



Fabricating large-area white OLED lighting panels via dip-coating



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ABSTRACT

Large-area lighting panels based on white organic light-emitting diodes (OLEDs) with emission area of $72 \times 72 \text{ mm}^2$ have been fabricated in air through dip-coating process. By studying the effects of the solution concentration, the panel withdrawal speed, as well as improving the panel structure, uniform hole transport layer and emitting layer are deposited with appropriate thickness. The 9-point luminance uniformity and the opto-electrical performance of the dip-coated OLED panel are as same as that of the spin-coated panel. However, the spin-coated panel exhibits dim emission at the corner region and the metal grid region due to panel rotation and material pile-up, which the dip-coated panel shows a uniform light emission across the whole panel.

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1. Introduction

Since the discovery of organic light-emitting diodes (OLEDs) [1,2], OLEDs have been successfully commercialized in the applications of flat-panel displays and solid-state lighting [3,4]. To date, most of the small-molecule based OLEDs are fabricated by thermal vacuum evaporation [5–9], leading to high production cost and low yield [10–12]. On the contrary, all the polymer based OLEDs are fabricated through solution process [11,13–21], such as spin-coating, ink-jet printing, screen printing, slot-die coating, dye diffusion, micro-contact printing, photolithographic process, etc. Compared to vacuum deposition, solution process is simple, cost-effective, and easy to scale up.

Among all the solution processes, dip-coating was first commercially applied to produce thin films in 1939 [22]. Dip-coating is generally carried out by immersing a substrate into a reservoir of solution, followed by withdrawing the substrate from the solution bath. It can deposit a thin film onto different substrates, such as plate, cylinder, or irregularly-shaped objects. In recent years, dip-coating has been utilized in nanotechnology to align the nanowire arrays [23,24], and in organic electronics to fabricate solar cells [25–28], field-effect transistors [29,30] and OLEDs [18,31]. However, dip-coating process has yet to be used in

the large-area OLED lighting panel fabrication.

In our contribution, we successfully fabricated large-area white OLEDs lighting panel through dip-coating process. The size of the panel is $86 \times 86 \text{ mm}^2$ with emitting area of $72 \times 72 \text{ mm}^2$ (4 inch in diagonal). The device architecture is bi-layer structure with poly(3,4-ethylenedioxythiophene):poly(styrene sulfonate) (PEDOT:PSS) as the hole injection layer (HIL), and a blend of three phosphorescent iridium complexes within a poly(*N*-vinylcarbazole) (PVK)/1,3-bis[(4-*tert*-butylphenyl)-1,3,4-oxadiazolyl] phenylene (OXD-7) host matrix as the emission layer (EL). By varying the solution concentration and withdrawal speed as well as improving the substrate structure, uniform HIL and EL layers are dip-coated in sequence. The film thickness of HIL and EL is respectively 40 nm, and 80 nm. The luminance uniformity reaches 83%. Compared to spin-coating process, dip-coating process significantly reduces the material pileup along the metal grids, and uses much less material. The light emission uniformity and performance of the 4 inch OLED lighting panel fabricated by dip-coating process are on a par with the panel fabricated by spin-coating.

2. Material and methods

2.1. Materials

PVK and CsF were purchased from Sigma Aldrich. Iridium bis(2-

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(4,6-difluorophenyl)-pyridinato- N,C_2') picolinate (Flrpic, blue emission), iridium tris(2-(4-tolyl)pyridinato- N,C_2') ($Ir(mppy)_3$), green emission), iridium(III)bis(2-methyldibenzo-[f,h]quinoxaline)(acetylacetonate) ($IrMDQ_2(acac)$, red emission) and OXD-7 were purchased from Luminescence Technology Corp. The PEDOT:PSS (CLEVIOSTTM P VP AI 4083) was purchased from Heraeus Electronic Materials Division. All chemicals and materials were purchased and used as received unless otherwise noted.

2.2. Device fabrication and characterization

The ITO glass substrates provided by Guangzhou New Vision Opto-Electronic Technology Co Ltd., have a 150 nm transparent indium-tin-oxide (ITO) layer on top with sheet resistance of 20 Ω per square. In order to improve the current injection from the edge of substrates to its central region, a 300 nm thickness and 40 μ m width auxiliary metal electrode Mo/Al/Mo with sheet resistance of 0.1 Ω per square was deposited and grid-patterned on the ITO surface. The metal grids were then covered with an insulating material to prevent electrical shorts. The thickness of the grids with the insulating material is about 1.5 μ m.

To fabricate lighting panels, the substrate was first cleaned by 20 min UV ozone followed by 5 min oxygen plasma. For spin-coated panels, a 40 nm-thick PEDOT:PSS layer was spin-coated onto the substrate followed by baking at 140 $^{\circ}$ C in nitrogen for 20 min. The EL layer was spin-coated from PVK:OXD-7:Flrpic: $Ir(mppy)_3$: $IrMDQ_2(acac)$ (weight ratio: 100:40:10:0.04:0.30) chlorobenzene solution to form a 80 nm-thick film and annealed at 120 $^{\circ}$ C in nitrogen for 30 min. For dip-coated panels, both PEDOT:PSS layer and EL layer were withdrawn at optimized speed (6.0 mm/min) in air. In order to obtain the same films thickness for both types of panels, the dip-coated solution had a higher concentration than the spin-coated solution. The annealing conditions of PEDOT:PSS and EL were same for both types of panels. After EL annealing, the electron injection layer (CsF, 1 nm thickness) and top electrode (Al, 150 nm thickness) were evaporated in sequence at a base pressure of 1×10^{-4} Pa. Before testing, the panels were encapsulated by a glass cap with desiccants inside. The small-area control devices used ITO-coated glass from the China Southern Glass Holding Corp. with a sheet resistance of 20 Ω per square. The fabrication process was as same as that for spin-coated panels. The area of the standard device is 0.15 cm² defined through a shadow mask.

The film thickness was determined by a Dektak 150 surface profiler. The optical microscopy images were obtained by a Nikon ECLIPSE E600 POL. The current density (J)-bias (V)-luminance (L) characteristics were measured using a Keithley 2400 source meter and a Konica Minolta Chroma Meter CS-200. The luminance of 9-point uniformity was measured by the Konica Minolta Chroma Meter CS-200. The EL spectra and CIE coordinates were taken using a Photo Research PR-705 spectrometer.

3. Results and discussion

3.1. Dependence of film thickness and uniformity on the withdrawing speed and the concentration of solution

The thickness and the uniformity of the film dip-coated from solution is determined by many factors, including the concentration of the solution, the withdrawing speed, the substrate's surface characteristics, the contact angle of the solution on the substrate, the solution's temperature, etc. [32] The thickness and uniformity of PEDOT:PSS films deposited on the 4 inch panel with different withdrawal speeds are shown in Fig. 1(a). The film thickness increases as the withdrawal speed increases due to the viscous drag

of the moving substrate. Since the viscous drag of the substrate is proportional to the withdrawal speed, at higher withdrawal speed, the larger drag takes more solution with the substrate, leading to a thicker film. The relationship between the film thickness and the withdrawal speed can be expressed by a power law in the form of $h = U^{\chi}$, in which h is the center film thickness, U is the withdrawal speed, and χ is the power [18]. The experimental results are well fitted by the empirical equation as shown in Fig. 1(a), with χ is around 0.66 for PEDOT:PSS film.

The uniformity of the films are characterized by 9-point non-uniformity [33], which is calculated by Non-Uniformity = $(1 - h_{\min}/h_{\max}) \times 100\%$, where $h_{\min}$ is the minimum thickness and $h_{\max}$ is the maximum thickness among the nine points. The Uniformity is therefore defined as $1 - \text{Non-Uniformity}$. As illustrated in Fig. 1(a), the non-uniformity increases as the withdrawal speed increases. When the withdrawal speed increases from 0.5 mm/s to 1 mm/s, the non-uniformity increases from 25% to 49%. Reducing the withdrawal speed to 0.06 mm/s, the non-uniformity could be lowered to 15%. As the withdrawal speed increases, the high speed induces more panel vibration. The panel vibration makes the solution unstable, which results in a worse uniformity.

Fig. 1(b) shows the dependence of the thickness and the uniformity of PVK film dip-coated at a fixed speed of 0.1 mm/s on the concentration of the solution. It's observed that the thickness monotonically increases with the solution concentration. As the solution concentration increases, so does the viscosity. A larger viscosity results in a greater viscous drag, thereby leading to a thicker film. Table 1 shows the viscosity of solution with different solution concentration. Besides the viscosity, the greater the concentration of the solution, the more the solute left on the substrate, which also makes the film thicker.

The film's non-uniformity does not change much as the concentration of the solution increases from 15 mg/mL to 25 mg/mL. However, increasing the concentration to 30 mg/mL sharply increases the non-uniformity from 22% to 37%. By carefully examining the coated film on the panel (Fig. 1(b) inset), it's found that there is material accumulation at the initial stage of dip-coating process causing the large non-uniformity. The material accumulation can be completely removed by improving the substrate layout design.

3.2. Improvement on the film uniformity by the substrate layout design

At the initial stage of dip-coating process, the solutes always accumulate at the top portion of the substrate. The material accumulation could be alleviated by reducing the withdrawal speed or lowering the solution concentration as shown in Fig. 1. Moreover, it can be eliminated by designing the substrate properly. To make the panel, we pattern four 4 inch panels on a 200 $\times$ 200 mm mother glass. Instead of cutting a 86 $\times$ 86 mm panel, we cut out a 86 $\times$ 129 mm panel, which sacrifice a panel on top of the studied panel. The panel picture is shown in Fig. 2(a). The additional half panel is used as the buffer zone along the withdrawal direction. At the initial stage of dip-coating, the material will accumulate on the buffer zone which guarantees the film thickness uniformity on the light emission area. Without the buffer zone, the material accumulation could be clearly observed on the upper part of the panel shown in Fig. 2(b). With the additional buffer zone, the active area exhibits a uniform film deposition shown in Fig. 2(a).

3.3. Morphologies of dip-coated and spin-coated organic films

Due to the limiting conductivity of the transparent anode ITO, the low-resistance auxiliary metal grids are necessary to improve

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