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Piezoresistive sensors on textiles by inkjet printing and electroless plating

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Abstract

We have printed arrays of strain sensors on textiles by inkjet printing of conducting lines and piezoresistive polymer (PEDOT) in order to provide detailed information about the response of a fabric in use. Conducting polymer has been printed onto polyamide and cellulose woven fabrics to form sensors using a modified HP inkjet print-head and X-Y linear positioning table. Good penetration and attachment is found on mercerized cotton but not on polyamide. Silver nitrate lines have been printed onto polyamide and converted to silver connectors by electroless plating. We observed that resistance of silver lines ranged from 0.7-1.5Ω/cm whereas for the conducting polymer it was 1-3 kΩ/mm by a four point probe method. The conducting polymer formed a surface coat on the fabric and also penetrated the weave. On stretching, the surface layer tended to crack but the embedded polymer acts as a strain gauge with a gauge factor of about 5. On the other hand the silver showed minimal change in resistance with stretching, as is required for connectors. Sensitivity towards temperature and humidity and the effect of orientation to stress and weave directions will be reported. Preliminary experiments show that these sensors attached to a sleeve could be effective for monitoring human joint motion.

Keywords

textiles, sensors, piezoresistive, inkjet, printing, plating, electroless

Disciplines

Medicine and Health Sciences | Social and Behavioral Sciences

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Piezoresistive Sensors on Textiles by Inkjet Printing and Electroless Plating

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ABSTRACT

We have printed arrays of strain sensors on textiles by inkjet printing of conducting lines and piezoresistive polymer (PEDOT) in order to provide detailed information about the response of a fabric in use. Conducting polymer has been printed onto polyamide and cellulose woven fabrics to form sensors using a modified HP inkjet print-head and X-Y linear positioning table. Good penetration and attachment is found on mercerized cotton but not on polyamide. Silver nitrate lines have been printed onto polyamide and converted to silver connectors by electroless plating. We observed that resistance of silver lines ranged from 0.7-1.5Ω/cm whereas for the conducting polymer it was 1-3 kΩ/mm by a four point probe method. The conducting polymer formed a surface coat on the fabric and also penetrated the weave. On stretching, the surface layer tended to crack but the embedded polymer acts as a strain gauge with a gauge factor of about 5. On the other hand the silver showed minimal change in resistance with stretching, as is required for connectors. Sensitivity towards temperature and humidity and the effect of orientation to stress and weave directions will be reported. Preliminary experiments show that these sensors attached to a sleeve could be effective for monitoring human joint motion.

INTRODUCTION

Electronic textiles or e-textiles are a new emerging field of research that brings together specialists in information technology, microsystems, materials and textiles [1]. Studies in the area of "electronic textiles" have recently captured researchers' attention worldwide because the fabrication of electronic systems on a flexible substrate such as textiles represents a breakthrough in many areas of military and civilian application [2].
A large number of studies have focused on the inkjet printing of organic molecules, metal nanoparticles, carbon nano material dispersions to form unique structures with piezoresistive sensing and actuation behavior. However, there are only few reports on the preparation of flexible piezoresistive strain sensors with defined and comparable gage factors using drop on demand thermal inkjet printing. Inkjet printing is considered to be one of the key technologies in the field of defined polymer deposition particularly in relation to the manufacturing of soft matrix based sensors, polymeric light emitting diodes displays and other polymer electronics.

The possibility of coating traditional textile fabrics with conducting polymers is quite recent. There are many types of conducting polymers such as polyacetylene, polypyrrole, polythiophene, polyphenylene, polyaniline, etc. Among these polypyrrole and polythiophene and their derivative show electrical conductivities that are stable at room temperature. Polythiophenes, in particular, serves as an obvious choice for research because of its processability and environmental stability. Polyethylene di-oxy thiophene (PEDOT) shows high conductivity, transparency and possess great environmental stability but is insoluble in water or any organic solvents. The insolubility of pure PEDOT can be overcome by using water soluble polyelectrolyte, poly (styrene sulfonic acid) (PSS), as a charge balancing dopant. PSS is added with PEDOT monomer during polymerization which leads to the formation of an aqueous suspension of PEDOT/PSS as shown in figure 1.

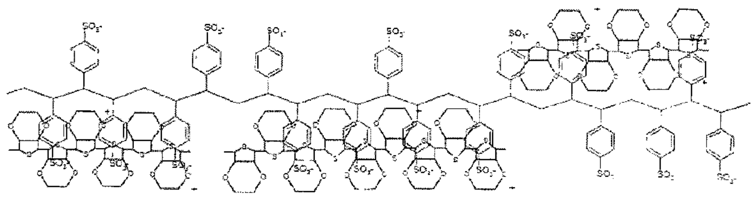

Figure 1. Segmented part of PEDOT/PSS

Another important property of polythiophene is that their electrical conductivity is sensitive to the surroundings which provide a basis for potential sensors.

The particular smart textiles effort here is aimed at stress and strain sensors printed on textiles to be the equivalent of proprioception in biology, providing information about the actions of the body for the purposes of controlling and monitoring muscle action.

In order to enhance our understanding of interactive textiles, we developed methods to inkjet print highly conducting lines to act as leads (connectors), printing more resistive conducting polymers to act as sensors and used them to provide semi-quantitative information about the motion of a human knee or wrist.

EXPERIMENTAL

<u>Formation of Connectors</u>

Formation of conducting leads was attained in two different steps. The first was to develop methods so as to inkjet print seed layers and second, to convert these seed layers into metallic lines by electroless plating.

0.32gms of silver nitrate, dissolved in 50ml of water was printed on the woven fabric (Nylon 6, 6 Semi-dull, Taffeta) by using a specially modified HP print head. The print head was used in conjunction with an X-Y positioned table which is driven by a Stepper motor and Velmex™. Figure1 shows an ink jet printing set up that is currently being used.

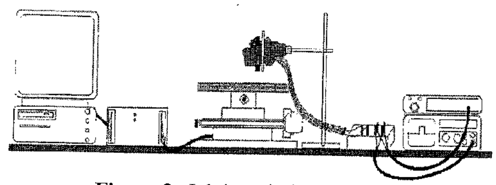

Figure 2. Ink jet printing assembly.

Since the ink, upon evaporation, leads to spreading on the substrate the woven fabric was mounted on a heating plate so as to avoid the spreading of the solution. This technique allows the individual droplets to fuse but hinders the formation of large droplets and thus prevents smearing of ink [7]. Once the lines, 3 cm long and 0.5 mm wide, are printed, the sample was dipped in an electroless bath of silver. The bath was maintained at a temperature of 50 degree Celsius and the hold time of 40 minutes. The pH of the bath was kept highly basic at around 12.5. After the sample is subjected to the required set of conditions, it is taken out of the bath and rinsed in hot water followed by a rinse in cold water along with mechanical action such as wringing.

In order to avoid precipitation of the compound in the electroless bath (because the plating is carried out in basic solution), ammonia is added to the electroless bath. This acts as a complexing agent [8].

The complex formation reactions are reported to be as follows:

$$2AgNO_3 + 2NH_4OH \rightarrow Ag_2O + 2NH_4NO_3 + H_2O$$

$$Ag_2O + 4NH_4OH \rightarrow 2[Ag(NH_3)_2]OH + 3H_2O$$

$$[Ag(NH_3)_2]OH + NH_4NO_3 \rightleftharpoons [Ag(NH_3)_2]NO_3 + NH_4OH$$

The reducing agent, glucose, in this case reduces the silver from its complex form to the silver atom and this silver in turn is deposited on to the already printed seed layer, proving the process to be autocatalytic [8, 9].

$$2[Ag(NH_3)_2]OH + RCHO \rightarrow 2Ag + 4NH_3 + RCOOH + H_2O$$

The focus was on attaining thick and uniform deposition of silver nitrate on the nylon woven fabric so as to form a continuous network of silver atoms on the printed regions during plating.

The resistance of the samples, after they have dried, was measured using Keithley 196 electrometer and also by 4-probe electrical measurements. The probes were kept on the printed region, and the corresponding reading from the front panel was taken down. The value of resistance ranges from 0.7- 1.5 ohms/inch. Upon calculating the resistivity, we obtained was about 4×10^3 Siemens/cm. This is off by the order of 10^2 from that of elemental silver. The conductivity of elemental silver is 6.25×10^5 Siemens/cm.

Formation of Sensors.

Suspension of Poly (3, 4 – ethylenedioxythiophene) - poly (4-styrenesulfonate) (PEDOT-PSS) from Bayer Scientific, 1.3 % by weight, was printed onto mercerized cotton fabric. The printed lines were about 5 cm long and less than 1 mm wide. The samples were annealed at 90 degree Celsius for about an hour. In this case the polymer penetrated the fabric and formed stable conducting lines. On Nylon 6, 6 it formed a surface layer which flakes off when the fabric is bent or stretched. The resistance drops as more ink is deposited. The conductivity of the PEDOT in the coatings is about 25 S/cm.

The conductivity is thought to involve quantum tunneling between particles and this is presumably the source of the piezo resistivity, the change in resistance with strain. The resistance is expected to be sensitive to temperature, humidity and the state of annealing of the polymer. For the strain sensing study, the fabric was clamped in jaw placed 2.54 cm apart on an Instron tensile testing machine and the corresponding resistance was recorded on a Keithley Multimeter 196 through a PC using General Purpose Interface Bus (GPIB) as a data acquisition mode.

The microstructure of the printed PEDOT and electroless silver was studied by JEOL JSM 5610 scanning electron microscope equipped with Oxford Energy Dispersive X-ray System operating at 8 kV.

Integration of Sensors and Connectors.

Resistance of connectors was 1/ 100 of that of sensor in order to avoid any interference with the data recording. Sensors were integrated with connectors by printing lines of PEDOT, 1 cm in length and about 0.5 mm wide, in between silver printed lines. The connectors were connected to an external device and the resistance was recorded on Keithley 196 electrometer along, interfaced with GPIB.

DISCUSSION

SEM and EDX observation

SEM study, in figure 3, shows coating of silver around every fiber through the thickness of the fabric. EDX analysis confirms silver on the fabric, figure 4. The second silver immersion step does also result in some silver being deposited on the unprinted regions and some staining but these do not become conducting. EDX also shows some silver penetrates into the individual Nylon fibers.

SEM of cotton fabric coated with conducting polymer, in figure 5, shows that part of the polymer forms a thick film on the upper surface of the fabric and cracks on elongation. This may not be true of the polymer penetrating deeper.

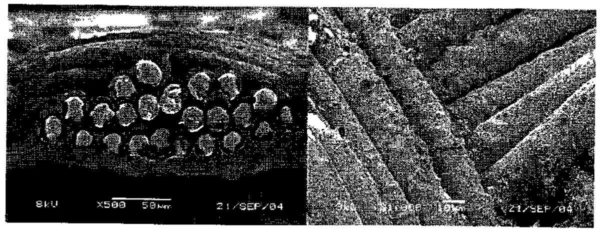

Figure 3. Cross section and top view of silver printed region of a Nylon fabric.

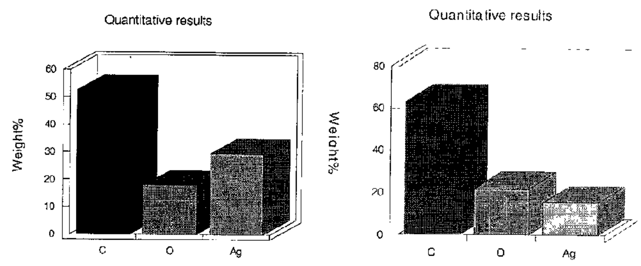

Figure 4. EDX of silver-treated fabric cross-section and EDX of a single Nylon filament showing silver penetration into the core of the fiber.

Strain Sensitivity

The reversible change in the electrical resistance of PEDOT-PSS printed fabrics, as with external mechanical strain relaxation reveals that these fabrics possess mechano-electrical properties. For this study 2.54 cm long PEDOT printed cotton fabric was subjected to an elongation of 5% at a rate of 5mm/min on an Instron tensile testing machine.

Figure 6 shows an increase in the resistance of the PEDOT printed fabric with strain and a decrease with relaxation. The gauge factor is positive and is about 1.5. This value is quite similar to those of metals. Metals have a gauge factor of about 2 and semiconductors have a gauge factor of an order or a couple of orders more [10, 11]. We can, therefore, say that resistance change may reflect some change in intermolecular bond length. Earlier work by De Rossi et al have shown that values of gauge factor of polymers similar to metals have potential application to be used as sensors in wearable applications [12].

However it is not possible to attain original resistance soon after the experiment because PEDOT is printed on cotton woven fabric which shows only 61% of recovery from strain as compared to Nylon fabric (91%) and some knit fabric (almost 100%),although with time original resistance is recovered..

Figure 5. PEDOT-printed cotton before and after 10% elongation

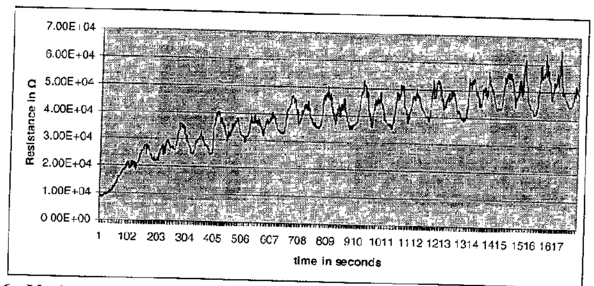

Figure 6. Variation in Resistance of PEDOT-PSS coated fabrics Vs time within 25 repeated cycles of strain and relaxation.

Identification of Human Motions.

The assembly of sensors and connectors was placed on human knee, as shown in figure 7, and wrist with the help of tape.

Figure 7. Bending of knee

Four trials for each motion, bending of knee and twisting of wrist, was carried out at both slow and fast speed and corresponding change in resistance was recorded with the help of Keithley 196 Electrometer and GPIB. Both time and frequency domain analysis was carried out of the readings as a part of digital signal processing.

For analysis, since the original data being noisy, it was necessary to take some procedure to reduce the noise. The mean filter was employed in this case. The mean filter is a simple sliding-window spatial filter that replaces the first value in the window with the average (mean) of all the data values in the window. The plots, shown in figure 9, are the two trials after removing running average and taking twice mean filtering in time domain. Pseudo-sinusoidal wave is showed in the data also after doing signal processing. Approximate 7 pseudo-sinusoidal cycles are contained in 50 sec. In frequency domain, existed data was used to estimate the power spectrum density of it. The power spectral density (PSD) function describes the distribution of power with frequency of the random process

Two reference trials for slow bending motion (Knee):
In Time domain:

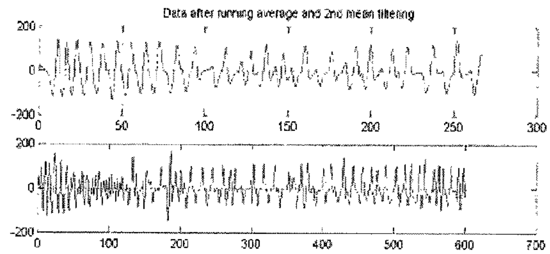

Figure 9. Resistance (k-ohms) Vs Time (seconds)

In frequency domain.

The plots, shown in figure 10, are the power spectral density for two trials. From the two trials, the frequency peaks are not fixed from trial to trial. It may be caused by the human motion data collection. However from trials, there exist three major frequency peaks in the PSD. Although the peaks are shifting, they all concentrate below normalized frequency 1. If there exists features for the slow bending motion, we can say that from the four trials that most of the power are concentrated below normalized frequency 0.5.

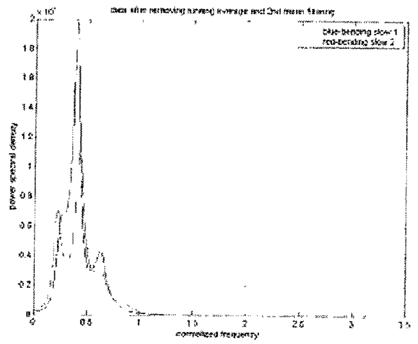

Figure 10. Data after removing running average and 2^{nd} mean filtering

Trial 1 – ratio = (power below w = 0.5 rad/sample)/ total power = 0.67498
Trial 2 – ratio = (power below w = 0.5 rad/sample)/ total power = 0.57909

Slow Speed: From the analysis including four trials from slow knee bending motion as well as four trials from slow wrist twisting motion, we were able to trace down some distinguishable features in both time domain and frequency domain.

In time domain, in general, there were approximate 7 pseudo-sinusoidal cycles contained in 50 sec for slow knee bending motion. For slow wrist twisting motion, approximate 12 pseudo-sinusoidal cycles were contained in 50 sec.

In frequency domain, one can set up a threshold for the power ratio to distinguish the bending and twisting motion in the fixed slow speed. Based on the eight trials, the power ratio threshold was set at 0.5.

Fast Speed: In time domain, the periodicity from the smoothed signals was used to separate the two motions. For fast twisting motion (wrist) the period was approximately 8 to 9 sec. per cycle, and for fast bending motion (knee) the period was about 3 sec. per cycle. In frequency domain, the peaks in each trial were not fixed and also the power ratio method was not working at the fast speed motion.

CONCLUSION

Controlled inkjet printing of PEDOT onto cotton fabric resulted in selective conductivity, which increased with curing of the printed sample. Inkjet printing and electroless plating lead to the deposition of silver on nylon 6, 6, as connecting leads, with width of 0.5 mm. SEM and EDX observations revealed thick deposition of silver on Nylon 6, 6 fabrics even after washing and rubbing which indicates reasonable adhesion between silver and fabrics. PEDOT-PSS printed fabrics showed a reversible change to applied external strain. Electrical analysis proved that human motions can be sense by the printed assembly of sensors and connectors.

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