



Ultrasonic spray coating as deposition technique for the light-emitting layer in polymer LEDs

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ABSTRACT

In this work the ultrasonic spray coating technique is introduced as an alternative wet solution process for the deposition of the (Super Yellow) light-emitting layer for polymer light-emitting diodes (PLEDs). An investigation on the use of this coating technique in ambient conditions is performed and a comparison with spin coated PLEDs in inert atmosphere is made. Uniform low roughness thin films with a typical thickness of 80 nm are obtained by varying the polymer–solvent mixture and spray coater parameters. PLEDs are produced and reach a luminous power efficacy in the order of 10 lm/W. Through the use of various optical and analytical techniques it is demonstrated that the applied ultrasonic atomization has no noteworthy influence on the original properties of the polymer and on the resulting PLED's efficacy. Ultrasonic spray coating is therefore a viable deposition technique for the production of PLEDs.

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1. Introduction

Organic light-emitting diodes (OLEDs) have the potential to be the next generation of solid-state lighting and flat panel displays [1,2]. These OLEDs can be produced by solution processing of polymers (PLEDs) [3], and by evaporation [4] or solution processing [5] of small molecules. Solution processable PLEDs hold great promise in terms of processing speed and cost, manufacturing simplicity, and roll-to-roll (R2R) and large-area production [6]. Spin coating is the laboratory standard deposition method to produce PLEDs. However, the spin coating technique is not scalable to large area production nor is it compatible with high throughput

and R2R production [7]. Although spin coating is a wasteful process, it is considered to be the most reliable and reproducible deposition method to yield uniform thin films. Spray coating [8] is a large-surface high-throughput technique used in a variety of sectors, e.g. medical, automotive, electronics, and food processing. In recent years, polymer-based organic solar cells (OSCs) were successfully (ultrasonically) spray coated [9–14]. Also transparent electrode materials like high conductive poly(3,4-ethylenedioxythiophene):poly(4-styrenesulfonate) (PEDOT:PSS) [15], zinc oxide (ZnO) [16], single wall carbon nanotubes (SWCNTs) [11] and nanoparticle-based silver top contacts [17] were ultrasonically spray coated to produce efficient OSCs. In this paper we investigate ultrasonic spray coating as an alternative deposition method toward fully printed PLEDs. We have developed a process for the ultrasonic spray deposition of thin and uniform films of the workhorse PLED material

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Super Yellow [18]. Furthermore PLED devices were made and characterized based on these findings. A set of analytical techniques was applied to determine the impact of ultrasonic spray deposition on the intrinsic polymer properties.

2. Experimental

All polymer LEDs presented in this article were prepared using Super Yellow (PDY-132), obtained from Merck, as the active polymer layer. The complete device structure, as illustrated in Fig. 1a, consists out of glass, indium tin oxide (ITO), PEDOT:PSS, Super Yellow, calcium and aluminum. The ITO coated (100 nm) glass substrates (Kintec, sheet resistivity 20 Ω/sq) were exposed to a standard cleaning procedure using soap, demineralized water, acetone and isopropanol, followed by a UV/O₃-treatment for 15 min. Hereafter, the PEDOT:PSS layer (~30 nm) was deposited by spin coating in ambient conditions and annealed in inert atmosphere for 10 min at 120 °C.

The Super Yellow emitting layer was either deposited by spray coating or by spin coating to compare the effects of the deposition techniques used. The spin coated active layers were prepared in an inert atmosphere glovebox, while the spray coated active layers were deposited in ambient conditions. The metal cathodes were deposited by evaporating Ca and Al, with layer thicknesses of ~30 nm and ~80 nm, respectively, at a pressure of 1×10^{-6} mbar in an inert atmosphere glovebox. The resulting polymer LEDs had an active area of 25 mm². The Super Yellow solutions were made in the glovebox and stirred overnight at 50 °C. The ultrasonically spray coated Super Yellow layers were deposited by a Sono-Tek ExactaCoat system using an Accu-mist spray nozzle, as illustrated in Fig. 1b. As the ultrasonic nozzle passes over the substrate (Fig. 1c) the individual droplets merge and form a full wet layer. The wet layer was then annealed in an inert atmosphere at 50 °C for 15 min. The Super Yellow solution used in our spray coated PLEDs had a concentration of 2.5 mg/mL in 1,2-dichlorobenzene (oDCB). The solution for the gel permeation chromatography (GPC) measurements was prepared by dissolving Super Yellow in tetrahydrofuran (THF) with a concentration of 1.0 mg/mL. The thicknesses of the polymer films were measured using a Dektak 3 ST Profilometer. The

PeakForce QNM measurements were done on a Bruker Multimode 8 atomic force microscope (AFM) in combination with a V series controller using a soft cantilever with a force constant of 0.4 N/m. The current, voltage and luminous flux characteristics were measured using a Keithley 2401 source meter and an absolute calibrated integrating sphere spectrometer from Avantes.

Analysis of the molar masses and molar mass distributions of the (pristine and ultrasonically treated) polymer samples was performed on a Tosoh EcoSEC System comprising of an autosampler, a PSS guard column SDV (50 \times 7.5 mm), followed by three PSS SDV analytical linear XL columns (5 μm , 300 \times 7.5 mm) and a UV detector (254 nm) using THF as the eluent at 40 °C with a flow rate of 1.0 mL min⁻¹. The UV–VIS absorption measurements were performed on an Agilent CARY 500 Scan UV–VIS–NIR spectrophotometer using Super Yellow spin coated films on glass substrates from pristine and ultrasonically atomized solutions. The IR transmission spectra were collected on a Bruker Tensor 27 with a resolution of 4 cm⁻¹ (16 scans) using films drop-casted on a NaCl disk from chlorobenzene solutions of the pristine and ultrasonically sprayed Super Yellow polymer. ¹H NMR spectra were acquired on a Varian Inova 400 spectrometer (Agilent Technologies, Santa Clara, California, US) in a 5-mm four-nucleus PFG probe. The ultrasonically sprayed Super Yellow sample was prepared by precipitation of the spray coated chlorobenzene solution in methanol followed by filtration and overnight drying under vacuum. Both the pristine Super Yellow and the obtained sample from spray coating were then dissolved in CDCl₃. NMR chemical shifts (δ , in ppm) were determined relative to the residual CHCl₃ (7.26 ppm).

3. Results and discussion

The formation of thin polymer films by the ultrasonic spray coating technique (Fig. 1b) is achieved by the spreading and merging of micro-meter sized droplets and subsequent solvent evaporation. Ultrasonic nozzles employ standing waves to atomize solutions. These standing waves are a result of the mechanical vibrations produced by the (ceramic) piezoelectric transducers inside the ultrasonic nozzle. As the solution is fed to and emerges onto the

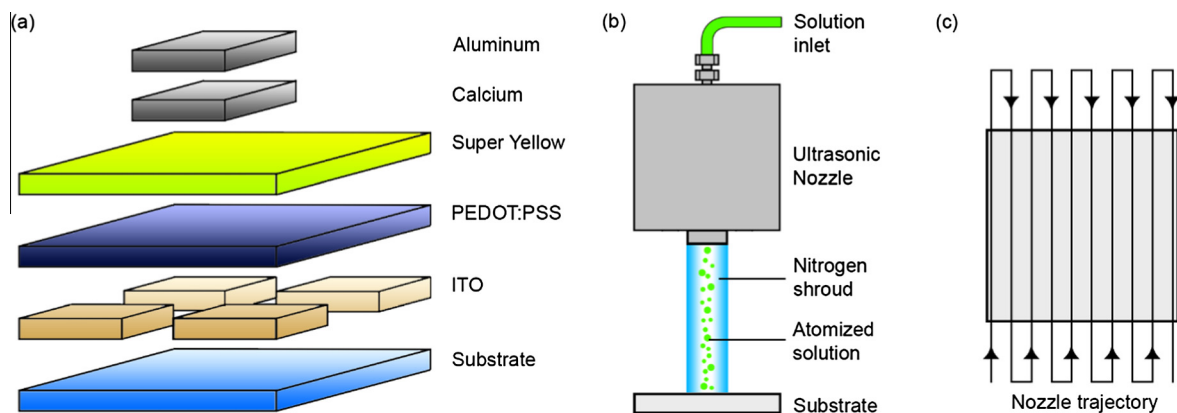


Fig. 1. (a) PLED device structure. (b) Ultrasonic spray deposition principle. (c) The nozzle trajectory over the substrate.

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