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Patterned Surface with Controllable Wettability for Inkjet Printing of Flexible Printed Electronics

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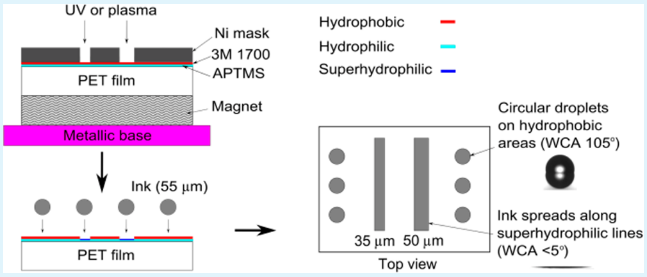
S *Supporting Information*

ABSTRACT: Appropriate control of substrate surface properties prior to inkjet printing could be employed to improve the printing quality of fine resolution structures. In this paper, novel methods to fabricate patterned surfaces with a combination of hydrophilic and hydrophobic properties are investigated. The results of inkjet printing of PEDOT/PSS conductive ink on these modified surfaces are presented. Selective wetting was achieved via a two-step hydrophilic−hydrophobic coating of 3-aminopropyl trimethoxysilane (APTMS) and 3M electronic grade chemical respectively on PET surfaces; this was followed by a selective hydrophilic treatment (either atmospheric O_2/Ar plasma or UV/ozone surface treatment) with the aid of a Nickel stencil. Hydrophobic regions with water contact angle (WCA) of 105° and superhydrophilic regions with WCA <5° can be achieved on a single surface. During inkjet printing of the treated surfaces, PEDOT/PSS ink spread spontaneously along the hydrophilic areas while avoiding the hydrophobic regions. Fine features smaller than the inkjet droplet size (approximately 55 μm in diameter) can be successfully printed on the patterned surface with high wettability contrast.



KEYWORDS: *inkjet printing, selective surface modification, controlled wettability, printed electronics, surface patterning*

1. INTRODUCTION

In addition to its conventional role in graphics output, inkjet printing has become a versatile deposition tool in various applications, especially in the fabrication of flexible electronic devices.[1−4] As this technique can produce drop-on-demand, a small amount of functional materials can be accurately deposited onto a wide range of substrates in well-defined patterns.[5] This noncontact method allows large area printing at low cost with minimal ink consumption and material wastage.

There are remaining challenges for inkjet printing, such as printing of high-resolution features and depositing droplets precisely. Pattern resolution is restricted by the size of the spread droplet on the substrate, which is controlled by the dimension of the generated droplet and wettability of the ink with respect to the substrate.[6] Because of drop generation limitations, the typical drop sizes currently attainable from inkjet print-heads are within the range from 10 to 100 μm in diameter (or 0.5 to 500 pL in volume).[7] In addition, clogging is more likely to happen with the use of small nozzle. To bypass these limitations, one promising strategy for printing fine features with size smaller than the limit imposed by the droplet size is by creating appropriate patterning on the substrate before printing to guide the spreading ink flow on a printed surface.[1] This can be achieved by either producing surface-energy texturing (hydrophilic/hydrophobic regions) via chemical surface modification or creating micropatterned walls as physical barriers for the ink, or a combination of both.

Numerous surface modification techniques have been proposed to pattern surfaces into different regions with well-controlled wetting behavior.[8] Sirringhaus et al. utilized surface-energy barrier produced by photolithography and oxygen plasma etching to guide the flow and confine the spreading of inkjet droplets for the application of all-polymer transistor circuits.[9] Low wettability contrast (∼50° difference in WCA) between different regions was obtained. Another approach is to create fluorinated self-assembled monolayer (SAM) on SiO_2 substrates by electron-beam lithography and plasma treatment.[10] These hydrophobic SAM lines aided the splitting of droplets (or "dewetting process") printed on top of them to achieve small width structures. However, the use of electron beam lithography is costly and complicated. An alternative to tune surface energy can be achieved with the aid of micro-contact printing, in which the substrate is modified to be hydrophilic and then locally printed with hydrophobic material. PDMS stamp coated with octadecyltrichlorosilane (ODTS) was kept in contact with plasma treated polyimide (PI) substrates.[11] Wettability contrast of up to 79° WCA difference between ODTS hydrophobic pattern and hydrophilic PI surface can thus be attained. Bessonov et al. applied similar method and obtained an abrupt change in surface energy of 84° difference in

Received: November 29, 2013
Accepted: February 26, 2014
Published: February 26, 2014

WCA.[12] By using shadow mask technique in electron cyclotron resonance (ECR) SF_6 plasma, Chuang et al. produced hydrophilic/hydrophobic contrast surface (100° difference) on polyethylene terephathalate (PET) substrate.[13]

In this study, we present a novel, but rather simple and versatile, approach to pattern polymer substrate with high wettability contrast (100° WCA difference). Hydrophilic patterns (wetting areas) are formed to be surrounded by hydrophobic surface barriers (non-wetting areas) by chemical coatings and atmospheric O_2/Ar plasma or UV/O_3 treatment. By inkjet printing technique, the functional ink (~55 μm in diameter) can then be printed on hydrophilic lines with 35 μm linewidth. The hydrophobic regions on the patterned substrate act as a combined physical and surface energy barriers for ink and thus facilitate the self-alignment of ink during spreading. This method, which employs simple coating chemical and atmospheric plasma treatment, can be potentially employed for mass production.

2. EXPERIMENTAL METHODS

PET, an excellent material for the fabrication of flexible printed electronics because of its low cost, high optical transparency and good thermal resistance,[14] was selected as a base substrate. The PET films (HK-31WF, thickness of 125 μm) were produced by Higashiyama Film Corporation. The coating chemicals, namely 3-aminopropyl trimethoxysilane (APTMS, $H_2N(CH_2)_3Si(OCH_3)_3$) and 3M electronic grade coatings (Novec 1700 and Novec 7100DL Engineered Fluid) were supplied by Sigma Aldrich (Singapore) and 3M (Singapore) respectively. The solvents (ethanol and acetone) were of high purity ACS grade purchased from Fisher Scientific (Singapore).

Patterned PET substrates with controllable wettability were prepared as illustrated schematically in Figure 1. First, pristine PET films were cleaned in ultrasonic baths of ethanol and acetone respectively for 2 minutes and subsequently rinsed in deionized (DI) water for 2 minutes to remove any surface contamination (type 1 PET). Next, the PET surfaces were made hydrophilic by forming a silica-like thin layer on their surfaces (see step 2 in Figure 1). A solution of APTMS 5 vol.% in acetone, which is named as the "precursor solution", was first left to age at room temperature for 10 days. The color of this mixing solution slowly changed from colorless to light yellow. The PET films were immersed in the precursor solution for 5 mins, and were then lifted slowly and baked in a vacuum oven at 120 °C for 3 mins (type 2 PET). Subsequently, the PET substrates were blanket coated with 3M chemical to create the hydrophobic surfaces. The as-received Novec 1700, which contains 2% fluoroacrylate in Novec 7100DL solvent was further diluted in Novec 7100DL to obtain a solution of 0.1% fluoroacrylate in Novec 7100DL. As such, the volume mixing ratio of Novec 1700 to Novec 7100DL is 19:1. The PET film was quickly immersed in the diluted Novec 1700 for 2 s and dried in a vacuum oven at 120°C for 10 s (type 3 PET).

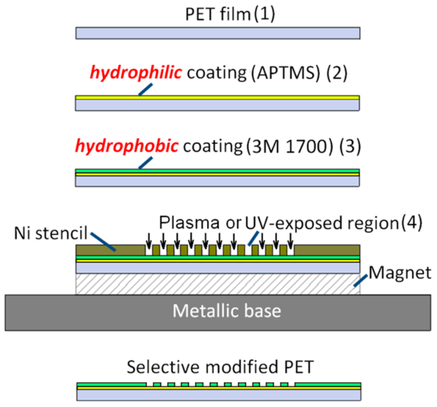


Figure 1. Process flow of selective patterning of PET film with controlled wettability: (1) cleaned virgin PET film, (2) formation of silica-like thin layer by dip coating PET film in aged solution of APTMS and acetone, (3) hydrophobic coating of 3M 1700 chemical, (4) selectively hydrophilic treatment (either atmospheric O_2/Ar plasma or UV/O_3) to create to-be-printed features with the use of a Ni mask.

Selective hydrophilic surface modification was conducted on the hydrophobic PET films with the use of a Nickel (Ni) electroformed mask fabricated by DEK International GmbH (Singapore). The PET films were sandwiched between the Ni stencil and a strong magnet attached to a metallic base (see Figure 1, step 4) and the whole assembly was placed in the machine chamber for treatment. Two different methods were used in this step, i.e. either atmospheric O_2/Ar plasma (type 4a PET) or UV/O_3 treatment (type 4b PET). Only regions exposed to plasma or UV (or unshielded areas) were rendered hydrophilic while the covered hydrophobic areas remained unchanged. For plasma treatment, the PET sample was subjected to 1.5 mins of gas plasma (Inspiraz Air plasma, RF power of 100 W, oxygen flow rate of 30 sccm, Argon flow rate of 6 sccm, 14 passes). For UV/O_3 treatment, the PET surface was placed in the UV ozone chamber (Senlight PL16, Japan, UV intensity of 10.2 mW/cm^2 for wavelength 185 nm, and 32.7 mW/cm^2 for wavelength 254 nm) for 35 min.

For convenience of discussion, the two different processes to produce textured PET surfaces and the various types of PET surfaces investigated are summarized in Figure 2. Figure 3 shows the design layout of the Ni mask (50 μm thick) and the enlargement of its measured features (parallel lines which are 5 mm long, 50 μm wide, and 100 μm in gap).

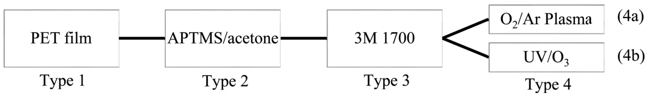
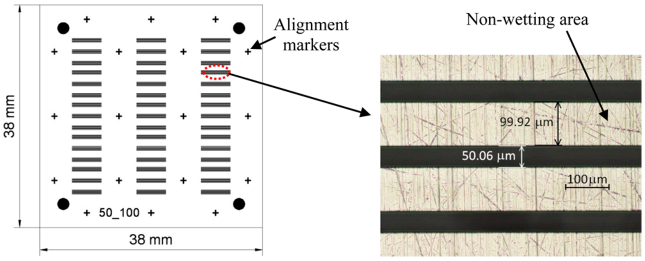


Figure 2. Various processes to pattern PET surfaces into different regions with controlled wettability.



(a) (b)

Figure 3. (a) Layout of Ni stencil mask and (b) optical enlarged feature image.

Hydrophilic wetting areas and hydrophobic non-wetting regions on PET surfaces were revealed after the removal of the Ni mask. To characterize the wetting behavior of modified films, static contact angle were measured under clean room conditions (temperature 25°C, 55% relative humidity). DI water droplets of 0.5 μL were placed onto various PET substrates by using a syringe system of Dataphysics OCA 40, Germany. For each sample, nine measurements were conducted and the reported values were averaged.

X-ray photoelectron spectroscopy (XPS) analysis using Thermo Scientific Theta Probe system (equipped with a hemispherical electron energy analyzer and a monochromatic Al Kα X-ray source $h\nu$ = 1486.68 eV, operated at 100 W, 15 kV, 6.7 mA) was conducted to determine the elemental composition on the treated PET surfaces. Step-scan intervals of 1 eV and 0.1 eV were used for wide scans and high resolution scans respectively. The pressure in the analysis chamber was kept at approximately 2.8 mbar during the scanning process. Analysis spectra were taken at 50° to the surface normal. All samples were mounted with double sided copper adhesive tape. All binding energies were referenced to the saturated C1s peak at 285 eV for compensation of surface charging. Peak fitting (Advantage software) was carried out using Shirley background.

Finally, inkjet printing was performed on the PET films with hydrophilic patterning (PET types 4a and 4b) using Microfab, Jet Lab system. The size of the inkjet nozzle is 50 μm. A water-based conductive ink PEDOT/PSS (poly 3,4-ethylenedioxythiophene/poly 4-styrenesulphonate, Clevios P Jet HC, Heraeus, Singapore) was chosen as the functional material for all printing experiments. According to the material datasheet provided by the supplier, the solids content, conductivity and viscosity ranges of the ink are 0.6−1.2%, 30−90 S/cm, and 5−20 mPa s, respectively.

3. RESULTS AND DISCUSSION

3.1. WCA Measurements.

Figure 4 shows the static WCAs captured immediately after surface modification for different types of PET films. For type 1 pristine PET film, WCA was 75.3 ± 0.4°. When immersed in the precursor solution of APTMS in acetone for hydrophilicity (type 2 film), the WCA reduced to 52.4 ± 0.9°. After hydrophobic coating with 3M 1700 chemical (type 3), the WCA increased to 105.7 ± 1.1°. Upon atmospheric plasma cleaning or UV/O_3 treatment, the PET surfaces became superhydrophilic (WCA <5°) as the substrates (types 4a, 4b) were completely wetted.

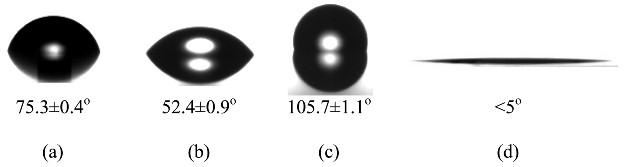


Figure 4. WCA on various PET surfaces measured immediately after treatment: (a) pristine PET film (type 1), (b) after hydrophilic coating of APTMS in acetone (type 2), (c) after hydrophobic coating of 3M Novec 1700 (type 3), (d) after selectively hydrophilic treatment by either O_2/Ar plasma or UV/O_3 (types 4a, 4b).

3.2. XPS Results.

Table 1 summarizes the surface elemental compositions of the five PET substrates obtained by XPS analysis. For ease of discussion, only five possible elements (C, F, N, O, and Si) are considered while other contaminated (trace) elements are ignored. Virgin PET film composed of 73.89% C and 26.11% O. After immersed in the precursor solution of APTMS in acetone, PET samples show a decrease in carbon percentage (53% C) but an increase in nitrogen (10.99% N) and silicon (14.1% Si). One possible chemical mechanism between APTMS and acetone to form a silica-like layer was reported elsewhere.[15,16] Amine group (−NH_2) in APTMS reacts with the carbonyl bond in acetone to form dimethylimino-propyltrimethoxysilane (DIPTMS) and water which subsequently supports the hydrolysis of alkoxides in DIPTMS to form hydroxyl (−OH) groups. By heating the PET film with a layer of DIPTMS, a Si−O−Si network is created. For type 3 PET, the coating of fluoro-based hydrophobic material resulted in a significant increase in fluoride percentage (39.16% F). Upon hydrophilic treatment by atmospheric O_2/Ar plasma or UV/O_3 modification, there were abrupt decreases in fluoride (1.07% F and 0.08% F for PET types 4a and 4b, respectively) together with considerable increases in oxygen (47.93% O and 51.8% O for PET types 4a and 4b, respectively) and silicon (25.37% Si for type 4a, and 28.68% Si for type 4b).

Table 1. XPS Analysis of Various PET Surfaces

PET type	process flow	atomic concentration (%)				
		C	F	N	O	Si
1	pristine PET	73.89	0	0	26.11	0
2	PET + APTMS	53.00	0	10.99	21.91	14.10
3	PET + APTMS + 1700	47.82	39.16	1.32	11.16	0.54
4a	PET + APTMS + 1700 + Plasma	23.83	1.07	1.80	47.93	25.37
4b	PET + APTMS + 1700 + UV/O_3	17.88	0.08	1.56	51.80	28.68

The similar XPS results of PET types 4a and 4b indicate that the hydrophobic layer of 3M 1700 (~50 nm thick) were removed by post plasma or UV/O_3 process to reveal the silica-thin buffer layers grafted (during APTMS coating). Indeed, by having longer exposure with either plasma or UV/O_3 than was necessary for the removal of the 3M layer, the exposed silica layer would become superhydrophilic (WCA<5°); see Appendix A of the Supporting Information for more details. High-resolution spectra of O1s and Si2p of types 4a and 4b provided additional evidence that the hydrophobic layer was removed with an increase in hydrophilicity of the exposed silica layer. Peak fitting results of collected O1s spectra (see Figure 5a, b) and Si2p spectra (Figure 5c, d) show that the plasma or UV treated surfaces composed of Si−OH at 532.6 eV (O1s) or 103.1 eV (Si2p). The high percentage of hydroxyl groups on the modified surfaces contribute to the superhydrophilicity obtained. During the plasma or UV process, organic substances are removed and replaced by −OH groups. Oxygen plays a critical role in forming active radical species to react with the surface. The step difference of ~45−126 nm (see Appendix B for detailed results) and a sudden change in surface energy (WCA difference ~100°) between the exposed areas and unexposed hydrophobic areas make the hydrophobic region a combined physical and surface-energy barrier. This is highly desirable for ink flow confinement and self-alignment for achieving high printing resolution. The physical step height can be adjusted by controlling the concentration of 3M fluoro-based chemical. Higher concentration solution leads to thicker hydrophobic layers and thus deeper walls.

3.3. Study of Hydrophilicity Stability.

The stability of superhydrophilicity on treated PET films after plasma or UV is important for practical applications. To investigate the optimum storage conditions for modified substrates, changes in WCA with time in various environments were observed for 6 days after treatments. Two environments were used including the normal clean room conditions (RH 55%) and dry environment inside a desiccator (RH 5%). Storage temperature for all cases was room temperature (25°C). As shown in Figure 6, the WCA recovery in normal storage condition is generally faster than that in the dry condition. In the high-humidity environment, the WCAs of UV/O_3-treated samples (type 4b) rose considerably (about 18° from 5 to 23°) during the testing duration. The WCAs of plasma-treated substrate under normal condition (type 4a) remained unchanged during the first two days and then significantly increased in the last four days. In the dry environment, it is found that all samples had slow degradation of hydrophilicity as WCAs increased slightly (from 5 to 7°). One potential reason is that water adsorption

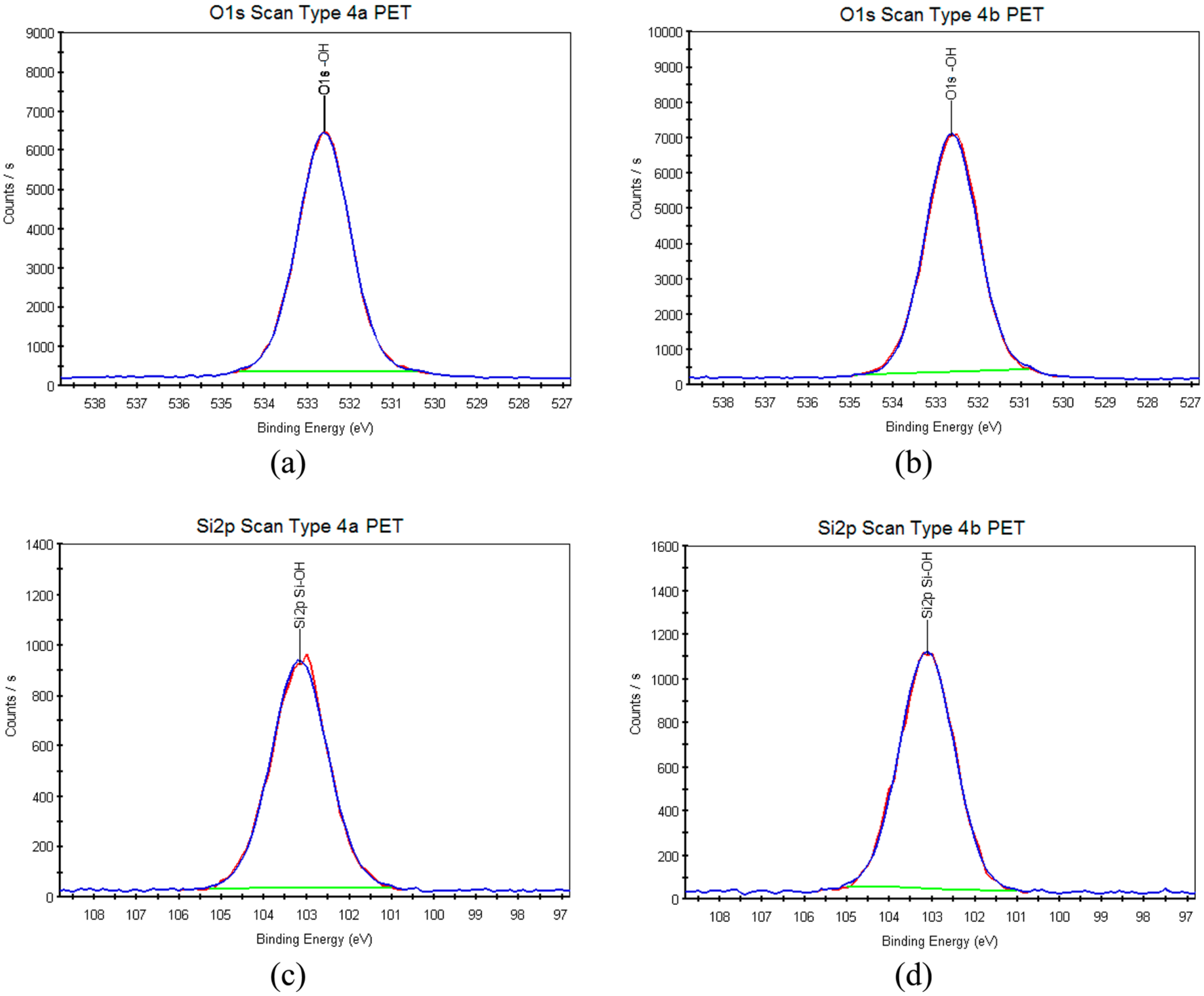


Figure 5. High-resolution spectra of PET types 4a and 4b. O1s spectra of (a) type 4a, (b) type 4b; and Si2p spectra of (c) type 4a, (d) type 4b.

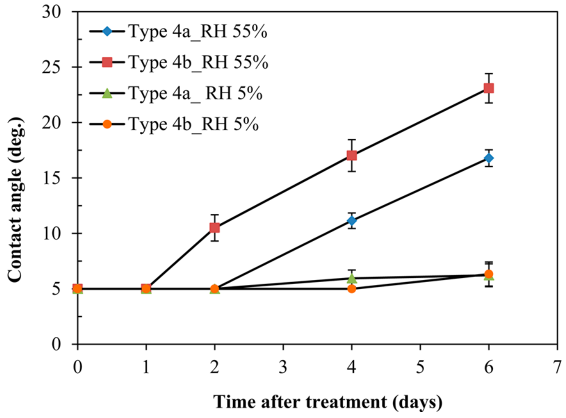
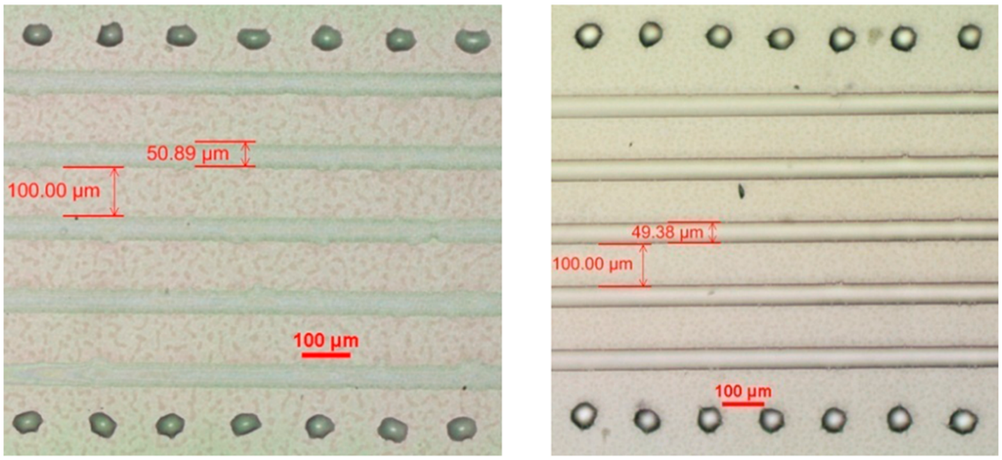


Figure 6. WCA recovery with time for various PET surfaces after plasma or UV treatment (types 4a and 4b, respectively) with different storage condition (humidity levels RH 55% or RH 5%).

to polymer surfaces stored under high humidity facilitates the hydrophilic losses. In brief, the plasma-treated sample in this study has a better hydrophilic stability than the UV-treated one. Proper storage condition with low humidity levels (RH 5%) is effective at reducing the rate of degradation.

3.4. Inkjet Printing Experiments. The wettability contrast between hydrophilic and hydrophobic regions is adjustable by varying the plasma or UV exposure time. In this study, the highest wetting contrast of 100° WCA difference attainable on PET films provided excellent surface conditions prior to printing. With this approach, hydrophobic regions on the modified substrates act as a combined physical and surface energy barrier for ink. Printing test was conducted to validate the feasibility of directing/confining the flow of ink by using surface-energy patterning. Ink-droplet-analysis showed that the generated droplets diameter was approximately 55 μm (or volume of 87 pL). The printing table moved at a speed of 20 mm/s. Printing distances between droplets in both x and y directions were set at 140 μm.

Figure 7 presents the printed results on the two types of PET films including types 4a and 4b. For all cases, conductive ink PEDOT/PSS only wet the hydrophilic lines while remaining almost circular on the hydrophobic nonwetting regions (refer to Appendix C in the Supporting Information for a discussion of the noncircular shape of the droplets). The ink can be self-

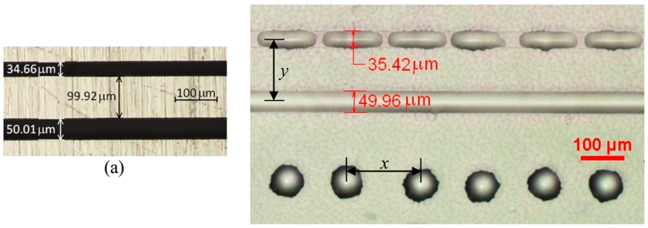


(a) (b)

Figure 7. Inkjet printed patterns on different treated PET substrates. Printed features are parallel lines (50 μm width, 100 μm spacing). Ink droplets spread on hydrophilic areas while remaining almost circular on hydrophobic nonwetting regions (a) type 4a PET and (b) type 4b PET.

aligned to spread only on the desirable wetting areas during the printing process and thus is capable of auto-compensating for small printing inaccuracy. This self-aligned feature may be of interest for the development of a complete roll-to-roll manufacturing process.

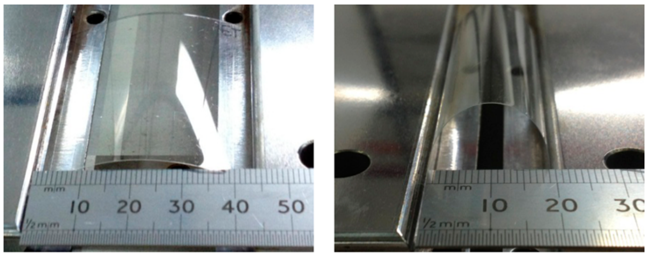
Another printing test was carried out on type 4b PET by using a new Ni mask (50 μm thick) having different line sizes (i.e. 35 μm and 50 μm linewidth as shown in Figure 8a). It is demonstrated in Figure 8b that the ink again successfully spread along the 35 and 50 μm lines. However, with the same printing pitch applied, the spreading length of ink droplets on the 35 μm line is shorter than that on the 50 μm and thus forms a 35μm wide interrupted line. One potential reason is that on a heterogeneous surface possessing a difference in wettability, a liquid drop is likely to move towards the areas of higher wettability. The driving force for this drop motion is proportional to the contact area.[17] The smaller contact area of the 35 μm line compared to the 50 μm one results in smaller driving force, which in turn leads to shorter spreading. However, by optimizing the printing pitch in the x direction, the spreading droplets should join to form a continuous line. This confirms that feature with dimensions less than the droplet size can be printed.



(a) (b)

Figure 8. Inkjet printing test on type 4b PET substrate patterned with different feature sizes (50 μm and 35 μm linewidth). (a) Optical image of structures on Ni mask. Darker areas are the exposed areas to plasma or UV. (b) Inkjet printed patterns on type 4b PET. Features with size smaller than the generated droplet size (~55 μm) can be successfully printed.

A bending test was conducted to evaluate the flexibility of the patterned PET substrates with coating layers (types 4a and 4b PET). The tested 50 × 50 mm^2 PET films were placed in the flexing machine (Starlight Tool Precision Engineering Ltd.) with an initial bending angle as described in Figure 9a. Each film was then bent by moving the film inwards automatically to the final position as shown in Figure 9b. After 10 000 bending cycles with a rate of 1 cycle/s, no cracks or delamination of the coatings could be observed.



(a) (b)

Figure 9. Bending test on PET films with coating layers (3 films each for types 4a and 4b PET): (a) initial position of the tested substrates freely supported with end to end distance of 45 mm, (b) final position of the bent substrates freely supported with end-to-end distance of 20 mm.

To confirm that the modified PET surface will not have a significant impact on the ink due to interaction between the modified surface and the ink, ink conductivity was measured after depositing on the surfaces of types 4a and 4b PET. However, it is difficult to measure directly and accurately the electrical properties of a small printed line of 50 μm line-width with a small pitch 100 μm, the measurement was conducted on a bigger line of 2 mm wide × 30 mm long. Tested PET samples were prepared as described for types 4a and 4b but using a new Ni mask having a line of 2 mm wide × 30 mm long. PEDOT:PSS ink was then deposited directly onto the lines by using the syringe system of Dataphysics OCA 40, Germany. The deposited ink was then left to dry in an oven at a temperature of 130°C for 10 mins. The average thickness of the resulting films was measured with a Dektak 150 surface profiler and the film resistance was measured by using the Fluke 287 multimeter. The average calculated conductivity of the deposited film was 38.7 ± 9.9 S/cm for type 4a and 42.9 ±

11.4 S/cm for type 4b. These values are in the range of the supplier specified values, demonstrating that the ink remains conductive on the modified substrates.

4. CONCLUSIONS

Controlling surface wettability of the substrate prior to printing is important to facilitate the printing of well-defined structures with the desired resolution. We presented here several methods to form selective wettability on patterned polymer surfaces. A silica-like layer was first formed on the substrate and served as a buffer layer for superhydrophilic treatment. This was followed by hydrophobic coating of fluoro-based chemical. Subsequently, the use of an electroformed Ni mask during plasma cleaning or UV/O_3 treatment patterned selectively the desired features. The hydrophobic layer of the exposed area to plasma or UV was removed, then the hydrophilic SAM layer behind was further modified to superhydrophilic conditions. As such, the remaining hydrophobic region covered by the Ni mask acted as a combined physical and surface energy barrier for ink. Between the two recommended processes, the approach with atmospheric O_2/Ar plasma is preferred as its overall process time is shorter and the WCA recovery in normal clean room condition is slower as compared to the UV/O_3 treatment. Inkjet printing was conducted to validate that surface-energy patterning can be used to control ink spreading. A high wettability contrast between printing and non-printing areas facilitates ink filling into the desired features and improves printing performance such as resolution and printing accuracy. In this study, a wetting contrast of up to 100° WCA difference can be achieved and feature less than the droplet size can be printed. The proposed processes utilize simple hydrophobic coating and atmospheric plasma treatment and thus show potential for mass production. Further studies on the smallest feature attainable with patterned PET film are currently being conducted.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

Superhydrophilicity of O_2/Ar plasma-treated APTMS layer and UV/O_3-treated APTMS layer. Measurement of step height between the hydrophobic regions and hydrophilic regions of PET types 4a and 4b. Effect of Novec 1700 layer thickness on the shape of printed PEDOT:PSS droplets. This material is available free of charge via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

The authors thank Dr Zhang Zheng from the Institute of Materials Research and Engineering for conduction XPS analysis of the PET samples. This project is funded by A*Star SERC Grant 102 170 0140.

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