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A Correlative TOF-SIMS & XPS Protocol For Analysis Of Organic Light-Emitting Diodes Layers

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INTRODUCTION

Since their invention in 1987 by Tang and Van Slyke [1], organic light-emitting diodes (OLEDs) have achieved tremendous progress leading to their commercialization twenty years later. Nowadays, research focuses on the improvement of the devices lifetime and efficiency by introducing new structures, molecules and dopants. Along these developments, the understanding of degradation mechanisms in the organic layers has become essential to increase the devices performances [2]. Composed of thin layers (~100nm in total), the analysis of OLEDs multilayers requires the use of instruments with good depth-resolution as well as a capacity to probe small changes from aging. Time of flight of secondary ion mass spectrometry (TOF-SIMS) and X-ray photoelectron spectroscopy (XPS) have been individually used to study the degradations in the devices [3, 4]. However, despite their complementarity, few studies correlate the results from each characterization techniques.

CORRELATIVE PROTOCOL

We developed a correlative protocol to obtain precise chemical characterization of OLEDs stacks and insight on their degradation mechanisms. By varying the argon cluster ion fluence over the surface, a bevel crater is formed. The analysis of the same crater by TOF-SIMS and XPS enables the correlation of the information from the two techniques. In TOF-SIMS, an image of the surface is obtained by scanning the surface of the crater, which allows the reconstitution of a profile of the different characteristic molecular ions. Then the sample is transferred to the XPS chamber using a transfer vessel under N₂ atmosphere; a line scan along the crater allows the identification of the different layers. High resolution core-level spectra acquired in each layer informs about the chemical environment of each elements. An easy correlation of the TOF-SIMS and XPS results as well as reduced analysis and sputtering damages on buried layers are achieved with this protocol.

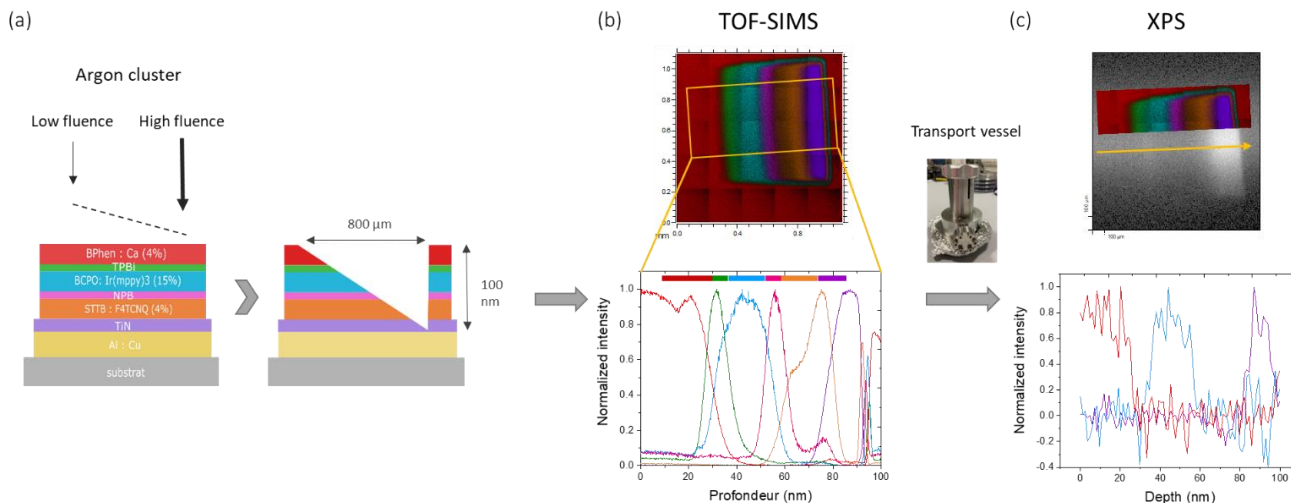


FIGURE 1. Correlative protocol for the analysis of thin multilayer OLED structures: (a) fabrication of the bevel crater by applying an argon cluster ion fluence gradient over the surface, (b) TOF-SIMS scan of the surface and the reconstituted profile and (c) XPS linescan over the surface of the bevel crater.

This protocol also allows tandem MS imaging to be used to resolve ambiguities in peak identification in the layers or to help identify the products of degradation [5]. Figure 2 shows a bevel crater in an OLED stack before and after a few minutes air exposure. The TOF-SIMS image is of the characteristic peak for BPhen. Before air exposure, the signal from this peak is located around the top of the crater that corresponds to its expected position in the stack (red layer in diagram). However, after air exposure this signal is much stronger at the bottom of the crater. To check that this signal really is BPhen and not a mass interference, tandem MS spectra were taken at the top and bottom of the crater and compared to a reference spectrum without air exposure. The similarity in these spectra confirms that at both locations the signal is coming from the BPhen molecule. This suggests that with air exposure the BPhen migrates over the surface of the crater. This is consistent with prior observations that this layer is particularly air sensitive, undergoing oxidation and crystallization rapidly upon air exposure [6]. This example highlights the need for transfer under a protective environment between XPS and TOF-SIMS instruments. When this experiment is repeated, replacing air exposure with a protected transfer between the two instruments there is no change in the position of the BPhen signal.

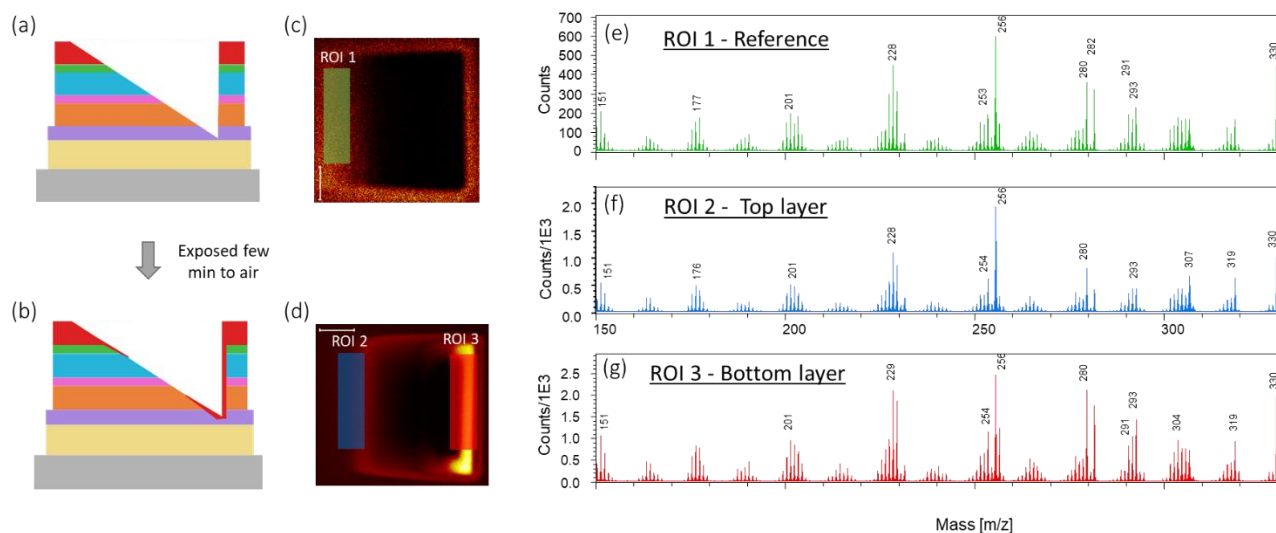


FIGURE 2. Tandem MS imaging of a bevel crater in an OLED stack before and after a few minutes exposure to air. Lateral scheme of the bevel crater (a) before and (b) after air exposure, TOF-SIMS image and ROI selected to compare the spectrum (c) before and (d) after air exposure, (e), (f) and (g) mass spectrum of the different ROI

CONCLUSIONS AND PERSPECTIVES

As well as facilitating the study of ageing in hybrid inorganic/organic stacks such as OLEDs, this bevel crater approach can also be applied to a wide range of applications where TOF-SIMS and XPS depth profiling are required on exactly the same sample area. For inorganic samples, a monoatomic ion beam (Ar, Cs, O, Xe, etc) can be used instead of the argon cluster beam.

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KEYWORDS

OLEDs, TOF-SIMS, XPS, correlative protocol, bevel crater