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CuPc buffer layer role in OLED performance: a study of the interfacial band energies

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Abstract

We present a UV photoemission (UPS) and topographic study of the indium–tin-oxide (ITO) anode used in organic light emitting diodes (OLEDs). The performance of these devices has been shown to be improved by introducing a thin layer of copper phthalocyanine (CuPc) between the anode and the hole-transport layer (HTL). While the device current at constant driving voltage increases with increasing CuPc thickness, the efficiency is optimized at 12–18 nm. In this article we address the issue of the charge (hole) injection process at the anode interface and demonstrate the importance of directly measuring the vacuum levels in quantitative study of the energy levels of OLED interfaces. As a result of this insight, many calculations relating to the relative energies of the bands at OLED interfaces may have to be revised and corrected. The interface morphologies were also studied using AFM to determine any changes with film growth.
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Keywords: OLED; CuPc; UPS; Vacuum level; Energy level; Morphology

1. Introduction

Organic light emitting devices (OLEDs) can be fabricated using small-molecule organic semiconductors deposited in vacuum on a wide variety of substrates [1]. This freedom of choice of materials may lead to the manufacture of inexpensive large-area displays, and much work has therefore been devoted to improving device structures and to understanding their operation. In OLEDs, light is emitted after opposite charges are injected from the electrodes into the organic layers, where they then recombine and form excitons which emit light after a transition from an excited state to the ground or an intermediate state. The injection efficiency is a critical parameter in such devices and depends in an important way on the work function of the electrode. For a given heterojunction between an organic material and indium–tin-oxide (ITO), the oxide work function will, to a first approximation, determine this energy barrier. Previous measurements of the absolute values of the vacuum work function of ITO thin films have yielded different values, ranging from 4.1 to 5.5 eV

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[2–4]. This discrepancy is well known for many metal oxide semiconductors, and it has been documented that the work function is extremely sensitive to the state of a surface [5]. Different surface treatments such as plasma treatments [6], or the grafting of molecules [7,8] induce significant changes in the work function. In recent years, particular attention has been given to the engineering of charge injecting contacts, whose properties are among the important parameters influencing device performance. An important enhancement of device stability was achieved by depositing a copper phthalocyanine (CuPc) layer on the ITO anode [9]. CuPc is a very well known dye pigment with a number of interesting properties; e.g. it is an organic semiconductor, it is chemically and thermally very stable, it can easily form compact and smooth thin films, and it exhibits photoconductivity and catalysis. It has been shown that a CuPc layer inserted between the anode (typically indium–tin-oxide (ITO) on glass) and the hole-transport layer (HTL) of an OLED structure [10,11] produces a significant improvement in device performance, particularly device lifetime [9]. A study of the systematic increase of the CuPc layer thickness from 5 to 25 nm in an ITO/CuPc/NPB/Alq_3/Ca device, has shown that the current density increases with increasing CuPc thickness, but the efficiency has a maximum value at 12–18 nm [12]. Despite this technical progress, the understanding of the basic mechanisms underlying charge injection and transport in CuPc are still very limited. The present study clarifies the role of energy level alignments in this system and possible dependence with CuPc film thickness and also presents a complementary study of the topography of the surfaces by atomic force microscopy (AFM).

The function of CuPc in the hole injection mechanism has been controversial. Some research groups concluded that a CuPc layer of a particular thickness lowers the drive voltage by reducing the effective barrier between the ITO substrate and the HTL (NPB or α-NPD) [13–16]. By contrast, another study has shown that a 15 nm thick CuPc layer, in fact, decreases the hole injection efficiency (which leads to an increase in operating voltage) resulting in an improved balance between the number of injected holes and electrons [17,18]. It was also noted in these studies, that the functioning of the devices depended on CuPc layer thickness. However, it has also been widely suggested that the "staircase" (or equivalently "ladder") arrangement of hole-transport molecular levels promotes charge injection. We have studied this multilayer, i.e. ITO/CuPc/NPB, system with combined ultraviolet photoemission spectroscopy (UPS) and AFM. UPS allows precise measurement of the energetic positions of Fermi and HOMO levels for metallic and organic surfaces, respectively, and therefore of changes in vacuum level position.

2. Experimental

The conductive glass samples of ITO films deposited on Float Glass were supplied by Delta Technologies with a sheet resistivity of 10 $\Omega/\square$. Our substrates were cleaned in the following way. They were initially degreased by scrubbing in detergent (Liqui-Nox®) and deionized water and then were cleaned by ultrasonic agitation successively in deionized water and detergent, deionized water, methanol, isopropyl alcohol and distilled acetone for 15 min each. The sample was then loaded into the load-lock chamber and was UV-ozone treated prior to evacuating the chamber assuring that the substrate was not exposed to atmosphere between ozone treatment and placement in vacuum. The films were deposited at a base pressure of 2×10^{-8} Torr in our home designed deposition and characterization system [5] which is pumped with turbomolecular (deposition chamber) and a sublimator/turbo combination in the characterization chamber. Evaporation rates and also film thicknesses were checked by a calibrated quartz crystal monitor. The organic thin films were deposited from buffered tantalum heating boats at rates ranging from 2 to 6 nm/min. Energy level measurements were carried out in the analysis chamber at a base pressure of better than 2×10^{-10} Torr. This chamber is connected to the evaporation chamber allowing in situ measurements.

The measured value of the work function of ITO strongly depends on its cleanliness and upon the measurement procedure. We have found two

main contributions to the variation in the measured values (4.3–5.35 eV) of the work function of ITO in our laboratory [5]. The range of values we observed in the present work is consistent with what Nüsch et al. [19] found earlier, i.e. 3.9–5.1 eV. The two main contributions are: (i) the presence or absence of hydrocarbon contamination (measured by XPS) which lowers the work function, and (ii) the fluence of the 21.2 eV UV photons in our UPS determination of the work function of ITO. Through elimination of the source of hydrocarbons by using only turbomolecular and sublimator pumping, we further showed that UV irradiation was the dominant source of work function reduction [5] and that to avoid this effect, the irradiation times had to be minimized. The effect probably arises from the ejection of oxygen ions by the energetic secondary electrons produced by the primary 21.2 eV photons. Attention to these details permitted the reproducible determination of stable ITO work functions over periods ranging from hours to days. In the present work, this requirement limited the S/N of the wide scan spectra, which are therefore “noisier” than desirable, but still illustrate the observations and interpretation that we wish to make.

The HeI line at 21.22 eV was used as the excitation source for ultraviolet photoelectron spectroscopy (UPS). The resolution of the UPS measurements was $\approx$200 meV, as determined from the width of the Fermi edge of a very pure Ni plate cleaned in situ by ion-sputtering; changes in WFs and IPs could be determined to about $\pm$50 meV. For the UPS measurements, the samples were biased at -12 V dc to separate secondary electrons from the spectrometer from the genuine secondary cut-off from the sample. Sputtered clean polycrystalline nickel and also in situ evaporated Al films were used to find the Fermi level as reference for ITO. AFM topography measurements were performed ex situ using the tapping mode of a Digital Instruments Nanoscope IIIA AFM.

UPS spectra of ITO substrates cleaned the way mentioned above, were studied to determine the Fermi edge and vacuum level [20]. 2 nm thick organic films were deposited on ITO or other surfaces of interest, to measure the shift in the valence band maxima and vacuum levels, which permits the determination of the barrier height. To measure ionization potentials we used organic films thicker than 10 nm.

3. Results

The configuration of the thin films studied, and the molecular structures of the materials used are shown in Fig. 1. As discussed above and reported in a companion paper [5], we have succeeded in preparing and testing high work function ITO. Typical stable values for ITO work function were 5.1–5.35 eV. Values as high as these are rarely reported due to the difficulty of keeping surface conditions unchanged in a typical experimental environment.

Fig. 2 shows typical UPS spectra for two ITO substrates with different work functions. The difference between the features of these spectra is obvious from the two plots and XPS scan of the substrates (not shown here) also indicates a higher level of carbon present on the low work function ITO [1] surface. (We again emphasize that the relatively poor S/N of the spectra arises from the need to minimize the fluence of UV photons.)

UPS spectra of CuPc films of successively increasing thickness, represented in Fig. 3, were acquired, indicating the validity of the assumption of dipole formation and vacuum level shift (VLS). [2] The ITO features are suppressed even after depositing only 2 nm of CuPc; this ensures that we are looking at the interface region. Also, since the UPS probing depth is about 1–2 nm, it is an indication that CuPc wets ITO very well. The valence band electron energy features (peaks around -6 and -9 eV) remain almost unchanged (within 0.1–0.2 eV) with increasing CuPc thickness, but the uncertainty introduced by the noise level prevents us from determining whether this shift is significant; it presumably indicated that a near-flat-band situation occurs at this interface. Also shown in Fig. 3 as the inserts, are narrow scan spectra (short data acquisition times) of

[1] For details refer to [5].

[2] A complete explanation can be found in [20].

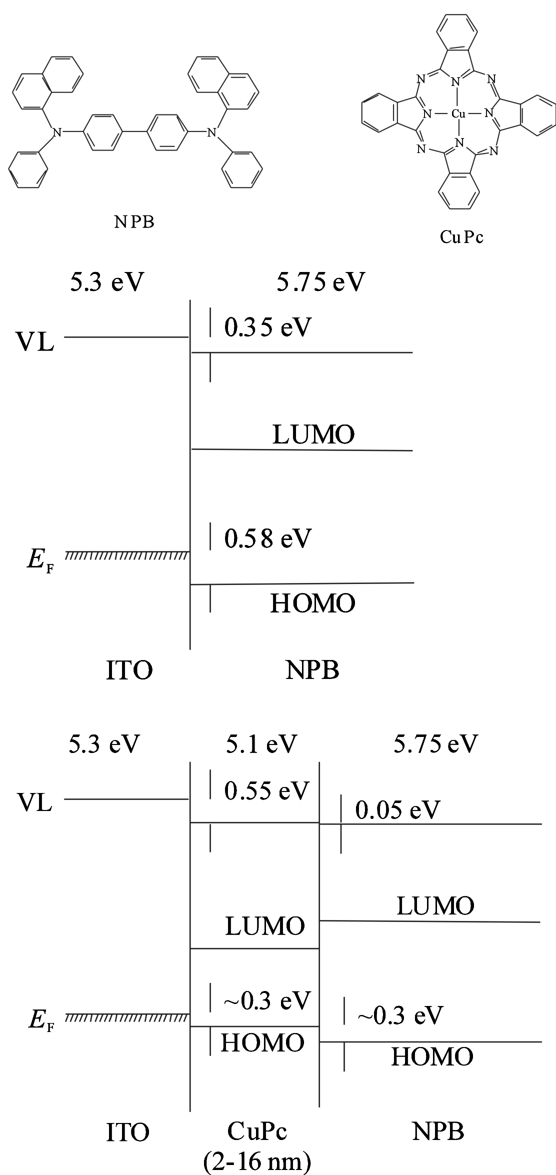

Fig. 1. Substrate configuration and molecular structures. (Top) Molecular structures of NPB and CuPc, (middle) anode without buffer layer, and (bottom) anode with varying buffer layer.

higher S/N, which illustrate the UPS data for the region around the Fermi level for ITO, 2 nm CuPc and 8 nm CuPc (a to c, respectively). These data allow us to define E_f more precisely. Equivalent narrow scan data around E_f were obtained for the spectra in Figs. 4 and 5, but are not illustrated.

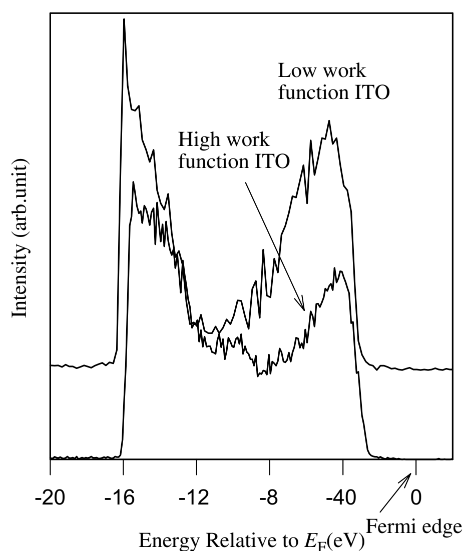

Fig. 2. HeI UPS spectra of high and low work function ITO. The low work function spectrum has been shifted vertically for clarity.

A typical UPS spectrum of ITO/CuPc/NPB structure is shown in Fig. 4 in which we can notice that the CuPc HOMO level is not visible anymore after deposition of 2 nm of NPB; however, a trace of CuPc feature around −5.8 eV can be seen by comparing with the thick NPB spectrum. This indicates that NPB does not wet CuPc as well as CuPc wets ITO, but we can presume a coverage of around 90% for a 2 nm thick film of NPB. This trend is almost the same for all CuPc thicknesses we have tried in this study.

The data in Fig. 5 for the deposition of NPB on ITO indicates that this interface exhibits the poorest wetting of the three studied. The HOMO level appears by a deposition of a 2 nm layer, enabling a consistent measurement of the energy barrier at the interface to be made. The offset between the HOMO edge in the organic overlayer and the Fermi energy in the conducting ITO determines the barrier for hole injection. All our observations of sticking properties for ITO/CuPc, CuPc/NPB and ITO/NPB are consistent with the findings obtained by AFM by another group [21].

Our careful and repeated measurements of CuPc films on ITO indicate a significant VLS upon deposition of ~2 nm CuPc on ITO. On average we

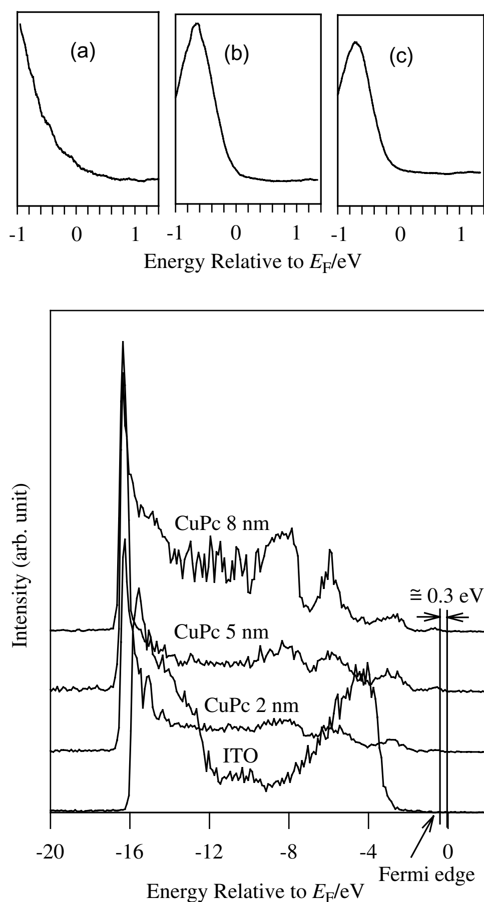

Fig. 3. HeI UPS spectra as a function of incremental CuPc deposition on ITO. Inserts (a), (b) and (c) show a blowup at region of Fermi level for ITO, 2 nm CuPc and 8 nm CuPc, respectively.

measure a VLS of ~0.55 eV after depositing CuPc on ITO. We have also measured a smaller but still substantial VLS for ITO/NPB. The VLS for CuPc/NPB is negligible. These results are summarized in Table 1.

An interesting result was obtained by measuring the VLS for deposition of CuPc on ITO surfaces with different amounts of adsorbed hydrocarbons. These ITO substrates were intentionally prepared by different treatments to achieve different initial values for the work function on nominally clean ITO. The values of the initial work function versus the vacuum level shift for 2 nm CuPc on ITO are shown in Fig. 6,

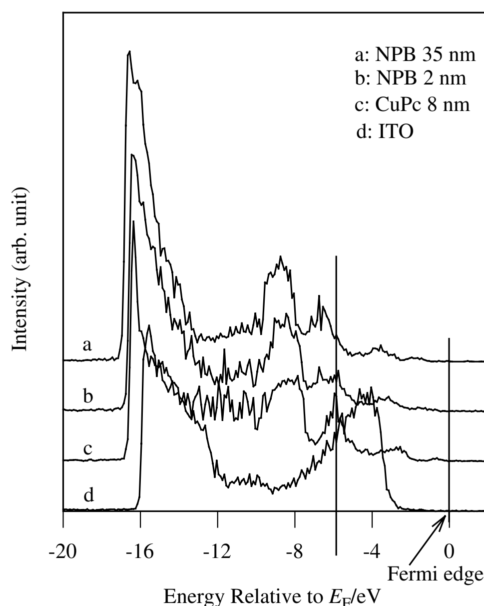

Fig. 4. HeI UPS spectra NPB on a typical ITO/CuPc substrate.

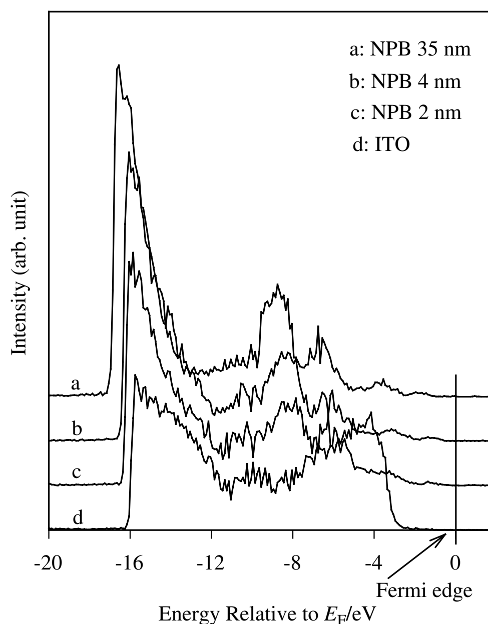

Fig. 5. HeI UPS spectra as a function of incremental NPB deposition on ITO.

and exhibit an almost linear relationship. The more hydrocarbon impurity on the ITO surface,

Table 1
VLS measured by UPS

Interface	Shift/eV
ITO/CuPc	0.55
ITO/NPB	0.35
CuPc/NPB	0.05

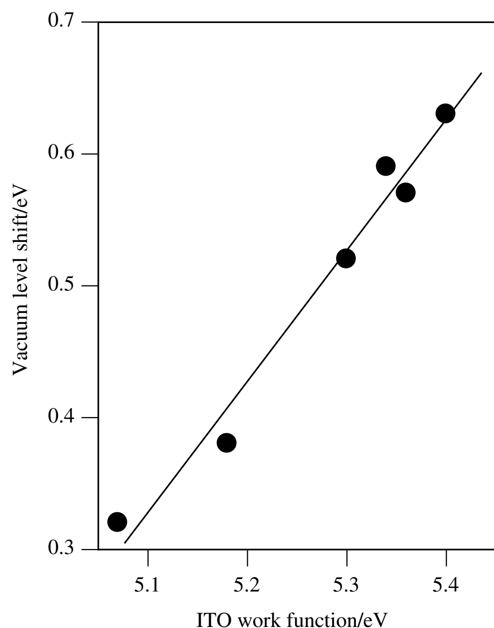

Fig. 6. Relationship between ITO work function and VLS after depositing CuPc.

i.e. the lower the work function, the smaller is the VLS for ITO/CuPc interface. The amount of hydrocarbon on ITO surface was checked regularly by examining the carbon peak in XPS spectra of the ITO substrates. While this conclusion for the relationship between the VLS and initial WF is correct for ITO/CuPc, it *cannot* be generalized for other metal/organic interfaces as each case must be measured individually. For the ITO/CuPc system, this behavior has the following consequence. Within the range of ITO work functions measured in this study, the energy barrier at the ITO/CuPc interface is independent of the ITO work function. This is illustrated in Fig. 7 where we have plotted the energy barrier at the ITO/CuPc interface versus ITO work function.

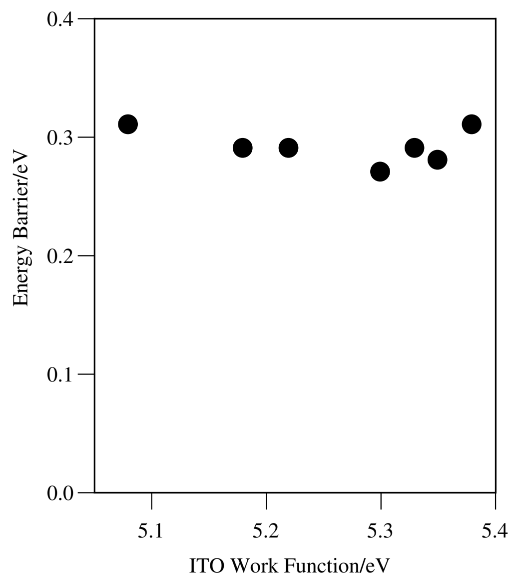

Fig. 7. Energy barrier at ITO/CuPc interface versus ITO work function.

We also have measured the energy barrier at the ITO/NPB interface and compared this single barrier to hole injection with the double barrier through the CuPc buffer layer. The values obtained are summarized in Table 2. The difference between the Fermi edge of ITO and the HOMO level of the organic, and also between the HOMO

Table 2
Energy barrier to hole injection at ITO/NPB and ITO/CuPc/NPB interfaces

Structure	Barrier at ITO/CuPc	Barrier at CuPc/NPB	Barrier at ITO/NPB
ITO/NPB	NA	NA	0.58
ITO/CuPc(2 nm)/NPB	0.31	0.28	NA
ITO/CuPc(5 nm)/NPB	0.29	0.29	NA
ITO/CuPc(8 nm)/NPB	0.29	0.28	NA
ITO/CuPc(11 nm)/NPB	0.31	0.31	NA
ITO/CuPc(14 nm)/NPB	0.27	0.27	NA
ITO/CuPc(16 nm)/NPB	0.27	0.27	NA

levels of the two organics was measured for a 2 nm thick organic layer deposited on ITO, and on a thick layer of organic, respectively. As can be found in the table, the hole injection barriers through the double interfaces of ITO/CuPc and CuPc/NPB have almost the same values independent of CuPc layer thickness. This effective barrier height is reduced to almost half value of the barrier height through ITO/NPB alone. The results for the energy level alignments for ITO/NPB and ITO/CuPc/NPB structures regarding hole injection barriers are illustrated graphically in Fig. 1.

3.1. Topography

The topography of CuPc on ITO was acquired ex situ using AFM. Figs. 8(a)–(f) show the topographical images for ITO and surfaces obtained after depositing 2, 5, 8, 11, 14, 16 nm of CuPc on ITO. These images reveal that ITO has a relatively smooth surface. There is no significant change in topography after deposition of 2 nm thick CuPc. However, for a thicker CuPc film, up to about 11 nm, the surface features become rather sharper and some of the sharp features are taller than before deposition. These sharp features become smoother and shorter again, and the topography is similar to the original ITO surface at a CuPc layer thickness of ∼16 nm.

4. Discussion

It has been widely assumed that the relative energy positions across interfaces involving molecular solids would be defined by the alignment of the vacuum levels of the two solids. This assumption was based on the presumed noninteractive nature of such interfaces due to the closed shell properties of molecules and the weak van der Waals bonds linking molecules to other solids. This assumption, however, was shown to be wrong for metal–organic interfaces where dipole barriers in excess of 1 eV have been recorded. [2] The assumption was also shown to be wrong for several organic–organic heterojunctions [22,23], although dipoles at these interfaces were generally found to be smaller than at metal–organic interfaces.

Our measurement of significant VLS upon deposition of CuPc on ITO is in clear contrast to the results reported by Hill et al. [24]. However, there have been other reports of comparable VLS for the ITO/CuPc interface [16]. It is essential to consider and measure the VLS to interpret the results of predicted band alignments. However, in the case of Hill et al., it is significant that the starting work function of their ITO was small, ∼4.5 eV, possibly indicating hydrocarbon contamination of the surface. This would create conditions for a small VLS. This effect can be seen in an extrapolation of the line in Fig. 6. We emphasize, that in our experience, it is build up of hydrocarbons on an ITO surface that is one of the major reasons for obtaining a low work function surface, provided the UPS irradiation time is kept short [5]. This monolayer (or even submonolayer) of hydrocarbon probably functions in a similar manner on ITO with the formation of dipoles at the organic/metal interface. Therefore, the absence of VLS upon CuPc deposition is an indication of hydrocarbon contamination (saturation) *prior* to organic deposition.

Previous work on single layer devices of ITO/CuPc/Al built on differently treated ITO substrates with different initial work functions, showed behavior consistent with *identical* effective work functions at the ITO/CuPc interface, despite the initial different ITO work function of the starting material [25]. This is in fact equivalent to having the same injection barrier height. This observation is in agreement with our direct measurements using UPS techniques.

Insertion of a CuPc is observed to enhance the device efficiency [12]. When the VLS is taken into account, the barrier at the CuPc/ITO interface is larger than if the VLS is ignored. However the insertion of CuPc reduces the barrier compared to the NBP/ITO interface. The observation of an increase in efficiency of the OLED accompanied by a shift in the *I–V* curve towards higher voltages, has led some researchers to believe that insertion of CuPc between the anode and the HTL reduces the hole injection rate. However, our measurements show a decrease in the overall

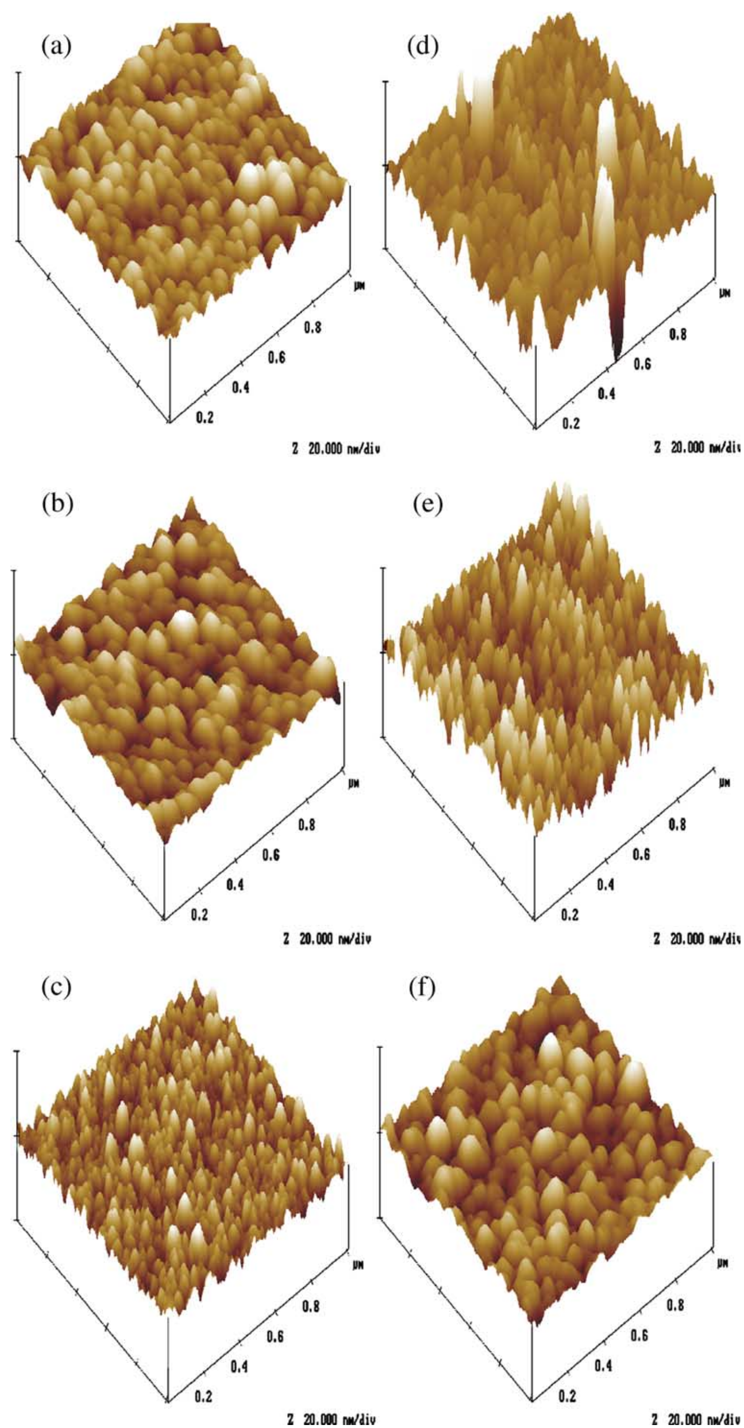

Fig. 8. Tapping mode AFM images for (a) bare ITO, (b) ITO/CuPc (2 nm), (c) ITO/CuPc (5 nm), (d) ITO/CuPc (8 nm), (e) ITO/CuPc (11 nm) and (f) ITO/CuPc (16 nm).

barrier height to hole injection independent of the CuPc thickness, while on the other hand, literature results demonstrate an improvement of the device efficiency for devices with a CuPc layer thicker than 10 nm. The possible oxygen diffusion from ITO into the CuPc layer may have a countereffect on the charge mobility. ITO has a well known tendency to release oxygen into organic layers. The amount of oxygen may be too small to cause any degradation of the device, but large enough to increase the trap level. Boudjema et al. [26] have shown that the negatively charged oxygen ions behave as shallow traps for positive charge carriers in organic materials. This effect might occur in the CuPc layer and result in lower mobility of holes which could bring the opposite carrier populations into balance as has been observed in devices built with a CuPc buffer layer [17]. These two facts bring us to the conclusion that a thickness dependent property must be responsible for the changes in device behavior. However, our observation seems to be in contrast to the dependence of energy barrier of a spin coated polymer layer and ITO on ITO WF [27]. This discrepancy can be explained as the following. The environment of fabrication for polymer LEDs is very different from OLEDs in cleanliness of the ITO surface (spin coating versus vacuum). While the starting WF in this reference is 4.5–4.8 eV, OLEDs can be built on clean ITO surface with a WF of 5 eV or higher. The WF dependence reported in this reference is also combined with the formation of indium chloride ($InCl_3$) at the polymer–ITO interface during the conversion process of precursor-PPV films on ITO: the HCl released in the conversion process interacts with the surface of the ITO substrate, leading to the formation of indium chloride, which diffuses into the polymer. Moreover, to measure the exact energy barrier at the interface, it is necessary to do the HOMO level measurement for a very thin top layer to be able to measure the energy barrier *at* the interface but this is impossible in spin-coating technique. It should also be noted that the interfaces of conjugated polymers on metals are ill-defined and ideal interfaces are unattainable in contrast to small molecules evaporated in a vacuum chamber.

It is well known that a significant problem in optimizing OLED structures is the mismatch of hole and electron mobilities for the HTL and ETL layers, and even for the same organic layer of a single layer OLED. A high hole mobility and injection rate, and a low electron mobility and injection rate (which is the case for a ITO/NPB/Alq3/cathode device) results in the recombination of the charges and the formation of excitons closer to the cathode. As a consequence, excitons have a much higher probability of decay without radiation. Values reported in the literature indicate that hole mobility and trapping concentration in CuPc can change by two orders of magnitude depending on the deposition conditions. The resulting magnitudes of hole mobilities in CuPc lie in a range which covers values which are smaller than the value for NPB to values larger than for NPB. Thus at lower substrate temperature [28], higher background pressure for deposition or even increased rate of evaporation, hole mobility can be lower and trapping concentration can be higher [28,29] in the CuPc layer than that for NPB [30]. If this effect of trapping and lower mobility of the holes occurs in CuPc, it could result in the prevention of holes from reaching the cathode, which in turn increases the percentage of holes that form excitons, thereby increasing the recombination efficiency. In the context of our results and previously published work, this occurs when the CuPc layer has an optimum thickness to balance the number of holes taking part in exciton formation. Our results clearly indicate that enhancement of hole injection remains unchanged with alteration of the CuPc layer thickness. The other factor which must be considered is a possible reduction in hole hopping frequency at the CuPc/NPB interface. More interestingly, I–V characteristic of a hole-only device produces a shift of the curve to a lower voltage [31] indicating that the observation of an opposite trend for a hole–electron device suffers from the combined effect of all the other parameters involved.

Our UPS spectra indicate good wetting of CuPc on ITO. The important enhancement of device stability reported by Van Slyke et al. [9], by insertion of a CuPc layer between ITO and NPB, may be partly also due to the improved wetting

property of organic materials on ITO, in addition to improved balances in electron and hole injection.

5. Conclusion

On the basis of UPS measurements under ultrahigh vacuum, we have determined that the CuPc HOMO level is situated between the ITO Fermi level and the NPB HOMO levels. Further, the UPS data show that the structure of the occupied CuPc molecular orbitals does not change for thicknesses up to 16 nm. From observations of the UPS feature with film thickness, we concluded that NPB wetting is significantly improved by using a CuPc interlayer.

In this article we have also shown that the difference in OLED behavior, with different thicknesses of CuPc buffer layers, is not caused by a variation in hole injection barrier height. In fact there is almost no change in the barrier height by changing the CuPc thickness from 2 to 16 nm. The observed behavior must have some other origin. At large CuPc layer thicknesses, the origin of the reduction of hole injection is undoubtedly the decrease in field at fixed operating voltage.

Our AFM investigation of ITO/CuPc topography reveals changes at the interface between CuPc and the hole-transport layer. A smoother CuPc(16 nm)/NPB interface versus thinner CuPc layers is a clear advantage for an OLED. Applying the idea of Seki (Ishii) et al. regarding the VLS, we showed first that it is a valid argument for organic/ITO interfaces, but not for the particular organic/organic junctions that we have studied. We conclude that careful consideration of the vacuum level shift is necessary for reliable quantitative assessment of the effect of energy level shifts associated with buffer layers.

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