ADVANCED FUNCTIONAL MATERIALS

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Investigation on the Low Luminous Efficiency in a Polymer Light-Emitting Diode with a High Work-Function Cathode by Soft Contact Lamination**

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This article reports the main origin of the low luminescent efficiency in hole-dominant polymer light-emitting diodes by controlling the hole injection and by chemically modifying the cathode by molecular monolayers. Since molecular modification of the top electrode is impossible when one deposits the electrode using a vacuum deposition method, this study was performed using a soft contact lamination technique to form electrical contacts on top of the emissive layer. The top electrode was chemically modified with an alkane thiol self-assembled monolayer (SAM) to act as an interfacial spacer layer between the emitting layer and the cathode. Herein, it is reported that, contrary to common belief, a high device quantum efficiency can be achieved from the dominantly hole-transporting device with a high work-function cathode (like Au) by facilitating more hole injection from the anode in the device with low population of exciton quenching channels near the cathode.

1. Introduction

Organic semiconductor materials have recently received a lot of interest because of the promising applications to electronic and optoelectronic devices. In particular, organic electroluminescent (EL) devices[1,2] have been developed toward ultrathin and power efficient mechanically flexible display systems with easy processibility. Electroluminescence in organic light-emitting diodes comes from the recombination of holes and electrons injected from the respective electrodes. The efforts to improve the luminescence efficiency of organic light-emitting diodes (OLEDs) were focused on balancing charge injection and transport of both carriers. It has generally been believed that the device quantum efficiency critically depends on the interface energy barrier caused by the band offset between the emitting layer and the electrodes.[3] For example, low work-function cathodes such as Ca, Ba, and Mg have been used to improve the electron injection efficiency.[3] Therefore, an Au electrode has not been utilized to fabricate practical OLEDs because of the high work-function that gives rise to a high electron injection barrier, even though the metal has high environmental stability. To date, in conventional thermally evaporated devices, there have been several methods employed to improve the electron injection in the device with an air-stable Al cathode by inserting electron injection layers (e.g., metal fluorides,[4] conjugated polyelectrolytes,[5] and ionic polymers[6]) before cathode metal deposition, by blending a surfactant-like additive with the emitting layer, by post-metal thermal annealing,[7] and by incorporating highly electron-deficient moieties (e.g., triazole)[8] or ionic moieties (e.g., quaternized ammonium salt)[5] into the emitting molecules. The quantum efficiency value has been known to be very poor in conventional thermal evaporated devices with an Au cathode (for example, a poly(*p*-phenylene vinylene) device with an Au cathode has a quantum efficiency in the order of 10^{-5} %).[3] However, it is demonstrated here that contrary to common beliefs, a high device quantum efficiency can be achieved from a dominantly hole-transporting polymer light-emitting diode (PLED) with a high work-function cathode (like Au) by facilitating more hole injection from the anode in the device with low population of exciton quenching channels near the cathode. For this study, the top electrode (i.e., cathode) was laminated conformally on the underlying EL organic layer to avoid the exciton quenching channels caused by metal vacuum evaporation as reported elsewhere.[9,10] The exciton quenching rate by the metal electrodes can be reduced by modifying the top Au electrodes with alkane thiol self-assembled molecules as a molecular spacer layer in the device. In this work, a high efficiency of ~1.0 cd A^{-1} was achieved for a green light-emitting polyfluorene device laminated with a molecularly modified Au contact by promoting the hole injection. Herein, it is demonstrated that hole space charges in hole-dominant devices can induce the injection of the opposite charge (i.e., electrons) near the Au cathode so that the luminescence efficiency can be highly enhanced when the exciton quenching is under control. This study reveals that the low quantum efficiency of EL devices with a thermally evaporated Au cathode reported to date does not stem mainly from the high interface injection barrier

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but from the quenching of the excitons mostly located close to the Au cathode. Herein the main origin of the low luminescent efficiency in this hole-dominant device is systematically investigated by controlling the hole injection and by chemically modifying the cathode by molecular monolayers. Since molecular modification of the top electrode is impossible when one deposits the electrode using a vacuum deposition method, this study was performed by using a soft contact lamination (ScL) technique[9] to form electrical contacts on top of the emissive layer. The top electrode was chemically modified with SAMs before lamination, which acted as an interfacial spacer layer between the emitting layer and the cathode.

OLEDs are usually fabricated in a standard way by sequentially depositing active layers and electrodes onto a substrate. The evaporation of metals onto organics leads to in-diffusion of the metal, changes in the morphology of the organic and, in some cases, disruption of chemical bonds,[11,12] which leads to the generation of luminescence quenching centers in OLEDs.[11–13] It is important to remove the quenching centers generated by vacuum deposition of the cathode in the case that most of the excitons are generated close to the metal cathode. To avoid the adverse effects by conventional metal deposition, OLEDs have been built based on a soft conformable physical lamination of thin metal electrodes supported by an elastomeric poly(dimethylsiloxane) (PDMS) substrate against an EL organic thin film (i.e., ScL).[9,14] Since ScL minimizes any chemical, physical, or morphological changes to the organic, it is suitable and useful for this study to investigate the effect of the non-radiative luminescence decay channels generated by metal evaporation. Even in the ScL devices, there is still a non-radiative energy transfer process of excitons adjacent to the bare metal electrode.[15] Therefore, molecular modification on the electrode was performed by treating the Au surface with alkane thiol molecule solutions in ethanol to form SAMs.

2. Results and Discussion

Figure 1a schematically illustrates how the OLED is fabricated by laminating the top Au electrode supported by a conformable transparent elastomeric substrate, PDMS, onto an EL organic (a green light-emitting polyfluorene derivative)[16] supported by an indium tin oxide (ITO)/glass substrate or by poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT-PSS)/ITO/glass substrate. The PEDOT-PSS layer was employed to promote the hole injection from ITO to the emitting layer. Au-coated PDMS was prepared according to the procedure in the literature.[9] The Au surface can be modified with thiol molecules in ethanol which easily react with metals such as Cu, Ag, and Au etc. In this study, hexadecanethiol ($CH_3(CH_2)_{15}SH$: hereafter C16) was utilized. The thickness of C16 SAMs in the literature is 2.2 nm on Au.[17] When the bottom and top pieces are brought together, van der Waals interactions pull them into intimate contact on a molecular scale to complete the device. Figure 1b shows the energy band diagram of the fabricated device. Judging from the energy band diagram, the electron injection should be very poor because of the large injection barrier (2.05 eV). Without the PEDOT-PSS layer, the hole injection barrier is as large as 0.75 eV. Therefore, the PEDOT-PSS layer was incorporated to improve the hole injection.

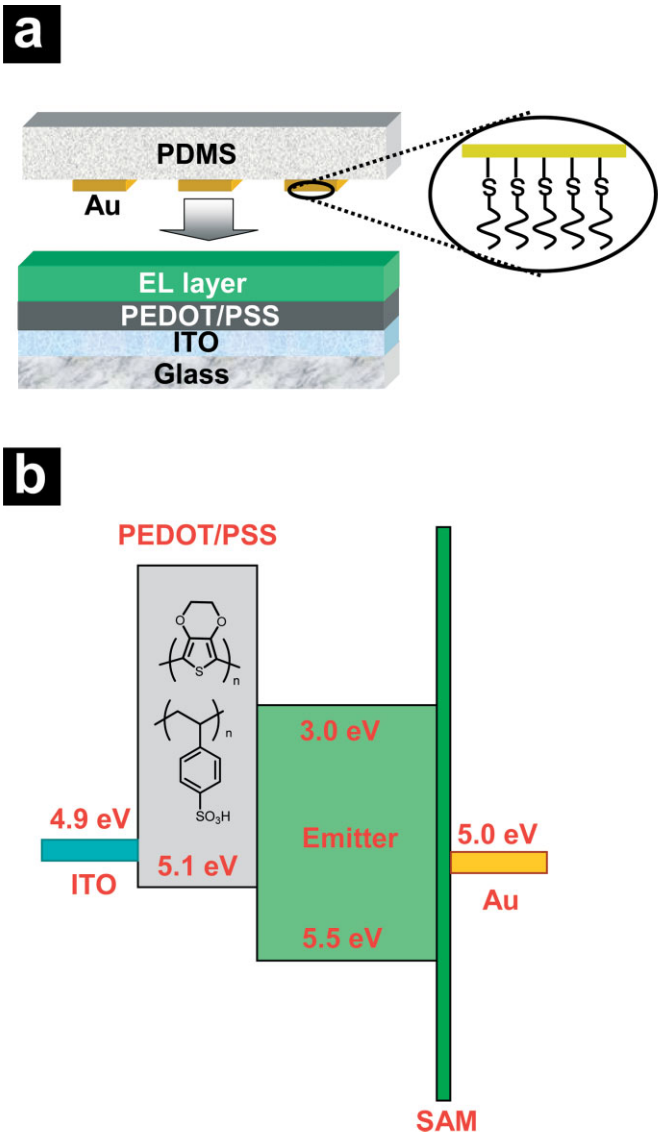

Figure 1. The schematic illustration of organic light-emitting devices laminated soft-conformably with a molecularly modified Au electrode deposited on the PDMS.

It has been widely understood that OLED devices with high work-function cathodes (e.g., Al: 3.7 eV, and Au: 5.0 eV) are dominantly hole-transporting because of the limited electron injection, which leads to low luminescence quantum yield.[3] Even if the hole injection is promoted by adding a hole-injection layer on top of the ITO, such as polyaniline doped with camphor sulfonic acid[18–20] or PEDOT-PSS,[21] the device quantum efficiency was very similar to the device without the hole-injection layer, as shown in the literature, and has been explained based on the unbalanced charge injection because of the poor electron injection.[19] The experiment in our lab also confirmed that there was no improvement in the device quantum efficiency by adding a PEDOT-PSS layer in the hole-dominant devices fabricated by conventional thermal evaporation of Au and Al. Figure 2 shows that the evaporated devices of ITO/PEDOT-PSS/polyfluorene/Au and ITO/PEDOT-PSS/polyfluorene/Al show very similar quantum efficiencies with the evaporated ITO/polyfluorene/Au and ITO/polyfluorene/Al, respectively. To date, it has been generally understood that the carrier injection and transport mostly determines the device quantum efficiency so that low work-function cathodes, such as Ca, Ba, and Mg, were essentially used for balanced carrier injection in the devices.[3] With this in mind, the results of the devices with evaporated cathodes in Figure 2 is generally understood based on the device energy band diagram because the minority carrier (i.e., electron) injection is still limited in the device despite the enhanced hole injection so that the hole-electron recombination rate is still low.[3] However, in this work, it was found that the light output and the quantum efficiency even in hole-dominant devices can be hugely improved by adding a hole injection layer (PEDOT-PSS) in ScL devices, unlike the hole-dominant devices formed by conventional metal evaporation. As Figure 2 shows, the luminescence efficiency of the ScL device with PEDOT-PSS is highly improved compared with that without PEDOT-PSS even if the cathode is Au so that the electron injection should be still limited.

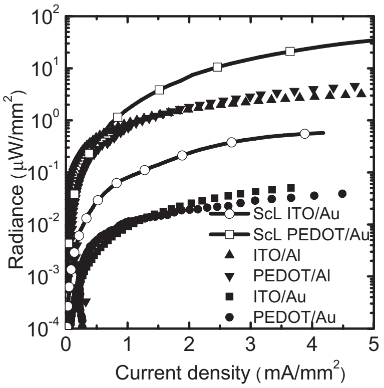

Figure 2. Log *L* vs. *I* characteristics of the thermally evaporated ITO/polyfluorene/Au (20 nm), ITO/PEDOT-PSS/polyfluorene/Au (20 nm), ITO/polyfluorene/Al (60 nm), and ITO/PEDOT-PSS/polyfluorene/Al (60 nm) devices as well as ScL ITO/polyfluorene/Au (20 nm) and ITO/PEDOT-PSS/polyfluorene/Au.

Figure 3 shows the current–voltage–luminescence (*I*–*V*–*L*) characteristics of conformally laminated polymer EL devices made of a green light-emitting polyfluorene derivative[22] in an ITO positive direction. The Au surface of the top piece was treated with C16 solution and then the excess thiol molecules were washed away with ethanol. A 100 nm thick layer was prepared by spincasting the EL polymer solution on top of the ITO/glass and PEDOT-PSS/ITO/glass. Figure 3a shows that the devices using a hole injection layer of PEDOT-PSS have remarkably enhanced current densities by at least two orders of magnitude, which is independent of the existence of thiol SAM molecules on the Au cathode. It was reported previously that the work-function of Au surfaces modified with alkane thiols was independent of chain length for molecules longer than octanethiol.[23] Hence, the effect of the alkane thiol chain length on the work-function is not likely to be the dominant factor and will not be considered in the discussions below. A thin hole-blocking and electron-injecting layer would result in increased current and luminescence, as was observed using ionic insulating polymers[6] or insulating Al_2O_3.[24] However, the *I–V* characteristics above light onset voltages with the SAM are almost the same as those without the SAM as shown in Figure 3a. The decay constant of the devices without the PEDOT-PSS layer was approximately 0.37 per CH_2,[10] which is consistent with reported values for tunnelling through alkane thiol molecules at high biases.[25] The SAM acts as a thin insulating layer that electrons

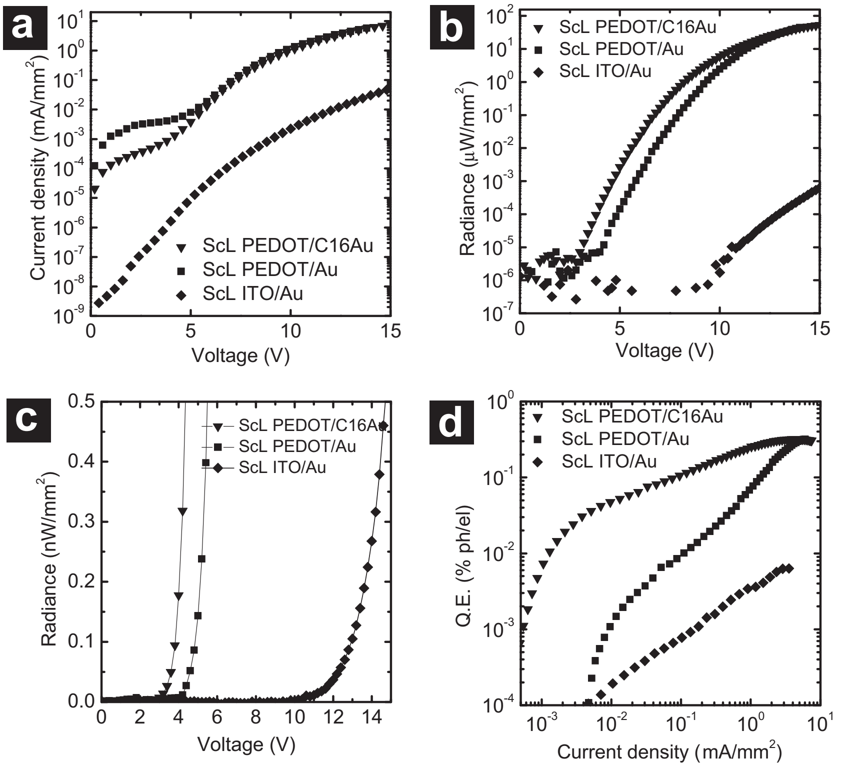

Figure 3. *I* (current)–*V* (voltage)–*L* (Luminescence) characteristics of polymer EL devices made of a polyfluorene derivative that are ScL with bare Au and C16-modified Au supported by PDMS. The EL layer was deposited on the PEDOT-PSS layer or directly on the ITO substrate. a) Log *I* vs. *V*, b) Log *L* vs. *V*, c) *L* vs. *V* (for turn-on voltage comparison), and d) Log QE vs *I* characteristics of ITO/PEDOT-PSS/polyfluorene/Au, ITO/PEDOT-PSS/polyfluorene/C16/Au, and ITO/polyfluorene/Au devices made by soft contact lamination.

and holes can tunnel through. It was also observed that the *I–V* characteristics of the devices with a PEDOT–PSS layer above the light onset voltage are independent of the chain lengths of the SAM (e.g., $CH_3(CH_2)_nSH$: $n = 7$, 11, and 15). This implies that the tunnelling efficiency through the SAM has a minor impact on the overall current of the devices with the PEDOT-PSS layer unlike the devices without the PEDOT-PSS layer.

Furthermore, Figure 3b shows that the devices with a PEDOT-PSS layer has a hugely enhanced luminance by about six orders of magnitude at 10 V. Since this remarkable result in a dominantly hole-transporting device was obtained by improving the hole injection capability instead of tuning the electron injection contact by lowering the cathode work-function[3] or incorporating an electron injection layer,[4,6,24] it strongly indicates that hole space charges accumulated within the emitting layer near the cathode can induce the injection of opposite charges (i.e., electrons) from the cathode even if the devices have a high electron injection barrier.[26,27] A previous report suggests that the holes can be promoted by accumulated electrons near the anode in electron-majority polyfluorene devices.[28] When the PEDOT-PSS (ionization potential: ~5.1 eV) was inserted as a hole injection layer on top of ITO (work-function ~4.9 eV), the hole injection barrier becomes as low as ~0.4 eV for the polyfluorene emitting layer (the highest occupied molecular orbital: ~5.5 eV, the lowest unoccupied molecular orbital: ~3.0 eV), while the electron injection barrier is still very high, as much as ~2.0 eV. The green polymer has a much lower hole mobility compared with electron mobility[29] so that even if holes are efficiently injected, hole space charges can be dominated as depicted in Figure 4. Since the accumulated hole space charges near the cathode can shift the vacuum level to reduce the energy barrier for electron injection, enhance the band-bending near the contact (see Figure 4), and enhance the built-in field, the effective energy barrier for the injection of electrons from the cathode is lowered and thus the electron injection is promoted. The large band-bending near the contact by accumulated space charges reduces the electron tunnelling width, which can also facilitate electron injection into the emitting polymer. As a result, the hugely improved light output implies that the electrons recombine with holes efficiently near the cathode without allowing the excitons to undergo non-radiative channels. However, this phenomenon in conventional evaporated devices has not been found. The reason is that even if the electrons are enhanced by accumulated holes, most of the excitons generated close to the cathode are quenched by the cathode as Figure 4a illustrates.[30]

The laminated device with the additional PEDOT-PSS layer also shows a much lower turn-on voltage (4.0 V) than the value (9.4 V) of the device without a PEDOT-PSS layer (Figure 3c). Again, among the devices with a PEDOT-PSS layer, the devices with an SAM on top of the Au have the lowest turn-on voltage (3.2 V) (Figure 2b and 2c). However, the *I–V* characteristics above the turn-on voltage were nearly same for all the ScL devices irrespective of the existence of an SAM as Figure 3a shows. This indicates that the SAM rarely affects the charge injection and transport in the devices but instead plays an important role as a spacer to prevent a luminescence quenching by non-radiative energy transfer and diffusion of excitons to the Au cathode in the device.[30,31] Since the recombination zone is located close to the Au cathode at low operating voltage, with the SAM more excitons can decay radiatively, and thus the luminescence is observed even at a lower voltage (3.2 V), at which luminescence without the SAM was not ob-

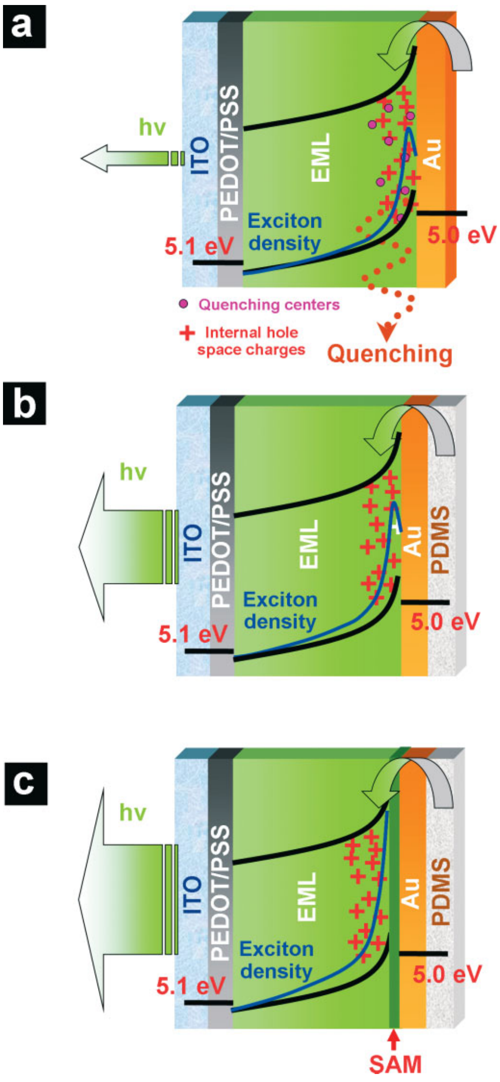

Figure 4. Schematic illustrations of exciton generation and decay close to the cathode in a) the Au-evaporated devices, b) Au-laminated devices, and c) molecularly modified-Au laminated device. In all the devices, most of the excitons are generated next to the cathode as electrons are injected from the Au cathode assisted by accumulated internal hole space charges. When an additional layer was used to promote hole injection such as PEDOT-PSS, hugely improved current density was observed. a) In the evaporated device, most of the excitons decay non-radiatively by quenching channels inside the emitting layer produced during the metal evaporation. Therefore, the luminescence yield can be very poor. b) In the ScL devices, some portion of the generated excitons (i.e., the excitons very close to the cathode) can decay non-radiatively by energy transfer from the exciton to the metal. Significantly fewer excitons go through the non-radiative process than those in the evaporated devices. c) In the ScL device with SAM-modified Au cathodes, the SAM layer acts as a molecular spacer to reduce exciton diffusion and non-radiative energy transfer to the Au cathode.

served. Therefore, the reduced quenching effect at the low voltages leads to a lower light-emission onset voltage. But at high voltages above 12 V, all the laminated devices show almost the same level of optical output and device efficiency irrespective of the existence of the SAM as Figure 3b and 3d show, which can be ascribed to the shift of the recombination zone away from the Au cathode as the electric field increases.[32] The strong dependence of the device quantum efficiency on the electric field as shown in Figure 3d (i.e., C16 device vs. bare Au device: ~34 times at 0.01 mA mm^{-2}, ~10 times at 0.1 mA mm^{-2}, and ~3.4 times at 1 mA mm^{-2}) implies that the exciton quenching takes place dominantly at the metal–organic interface. The maximum efficiency of the laminated device with an SAM-modified Au electrode showed as good as 0.32 % photons/electron (ph/el) (~1.0 cd A^{-1}), which is three orders of magnitude higher than the thermally evaporated ITO/PEDOT-PSS/polyfluorene/Au device (5×10^{-4} % ph/el). Furthermore, such a high value has not usually been observed from devices that use a high work-function cathode of Au to date, even without modifying the emitting layer chemically and without adding an electron injection layer. The remarkable difference in the output luminescence and the efficiency between the evaporated devices and the laminated devices is amplified by the fact that the exciton densities in the hole-dominant devices are mostly populated next to the cathode where most of quenching channels exist. In the laminated devices, the exciton quenching by exciton diffusion toward Au and non-radiative dipole coupling with bare Au still occurs. Therefore, the SAM layer can further reduce the exciton quenching as Figure 3d shows.

Figure 4 summarizes the exciton quenching phenomena dependent on the method to form the top electrode in the device. The major non-radiative recombination pathways for excitons includes scattering by the quenching centers in the polymer created by metal evaporation, exciton diffusion toward the metal, and non-radiative long-range dipole–dipole coupling with metal.[31,33] This work demonstrates that even if the excitons in the ITO/PEDOT/polyfluorene/Au device formed by a conventional evaporation method are generated much nearer the cathode by assistance of accumulated holes compared with conventional ITO/polyfluorene/Au device, the increased exciton population can not be observed by electroluminescence because most of the excitons are quenched as a result of the quenching channels generated by metal evaporation (Figure 4a). Since ScL devices (Figure 4b) based on bare Au/PDMS do not have the same exciton quenching channels that are generated by metal evaporation, the luminescence efficiency is hugely improved, as shown in Figure 3d. In this case, the electron injection from the cathode can be facilitated mainly by lowering the effective energy barrier by internal hole space charges in the device, and the recombination takes place close to the metal–organic interface. When the electric field is low, the exciton quenching can take place by non-radiative energy transfer through dipole coupling to the cathode. However, when the electric field is high enough to shift the recombination zone far from the Au, most of the generated excitons will decay radiatively. The exciton quenching by dipole–dipole coupling with Au can be reduced by adding a molecular spacer layer (i.e., C16 SAM). The dominantly hole-transporting device has a much lower population of exciton quenching channels near the cathode because of the formation of an interfacial spacer layer using SAM-modified Au/PDMS (Figure 4c). The turn-on voltage is further lowered and the device quantum efficiency can be greatly enhanced by 340 times at 4.5 μA mm^{-2} which is the light onset point of the ScL device without the C16 SAM layer. The effect of SAM layers on the turn-on voltage and the quantum efficiency are much more obvious when the driving voltage is low and thus most of excitons are located close to the cathode.

3. Conclusion

In conclusion, this study suggests that when non-radiative energy transfer and diffusion of excitons to metal is mostly prohibited under control, highly efficient PLED devices can be realized even with an air-stable high work-function cathode by improving the hole injection rather than by engineering the cathode contact (e.g., by using a low work-function metal or an additional electron injection layer). The electron injection even in hole-dominant devices can be assisted by hole space charges near the cathode. Therefore, the low efficiency of the hole-dominant polyfluorene devices mainly comes from the quenching of excitons close to the cathode rather than the poor electron injection owing to the high electron injection barrier as previously thought. This study will give an important insight for the fabrication of roll-to-roll laminated flexible OLED displays with good performance, which currently require an environmentally stable cathode.

4. Experimental

OLEDs were fabricated by laminating the top Au electrode supported by a conformable transparent elastomeric substrate, PDMS (Sylgard 184, Dow Corning Inc.), onto the EL organic (a green light-emitting polyfluorene derivative, Dow Green B) supported by an ITO/glass substrate or by a PEDOT-PSS/ITO/glass substrate. The PDMS precursor was cast and cured (60 °C for 3 h) against the flat surface of a silicon wafer. Exposing the PDMS to an oxygen plasma (~2 s, 30 sccm, 30 mT, 100 V; Plasma-Therm reactive ion etcher) followed by electron beam evaporation (evaporator: Temescal BJD 1800) at ~5×10^{-7} torr of ~1 nm Ti (adhesion promoter; 0.3 nm s^{-1}) and 20–60 nm Au (1 n m s^{-1}) generates thin electrically continuous metal films that are strongly bonded to the PDMS. The Au surface was modified with hexadecane thiol ($CH_3(CH_2)_{15}SH$). A 2×10^{-3} M alkane thiol ethanol solution was put on the Au electrode supported by PDMS and then left for 15 min, followed by blow-drying with N_2 and rinsing with ethanol.

The ITO was cleaned with detergent, acetone, and isopropyl alcohol in sequence, and then O_2 plasma treatment was performed for 5 min. The PEDOT-PSS (Baytron P, H. C. Starck, GmbH) layer was spincast to be 35 nm and baked at 110 °C in a vacuum oven for 50 min. Spin casting formed a 100 nm uniform film of the electroluminescent material on the ITO (~15 Ω □$^{-1}$)/glass substrate or PEDOT-PSS/ITO/glass substrate. When the bottom (ITO/emitting layer) and top pieces (Au/PDMS) were brought together, van der Waals forces pulled the electrodes into intimate contact with the EL layer at room temperature, without application of external pressure. Typically, this contact initiates on one side of the structure, a wetting front then progresses naturally across the sample until the entire surface is in contact. The complete lamination over the entire surface was observed through a microscope after laminating the top electrode over the electroluminescent materials on an ITO glass substrate.

For comparison, thermally evaporated devices of ITO/PEDOT-PSS/polyfluorene/Al and ITO/PEDOT-PSS/polyfluorene/Au were fabricated. The thermally evaporated devices were fabricated by evaporating the Au and Al at a rate of 1 Å s^{-1} and 1–3 Å s^{-1}, respectively, on the same materials described above using an Edward Auto 306 thermal evaporator with distance of 11.5 inches between the samples and the metal source.

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