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HP Laboratories
HPL-2010-170

Keyword(s):

Display, printed electronics

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External Posting Date: October 21, 2010 [Fulltext] Approved for External Publication
Internal Posting Date: October 21, 2010 [Fulltext]

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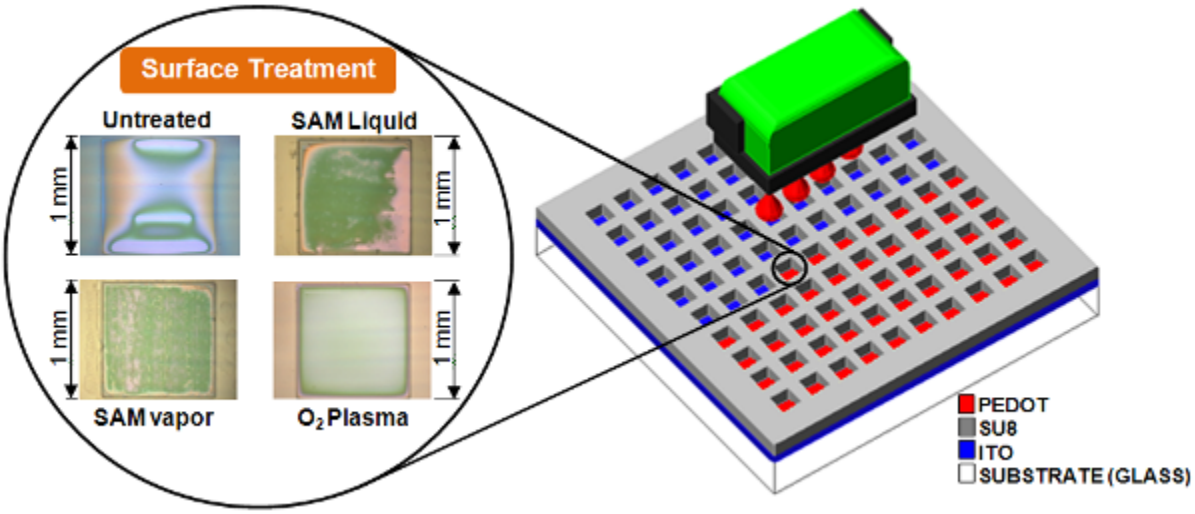
Abstract

In this contribution, we describe the development of inkjet printable PEDOT:PSS polymer-based inks for fabrication of polymeric organic light-emitting devices (OLEDs). By using a 10 x 10 array of SU8 wells on ITO/glass substrates, guided deposition of PEDOT in a simulated OLED pixel structure was possible. The quality of the printed patterns was controlled by fine tuning of the surface wetting properties using self-assembled monolayers (SAMs) and/or oxygen reactive plasma. All the investigated surface treatments improved the quality of the printed pattern. However, the O_2 plasma treated surfaces, which had the highest free energy, resulted in smoother and more uniform PEDOT films than did the SAM-coated surfaces. Rainbow-like features observed at the edges were attributed to the well-known coffee stain effect. An impressive reduction of such features was achieved by decreasing PEDOT content in the inks.

Graphical Abstract

Organic Electronics, Volume xx, Issue x, Pages xxx-xxx (x xxxx 2010). Patterning quality control of inkjet printed PEDOT:PSS films by wetting properties. Fernando Ely, César O. Avellaneda, Celso Molina, Pablo Paredez, Viviane C. Nogueira, Thebano E. A. Santos and Victor P. Mammana.



1. Introduction

One of the driving forces for flexible electronics is the promise of low-cost production with high yield and throughput using roll-to-roll (R2R) processes. Deposition methods based on solution-processable materials such as ink-jetting, screen and flexo/gravure printing, and spray coating are more easily adapted to a R2R environment [1-2]. One of the most straightforward approaches towards achieving locally patterned functional materials involves depositing them directly in a pattern using inkjet printing (IJP)[3-7]. For most applications in electronic devices, the drop-on-demand (DOD) jetting mode is the best choice due to its smaller drop size and higher placement accuracy. The feature size and the quality of the printed pattern are ultimately determined by drop volume and the interaction of the jetting fluid with the substrate. Figure 1 illustrates the influence of the substrate on drop spread. Dot gain is calculated as the ratio in diameter of the spread drop (d_c) and drop diameter (d_a). The smallest printed feature would have a width equal to the diameter of the drop. Drop spread is influenced by fluid properties such as surface tension and viscosity plus the relative surface energy and roughness of the substrate. So it is possible to limit drop spread by carefully modifying the surface energy of the substrate and/or confining the fluid by creating cavities or wells on the substrate [8,9]. The last is the approach usually taken to define pixels in polymeric OLED displays and in color filters for LCDs.

INSERT FIGURE 1 HERE

In this contribution, we describe the development of inkjet printable PEDOT:PSS polymer-based inks for fabrication of polymeric OLED devices. The quality of the printed patterns was controlled by fine tuning of the surface wetting properties using self-assembled monolayers (SAMs) and/or oxygen reactive plasma. The remarkable role of the surface treatment on the chemical and physical properties of ITO and SU-8 photoresist films was evaluated by contact angle and surface energy measurements, atomic force microscopy (AFM), optical profilometry and microscopy.

2. Experimental

General: PEDOT:PSS (Clevios P VP AL4083, 1.3 to 1.7 % solid content) from H.C. Starck was used as received in the preparation of inkjet inks 1-3. Other chemicals were acquired from Sigma-Aldrich Co. and used without further purification. ITO coated glass substrates (R_S = 10-12 Ω/□, PGO-Praezisions Glas & Optik GmbH) were cleaned in a class 10.000 clean room using a sequence of detergent, acetone and isopropanol ultrasonic baths. AFM and optical profilometry images were taken using a Nanosurf EasyScan 2 (Nanosurf AG, Liestal Switzerland) microscope and Zygo Optical Profilometer New View 500 (Zygo Co., Middlefield, USA), respectively. The AFM images were analyzed using the WSxM 5.0 Software from Nanotec Electronica S.L [10]. Contact angle measurements were conducted to determine the wetting angle and surface energy of the samples. We employed a OCA15 plus (Dataphysics Instruments GmbH, Filderstadt, Germany) contact angle system using glycerol and *n*-dodecane [11] under ambient conditions (22 °C; RH 45–55 %). For jetting experiments a commercial research grade inkjet printing, Dimatix DMP 2810, was used (Fujifilm-Dimatix, Santa Clara, USA).

Polymer wells: The polymer wells were defined by wet photolithography using negative-tone SU8 25 photoresist from Microchem Co. SU8 25 was spin-coated on 40 mm x 40 mm ITO coated glass slides in two steps at 600 rpm (5s) and 5000 rpm (40s). A pre-bake step was carried out for 3 min at 65 °C and then at 95 °C for 8 min on a hotplate. Then, the substrates were exposed in a near UV (350-400 nm) contact printer (Model 155, Tamarack Scientific Co., USA) for 16 sec (200 mJ/cm^2) using a soft contact mask to define a 10 x 10 array of wells (each well had 1 mm x 1 mm size and 1 mm pitch). After exposure the substrates were submitted to a post-bake step for 2 min at 65 °C and then 95 °C for 5 min on a hotplate. The photoresist was developed in SU8

developer for 1 minute then rinsed in isopropanol for 30 seconds and finally hard-baked at 150 °C for 5 minutes. The final SU-8 thickness obtained was 6.7 µm as confirmed by optical profilometer. **Surface treatment**: a) Oxygen plasma treatment was done by using O_2 plasma RIE (Plasma Technology, Erlanger, USA). An oxygen flow of 20 sccm at a pressure of approximately 26.7 Pa was maintained resulting in a plasma power of 100 W. The plasma treated SU-8 samples are stable for 40 days without significant changes in terms of surface free energy [12]. b) APTS ((3-aminopropyl) triethoxy silane) SAM in solution: First the 10 x 10 array of wells was activated by oxygen plasma treatment as described above. Then the activated substrates were immersed in a 6 mM APTS solution in ethanol for 10 seconds and transferred to an oven for 15 minutes at 60 °C. b) APTS SAM in vapor: surface oxygen plasma activated 10 x 10 well arrays were placed in a dessicator together with about 0.3-0.5 mL of APTS. The dessicator was pumped down to a pressure of 200 Pa. After 15 minutes the dessicator was vented with air. The substrate is then inserted into a preheated oven at 60 °C for 45 minutes.

3. Results and Discussion

PEDOT:PSS is commercialized as a dispersion in different grades. In semiconducting grades used for OLEDs and organic photovoltaics (OPV) water is the solvent, while the electrically conducting grades usually have alcohols or DMSO as a co-solvent to improve the conductivity. In this work the water-based semiconducting grade Clevios P VP AL4083 (1.3 to 1.7 % solid content) from H.C. Starck was used. For deposition methods such as spin-coating and drop-casting this formulation is able to provide very good thin films but for inkjet application additives are necessary to adjust the viscosity and surface tension. Consequently, for jetting experiments three different PEDOT:PSS inks were developed as summarized in table 1.

INSERT TABLE 1 HERE

All the PEDOT inks from table 1 presented a viscosity of 10 cps and surface tension of 31 $dyn.cm^{-2}$, which fit the requirements of the jetting equipment used. In inks 2 and 3 the concentration of PEDOT was reduced by diluting twice and three times with deionized water, respectively. When using a piezoelectric inkjet head, the applied voltage waveform is critical to the jettability of the ink. A waveform was developed for this PEDOT jetting studies and a stable single drop regime was achieved. The PEDOT droplets ejected from the 16 nozzles presented a constant velocity (v = 8 m/s) with no variation of their in-flight trajectory. The measurements indicated a droplet volume (V) of 6 pL (see figure 2a).

INSERT FIGURE 2 HERE

Figure 3 shows schematically the structure created to guide the deposition of PEDOT by jetting.

INSERT FIGURE 3 HERE

On ITO coated glass slides a 10 x 10 array of square wells were defined by photolithography using SU8 photoresist. In the inset of the figure 3 three possible behaviors for the PEDOT spread into the wells are suggested (I-III) based on the wettability differences between the fluid and the SU8 and ITO surfaces. In situation I, where the contact angle on SU8 (θ_{SU8}) is significantly higher than that on ITO (θ_{ITO}), the thickness of the PEDOT film should be less near the SU-8 interface. The reverse profile (III) is expected if θ_{ITO} is much higher than θ_{SU8}. Finally, a more homogenous and flat PEDOT film can be obtained if the wettability is similarly high for both ITO and SU-8 (II, $\theta_{SU8} \approx \theta_{ITO}$). The figure 2b shows a set of 4 wells after the deposition of PEDOT-ink-1 without any previous surface treatment. A highly non-uniform PEDOT film was obtained under this condition as evidenced by the apparent color variation. This color profile may be directly correlated to a thickness variation of the film within the well area. Another observed feature observed is the empty spaces at the borders, which indicate that PEDOT did not completely fill the wells. The optical image shown in Figure 2a fits the profile suggested in Figure 3. Such observations motivate us trying to control the wetting properties by surface treatments aimed at achieving a film profile as close as possible to II. Four different surfaces treatments were considered: a) vapor or solution deposition of a SAM of APTS; b) exposure to reactive O_2 plasma; c) vapor deposition of a SAM of fluorosilane (Trichloro (1*H*, 1*H*, 2*H*, 2*H*) perfluorooctyl) silane) [13] and d)

soaking in phosphoric acid [14]. The treatments c) and d) are not discussed in this paper because they resulted in worse behavior than untreated surfaces in IJP experiments.

Contact angle and Free Surface Energy (SFE) Analysis

As mentioned afore, the surface energy is one of the fundamental parameters controlling the inkjet printed fluids. The contact (wetting) angle determines the spread of a liquid drop on the surface and depends on the relative surface free energy (SFE) of the solid-liquid, solid-vapor, and liquid-vapor interfaces. The well-known Young´s equation describes the interfacial interactions.

$$\gamma_{sl} = \gamma_s - \gamma_l cos(\theta) \qquad (1)$$

One can separate the surface energies γ_l and γ_s into two interaction components: the dispersive part γ_d representing the van der Waals interaction and the polar part γ_p. The quantities γ_l and θ appearing in Eq. 1, can be easily measured. The interfacial energy γ_{sl} can then be calculated by subtracting the geometric mean of both polar (permanent dipoles) and dispersive (induced dipoles) contributions (OWRK – Owens-Wendt-Rabel and Kaelble method) [15] leading to the equation

$$\frac{1+\cos(\theta)}{2}\frac{\gamma_l}{\sqrt{\gamma_l^d}} = \sqrt{\gamma_s^p}\sqrt{\gamma_l^p/\gamma_l^d} + \sqrt{\gamma_s^d} \qquad (2)$$

Because the surface tension parameters of the test liquids are known and the contact angle can be measured, both parameters γ_s^p and γ_s^d can be obtained from a linear least-square fit. The surface energy of the solid is given by $\gamma_s = \gamma_s^p + \gamma_s^d$. Table 2 summarizes the calculated SFE for the ITO and SU-8 surfaces before and after treatment. In this study, the surface energy was

determined by contact angle measurements of sessile drops on solid surfaces in conjunction with the OWRK geometric mean method.

INSERT TABLE 2 HERE

On ITO, the APTS SAM surface treatments decreased the SFE relative to the pristine surface. This reduction was more significant for the SFE polar component than for the disperse one which indicates preferential neutralization of permanent dipoles at the surface. The APTS SAM seems to have no effect on the SFE of SU-8. For both SU8 and ITO the SFE increased upon O_2 plasma treatment (ΔSFE_{SU8} = +9.8 $mN.m^{-1}$) and (ΔSFE_{ITO} = +7.7 $mN.m^{-1}$).

Surface Roughness Analysis

Along with the SFE, surface roughness is another important characteristic that impacts the drop spread and uniformity of the resulting printed film. AFM was used to evaluate the topography of the modified surfaces of ITO and SU8, see table 3.

INSERT TABLE 3 HERE

No significant alterations in terms of topography and roughness were observed for ITO upon surface treatment. For SU8 a small roughness increase occurred from R_{rms} = 0.4 nm (pristine) to R_{rms} = 2.0 nm (APTS liquid) and R_{rms} = 1.5 nm (O_2 plasma treated sample) which is in good agreement with previously reported analysis [12]. 10 x 10 arrays of square wells pre-treated with APTS SAM or O_2 plasma were used to deposit the PEDOT-ink-1. The results are shown in table 4. If no surface treatment is used then the PEDOT overspreads the wells (entry 1, table 4).

INSERT TABLE 4 HERE

Among the studied surface treatments, O_2 plasma shows the best results in terms of uniformity of the printed PEDOT film (entry 2, table 3). This treatment improved the wetting properties by reducing the contact angle for PEDOT on both ITO and SU-8 surfaces. The APTS SAMs made the ITO surfaces slightly more hydrophobic (entries 3 and 4, table 4). These achievements are in agreement with the general observation that high energy surfaces (e.g. SU-8 and ITO after O_2 plasma treatment) result in small wetting angle and higher drop spreading [16].

Using PEDOT-ink-1 after an O_2 plasma surface treatment resulted in a uniform film approximately 300 nm thick. However, rainbow-like features are observed at the edges (entry 2, table 4) indicating a gradual increasing of the film thickness. We believed this drying pattern is result of the well-known coffee stain effect commonly noted on high energy surfaces. These coffee stains are due to evaporation of the solvent causing a difference in temperature and concentration between the edge and interior of a drop. This leads to a difference in surface tension and creates a capillary flow. Consequently, the solute particles are carried to the edge

and deposited at the contact line, typically forming a ring. In order to minimize this effect and obtain thinner films the PEDOT content in the ink was diluted with water. This strategy is the reason for ink-2 and ink-3 described earlier in table 1. Table 5 summarizes the printing results using the three PEDOT-inks of different concentration. As expected, diluting the inks resulted in a thickness reduction from about 300 nm (ink-1) to 110 nm (ink-3). Moreover, the rainbow-like features at the film edges almost disappeared by using PEDOT-ink-3 as evidenced by polarized optical microscopy (POM) images and confirmed by surface profilometry.

INSERT TABLE 5 HERE

4. Conclusion

In summary, three semiconducting PEDOT-based inks suitable for IJP deposition of polymeric OLED and OPV devices were developed. By using a 10 x 10 array of SU8 wells on ITO/glass substrates it was possible to guide the deposition of PEDOT in simulated OLED pixel structures. The ink spread within the wells was controlled by carefully modifying the surface free energy (SFE) of ITO and SU8 with self-assembled monolayers and/or oxygen reactive plasma. All surface treatments improved the quality of the printed pattern. However, surfaces having the highest SFE, as a result of O_2 plasma treatment, rendered smoother and more uniform PEDOT films than those coated with a SAM. Rainbow-like features observed at the edges were attributed to the well-known coffee stain effect. An impressive reduction of such features was achieved by decreasing the PEDOT content in the inks.

5. Acknowledgements

This work was supported by Hewlett-Packard R&D Brasil and FAPESP agency. The authors thank Maria das Graças Almeida and Elaine F. von Zuben for the photolithography work and optical profilometry analysis and also de Atomic Force and Tunneling Microscopy Lab. - MTA/LNLS for AFM analysis.

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Figure Captions

Figure 1. Influence of non-absorbent substrate on the drop spread. The d_a, d_b and d_c represent the drop diameter after jetting and in two stages after hitting the substrate.

Figure 2. (a) Frame capture during PEDOT jetting using ink 1 (table 1) at 12 V firing voltage (23 kHz) and 30 °C cartridge temperature. (b) Optical microscopy (OM) showing a set of four SU8 wells filled with PEDOT-ink-1 using a 20µm drop spacing without previous surface treatment.

Figure 3. Schematic representation of the 10 x 10 array of wells created to guide the deposition of PEDOT by inkjet. Each SU8 well has 1 mm x 1 mm size and 6.7 um deep, on a 1 mm pitch. The inset presents a single well in detail with the three possible profiles for the PEDOT film depending on the magnitude of the contact angle for the SU-8 and ITO surfaces.

Table Captions

Table 1. PEDOT inks developed for inkjet deposition.

Table 2. Surface Free Energy (SFE) evaluation for ITO and SU8 before and after surface treatment.

Table 3. AFM images of ITO and SU8 films before and after liquid APTS treatment[a].

Table 4. Polarized optical microscopy images of PEDOT:PSS deposited by IJP in SU-8 wells on ITO coated glass substrates after surface treatment with APTS SAM or oxygen plasma.

Table 5. POM and optical profilometry analysis of different PEDOT:PSS inks deposited in SU8 wells on ITO coated glass substrates after surface O_2 plasma treatment[a,b,c].

List of Tables

Table 1:

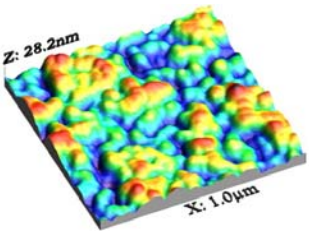
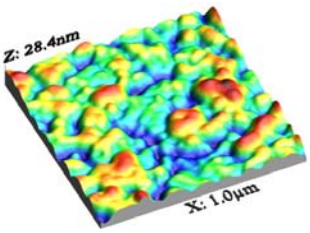
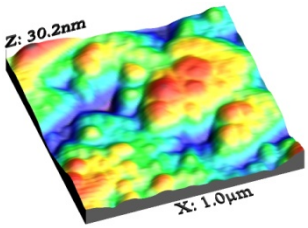
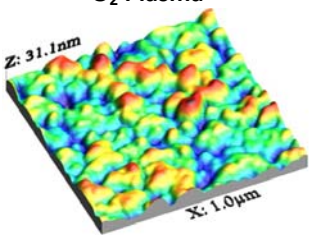
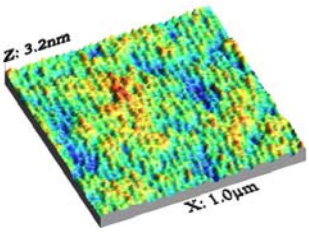
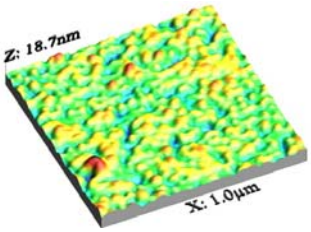
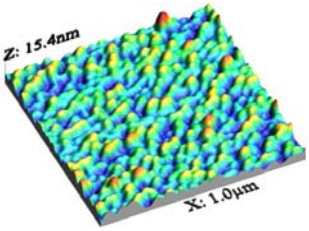
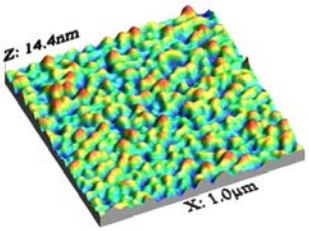
PEDOT ink	Clevios P VP AL 4083 (g/mL)	% Glycerin	% Triton X-100
1	1.00	3.0	0.2
2	0.50	3.0	0.2
3	0.34	3.0	0.2

Table 2:

ITO	Surface Treatment			
	Pristine	**APTS liquid**	**APTS vapor**	**O_2 Plasma**
θ Glycerol (°)	65±2	88.4±0.5	74±1	47±2
θ *n*-Dodecane (°)	10.2±0.4	7.7±0.1	7.4±0.5	25.0±0.4
γ_p (mN/m)	24.7	2.1	7.0	23.1
γ_d (mN/m)	14.8	24.9	24.9	22.8
SFE (mN/m)[a]	36.04	26.63	31.84	45.85
SU-8	**Surface Treatment**			
	Pristine	**APTS liquid**	**APTS vapor**	**O_2 Plasma**
θ Glycerol (°)	79.3±0.5	86.6±0.2	79.1±0.5	63±4
θ *n*-Dodecane (°)	10.9±0.5	9.9±0.5	10.4±0.5	10.2±0.3
γ_p (mN/m)	4.9	2.6	5.0	12.6
γ_d (mN/m)	24.6	24.7	24.7	24.7
SFE (mN/m)	29.6	27.3	29.7	37.3

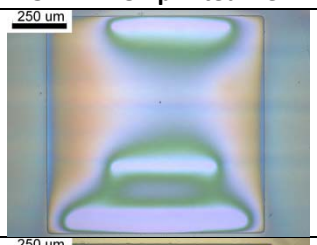
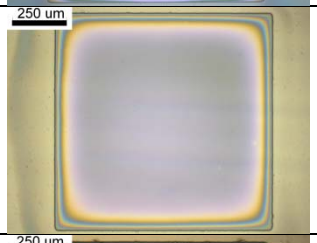
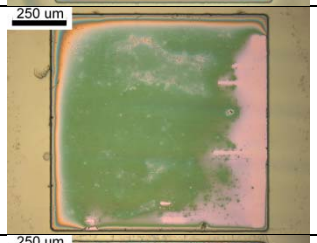
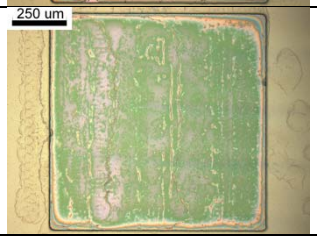
a) θ = contact angle; SFE = Surface Free Energy; γ_p = polar component of SFE; γ_d = disperse component of SFE. SFE and its polar and disperse components were calculated based on the geometric mean Owens-Wendt-Rabel-Kaelble method (OWRK).

Table 3:

ITO Surface Treatment			
Pristine	**APTS liquid**	**APTS vapor**	**O_2 Plasma**
			
R_{rms} = 4.7 nm	R_{rms} = 4.4 nm	R_{rms} = 4.8 nm	R_{rms} = 4.6 nm
SU8 Surface Treatment			
Pristine	**APTS liquid**	**APTS vapor**	**O_2 Plasma**
			
R_{rms} = 0.4 nm	R_{rms} = 1.5 nm	R_{rms} = 2.0 nm	R_{rms} = 1.5 nm

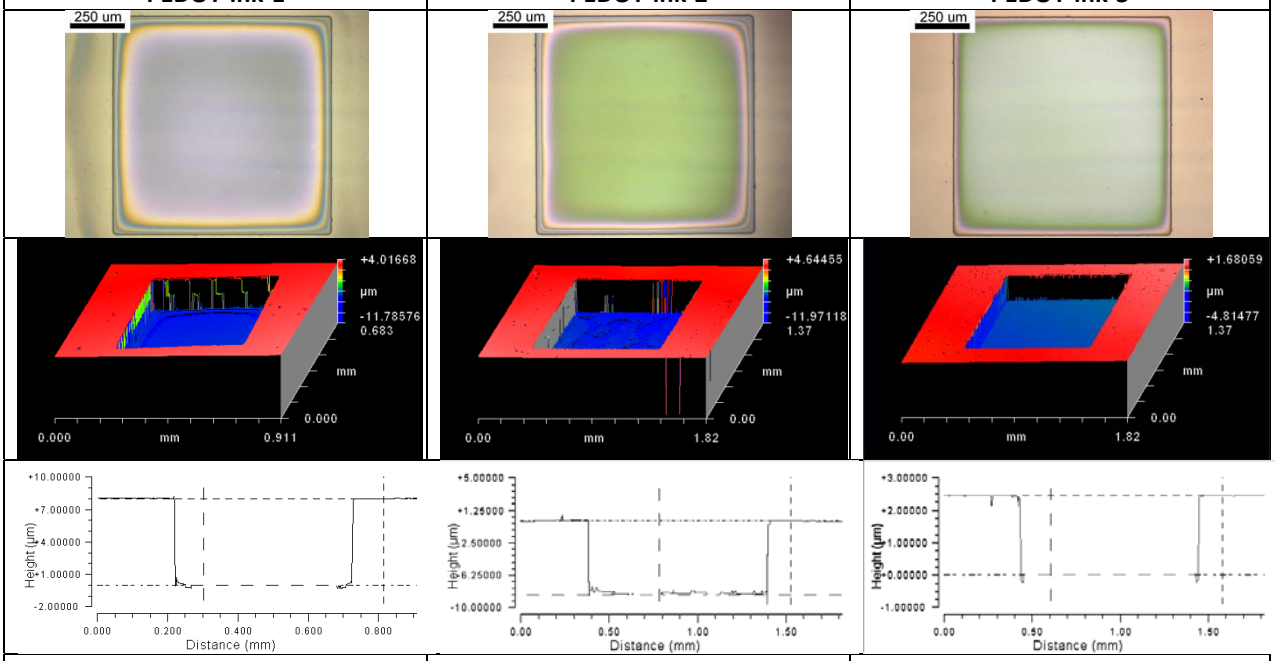
a) R_{rms} refers to the Root Mean Square (RMS) roughness parameter. b) Z refers to peak-valley distance.

Table 4:

Entry	Surface treatment	POM PEDOT printed well[a]	θ ITO (°)[b]	θ SU-8 (°)
1	None		28.8	30.8
02	O_2 plasma		20.3	9.8
3	APTS liquid		32.1	41.3
4	APTS vapor		31.3	10.7

a) Printed using PEDOT-ink-1 at 12 V firing voltage (23 kHz) at 30 °C cartridge and substrate temperature. After printing the films were baked at 100 °C on a hotplate for 1h. b) Contact angle measurements were taken by sessile drop method on samples of ITO coated glass and 8.2 µm thick SU-8 films on ITO coated glass slides. The values shown represent an average of at least 3 drops.

Table 5:

PEDOT-ink-1	PEDOT-ink-2	PEDOT-ink-3
		
t = 300 nm R_{rms} = 6.8 nm	**t = 174 nm R_{rms}** = 6.1 nm	**t = 110 nm R_{rms}** = 4.5 nm

a) POM images (100x) of PEDOT:PSS films printed at 12 V firing voltage (23 kHz) at 30 °C cartridge and substrate temperature before O_2 plasma treatment. After printing the films were baked at 100 °C on a hotplate for 1h. b) Oblique plot and surface profile taken by optical profilometer. c) Roughness measurements taken by AFM probing on 10 µm x 10 µm area.

List of Figures

Figure 1

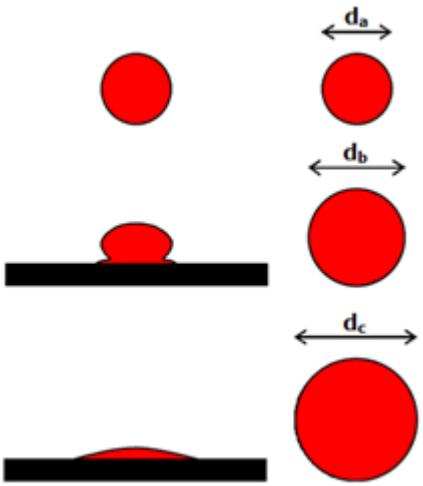


Figure 2

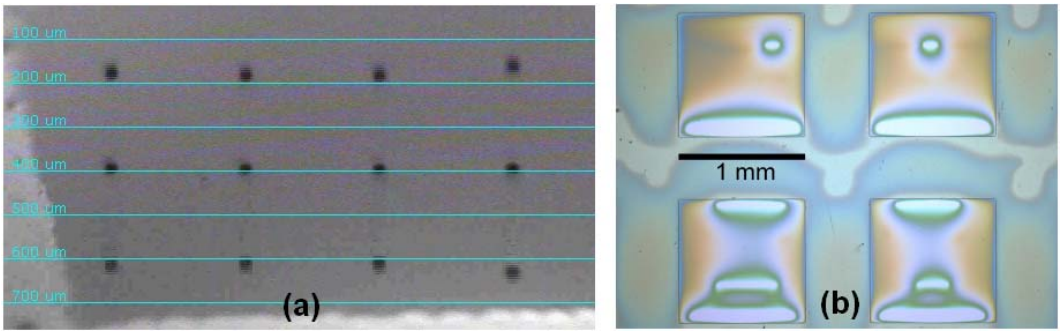
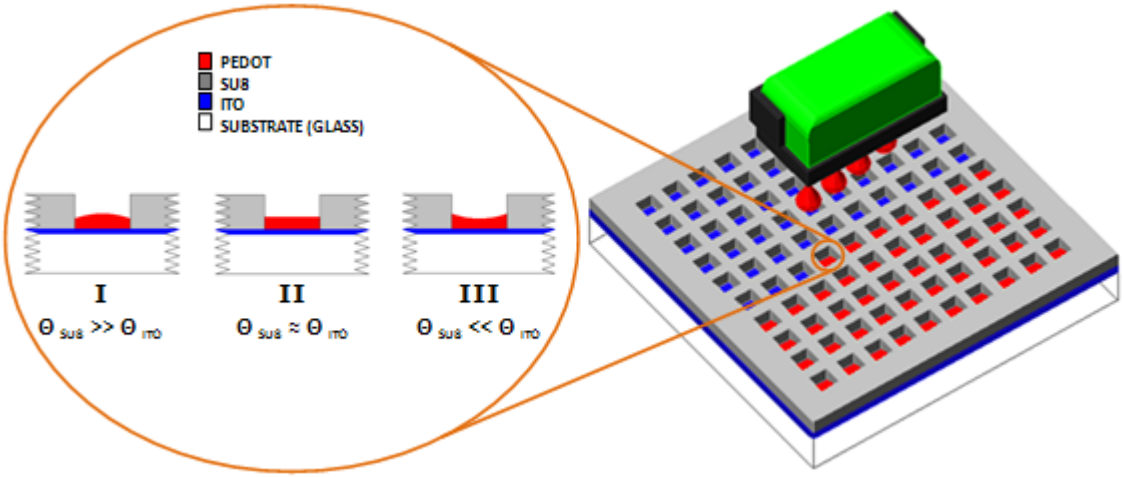


Figure 3


PEDOT
SU8
ITO
SUBSTRATE (GLASS)
I
θ SU8 >> θ ITO
II
θ SU8 ≈ θ ITO
III
θ SU8 << θ ITO