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Effect of lithium and silver diffusion in single-stack and tandem OLED devices

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ABSTRACT

A study of metal (Li, Ag) diffusion has been carried out in an archetypal OLED device based on *N*,*N*'-di(1-naphthyl)-*N*,*N*'-diphenyl-(1,1'-biphenyl)-4,4'-diamine|tris(8-hydroxyquinolinato) aluminum|4,7-diphenyl-1,10-phenanthroline (NPB|Alq₃|Bphen). Using single-stack and two-stack tandem OLED structures with variations of layer thicknesses and metal layer placements, we have found that Ag vapor-deposited on Alq₃ layer can diffuse or penetrate deep into Alq₃, up to ~2,000 Å, causing luminescence quenching. This diffusion can be substantially prevented by a thin layer of Li or Bphen deposited on Alq₃ prior to the deposition of Ag. In contrast, Li diffusion in either Alq₃ or Bphen is limited to about 50 –100 Å. Li appears to be able to diffuse into Bphen irrespective of the order of Li and Bphen depositions. © 2016 Published by Elsevier B.V.

1. Introduction

Lithium is commonly used in OLED devices as the cathode and as an n⁺ type dopant in an electron-acceptor material in forming the electron-injecting layer. Early studies have shown that lithium can diffuse in various electron-acceptor materials and its effects on the OLED performance are varied but not well understood in most For instance, in 2,9-Dimethyl-4,7-diphenyl-1,10cases. phenanthroline (BCP), a classic electron acceptor, a large diffusion length of 70 nm has been estimated [1]. However, a diffusion length from 2 to 17.5 nm has been found in 4,7-diphenyl-1,10phenanthroline (Bphen), another common electron acceptor that is similar to BCP [2]. Silver, which is often used as the electrode, has also been found to diffuse in OLED materials. Some reports indicate that silver can diffuse through a 60 nm layer of tris(8hydroxyquinoline) aluminum (Alq₃), an archetypal emitter for OLED devices [3,4]. Other research results give a diffusion length for silver in Alg₃ of about 30 nm [5]. However, a short Ag penetration into Bphen has been reported, depending on the deposition rates and techniques used [6]. Since both lithium and silver can severely affect light emission in an OLED device through either plasmonic or non-radiative quenching effects [7–9], their presence in or near the

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emission layer due to diffusion from the adjacent transport layers or the metallic electrode is undesirable. Furthermore, metal diffusion in tandem OLED devices can be more problematic due to the presence of one or more charge-generating layers (CGL), which often comprise a thin metal layer such as Li. Tandem OLED devices promise much higher current efficiencies and brightnesses [10,11], however their operational lifetimes can be compromised by the instability of the CGL related to metal diffusion. In this study, we have examined the effects of lithium and silver on OLED devices with a simple layer structure consisting of a bi-layer N,N'-di(1naphthyl)-*N*,*N*'-diphenyl-(1,1'-biphenyl)-4,4'-diamine (NPB)|Alg₃ stack or a tri-layer NPB|Alq₃|Bphen stack. We will show that much of these effects can be attributed to metal penetration into the organic films during vapor deposition of the metal. As the metal penetration depths can vary with the deposition rates [6,12–14], these were kept constant to limit its effect. Tandem OLED devices were used to further confirm the effect of metal diffusion on OLED devices.

2. Experimental

OLED devices were fabricated on pre-patterned ITO (~1000 Å) glass substrates (1.5×1.5 inches) with a surface resistance of ~15 Ω / sq and an optical transparency of ~90%. The ITO substrates were batch-wise cleaned by scrubbing with detergent solution, then rinsed, and subjected to 10 min agitation in ultrasonic baths of DI







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water, acetone, and isopropanol (IPA), sequentially. Following a final IPA rinse, the substrates were dried with nitrogen and treated with oxygen plasma (2:15 min, 400 W and 5×10^{-1} mbar). All films were prepared by vapor deposition in a vacuum chamber $(<5.0 \times 10^{-6} \text{ Torr})$ without breaking vacuum until the OLED device was completed with a 1000 Å top silver electrode, at a constant rate of 5.0 Å/s for silver. A typical layer sequence is as follows: A 20 Å laver of molybdenum oxide (MoO_x) was deposited at about 0.3 Å/s on ITO as the hole-injecting layer (HIL), N,N'-di(1-naphthyl)-N,N'diphenyl-(1,1'-biphenyl)-4,4'-diamine (NPB) was the hole transport layer (HTL), tris(8-hydroxyquinolinato) aluminum (Alq₃) was the electron transport layer (ETL) as well as the light emitter, and 4,7diphenyl-1,10-phenanthroline (Bphen) was the optional ETL. The deposition rates for the organic layers were typically 4 Å/s. Metallic lithium was deposited from heating lithium nitride, Li₃N, to a threshold temperature of 400 °C where it decomposes into metallic Li and N₂ gas, causing an initial pressure rise before settling at a pressure below 1.0×10^{-5} torr before deposition. Stable Li deposition rates from 0.1 Å/s to 4.0 Å/s can be obtained with careful control of the source temperature [15]. For Li thickness dependence experiments, 0.1 Å/s and 1.0 Å/s were used for thicknesses below and above 1 Å of Li, respectively. Four identical OLED devices with an active area of 0.1 cm² were produced on a single substrate. The electroluminescent properties of the devices were recorded with a Photoresearch PR650 SpectraScan Colorimeter and a Keithley 2400 source meter.

3. Results and discussion

3.1. Single-stack devices

We first examined a series of single-stack OLED devices -ITO|MoOx(20 Å)|NPB(600 Å)|Alq3(400 Å)|Li(x)|Ag, with Li|Ag as the cathode where the Li layer was varied from 0 to 500 Å. In fabricating these devices, all the layers were deposited in a vacuum chamber without vacuum breaks to ensure exposure only to the chamber environment. Fig. 1A shows a plot of the external quantum efficiency (EQE) and drive voltage Vs Li thickness, all measured at 20 mA/cm^2 . It can be seen that the EQE is low (<0.1%) for the device without Li (Ag-only) and remains so for devices with Li below 1 Å. Above 1 Å of Li there is a sharp rise in EQE to ~0.9%, which remains more or less unchanged up to ~200 Å of Li. The drive voltage is ~6.5 V for all these devices with Li < 1 Å, including the Ag-only device. It decreases gradually to a minimum of ~5.0 V at about 8 Å Li before rising steadily to more than 10 V at 200 Å Li. At 500 Å Li, the OLED device fails catastrophically, resulting in little or no light emission even as the drive voltage continues to increase. During Li deposition, Li atoms (from the vapor phase) impinging on the surface of the Alg₃ layer are expected to penetrate or diffuse into the bulk of the Alq₃ layer, producing a chemically doped layer consisting of Li⁺-Alq₃ charge-transfer complexes [16,17]. This Li doped Alq₃ or n⁺ layer presumably forms an electron-injecting contact to the undoped Alq₃ layer immediately underneath, assuming that Li penetration is much less than 400 Å - the thickness of the entire Alg₃ laver. Likewise, the deposition of Ag electrode can also result in Ag penetration into the Alq₃ film [3,5]. With Li as the interlayer, the depth of Ag penetration is expected to depend on the Li layer thickness. The low EQE values (<0.1%) for the Ag-only device and devices with <1 Å Li suggest that Ag can penetrate at least as deep as 400 Å into the Alq₃ layer to result in quenching of the Alq₃ emission from the NPB|Alq₃ interface. Alternatively, these low EQE values can be attributed to charge imbalance and shifting of the recombination zone toward the cathode due to poor electron injection from the cathode, although this will be shown to be a minor pathway. Devices with Li > 1 Å attain substantially higher EQE values of ~0.9%, indicating the emission quenching at the NPB|Alq₃ interface is much reduced. The device with 500 Å of Li is an exception as the Li|Ag electrode with such a thick Li layer appeared non-uniform and the device was not emissive. The initial drop in the drive voltage in 1 to 10 Å range of Li suggests the formation of Li⁺-Alq₃ electron-injecting contact. Above 10 Å Li, the driving voltage gradually increases, which can be due to the presence of an insulating layer, possibly LiO_x and $Li(OH)_x$, due to the reaction of excess Li with residue H₂O and O₂ in the vacuum chamber.

Next we investigated the penetration of Ag into Alq₃ using a NPB|Alq₃ bi-layer stack, where the Alq₃ layer is varied from 100 to 5,000 Å and Ag-only is the cathode. Fig. 1B shows that the drive voltage increases gradually with Alq₃ thickness due to the increasing bulk resistance of Alq₃. The EQE, however, is essentially zero for devices with Alq₃ thickness less than 200 Å. It increases gradually to a maximum of 0.7% at 2,000 Å of Alq₃, which is considerably larger than the EQE of 0.1% obtained for device with a 400 Å Alq₃. These results suggest that Ag is capable of penetrating deep into Alq₃ during vapor deposition to cause luminescence (exciton) quenching at the NPB|Alq₃ interface, where electron-hole recombination takes place. The minimum Ag penetration depth is at least about 200 Å, but the effective penetration depth could be even larger than 2000 Å since the maximum EQE of 0.7% (at 2000 Å of Alq₃) is still low compared to the devices with Li|Ag cathode. The drop in EQE at Alq₃ thickness greater than 2000 Å can be attributed to optical effects (e.g., microcavity effects). Comparing the EQE values in Fig. 1A and B, it can be seen that OLED devices with Li|Ag are much more efficient. These results indicate that Li penetration



Fig. 1. Drive voltage and EQE dependence on (A) Li thickness for: ITO|MoO_x(20 Å)|NPB(600 Å)|Alq₃(400 Å)|Li(x)|Ag devices, and (B) Alq₃ thickness for: ITO|MoO_x(20 Å)|NPB(600 Å)| Alq₃(x)|Ag devices. The voltage and EQE were measured at 20 mA/cm².

into Alq₃ is limited, probably less than 50-100 Å, and that Ag penetration into Alq₃ can be effectively blocked by an interlayer of Li as thin as ~ 1 Å or a Li-doped Alq₃ layer.

We investigated in more detail the effect of Li penetration into Alq₃ using a device structure - ITO|MoOx|NPB|Alq₃(400 Å-x)| Li(4 Å)|Alq₃(x)|Ag - where Li is deposited in between two separate Alg₃ layers. As shown in Fig. 2, the EQE remains essentially constant (~1.0%) as long as the Li laver is spaced about 100 Å from the NPB|Alq₃ interface, below which the EQE drops sharply. This sharp loss of EQE with the proximity of the Li layer near the NPB|Alq₃ interface can only be due to quenching from the Li layer. In addition, the Ag electrode can also contribute to the quenching as the Li layer near the NPB|Alq₃ interface may not be able to shield the effect of Ag completely. The linear rise in drive voltage from about 6 to 12 V can be attributed to the formation of an additional Li|Alq₃ interfacial barrier opposing the electron transport from the Ag cathode. These results suggest that Li penetration into Alq₃ by vapor deposition is limited to at most 50-100 Å, in agreement with the previous results [1,2].

The difference in the penetration depths of Ag and Li into Alq₃ can be understood in terms of the various degrees of chemical interactions and formation of species between the metals and the organic layer. Calculations haven shown that Alq₃ interacts with Li strongly to form Li⁺-Alq₃ [17], resulting in a short penetration depth. The chemical affinity between Alq₃ and Ag is known to be



Fig. 2. Drive voltage and EQE for: ITO $|MoO_x(20 \text{ Å})|NPB(600 \text{ Å})|Alq_3(400 \text{ Å}-x)|Li(4 \text{ Å})|$ Alq_3(x)|Ag devices. The voltage and EQE were measured at 20 mA/cm².

low, which would explain the deep penetration of Ag into Alq₃ [3–5]. For Ag deposited on Bphen, an Ag⁺-Bphen⁻ species has been identified, indicating chemical interaction and low Ag penetration on Bphen [6,18]. Ag clusters are formed as excess of Ag is deposited on top of Bphen with the effect of preventing Ag penetration into the Alq₃. A similar behavior is expected for Li, where the Li clusters forms the barrier layer to prevent the penetration of Li or other subsequent metal, such as Ag, to diffuse into the Alq₃ layer. For example, Ag penetration into trimethylcyclohexane polycarbonate can be prevented by inserting a Cr layer, which has a higher affinity with the polymer than Ag and the tendency to form Ag cluster from the subsequent metal atoms [19,20].

Following the study of Li and Ag penetration on Alq₃ in OLEDs with a NPB|Alq₃ bi-layer structure, we examined such an effect on OLEDs with a NPB|Alq₃|Bphen tri-layer structure, where Bphen is an additional electron-transport layer. Two sets of devices were prepared: A) ITO|MoO_x|NPB|Alq₃|Bphen(100 Å)|Li(x)|Ag, where Li was varied from 0 to 500 Å; and B) ITO|MoO_x|NPB|Alq₃|**Bphen(x)**| Li(4 Å)|Ag, where Bphen was varied from 0 to 100 Å. NPB and Alq₃ were fixed at 600 Å and 400 Å for all the devices, similar to previous experiments. As shown in Fig. 3A for Set A devices, EQEs as high as 1.2% are obtained with 100 Å Bphen even with a Ag-only (without Li) cathode. This result alone suggests that the penetration of Ag into Bphen is quite limited, compared to Ag into Alg₃. The formation of the Ag⁺-Bphen⁻ species and subsequent Ag clusters at the Bphen|Ag interface prevent the metal penetration [6,18]. It can also be seen that the effect of Li on these tri-laver NPB|Alg₃|Bphen devices is marginal as the EOE remains relatively flat at ~1.2% from 0 to ~50 Å of Li. Beyond this range, both voltage and EOE deteriorate due to Li reactions with residue oxygen and moisture as observed in previous experiments. Results of the Bphen thickness dependence for Set B devices with Li|Ag and Ag-only electrodes are shown in Fig. 3B. It can be seen that for the Ag-only devices the drive voltage first decreases with increasing Bphen thickness (from 7.1 to 5.7 V in the 0 to 20 Å range) and then increases to 8.3 V at 100 Å Bphen. In comparison, the drive voltages of Li|Ag devices are lower and exhibit much less dependence on the thickness of Bphen. At 100 Å Bphen, the drive voltage remains at 6.5 V. The behavior of EQE is quite different. The presence of Bphen, even as little as 10 Å, is needed to recover the high EQE values for the Ag-only devices. With Li|Ag as cathode, high EQE is obtained even for devices without Bphen, as previously shown for the bi-layer NPB|Alq₃ devices. In a sense, Bphen or Li may be considered as a "buffer" layer with the effect of moderating the penetration of Ag into Alq₃, which is seen as the root cause of EQE depression in NPB|Alq₃ devices. Li, in addition, facilitates electron injection into Bphen by pushing the Fermi level towards the LUMO, resulting in a reduced drive voltage



Fig. 3. Drive voltage and EQE dependence on (A) Li thickness for: ITO $|MoO_x(20 Å)|NPB(600 Å)|Alq_3(400 Å)|Bphen(100 Å)|Li(x)|Ag devices, and (B) Bphen thickness for: ITO<math>|MoO_x(20 Å)|NPB(600 Å)|Alq_3(400 Å)|Bphen(x)|Li(4 Å)|Ag devices.$ The voltage and EQE were measured at 20 mA/cm².

compared to the devices without Li [1]. The formation of Li⁺Bphen⁻ and Ag⁺Bphen⁻ would explain the buffer behavior of Li and Bphen with respect to Ag penetration.

The diffusion of Li in Bphen was further evaluated using a device structure -ITO|MoO_x|NPB|Alq₃|**Bphen(100** Å-x)|Li(4 Å)|**Bphen(x)**| Ag, where a layer of Li is deposited between two layers of Bphen. As shown in Fig. 4, both the drive voltage and EOE are relatively unaffected by the position of the Li laver in Bphen, whether it is deposited on top of the Alq₃ or anywhere in the Bphen layer. There is only a small rise in voltage (~1 V) with Li directly on the Alq₃ layer, as the Li-doped Bphen layer formed is supposedly located further away from the Ag cathode. This result differs from the Li deposited between two layers of Alq₃ experiment in Fig. 2, where the voltage increased linearly, as the Li layer is located closer to the NPB interface. The fact that the EQE is almost constant (~1.2%) further supports the role of Li blocking Ag diffusion or penetration during deposition, forming a Li⁺-Bphen⁻ complex and subsequent Li clusters [1,6,18]. The results also suggest that Li is more diffusive in Bphen than in Alq₃. Li apparently can diffuse into Bphen even as the Bphen layer is deposited on top of it. A diffusion length of 50–100 Å of Li in Bphen can be estimated.

3.2. Tandem devices

Li has been used in tandem OLEDs as an efficient electron-



Fig. 4. Drive voltage and EQE dependence on the location of Li layer for: ITO $|MoO_x(20 \text{ Å})|NPB(600 \text{ Å})|Alq_3(400 \text{ Å})|Bphen(100 \text{ Å}-x)|Li(4 \text{ Å})|Bphen(x)|Ag devices. The voltage and EQE were measured at 20 mA/cm².$

injecting component in the charge-generation layer (CGL). However, its formulation and diffusion characteristics in the adjacent layers, which could affect the tandem device performance including stability, have not been studied in detail. Here in this study we used Li|MoOx as the CGL in a tandem device -ITO $|MoO_x(20 \text{ Å})|NPB|Alq_3|Bphen(100 \text{ Å})|Li(x)|MoOx(20 \text{ Å})|$ NPB|Alq₃|Bphen|Li(4 Å)|Ag, where Li is varied from 0 to 500 Å. As shown in Fig. 5A, a strong Li thickness dependence is observed. The drive voltage continuously decreases from 23.3 V without Li to 12.8 V with 50 Å of Li. Beyond 50 Å, the voltage begins to rise due to the formation of an insulating LiO_x or Li(OH)_x layer as explained previously for the single-stack devices. The EQE remains mostly constant at ~1.2% (similar to what was observed for the single-stack devices) for Li thicknesses below 1 Å, indicating that Li at this thickness is not effective as an electron-injecting component of the CGL. Above 1 Å and up to 100 Å, the EQE reaches a value between 1.9% and 2.2%. In the 5-50 Å range, the drive voltage and the EQE of the tandem OLEDs are approximately twice those of the singlestack devices, indicating that the CGL is functioning properly as expected.

Our experiments on single-stack OLEDs have shown that Li can diffuse approximately 50–100 Å into Bphen, irrespective of the order of Li and Bphen depositions. This is also confirmed in tandem OLEDs according to Fig. 5B, which plots the thickness dependence of Bphen for a set of tandem devices – ITO|MoO_x(20 Å)|NPB|Alq₃|Li(50 Å)|**Bphen(x)**|MoO_x(20 Å)|NPB|Alq₃|Bphen|Li|Ag, where the Bphen layer is sandwiched between the Li layer of the front subcell and the MoO_x layer of the back subcell. With Li|B-phen|MoO_x as the CGL, no significant variations in both the drive voltage and EQE are observed as long as the Bphen layer is kept below 100 Å – the diffusion length of Li in Bphen. The steep rise in the drive voltage with 200 Å Bphen is due to the presence of an undoped Bphen layer separating the electron-injecting Li-doped Bphen layer and the hole-injecting MoO_x layer.

4. Conclusions

Through studies of single-stack and two-stack tandem NPB|Alq₃|Bphen OLED devices, we have found that Ag can diffuse or penetrate deep into Alq₃ layer during vapor deposition of Ag on Alq₃ and cause luminescence quenching. The penetration depth can be as high as 2000 Å. This penetration can be substantially prevented by a thin layer of Li or Bphen deposited on Alq₃ prior to the deposition of the Ag electrode. We also found that Li diffusion in either Alq₃ or Bphen is limited to about 50–100 Å. Li also appears to be able to diffuse into Bphen irrespective of the order of Li and



Fig. 5. Drive voltage and EQE dependence on (A) Li thickness for: ITO|MoO_x(20 Å)|NPB(600 Å)|Alq₃(400 Å)|Bphen(100 Å)|Li(**x**)|MoO_x(20 Å)|NPB(600 Å)|Alq₃(400 Å)|Bphen(100 Å)|Li(4 Å)|Ag tandem devices, and (B) Bphen thickness for: ITO|MoO_x(20 Å)|NPB(600 Å)|Alq₃(400 Å)|Li(50 Å)|Bphen(**x**)|MoO_x(20 Å)|NPB(600 Å)|Alq₃(400 Å)|Bphen(100 Å)|Li(4 Å)|Ag tandem devices. The voltage and EQE were measured at 20 mA/cm².

Bphen depositions.

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