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Performance and lifetime of vacuum deposited organic light-emitting diodes: Influence of residual gases present during device fabrication

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Understanding the influence of residual gases present during vacuum deposition of organic light-emitting diodes (OLEDs) and their effect on the device lifetime and the electrical characteristics of OLEDs is crucial for advancing industrial fabrication. In order to gain a more in-depth understanding, the influence of residual nitrogen, oxygen, and water vapor on lifetime and electrical characteristics is investigated. This is achieved by introducing the residual gases into the evaporation chamber through a needle valve while monitoring the partial pressures with the help of a mass spectrometer. We find that water vapor introduces a series resistance to the OLED while the other gases do not influence the electric characteristics. The presence of oxygen or nitrogen impacts the lifetime of the OLEDs by the same amount. Water vapor introduces an additional, even faster degradation process within the first hours of OLED operation. The electrically stressed OLEDs are analyzed by laser desorption/ionization time-of-flight mass spectroscopy. We identify the dimerisation of BPhen as well as the complexation reaction of α -NPD with an $Ir(piq)_2$ fragment as sources of device degradation.

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1. Introduction

Due to their applications in lighting and display technology, organic light-emitting diodes (OLEDs) have been attracting considerable attention [1-3]. However, it remains challenging to fabricate OLEDs with sufficient long-term stability in a reproducible manner and being compatible with industrial manufacturing standards. Revealing the degradation processes, especially the chem-

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http://dx.doi.org/10.1016/j.orgel.2014.07.014 1566-1199/© 2014 Elsevier B.V. All rights reserved. ical degradation involved in the aging of OLEDs, is therefore of great interest [4]. In particular the processing conditions, such as the amount of residual gases during the deposition of the materials have considerable influence on the OLED lifetime.

The influence of the chamber pressure during the fabrication of vacuum deposited OLEDs is reported in several publications [5–8]. Helander et al. [5] showed that the background pressure during device fabrication can significantly alter the energy-level alignment at metal/organic interfaces. Böhler et al. [6] found that samples fabricated at high base pressures(1.10^{-6} mbar) show increased formation of black spots, reduced quantum efficiency, and increased turn-on voltage. Although no black spot formation was found for Alq₃ based OLEDs, Ikeda et al. [7]





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reported a significant increase in operation voltage and a drop in luminance during electrical operation of OLEDs produced at higher base pressure values. They suggest that the Alq₃ molecules decompose by an electrochemical reaction with water molecules intercalated during OLED production. Yamamoto et al. [8] analyzed the effect of an initial drop in device luminance during the first five hours of OLED operation compared to reference devices produced under lower base pressure values. A possible correlation of the initial luminance drop to the amount of water molecules was investigated. However, this has been hampered by the fact that multiple types of residual gases were present in the OLED processing chamber.

In this work, we separately investigate the influence of residual nitrogen, oxygen, and water during OLED preparation on the performance and lifetime of p-i-n OLEDs based on the stable red triplet emitter tris(1-phenylisoquinoline) iridium(III) ($Ir(piq)_3$) [9]. In order to control the partial pressures during evaporation, nitrogen, oxygen, or water vapor is added to the processing chamber. This enables us to intentionally contaminate the chamber with these gases. The chemical degradation processes occurring during electrical operation of the OLEDs are investigated by laser desorption/ionization time of flight mass spectroscopy (LDI-TOF–MS).

2. Experimental

A long-living red OLED based on the red triplet emitter tris(1-phenylisoquinoline) iridium(III) (Ir(piq)₃) [10,11] is used as model system for this study. The p-i-n OLED stack consists of 90 nm indium tin oxide (ITO) pre-deposited on glass substrates, a hole transport layer (HTL, 75 nm) of 2,2',7,7'-Tetrakis-(N,N-di-methylphenylamino)-9,9'-spirobifluorene (Spiro-TTB) doped with 4 mol% 2,2'-(perfluoronaphthalene-2,6-diylidene) dimalononitrile (F₆-TCNNQ), an electron blocking layer (EBL, 10 nm) of N,N'-Di(naphthalen-1-yl)-N,N'-diphenyl-benzidine (α -NPD), an emission layer (EML, 20 nm) composed of N,N'-di(naphthalen-1-yl)-N,N'-diphenyl-benzidine (α -NPD) doped with 20 wt% Ir(piq)₃, a hole blocking layer (HBL, 10 nm) of aluminum(III) bis(2-methyl-8-guninolinato)-4-phenylphenolate BAlq₂, an electron transport layer (ETL, 45 nm) of 4,7-diphenyl-1,10-phenanthroline (BPhen) doped with Cesium (Cs), and a 100 nm Al top electrode. The schematic layer structure of the p-i-n OLED and chemical structures of the used materials are shown in Fig.1.

The devices are prepared by thermal evaporation at a base pressure between 1.10^{-7} mbar and 5.10^{-8} mbar. In order to introduce different impurity gases and to adjust the chamber pressure, a needle valve is attached to the evaporation chamber. Oxygen and nitrogen impurities are produced by connecting the needle valve to corresponding gas containers. To intentionally contaminate the chamber with water vapor, nitrogen is fed through a gas-washing bottle which is connected to the needle valve. Due to the fact that the effective cross section of the pumping system is higher for nitrogen than for water, water molecules then accumulate in the evaporation camber. Immediately after preparation, the devices are encapsulated under nitrogen atmosphere using two-component



Fig. 1. Schematic layer structure of the red p-i-n OLED used in this study and the chemical structures, acronyms and molar masses of the used materials.

epoxy glue and cavity glass lids with an implemented hydrophilic getter material.

The exposure of the vacuum chamber to the different impurity gases is limited to the time during which the organic layers are deposited. To exclude the known degradation effects induced by oxide formation at the organic metal interface, the aluminum cathode is evaporated at the same pressure of 5.10^{-7} mbar and approximately the same background gas conditions (nitrogen dominated atmosphere) for all devices [12]. Four production runs with identical layer sequence but different vacuum conditions are conducted. In each production run multiple devices with the same structure are obtained for each condition. Sample-to-sample variation is found to be negligible compared to the effect of different residual gas exposure.

Device 1 is the reference device produced under the best possible vacuum conditions, the mean pressure during evaporation is 5.10^{-8} mbar. Device 2 was exposed to nitrogen while keeping the chamber pressure at a constant value of 1.10^{-7} mbar. Device 3 was exposed to oxygen and Device 4 to water vapor, both at the same mean pressure of 1.10^{-7} mbar.

In order to ensure that the injected gas is the dominating residual gas in the chamber, the composition of the residual gas is monitored with a quadrupole mass spectrometer (QMG220F3 PrismaPlusTM), which records the ion currents at the m/z ratio signals related to H₂O (m/z = 18 u), N₂ and/or CO (m/z = 28 u), O₂ (m/z = 32 u), Download English Version:

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