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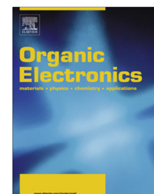

Letter

High-performance barrier using a dual-layer inorganic/organic hybrid thin-film encapsulation for organic light-emitting diodes

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ABSTRACT

We report an inorganic/organic hybrid barrier that combines the alternating deposition of a layer of ZrO_2 using low temperature atomic layer deposition and a 16-μm-thick layer of UV-curable NOA63 epoxy using spin-coating. The effective water vapor transmission rates of single ZrO_2 film was improved by adding solution epoxy from 3.03×10^{-3} g/m^2 day to 1.27×10^{-4} g/m^2 day in the hybrid NOA63/ZrO_2/NOA63/ZrO_2 films at 20 °C and a relative humidity of 60%. In consequence, the organic light-emitting diodes encapsulated with inorganic/organic hybrid barriers were undamaged by environmental oxygen and moisture and their luminance decay time improved by a considerable extent.

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1. Introduction

In recent years, organic light emitting diodes (OLEDs) have become increasingly poised to play a significant role in future technologies due to their various potential applications in solid-state lighting and flat panel displays [1–6]. However, one of the primary technical challenges that remain in the implementation of OLEDs in practical applications is to achieve long lifetimes and high reliability [7,8]. It is a common property of most small molecule materials to experience degradation of their electrical properties upon exposure to moisture and/or oxygen found in the atmosphere [9]. In addition, electrical contacts to the active layers are also susceptible to degradation when exposed to water vapor and oxygen, resulting in decreased charge injection or collection in the devices. Thus, when OLEDs are operated under ambient atmosphere, the formation of dark spots leads to complete device degradation within a few hours [10].

Since OLEDs are vulnerable to moisture and oxygen, ensuring a reasonable device lifetime requires encapsulation of the device to protect it from water and oxygen. Thin film encapsulation (TFE) is a superior technology to the presently used glass encapsulation method, since it can produce thinner devices and does not require the use of getter and sealant materials [11]. TFE is often created using high-performance inorganic film barriers or alternating multilayers consisting of organic and inorganic materials. However, for organic active layers with low glass-transition temperatures and thermal stability, the temperatures at which the barrier layers can be deposited are limited. This inevitably requires that the inorganic layers used in TFE be deposited at low temperatures if they must be deposited directly on the device [12]. However, processing at low temperature may lead to more defects in the films, limiting the overall performance of the barrier [13]. Thus, the development and

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integration of high-performance barrier encapsulation films with organic electronics remains a challenging endeavor.

Atomic layer deposition (ALD) is a technique consisting of a series of self-limiting, surface-saturated reactions that form thin dense films at low temperatures (<200 °C) [14,15] and appears to be a promising technique for the preparation of encapsulation layers on top of organic electronic devices. Al_2O_3 films are the most commonly used material in TFE for organic devices because they are easy to fabricate at low temperatures [15]. However, it has been recently reported that corrosion of neat Al_2O_3 layers occurs in the presence of H_2O, which causes deterioration and affects barrier functionality. Based on the concepts of Al_2O_3/SiO_2 [16] and Al_2O_3/ZrO_2 [17] nanolaminates, the authors were able to use SiO_2 and ZrO_2 to protect Al_2O_3 and reduce corrosion substantially. Among the existing high-performance barrier inorganic materials, ZrO_2 is considered to be one of most promising material because it exhibits a relatively high chemical stability when compared to Al_2O_3 [18], is amenable to low-temperature, simple fabrication by ALD, and is compatible with the manufacturing of integrated circuits.

It especially is well known that most single inorganic compounds cannot satisfy the stringent encapsulation requirements of organic devices and are often used in complex multilayer architectures [19,20]. The multilayer film approach was developed to circumvent the defect issues that limit the barrier performance of single-layer films. By applying multilayer films with alternating materials, defects that span the entire thickness of individual inorganic layers are rescued by the addition of another layer and do not channel continuously through the film structure [21].

In a previous study [22], we made a UV-curable epoxy (NOA63 from Norland Optics) film as a passivation layer for the encapsulation of OLEDs. We observed that single NOA63 film layers deposited by spin-coating technology that were microns in thickness were able to act as water barriers. 16 μm-thick layer possessed low shrinkage and an average root-mean-square roughness of 1.2 nm, and the maximum difference in height (peak-to-valley) is about 1.9 nm. The corresponding diffusion and solubility coefficient derived from lag time were 9.36×10^{-12} cm^2/s and 0.527 g/cm^3/atm, respectively. The water vapor transmission rate (WVTR) of 16 μm-thick NOA63 film was 0.031 g/m^2 day at 20 °C, under 50% relative humidity (RH) and standard atmospheric pressure. This WVTR is among the lowest obtained using a single organic or polymer passivation layer.

In the present study, we investigated inorganic/organic hybrid multi-layer barriers for the encapsulation of OLEDs using alternative layers of ZrO_2 deposited by ALD and NOA63 film. Here, we adopted epoxy NOA63 as an organic layer to extend the diffusion path of vapor permeation [21]. We demonstrate that inorganic/organic hybrid thin film encapsulation (TFE) delivers a considerably lower WVTR and longer OLED lifetime when compared with a single ALD inorganic layer.

2. Experimental materials and methods

The ZrO_2 layers were deposited using the ALD method using a LabNano 9100 ALD system (Ensure Nanotech Inc.). The chamber pressure was 3×10^{-2} Pa. Tetrakis (diethylamide) zirconium (IV) and H_2O were used as the precursors of Zr and O, respectively. The warm-wall reactor was operated at 80 °C. High-purity N_2 was used as the carrier gas for the precursors at a flow rate of 20 sccm. NOA63 film was used without further purification and was directly dropped onto the samples, which were mounted on Laurell's WS-650 spin processor. The coating preparation process was as follows. First, the NOA63 viscous solution was spun slowly (500 rpm, 10 s) to distribute it over the entire surface, the solution was then spun quickly at 2000 rpm for 20 s to place a film with final thickness of 16 μm on top of the device. Second, the film was irradiated to form solid passivation using a high-pressure mercury (Hg) lamp (exposed light energy 50 mW/cm^2 at 370 nm). Inorganic/organic hybrid multi-layer barriers were made based on sandwiching repeating layers of the ALD ZrO_2 and spin-deposited NOA63 films. X-ray photoemission spectroscopy (XPS) was performed using a Scienta ESCA 200 spectrometer in ultra-high vacuum with a base pressure of 1×10^{-10} mbar. The measurement chamber was equipped with a monochromatic Al KR X-ray source to provide photons with 1486.6 eV. The scanning electron microscopy (SEM) was performed using a field-emission SEM (JSM-6700F, JEOL) operated at an accelerating voltage of 10 kV. Samples were coated with a thin layer of gold (5 nm) prior to analysis. The phase and crystallinity of the films were determined using X-ray diffraction (XRD, model D/max 2400). High-angle XRD data were collected at 0.05° increments and 15 s count times using films deposited on silicon (Si) substrates.

3. Results and discussion

The chemical bonding of ZrO_2 film on Si substrate was examined using XPS. Fig. 1(a) shows two peaks in the ranges of 181.3 eV and 183.7 eV, which correspond to features observed with Zr $3d_{5/2}$ and $3d_{3/2}$, respectively. The ZrO_2 films showed a Zr $3d_{5/2}$ peak at 181.3 eV, indicating a dominant formation of ZrO_2 [23]. Fig. 1(b) shows the O1s XPS spectra of the ZrO_2 films. The O1s peak was deconvoluted as the sum of two contributing peaks. The main O1s peak component at 529.9 eV was assigned to the lattice oxygen in ZrO_2, whereas the second peak at 531.2 eV was assigned to the surface carbonate/hydroxyl species and adsorbed H_2O [24]. These data indicate that the ZrO_2 film deposited at 80 °C exhibits non-lattice oxygen atoms, which may induce the formation of a non-homogeneous and non-stoichiometric ZrO_2 film. To obtain crystalline information of ZrO_2 films, we performed XRD analysis of 80-nm-thick ALD ZrO_2 films (Fig. 2). As reported by Hausmann and Gordon [25], crystalline ZrO_2 forms when higher deposition temperatures (>100 °C) are used, where both monoclinic and cubic phases exist during the ALD process. In our experimental setup, at a deposition temperature of

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