CHAPTER VIII

DEPOSITION OF PEDOT: PSS NANOPARTICLES AS A CONDUCTIVE MICRO-LAYER ANODE IN OLEDS DEVICE BY DESKTOP INKJET PRINTER

8.1 Abstract

A simple micro-fabrication technique for delivering macromolecules and patterning microelectrode arrays using desktop inkjet printer was described. Aqueous solution of nanoparticle of poly (3, 4-ethylenedioxythiophene) (PEDOT) doped with polystyrene sulfonic acid (PSS) was prepared while its particle size, the surface tension and the viscosity of the solution were adjusted to be suitable for deposition on a flexible cellulose nanocomposite substrate via inkjet printer. The statistical average of PEDOT: PSS particle size of 100 nm was observed. The micro-thickness, surface morphology and electrical conductivity of the printed substrate were then characterized by profilometer, atomic force microscope (AFM) and four point probe electrical measurement, respectively. The inkjet deposition of PEDOT: PSS was successfully carried out, whilst retained its transparency feature. Highly smooth surface (roughness ~ 23-44 nm) was achieved.

8.2 Introduction

Reliable, low cost and optimized properties are fundamental prerequisites for researches in advanced surface coating and technology. Especially, in the field of electronic and photonic devices, there have been numerous publications on thin film formation via chemical vapor deposition (CVD) [172-174], physical vapor deposition (PVD) [95], vacuum deposition [130, 175] and plasma technique [17]. Even though these methods are excellent for thin film deposition, their major drawbacks are their requirement for special instrument accessory, high cost, and high energy consumption.

In contrast, inkjet printing [2, 176-180] is a commonly-used deposition technique for direct patterning or row-column pixel assemblies. It is rapid and simple, with lower operating cost than any vapor deposition technique. In addition, it offers a non-contact patterning feature. This technique has been applied in a variety of materials including ceramics, metals, polymers and composite. A standard desktop computer and a standard inkjet printer can control the delivery of small volume of ink for precise patterns. Inkjet printing technique has attracted interests of many academia and industries for commercial market of electronic device such as organic light emitting diodes (OLED) [146] , thin film transistors [181] , solar cells [182]. To effective apply the inkjet printing technique, parameters such as substrate properties, process parameters and ink solution properties have to be optimized. In general, inkjet printers often require conductive polymer size below 200 nm in order to prevent the clogging of print-head nozzles and to avoid the agglomeration among particle, as suggested by inkjet printer supplier.

Furthermore, ink formulation is also a key parameter. Several literatures propose conductive inks dedicated to inkjet printing process by using conductive polymer such as PEDOT:PSS (poly (3,4-ethylenedioxythiophene) poly(styrenesulfonate)) [91, 183]. This conductive polymer is commonly used in optoelectronic device due to its solution processability, high transparency in the visible wavelength and excellent thermal stability. PEDOT: PSS is a promising candidate to replace indium tin oxide (ITO) as a transparent electrode in optoelectronic device. There is an imminent need to find new transparent conductive materials to replace ITO, since indium is a scarce material in earth while market for optoelectronic devices has been rapidly expanding [184]. In addition, as PEDOT: PSS has high mechanical flexibility; it could be used in flexible electronic devices that are expected to be the next-generation of electronic devices. However, PEDOT: PSS has a problem of low conductivity. The film obtained from the neat PEDOT: PSS aqueous solution usually has remarkably lower conductivity than ITO. Therefore, it is important to significantly enhance the conductivity of PEDOT: PSS. According to several literatures, the conductivity enhancement can be achieved by adding small amount of solvent to the ink formulation [17, 185, 186]. The ink solution properties such as viscosity and surface tension also have to be controlled by appropriate solvent and surfactant.

Our earlier work has preliminarily evaluated the physical and electrical properties of flexible cellulose nanocomposite for its potential use as a substrate for optoelectronic device, especially for OLED application. Additional information on the preparation of flexible nanocomposite substrate, its characterization and scientific properties has been reported elsewhere [9]. In this work, the deposition of PEDOT: PSS solution on flexible cellulose nanocomposite substrate was evaluated. Process parameters and ink solution properties were investigated for printed electronic applications. Conductive patterns were printed using a PEDOT: PSS solution modified with organic solvent for electrical conductivity enhancement.

8.3 Experimental

8.3.1 Materials

Poly (3, 4-ethylenedioxythiophene) (PEDOT) doped with polystyrene sulfonic acid (PSS) and Triton X-100 were purchased from Sigma-Aldrich Chemical Company (Oakville, ON, Canada). They were used as ink suspension and surfactant, respectively. Surfynol DF-1100 defoamer was from Air Products Company (Allentown, PA, USA). Glycerol in reagent grade (>99% Reagent Plus) was purchased from Bioshop Canada Inc. (University of Toronto, Canada)

The flexible substrate preparation was successfully prepared by Wu [9]. The nano-scale cellulose was reinforced by polyurethane based resin (Sartomer Company Inc., Exton, PA, USA) as nano-composite substrate. The polyurethane based resin was stored in the darkroom in order to preferably avoid light sensitive problem prior to use. All of them were used as received.

8.3.2 Instruments

- Desktop inkjet printer

The Dimatix DMP-2800 inkjet printer (Fujifilm Dimatix, Inc., Santa Clara, CA, USA) was used to deposit conductive solution on 50x50 mm^2 substrate with a disposable piezo inkjet cartridge. The cartridge reservoir contained 2 ml of conductive polymer solution. The temperature of vacuum plate, which secured the substrate in place, was adjusted to 60 °C. The conductive solution was diluted with 50 wt% deionized water and filtered by nano-size filter before usage.

- Transmission electron microscope (TEM)

The particle size of conductive polymer was investigated by Transmission Electron Microscopy (TEM) Hitachi H-7000. The image obtained was processed with computer for identification of the domains in which certain lattice fringes appear.

- Profilometer

The film thickness was measured by Talysurf-10 profilometer, (Taylor Hobson Ltd., Leicester, UK) equipped with a diamond cursor running on the object surface and following local surface irregularities. The cursor was linked with an optical transductor. The vertical displacements were recorded by a photoelectric cell. The diamond point was 2.5 μm in thickness. Each measurement was 2.5 mm in length with an interval of 50 μm separating two parallel measurements. For the give test line, the roughness was defined in micrometer as the arithmetical mean of peaks above and under baseline. The mean thickness was an average of five successive measurements.

- Four point probe technique

The electrical conductivity was measured by the four point probe technique. This device was specified to have in-line fixed four probes made of solid tungsten carbide needles with 0.4 mm diameter. The nominal radius of a probe tip was 100 μm. Equal probe spacing, S, was 1 mm and downward force was approximately 0.2 kg per probe. A constant current, I, was applied to the bar specimen through two outside probes, V, was then determined. A DC precision power source and nanovoltmeter were used for all measurements. The electrical current I = 1 mA was maintained in all measurements.

- Tensiometer

The surface tension was measured by tensiometer technique with Wilhelmy plate. The paper was cut into 10 mm wide and 1 mm thick. For each withdrawing velocity of the Wilhelmy plate, at least ten times of dynamic meniscus were recorded and the coordinates of their contours were obtained.

- Viscometer

The viscosity was investigated by Ubbelohde canon type viscometer. The measurement used a capillary based method of measuring viscosity. The measurement was repeated five times and the statistical average was reported. This experiment was conducted at room temperature. Water was used as reference.

- Atomic force microscope (AFM)

The surface morphology and topology were investigated by AFM (Digital Instruments Nanoscope III Scanning Probe Microscope, Digital Instruments, CA, USA). The instrument was equipped with silicon nitride tip and operated in the non-contact mode. The scanning parameters were as follows: scanning rate 5 μm/s, resolution 300 and scan range at 50x50 μm. The image was taken from the optimized force between tip and sample at ambient temperature.

8.3.3 Methods

- Physical Chemistry of PEDOT: PSS solution

Poly (3, 4-ethylenedioxythiophene) (PEDOT) doped with polystyrene sulfonic acid (PSS) in aqueous solution as received was diluted with 50 wt% of deionized water. 5 wt% of Triton X-100 was added into the solution as a surfactant, followed by 1 wt% of Surfynol DF-1100 as a defoamer. Glycerol in reagent grade was consequently dropped in 1 wt% to enhance the conductivity of PEDOT: PSS. Then, the conductive polymer solution was homogeneously stirred and stored in the refrigerator. TEM, tensiometer and Ubbelohde canon type viscometer were employed to investigate particle size, surface tension and viscosity, respectively.

- PEDOT: PSS film formation by desktop inkjet printer

1 ml of the ink solution was loaded into inkjet cartridge reservoir. The plate temperature was set to 50 °C. 1, 3, 5 and 10 layers were deposited on substrate. Film thickness and electrical conductivity were subsequently determined by profilometer and four point probe measurement, respectively.

8.4 Results And Discussion

- Physical Chemistry of PEDOT: PSS solution

Nano-scale size of PEDOT: PSS particle in aqueous solution was observed by TEM. Figure 8.1 exhibits the particles in irregular shape with good homogeneity in aqueous solution. After filtering, the particle size is in the range of ~ 100-200 nm as the nozzle size of cartridge printhead is 200 nm in diameter [187]. In general, the wide particle size distribution was preferred to fulfill the free space between each particle. Higher packing efficiency of particle with different sizes offers higher electrical conductivity [188]. Higher packing efficiency will also help to avoid the problem of crack phenomena [189] in electronic device fabrication.

Figure 8.1 TEM image of PEDOT: PSS nano-particle in aqueous solution.

Table 8.1 Contact angle, viscosity, surface tension and conductivity of PEDOT: PSS Ink formulation.

Ink Formulation	**Contact angle (°)**	**Viscosity (cP)**	**Surface tension (mN/m)**	**Conductivity (S/m)**
PEDOT: PSS as received	44	15	62	0.1
PEDOT: PSS diluted with 50 wt% of deionized water	40	8	71	0.05
PEDOT: PSS diluted with 50 wt% of deionized water + 5wt% Triton X + 1wt% Surfynol DF-1100 + 1 wt% Glycerol	38	6	48	3.4

Remark: 1 wt% of Surfynol DF-1100 was added as defoamer in order to be well compatible with substrate and to prevent form during load into cartridge reservoir, suggested by supplier.

Table 8.1 shows contact angle, viscosity, surface tension and conductivity of PEDOT: PSS in 3 different formulations. The deionized water was added in order to dilute conductive polymer solution, following the PEDOT: PSS supplier's suggestion, as well as to adjust the viscosity of the ink for inkjet printing. On the other hand, glycerol was added to improve electrical conductivity [17, 186, 190]. Kim et al [17] suggested that adding small amount of organic solvent could make PEDOT chain shifted from a coil structure to an expanded-coil or a linear structure, based on electron spin resonance (ESR) microscopy and electrochemical experiments. Such conformational changes may lead to a change in the morphology of the PEDOT: PSS film. The free ion generated from organic solvent can be trapped with polymer chain, so called "polyelectrolyte", enhancing the mobility of free ion along polymer main chain [93]. However, it is important to note that only the appropriate amount organic solvent can be added for conductivity enhancement. Excessive solvent can lead to network structure formation among linear polymer chain and reduce the overall conductivity.

The surface tension of PEDOT: PSS decreased from 71 to 48 mN/m after adding surfactant, defoamer and conductivity-enhancer. In general, adding low concentration of surfactant is useful where micelle formation or micro-emulsion dominates. In this experiment, PEDOT was preferentially dissolved in the micelle assembly because of its hydrophobic nature. Adding the surfactant led to PEDOT particle connection as a consequence of the decrease in surface energy and interfacial tension between PEDOT: PSS solution and substrate.

The decrease in the surface tension of PEDOT: PSS was supported by the decrease in its contact angle. In this experiment, a small liquid was dropped on a flat horizontal solid glass surface. The shape of the droplet is determined by the Young's relation [191, 192]. The contact angle of PEDOT: PSS in aqueous solution at ambient temperature decreased from 44 to 38°. The liquid strongly attracted to the solid surface, and completely spread on the solid surface.

On the other hand, the viscosity decreased from 15 to 6 cP after the surfactant, the defoamer and the conductivity enhancer were added to PEDOT: PSS solution. The as-received PEDOT: PSS solution offered viscosity of ~ 15 cP while the acceptable range of viscosity of ink for use in our particular inkjet printer is between 2-10 cP, as suggested by Dimatix DMP-2800 supplier. The role of deionized water as diluent was added to decrease the viscosity of fluid. The viscosity of the ink solution was in the range of acceptable viscosity for Dimatix DMP-2800.

- PEDOT: PSS film formation by desktop inkjet printer

Table 8.2 The numbers of layer and film thickness measured by profilometer

Number of layer	Thickness (micron)
1	1.45
3	4.94
5	8.23
10	15.23

Table 8.2 exhibits the film thickness as a function of printing layers. Here, PEDOT:PSS diluted with 50 wt% of deionized water + 5wt% Triton X + 1wt% Surfynol DF-1100 + 1 wt% Glycerol was used as this formulation offered the highest conductivity. The film was successfully deposited by inkjet printer. It is a general practice that the film thickness is controlled to be as thin as possible to the prevent the accumulation of electric charge mobility at the interface layer [193, 194]. As traditional electronic devices are fabricated on rigid material [195, 196], the preferred nano-scale thickness can be achieved to avoid accumulation of electric charge mobility. Our fabrication was expected to find application in flexible display device. Therefore, the thickness of anode layer was increased from nano to micro scale range to maintain the device lifetime and to avoid crack phenomena when the device is bended [189].

Furthermore, electrical conductivity samples with different thickness were determined by four-point probe method [197, 198]. The schematic diagram of the apparatus is exhibited in Figure 8.2. By passing a current through two outer probes and measuring the voltage through the inner probes, the substrate resistivity can be determined.

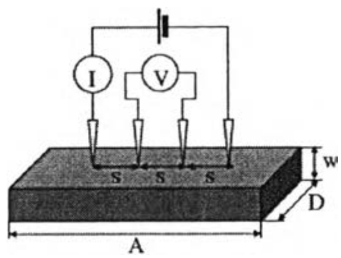

Figure 8.2 Schematic diagram of test circuit for measuring bar specimen resistivity with the four-point probe method

Conductivity, denoted by σ, was determined from sheet resistance data according to the definition of resistivity (ρ) [199].

Conductivity = 1/ (Resistivity)

Table 8.3 The relationship of numbers of layer and resistivity and conductivity

Numbers of layer	**3**	**5**	**10**
Resistivity (Ω-cm)	25	0.76	0.07
Conductivity (S/cm)	0.04	1.31	14.89

Table 8.3 illustrates the electrical resistivity and conductivity of samples printed with 3-10 layers of PEDOT: PSS. Electrical conductivity of PEDOT: PSS anode layer depends on a number of factors including conformation structure of polymer chain, content and distribution as well as the processing method and conditions. However, the most significant parameter determining the properties of PEDOT: PSS anode is the number of deposited layer. As a rule, the conductivity of film increases with increasing the number of deposited layer. In our experiment, in case of one deposited layer, the resistivity was beyond the capability of the four-point probe apparatus as the deposition was not sufficient. Three, five and ten deposited layers were successfully measured the electrical properties.

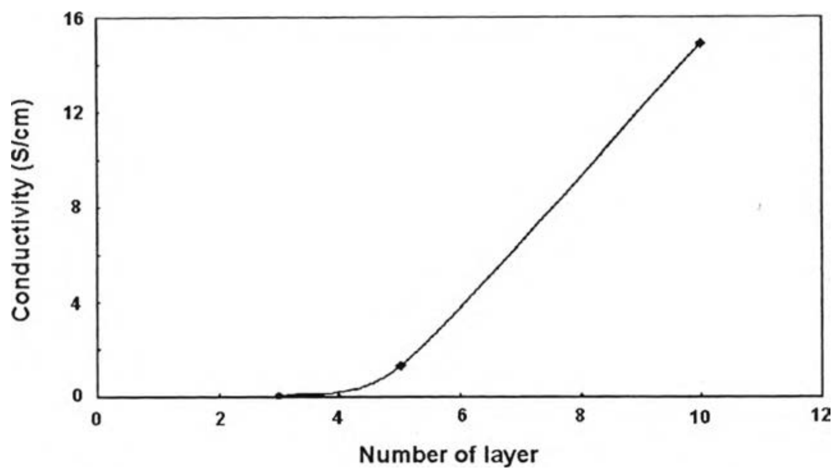

Figure 8.3 The linear relationship between number of layer and conductivity value

Figure 8.3 exhibits the number of layer and conductivity relationship. In the case of OLEDs device [146], the optimization between the small amount of number of layer and conductivity must be considered in order to preserve high transparency. In OLEDs theory, light is generated after the combination of positive and negative charge at emissive layer. The direction of light is passed though the transparent anode. According to Figure 8.4, the photograph exhibited that 3 and 5 deposited layers still yielded transparent properties. In the case of 1 deposited layer, the transparency is clear, although its electrical conductivity is to low to be detected. As for 10 deposited layers, the electrical conductivity is high, but the transparency became too low to be useful for OLEDs application.

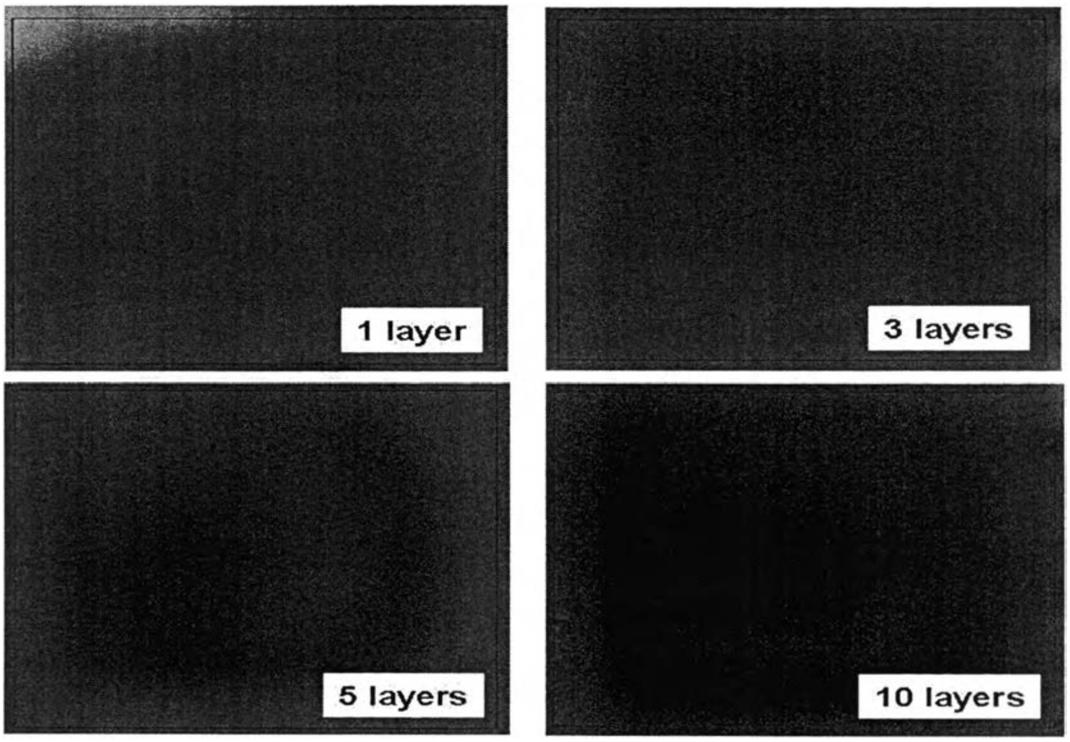

Figure 8.4 The photograph of PEDOT: PSS as anode by Desktop inkjet printer

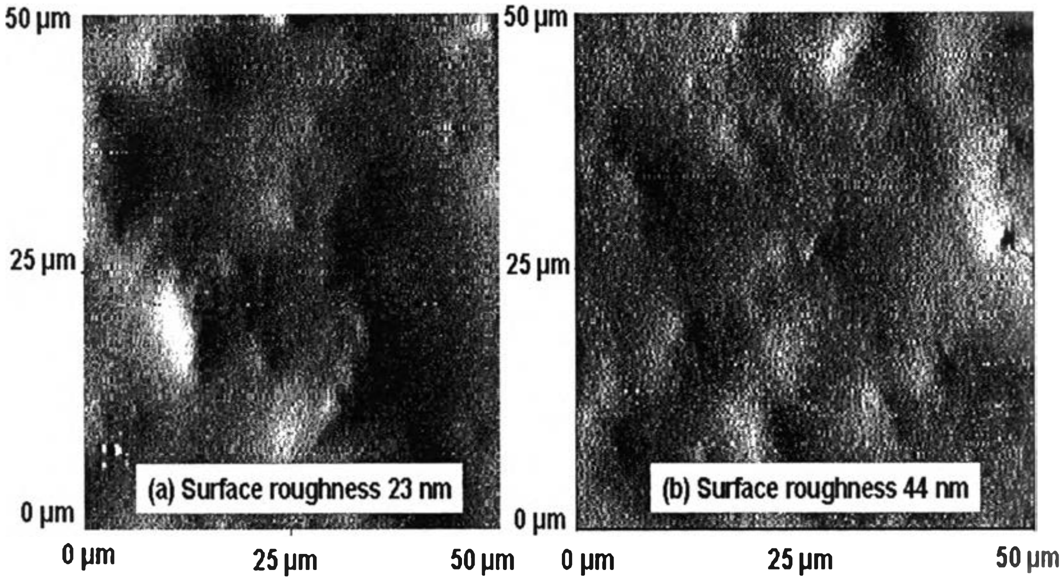

Figure 8.5 AFM investigation of PEDOT: PSS coated on resin in (a) 3 and (b) 5 layers, respectively.

Figure 8.5 illustrates AFM image taken with lateral contact mode. AFM image with scan size of 50x50 μm^2 was acquired for the surface morphological observation. In Figure 8.5, three and five layers of deposited PEDOT: PSS film was investigated. The root mean square (rms) surface roughness values of these 3 and 5 layers are 23 and 44 nm, respectively, indicating that smooth surface of PEDOT: PSS can be deposited on cellulose nanocomposite substrate. Legnani et al [5] reported the roughness of bacterial cellulose sheet as ~ 223.5 nm with scan size of 5x5 μm^2; therefore, the PEDOT:PSS deposition has significantly improved the smoothness of the substrate surface. It can be concluded that desktop inkjet printer was successfully applied to carry out the coating of uniform surface on anode for OLEDs device.

8.5 Conclusion

The excellent uniformity of PEDOT: PSS thin film has been successfully deposited on cellulose nanocomposite substrate by desktop inkjet printer. The conductivity of adjusted PEDOT: PSS solution with adding 1 wt% of glycerol can be enhanced up to 10 S/cm when multilayer was deposited, indicating the benefit of desktop inkjet printing technique in anode deposition for a flexible OLED device.

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