs fabrication:** The buffer layer PEDOT:PSS (Clevios P AI4083, 35 nm) was spin-coated onto ITO substrates, and then

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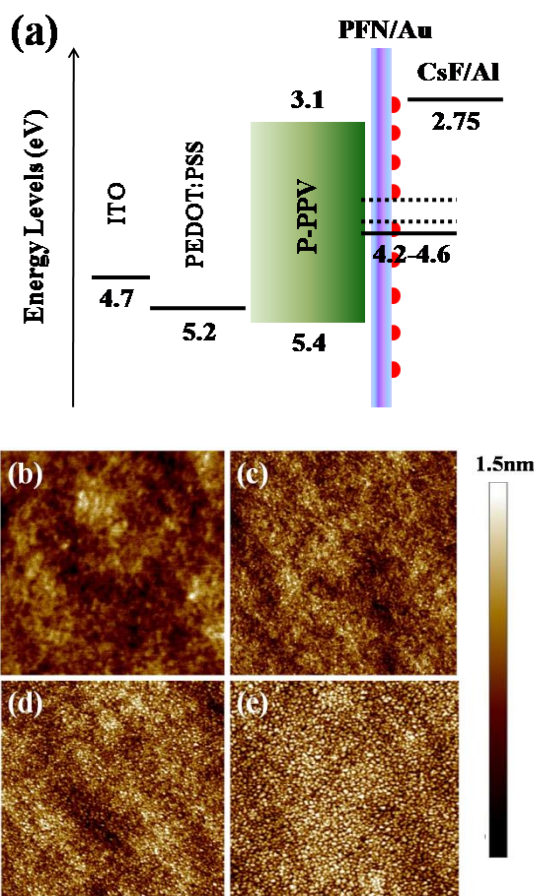
subjected to thermal annealing at 120 °C for 20 min in air. The light emitting layer, P-PPV was prepared by spin-coating method (1400 r/min from 6 mg/mL p-xylene solution as active layer (80 nm)). PFN was spin-coated as 5 nm. Au was evaporated by different thickness 0.5 nm, 1 nm, 2 nm. Finally, a 1.5 nm CsF film as an electron injection layer and a 120 nm Al cathode layer were thermally evaporated with a base pressure of  $3 \times 10^{-4}$  Pa. Overlap between the cathode and anode defined the  $16.5 \text{ mm}^2$  pixel area. Except deposition of the PEDOT:PSS layer from aqueous suspension, all other processes were carried out in the controlled atmosphere of a nitrogen dry-box (Vacuum Atmosphere Co.) containing less than 10 ppm oxygen and moisture.

**Organic field-effect transistors (OFETs) fabrication:** OFETs were fabricated in a top contact geometry using Ag as the source and drain electrode. Highly *n*-doped silicon and thermally grown silicon dioxide were used as the back gate and gate dielectric, respectively. An additional polymer dielectric, 1.2 wt% polymethyl methacrylate (PMMA) ( $M_w \sim 350,000$ , Aldrich) in butyl-acetate was spin cast 30 s on top of the  $\text{SiO}_2$ . Coated substrates were baked under 220 °C for at least 30 min in order to crosslink the PMMA.<sup>26</sup> The Poly[[4,8-bis[(2-ethylhexyl)oxy]benzo[1,2-b:4,5-b']dithiophene-2,6-diyl][2-[[2-ethylhexyl)oxy]carbonyl]-3-fluorothieno[3,4-b]thiophenediyl]] (PTB7) (1-Material) film (80 nm) was spin-coated on the cross-linked PMMA treated substrates from a dichlorobenzene solution, 1.5%, 700 r/min. Then silver electrode (60 nm) was deposited under vacuum as the source and drain electrodes. The OFETs device has width to length ratio  $W/L=60/1$ .

Current density–brightness–voltage ( $I$ – $B$ – $V$ ) characteristics were measured in the nitrogen dry-box using a Keithley 236 source measurement unit and a calibrated silicon photodiode in the  $\text{N}_2$  atmosphere dry-box. Then the brightness was calibrated using a PR-705 SpectraScan spectrophotometer (Photo Research), which was aligned normal to the direction of emitting surface. The OFETs characterizations were performed using a probe station and a 4 semiconductor parameter analyzer (Agilent 4155C). The film thickness was measured by Dektak 150. The atomic force microscopy (AFM) images were recorded on a Seiko SPA 400 with an SPI 3800 probe station in tapping mode.

## Results and discussion

In the present report, green emitting P-PPV was applied as the emissive layer, and we fabricated the PLEDs with a structure comprised of a ITO/PEDOT:PSS (40 nm)/P-PPV (80 nm)/PFN (5 nm)/Au NPs ( $\delta$  nm)/CsF (1.5 nm)/Al (120 nm), which is different with Au NPs thickness and as shown in the Fig. 1a. PFN is a classic electron injection layer, which can promote the device performance.<sup>1,27–28</sup> Because Au NPs direct contacting with the emissive layer will quench the fluorescence, the ultra-thin gold nanoparticles layer was evaporated onto PFN layer before the evaporation of CsF/Al cathode, in order to avoid the direct contact between Au NPs and emitting layer (Fig. 1a). The thicknesses of gold nanoparticles layer used is 0 nm, 0.5 nm,



**Fig. 1.** (a) Architecture and energy levels of the PLEDs. AFM micrographs of Au NPs prepared by thermal evaporation to a nominal thickness of (b) PFN surface, (c) 0.5 nm, (d) 1 nm, and (e) 2 nm on the PFN with a scan of  $500 \text{ nm} \times 500 \text{ nm}$ .

1 nm, 2 nm measured by the Filtech during evaporation. Actually the gold nanoparticles layer is incomplete coverage on the PFN layer as shown in Fig. 1b, c, d, e, where the coverage density and particle size are increased with the Au thickness. The reality diameter size of Au NPs cannot be determined because the thin PFN surface is not smoothness. Initially, small and sparsely distributed Au NPs were gradually coarsened and distributed more closely. The work functions of ITO/PEDOT:PSS/P-PPV/PFN/Au NPs ( $\delta$  nm) are measured by Kelvin probe as 4.2 eV, 4.4 eV, 4.6 eV, 4.6 eV for the 0 nm, 0.5 nm, 1 nm, 2 nm Au NPs thickness respectively. The work function of gold film is 5.2 eV, therefore it is logical to observe the increase of work function with increase in gold nanoparticles layer thickness. We observed only 0.4 eV increase because of the ultra-thin gold film and incomplete coverage on the PFN layer.<sup>29–30</sup>

By changing the thickness of Au NPs, we obtained the optimum device performance. Fig. 2a, b, and c show the device performances and their data are summarized in Table 1. Even though the work function keeps increase from gold thickness from 0 nm to 2 nm, the current density keeps decrease from 0 nm to 0.5 nm and then changes little with different Au thicknesses. However the performance of devices including brightness and efficiency has been enhanced

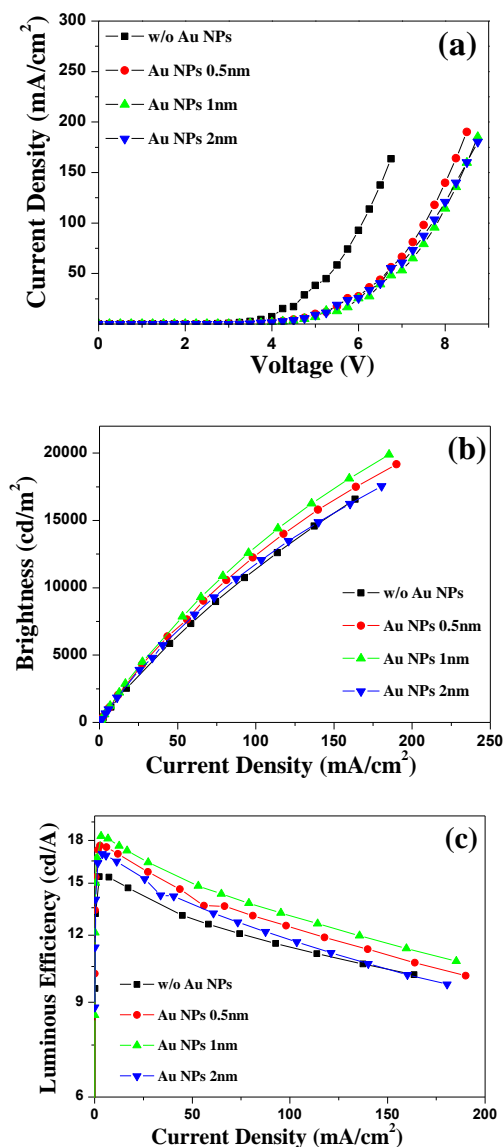


Fig. 2. (a) Current density vs. applied voltage (I–V), (b) brightness vs. current density (B–I) (c) luminous efficiency vs. current density (LE–I) curves as a function of Au NPs thickness.

Table 1 Device performance of PLEDs

Au NPs Thickness (nm)	Maximum Brightness [ $\text{cd}\cdot\text{m}^{-2}$ ]	Maximum LE [ $\text{cd}\cdot\text{A}^{-1}$ ]	Brightness [ $\text{cd}\cdot\text{m}^{-2}$ ]	LE [ $\text{cd}\cdot\text{A}^{-1}$ ]	$V_{\text{on}}^{\text{a}}$ (V)
0	17k	15.4	11k	11.4	2.75
0.5	19k	17.6	12k	12.5	2.75
1	20k	18.3	13k	13.1	2.75
2	18k	17.0	12k	11.8	3.00

a) The  $V_{\text{on}}$  at which luminescence reach  $0.1 \text{ cd}\cdot\text{m}^{-2}$ . Maximum brightness and maximum LE are the maximum brightness [ $\text{cd}\cdot\text{m}^{-2}$ ] and luminous efficiency [ $\text{cd}\cdot\text{A}^{-1}$ ] values recorded for each device. Brightness, LE are the brightness and luminous efficiency values recorded at a current density  $100 \text{ mA}\cdot\text{cm}^{-2}$ .

effectively, with the best optimized performance achieved as the 1 nm gold nanoparticles layer. From brightness vs. current density characteristic in Fig. 2b brightness increases obviously

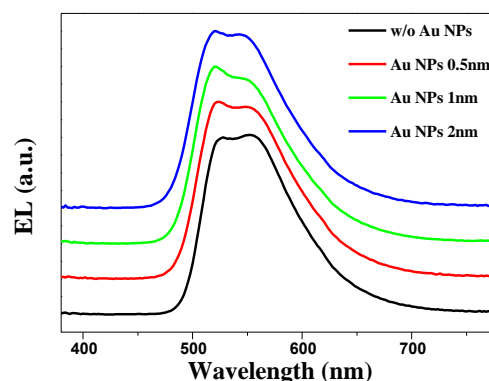


Fig. 3. The EL spectra of devices with different Au NPs thickness.

with the increased thickness of Au NPs, and the brightness maximum values increased from  $17 \text{ k cd}\cdot\text{m}^{-2}$  to  $19 \text{ k cd}\cdot\text{m}^{-2}$ ,  $20 \text{ k cd}\cdot\text{m}^{-2}$  and  $18 \text{ k cd}\cdot\text{m}^{-2}$ , for the Au NPs thickness of 0.5 nm, 1 nm, and 2 nm, respectively. At a current density of  $100 \text{ mA}\cdot\text{cm}^{-2}$  under different Au NPs thickness, the brightness is increased from  $11 \text{ k cd}\cdot\text{m}^{-2}$  to  $12 \text{ k cd}\cdot\text{m}^{-2}$ ,  $13 \text{ k cd}\cdot\text{m}^{-2}$  and  $12 \text{ k cd}\cdot\text{m}^{-2}$ . At the same time, we observe a noticeably upward trend in the values of luminous efficiency after decorating gold with different thickness. For the devices with Au NPs thickness 0.5 nm, 1 nm, 2 nm the maximum LE values are improved from  $15.4 \text{ cd}\cdot\text{A}^{-1}$  to  $17.6 \text{ cd}\cdot\text{A}^{-1}$ ,  $18.3 \text{ cd}\cdot\text{A}^{-1}$ ,  $17.0 \text{ cd}\cdot\text{A}^{-1}$ , respectively. And at a current density of  $100 \text{ mA}\cdot\text{cm}^{-2}$  the luminous efficiency is increased from  $11.4 \text{ cd}\cdot\text{A}^{-1}$  to  $12.5 \text{ cd}\cdot\text{A}^{-1}$ ,  $13.1 \text{ cd}\cdot\text{A}^{-1}$  and  $11.8 \text{ cd}\cdot\text{A}^{-1}$ . The turn-on voltage remained constant until the Au NPs thickness increased to 2 nm. That is, the driving voltage of the devices with Au NPs would be higher than that of the reference device. It may decrease the power efficiency of the device. However, OLEDs is a current driving device, and the devices show an observable enhancement in brightness and luminous efficiency at the same current from the control one in Fig. 2b, c.

Fig. 3 shows the EL spectra of PLEDs operating at 2 mA as a function of gold nanoparticles layer thickness. The EL spectra of P-PPV show two vibration peaks, at about 520 nm and 550 nm respectively. The relative intensity of the 0-0 peak at 520 nm increases from 0 nm to 1 nm and decreases back for 2 nm Au nanoparticles layer thickness. Based on the re-absorption coming from the overlap between absorption and EL emission of P-PPV, the evolution of EL spectra indicates the light emitting profile shifting in presence of gold nanoparticles layer.<sup>22</sup> The higher intensity of 0-0 peak means a decrease in re-absorption, attributed to the shortening of light path. In this case, the light emitting profile shifts to anode when gold nanoparticles layer increase from 0 nm to 1 nm and the corresponding brightness/efficiency increases. This is the typical characteristic of charge transport balance induced performance improvement. As shown in our previous report for inverted PLEDs, local surface plasma resonance (LSPR) induced performance improvement would show a totally opposite phenomenon, in which brightness/efficiency increase corresponds to the light emitting profile shift to the cathode.<sup>22</sup> It is because of the rapid decay of LSPR enhancement profile



from the cathode to the active layer. Thus the EL spectra analysis support the performance improvement by introducing gold nanostructures in this work (conventional PLEDs) mainly comes from charge transport balance but not LSPR induced enhancement (typical mechanism in inverted PLEDs). We suggest it is because the charge transport is more balance in conventional PLEDs than inverted PLEDs, where there is no effective overlap between LSPR field and light emitting profile. Therefore all the results indicate the complexity of electrical effect introduced by Au NPs, in which similar I–V behaviour has different brightness, efficiency and light emitting profiles at different gold thicknesses. It depends on the corresponding contribution of holes and electrons. Thus the electrical effect is focused on the enhancement mechanism in conventional PLEDs.

For the detailed evaluation of the electrical effects, the contribution of holes and electrons should be determined separately. Firstly we prepared the electrons-only devices with the device structure of ITO/Al (30 nm)/P-PPV (80 nm)/PFN (5 nm)/Au NPs (1 nm)/CsF (1.5 nm)/Al (120 nm) to study the effect of Au NPs on electrons transportation (Fig. 4a). The electrons-only current density increased when the thickness of gold nanoparticles layer from 0 nm to 0.5 nm, 1 nm, which indicated that the gold nanoparticles layer helps the electrons injection. Gold normally shows little interaction with the electrons due to the high work function. It is very interesting to observe that the gold nanoparticles layer helps the electron injection. Here we suggest the changes in electrons-only devices originate from the incomplete coverage of Au NPs enlarge the PFN/cathode interfacial area, leading to the improvement of electrons injection efficiency.<sup>21</sup> Yang also has reported that the LiF/Ag cluster/LiF structure could resolve the carrier injection problem between the cathode and organic layer, since Ag was deposited on the organic layer in clusters.<sup>25</sup> However, the electrons-only current density decreased from Au NPs 1 nm to 2 nm with similar work functions, which could be attributed to the better continuous morphology of the Au surface approaching the Au film.

The holes transport behaviour effected by Au NPs is determined in related OFETs with device structure of Si/SiO<sub>2</sub>/Au NPs (1 nm)/PMMA (30 nm)/PTB7 (80 nm)/Ag (60 nm), because the classic holes-only device using Au or MoO<sub>3</sub>/Al as electrode (the work function is too similar to distinguish from Au NPs layer to electrode).<sup>6</sup> When suitable gate voltage is applied, only holes are induced in transporting channel, which makes the possibility for observation the relationship between ultra-thin Au nanoparticles layer and holes transportation. Transfer curves for Si/SiO<sub>2</sub>/Au NPs (δ nm)/PMMA (30 nm)/PTB7 (80 nm)/Ag (60 nm), with δ = 0, 1 nm have been compared (Fig. 4b). The transfer curves exhibit a typical *p*-channel field-effect transistor behaviour, which means the holes are the main carries in the PTB7 active layer. After introducing Au NPs nanoparticles layer, the I<sub>ds</sub> decreases when compared with the control device structure, which indicates that the presence of Au NPs can decrease the current density.<sup>31-32</sup> This is a typical phenomenon for Au NPs in the dielectric layer of OFETs, which is explained as holes-trapping

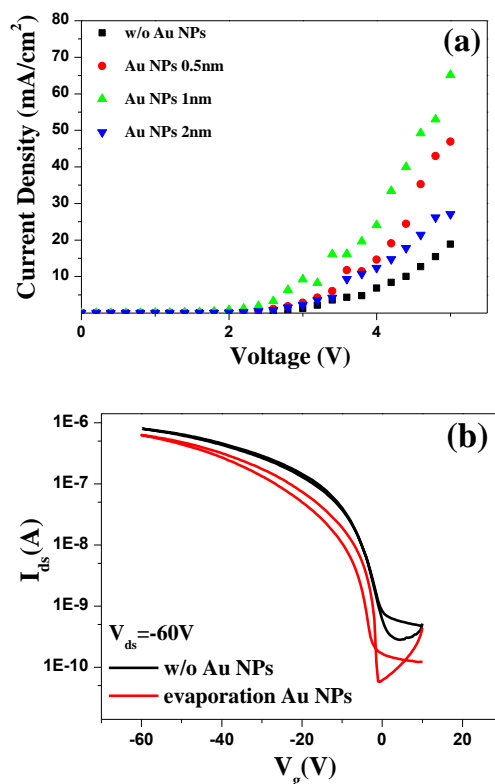


Fig. 4. (a) The current density vs. applied voltage characteristics of the electrons-only devices ITO/Al (30 nm)/P-PPV (80 nm)/PFN (5 nm)/Au NPs (δ nm)/CsF (1.5 nm)/Al (120 nm), (b) Transfer curves for Si/SiO<sub>2</sub>/Au NPs (δ nm)/PMMA (30 nm)/PTB7 (80 nm)/Ag (60 nm), without Au NPs device and with evaporation 1 nm Au NPs.

through tunnelling effect.<sup>33</sup> We propose a similar process in PLEDs for the decrease of current density. The trapped holes normally caused hysteresis window at the same time in Fig. 4b, which depends on the particle size and surface properties. It indicates that the charge could be stored in Au NPs, which is not positive in PLEDs. We suggest the performance decrease at 2 nm thickness of Au nanoparticles layer is related to this effect, because the bigger Au NPs can show the better charge storage ability.<sup>30</sup> On the other hand, the addition of ultra-thin gold nanoparticles layer decreases the total current density (Fig. 2a), which also indicate that the decreased current density originates from the combination of holes and electrons current). The conjugated polymer P-PPV used in this work is a typical *p*-type semiconductor, in which the hole-mobility is much higher than the electron mobility. Thus the decreased hole current and the increased electron current strongly support enhancement of the device performance originating from the balanced charge transport and the shifted light emitting profile towards the anode.

## Conclusions

In conclusion, we have demonstrated that the insertion of ultra-thin gold nanoparticles layer modified cathode into the

devices could improve the performance of solution-processable PLEDs. The brightness and luminous efficiency could be enhanced simultaneously due to Au NPs. The mechanism of performance enhancement is ascribed to the decreased holes and the increased electrons, resulting in the balanced electron-hole recombination and which shifted the light emissive profile towards the anode, rather than LSPR effect. The unbalanced electron-hole transportation is the basis of this type of enhancement, which is common in organic semiconducting polymers and molecules. Thus we believe that Au NPs constitute a versatile and effective route to achieve high-performance and this is of great significance of Au NPs using in the development of high performance organic optoelectronic devices.

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Graphical Abstract for:

## Ultra-Thin Gold Nanoparticles Layer Modified Cathode for the Enhanced Performance of Polymer Light Emitting Diodes

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Ultra-thin gold nanoparticles layer modified cathode is applied in PLEDs to improve devices performance, originating from the balanced electron-hole recombination.

