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Direct printing of conductive polymer PEDOT:PSS for foldable transient electronics

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Abstract

With the increased demand on portability, electronics have progressed from rudimentary flexible electronics to foldable electronics. Poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS), a conductive polymer, is a promising material for achieving foldable electronics, due to its mechanical stability. In foldable electronics, however, inadequate physical adhesion between electrodes and substrates under folding deformation has been a challenge. It can cause interfacial delamination and electronic failure during the folding and unfolding processes. In this study, electrohydrodynamic (EHD) printing is utilized for the fast, low-cost, and high-resolution fabrication of PEDOT:PSS circuits onto polyvinyl alcohol (PVA) films to improve the interface binding force for foldable electronics. The morphology and electrical properties of PEDOT:PSS patterns with different printed conditions were experimentally investigated . The adhesion between the printed PEDOT:PSS circuits and the PVA film was characterized by tape adhesion test, and the electrical property remained almost unchanged after 50 peeling tests. We demonstrated excellent foldability of the printed electronics. After 4 folds (16 layers), the resistance of PEDOT:PSS circuits varied minimally, and the external LED lights remained operational while folding and unfolding. Moreover, using the water soluble and degradable PVA substrate, the printed circuits can be simply dissolved in water, which provide a promising approach toward transient electronics and green electronics, and reduce the electronic waste.

Keywords: EHD printing; Foldable electronics; PEDOT: PSS; Transient electronics

1. Introduction

Over the past two decades, electronic devices have started to move from traditional bulky rigid electronics to light flexible electronics to provide convenient interfaces with non-flat surfaces and for wearable applications [1-4]. The elementary flexibility is not sufficient to meet the growing demand for device portability. These days, there has been a lot of focus placed on the development of bendable, rollable and even foldable electronics [5-10]. Conventional metallic electrodes exhibit excellent electrical conductivity. However, foldable electronics require an extraordinarily high level of mechanical stability, and these materials are not suited for foldable electronics because of their stiffness and fragility, which causes device circuit failure and susceptibility to interfacial delamination between device component layers. [11-13].

Poly(3,4-ethylenedioxythiophene):poly(styrenesulfonate) (PEDOT:PSS), as a conducting polymer, has been developed and widely used as an alternative to conventional metallic electrodes in flexible electronics [14-17]. PEDOT:PSS is a polymer electrolyte incorporating conducting conjugated PEDOT with positive charges and insulating PSS with negative charges. Polar solvents (such as DMSO, ethylene glycol, co-solvents, etc.) are normally used to increase the conductivity of PEDOT:PSS films [18]. Due to the fact that the mechanical property of pure PEDOT:PSS is reliant on the environment's relative humidity [19], the incorporation of stretchy polymers or small molecules into PEDOT:PSS is regarded to be an effective and efficient method for enhancing the mechanical stability. [16, 17, 20, 21]. PEDOT:PSS has the potential to be used as a conductive material for foldable electronic devices in this regard.

Achieving the required level of mechanical stability for foldable electronics is a major challenge for material selection and device design [22-24]. Compared to layer-by-layer deposition, embedding electrical components into a flexible polymer was demonstrated to be a promising method for creating a larger interface adhesion force [19, 25-28]. Traditionally, conductive materials were printed on rigid substrates, such as glass slides or wafers, and then the liquid polymer was cast onto the conductive material. After polymer curing, the conductive material was transferred and embedded into the polymer with a strong interface binding force. This typical approach requires various fabrication steps, which complicates the fabrication process. In addition, the likelihood of erroneous transfer leads to conductivity losses and makes precise patterning difficult.

The ever-expanding field of flexible electronics is being revolutionized by printing technologies, which enable more efficient and affordable methods for processing a wide variety of materials on many different substrates. Electrohydrodynamic (EHD) printing is a high resolution printing method that makes use of an applied voltage to print materials in a controlled manner on a wide variety of substrates. The application of an electric field causes the meniscus at the tip of the printing nozzle to deform into a cone shape (also known as a Taylor cone), which then ejects a thin jet or droplets [29-33] for material transfer. This printing mechanism allows for the printing of small patterns using a large nozzle. In EHD printing, fluid is dragged out of the nozzle tip with the assistance of an applied voltage, requiring less energy than in piezoelectric and thermoelectric printing. Many studies have been done on EHD printing of PEDOT:PSS [34-36]. With proper materials selection and printing process conditions, EHD printing makes it possible to directly print and embed PEDOT:PSS into flexible polymer substrates while maintaining a high level of resolution.

In this paper, we reported the EHD printing of PEDOT:PSS circuits embedded into the polyvinyl alcohol (PVA) substrate for the fabrication of foldable transient electronics. The morphology and electrical properties of printed PEDOT:PSS traces was investigated. The interface binding force between the PEDOT:PSS and PVA was evaluated. Moreover, a complex pattern of PEDOT:PSS was directly printed into the PVA as a foldable transient electronic which shows the mechanical stability during folding and unfolding.

2. Materials and Methods

2.1. Materials Preparation

The 4 wt. % aqueous polyvinyl alcohol (PVA) solution (Innovating Science) was deposited on the glass slides to form the heterogeneous film with ~90 μm thickness as the target substrate.

Ethylene Glycol (Sigma-Aldrich) was added into PEDOT:PSS (1.3 wt. % in water, PEDOTinks) at 5 wt. % to enhance the conductivity and Capstone FS30 (DuPont) was added at 3 wt. % to get better ink wettability. The mixture was stirred at 500 rpm for 5 hrs to obtain the ink for printing.

2.2. EHD printing

As shown in Fig. 1a, the EHD printing system was illustrated. It has three subsystems: the pneumatic dispensing system, the high voltage supply system, and the precise three-axis motion system. The pneumatic dispensing system is controlled by a pressure regulator, which provides pneumatic pressure to aid ink flow to the nozzle tip. The printing voltage is provided by a high-voltage supply with a maximum voltage of 10 kilovolts that is connected to the printing nozzle and the ground electrode. The precision motion stage consists of three linear stages in the XYZ direction and has a repeatability and accuracy of 100 nm. A camera with a high resolution is utilized to watch the printing process. Printed patterns were designed using CAD software before being translated into computer code. For EHD printing, a nozzle with an inner diameter of 234 μm and an outer diameter of 305 μm was used for printing. The printing voltage was selected to be 2000 V. The printing speed was 4 mm·s^{-1}.

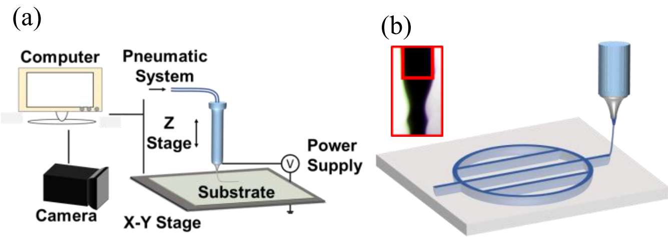

Fig. 1. (a) Schematic illustration of EHD printing system; (b) EHD printing process of PEDOT:PSS into the PVA film and the formed Taylor Cone with applied voltage (insert).

2.3. Printing process

In this study, the modified PEDOT:PSS ink was printed into the PVA film using the stable cone-jet mode of EHD printing, as shown in Fig. 1b, in which a continuous jet was ejected from the Taylor cone for the ink transfer. During the printing process, due to the water-solubility of PVA, the PVA underneath the printed PEDOT:PSS patterns is dissolved by the water in the PEDOT:PSS aqueous ink. A channel was created while printing the initial line and more PEDOT:PSS ink was printed into the channel as number of lines increases. The dissolved PVA and the printed PEDOT:PSS formed the PEDOT:PSS/PVA composite after annealing. The structure of embedded PEDOT:PSS in the PVA film was obtained. The interface binding force between the PEDOT:PSS and PVA could be extremely high in this way. By moving the printing nozzle in a preprogrammed manner, the feature was defined on the substrate. In EHD printing, the ink was continually printed into the substrate through a stream of ink. The formed cone shape and ejected filament have a smaller size than the nozzle diameter, which can print high-resolution features. After printing into the PVA, the samples were placed on a hot plate and annealed at 120 °C for 15 min to enable the curing process and improve the conductivity.

3. Characterization of printed PEDOT:PSS circuits

The performance of the PEDOT:PSS device is significantly impacted by both the morphology and electrical properties of the printed pattern. When it comes to foldable electronics, the interface adhesion has a considerable impact on the electrical robustness during folding and unfolding.

3.1. Morphology and electrical properties

In printed electronics applications, it is often needed to print multi-layers on the same trace to achieve the desired feature thickness and conductivity. The number of repetitively printed overlaid lines/layers along the same trace will affect the morphology and electrical properties of printed patterns. When printing PEDOT:PSS traces on the glass (Fig. 2a), the spreading of ink on the substrate after printing is very significant. The total number of printing along one trace indicates the volume of ink deposit onto the same area. As the number of overlaid lines/layers increases, the amount of printed ink onto the same area increases, resulting in more ink spreading. As a result, the line width of the printed features on glass becomes wider. When printing 5 layers along one trace, the resulting line width changed from about 500 μm to about 835 μm.

In contrast to printing on glass, printing PEDOT:PSS into PVA substrate provides controlled ink spreading, as shown in Fig. 2b. The PVA underneath the printed PEDOT:PSS was partially dissolved, and sank into the PVA substrate with limited spreading along the lateral direction. This effect regulated the shape of the printed features and prevented the lateral diffusion of ink with improved line width resolution.

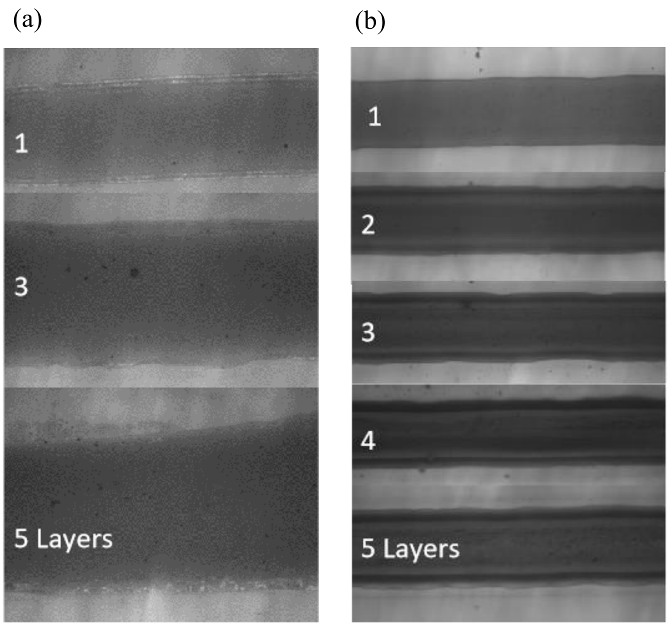

Fig. 2. Optical images of printed PEDOT:PSS traces with different overlaid layers at printing speed of 4 mm s^{-1} (a) on the glass slide and (b) into the PVA film Scale bars 200 μm.

After 5 layers were printed along the same trace into PVA, the line width did not vary considerably while the printed line became darker, indicating an increased feature depth and thickness. We measured the line width at 5 locations and calculated the average and standard deviation. The relationship between the line width and the number of printed layers is depicted as a scatter plot in Fig. 3a. When compared to the line width of a single line, the line width after printing 5 layers was only increased by about 20 μm. Printing PEDOT:PSS on PVA film exhibited advantages in preventing ink spread and keeping pattern consistency, particularly with increasing ink deposition volume.

After printing, heat treatment was performed to cure the ink, and increase the conductivity of printed PEDOT:PSS circuits. The sheet resistance of printed PEDOT:PSS of different lines/layers was measured and calculated. A 4-probe approach was utilized to measure the resistance (R) of these printed lines to get their sheet resistance, R_S. R_S was determined using the formula $R_S = R \times W/L$, where W and L are the width and measured length (set as 10 mm when tested) of the lines, respectively.

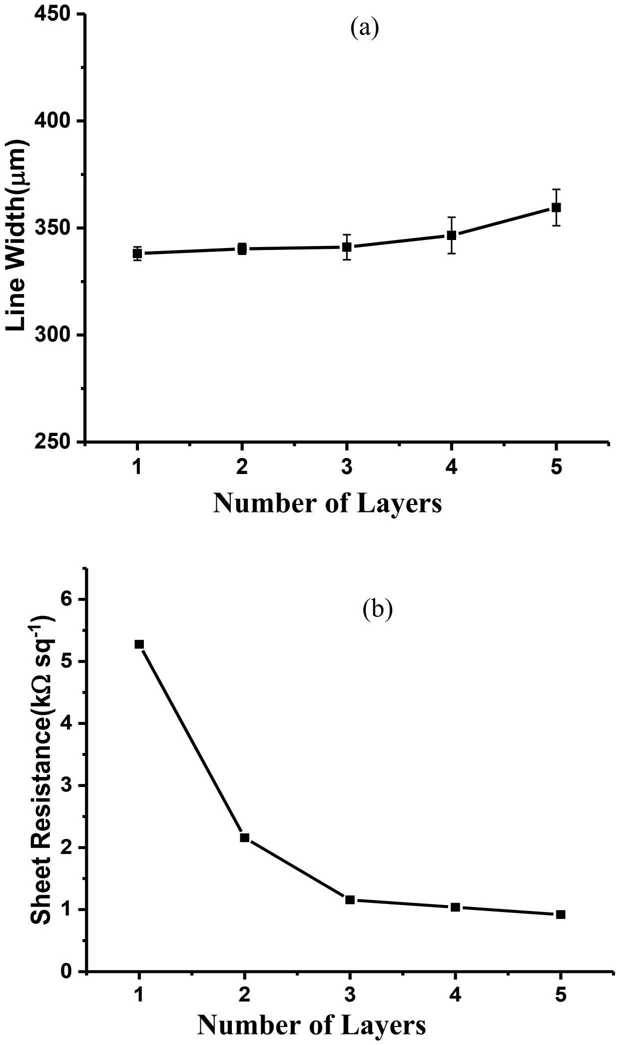

Fig. 3. (a) Line width vs. number of printed layers of PEDOT:PSS printed into the PVA; (b) Sheet resistance vs. number of printed layers of PEDOT:PSS printed into the PVA.

Since the printed PEDOT:PSS dissolves PVA on contact and forms a PEDOT:PSS/PVA composite along the printed trace following heat treatment, the electrical conductivity of the printed PEDOT:PSS into PVA could be reduced from that of pure PEDOT:PSS. From Fig. 3b, it can be seen that the sheet resistance of PEDOT:PSS printed with a single line was quite high, up to 5.37 kΩ sq^{-1}. When the number of overlaid layers increases from 1 to 3, the sheet resistance decreases sharply, and when the number of layers continues to increase to 5, the sheet resistance decreases slightly. Here, as the number of printing layers increases, the channel depth and the thickness of PEDOT:PSS/PVA increases, while the content of PVA in the composite decreases. The ratio of PEDOT:PSS and PVA affects the electrical properties of the composite [37]. At the first few layers, the PEDOT:PSS content increases quickly in the PEDOT:PSS/PVA composite that results in a quick drop of resistance. After a few overlaid layers, the PEDOT:PSS content in the PEDOT:PSS/PVA composite is already very high , the reduction of resistance become less significant. When the number of lines/layers increased to 5, the sheet resistance reduced below 1 kΩ sq^{-1}. In this way, we can achieve a controllable electrical property by printing multiple layers along the same trace without changing the feature's geometry. Since the sheet resistance of printed lines with layers from 3 to 5 doesn't change too much, we chose 3 as the number of layers for the following samples printing.

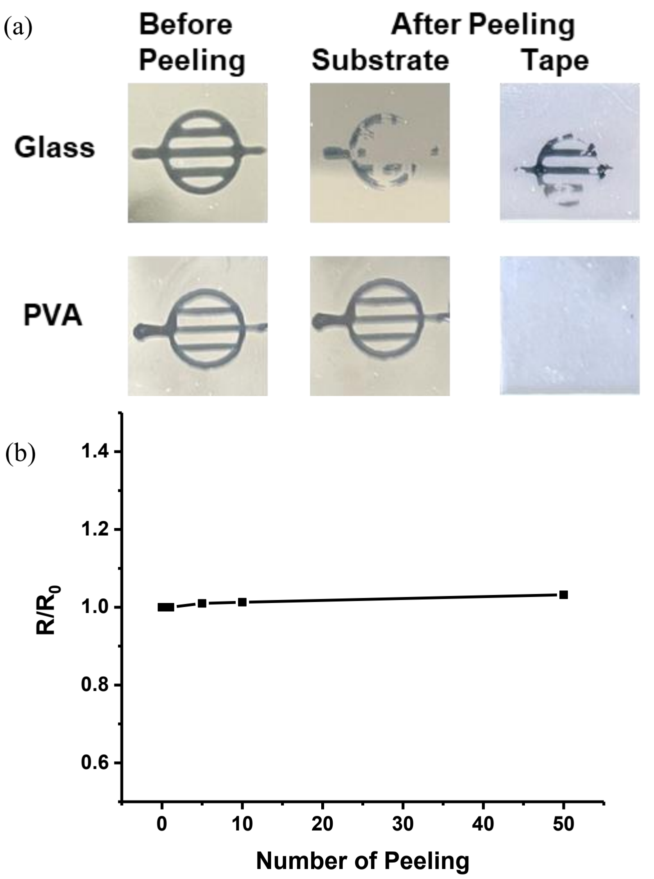

Fig. 4. (a) Adhesion test of printed PEDOT:PSS onto the glass (top) and into PVA film (bottom); (b) R/R_0 of 50 peeling in adhesion test.

3.2. Interface adhesion property

Besides the morphology difference when printing PEDOT:PSS onto different substrates (PVA vs. glass), the interface adhesion force between the printed PEDOT:PSS and substrates also changed significantly. As illustrated in Fig. 4a, during the Scotch tape adhesion test, the printed PEDOT:PSS on the glass was easily removed, indicating a poor physical adhesion between the printed PEDOT:PSS and the glass substrate. In contrast, a peeling test conducted on the PEDOT:PSS printed into PVA substrate showed that the Scotch tape removed very little to no material; that is, the PEDOT:PSS circuits embedded in the PVA film resulted in a strong interface adhesion between the printed PEDOT:PSS and the PVA substrate. This was owing to the high level of physical bonding as well as the chemical reactions that occurred. The resistance of printed PEDOT:PSS on PVA before peeling, R_0, and after peeling by Scotch tape, R, were measured at the two ends of the conductive line by a multi-meter. After 50 peeling tests, there was little difference in the resistance (Fig. 4b).

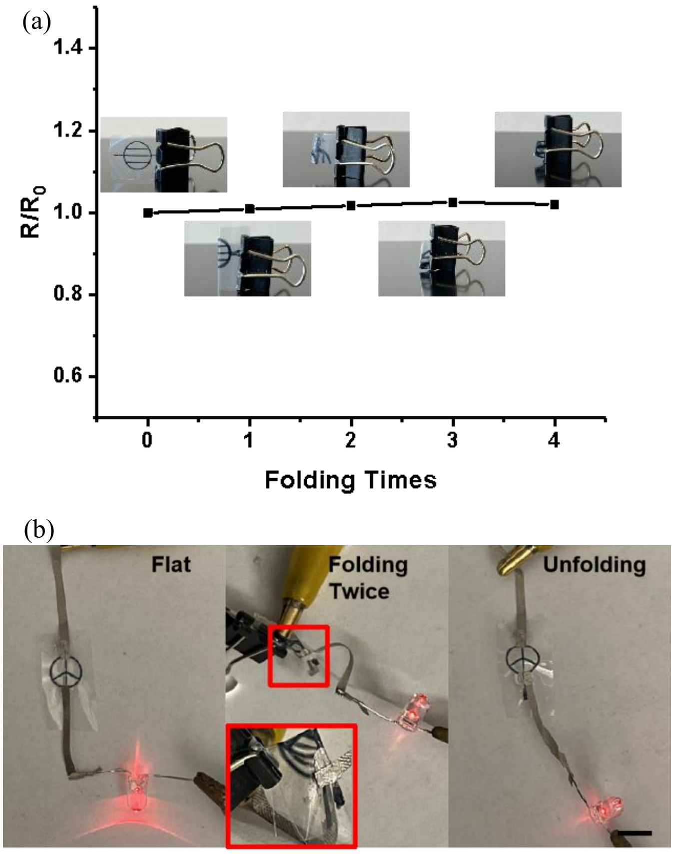

Fig. 5. (a) R/R_0 of the printed foldable transient electronic in the state of 0, 1, 2, 3, and 4 folds into 1, 2, 4, 8, and 16 layers, respectively. (b) The printed foldable transient electronic was connected with an external LED light and remained stable when flat, folded twice into 4 layers and unfolded. Scale bar, 2 cm.

4. Fabrication of Foldable Transient Electronics

The significant interface binding force between the PEDOT:PSS and the PVA film provides the excellent mechanical stability for foldable electronics. We demonstrated the mechanical foldability of the printed electronics by folding the printed PEDOT:PSS circuits 180° for several times into multiple layers. The initial resistance R_0 , and the resistance after folding R, were measured. The normalized resistance, R/R_0, was averaged using the results of 5 samples. As shown in Fig. 5a, R/R_0 stayed almost consistent when we folded the electronic once into 2 layers, twice into 4 layers, 3 times into 8 layers, and even 4 times into 16 layers.

To illustrate the robustness of the printed foldable electronic, an external LED light was connected to the circuit. As seen in Fig. 5b, the LED light remained the same regardless of the state of the foldable electronic device: flat, folded twice into two layers, or unfolded. This reveals that the foldable electronic is capable of maintaining its electronic response in both folded and unfolded states, which opens the way for the manufacture of more complicated portable electronics.

The water solvable PVA substrate also enables the easy dissolution of the printed electronics as transience electronics to reduce the E-waste. To illustrate the transience of foldable electronics, an evaluation of their dissolution/dispersion capability in Deionized water was conducted at ambient temperature by adding a small amount of water to the foldable electronic (Fig. 6). PVA is soluble in water, although its solubility in water at room temperature is quite low. For our printed foldable electronics, a thin layer of PVA allowed rapid dissolution with a small amount of DI water. After being submerged in DI water for 5 minutes, the PVA film disappeared completely. In spite of the fact that PEDOT:PSS is insoluble in water, the PEDOT:PSS film was able to be broken down into small particles and subsequently dispersed in DI water after 45 minutes of submersion in water and recycled differently.

Fig. 6. Transience of the foldable electronic.

5. Conclusion

In this work, we investigated the EHD printing of aqueous PEDOT:PSS ink onto PVA thin film for the fabrication of low-cost foldable transient electronics. The water solubility of PVA made it possible to print PEDOT:PSS to form an embedded trace on PVA, which assisted in maintaining the morphology of printing features under multi-layer printing. When 5 lines/layers were printed along the same trace, the line width only slightly increased by about 20 μm, while the sheet resistance was decreased as the number of printed lines increased. The electrical property of the printed PEDOT:PSS can be tuned without altering the feature dimension. The printing of PEDOT:PSS into PVA film also showed a high interface binding force, which is a critical requirement for foldable electronics. The printed PEDOT:PSS circuits maintained stable electrical resistance under severe folding and unfolding operations. Moreover, the foldable electronic exhibited the transient behavior of dissolution/dispersion in DI water. The electronic by printing PEDOT:PSS into PVA has demonstrated its cost-efficiency, foldability and transient properties, which provide great potential for high-resolution and scalable printing of foldable and transient electronics.

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