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Polypyrrole-coated Fabric Strain Sensor with High Sensitivity and Good Stability

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***Abstract*—The sensitivity and stability are mainly factors to hold back the practical applications of Polypyrrole coated fabrics. In this paper, a flexible fabric strain sensor with high sensitivity, good stability and large deformation is reported. It is fabricated by depositing a nano-layer (200nm to 300nm) of polypyrrole on the fabric substrate at low temperature. Thickness and morphology of the conducting thin film on the surface of the fibers were examined by scanning probe microscopy (SPM) and scanning electron microscopy (SEM). The measurement of the conductivity change with strain shows the fabrics so prepared exhibits a high strain sensitivity of ~160 for a deformation as large as 50%, while its good stability is indicated by a small loss of conductivity after the thermal and humidity aging tests, and supported by the slight change in conductivity and sensitivity over a storage of eighteen months. The flexible strain sensor is expected to be a promising "soft" smart material with good sensing properties in the preparation of smart garment, wearable hardware and biomedical applications.**

Keywords- fabric strain sensor; polypyrrole; stability

I. INTRODUCTION

Polypyrrole (PPy) has been widely investigated as actuators, chemical sensors, biomaterials, batteries, gas separation films, etc. [1]. When it forms composites with fabrics, the obtained conductive fabrics can also be used in electromagnetic insulation, microwave absorbance, etc. due to the combination of both the mechanical properties of the flexible fabrics and the electrical, microwave properties, and biocompatibility of the PPy coatings [2, 3]. In recent years, it was found the conductivity of the PPy-coated fabrics is also sensitive to strain, thus the conductive fabrics can be used to measure and control the movement of human body and construct the wearable devices [4].

However, these conductive fabric strain sensors exhibit low sensitivity and unsatisfying stability. D. Rossi et al. developed a sensorized glove based on the PPy-coated Lycra/cotton, but the sensor was aged severely in air and the conductivity decreased continuously. Furthermore, the saturation of the sensor occurred at a small strain of about 6% in [4]. K. W. Oh et al. reported the PPy-coated Nylon-spandex was sensitive to strain change until a deformation of 50%, but the sensitivity is as small as no more than 2 in [5]. X. P. Jiang et al. also proposed the PPy-coated PET/Sapndex can be used as a strain senor for a large deformation of up to 50%, while the sensitivity is only 3 in [2].

We report a PPy-coated flexible fabric strain sensor which was prepared by depositing a nano-layer (200nm to 300nm) of polypyrrole on the fabric substrate at low temperature. The sensor so fabricated shows both high strain sensitivity for a large deformation and a good environmental stability.

II. EXPERIMENT

A. Preparation of the Fabric Strain Sensor

PPy-coated fabric strain sensor was prepared by chemical vapor deposition (CVD) at the low temperature. The typical procedure is as follows: Fabrics composed of 83% Tactel and 17% lycra (195g/m^2, from Sunikorn Knitters Limited) was first immersed into the solution containing sodium dodecyl benzene sulfonate (0.011 mol/L) and $FeCl_3$ (0.1 mol/L), and the wet take-up was controlled by using a padding machine (PA-0, Rapid Labortex Co. Ltd). The fabrics and a beaker containing 10 mL pyrrole monomer were transferred into a desiccator, which was evacuated under vacuum and flushed with N_2 repeatedly and put in a fridge where the temperature is kept below -10 ^{o}C. The vapour phase polymerization of polypyrrole proceeded on the surface of the fabrics under vacuum. The black fabrics so obtained were then washed with deionized water and ethanol, respectively, and dried at 40^{o}C under vacuum. The annealing was carried out by heating the dried fabrics at 60^{o}C for 40 hours under vacuum.

B. Measurement

The strain-stress properties of the PPy-coated fabrics were obtained using an Instron testing Instrument (Model 4466) under the standard testing conditions (T = 25^{o}C and RH = 65%). The fabrics were repeatedly stretched and relaxed for 10 cycles with the maximum extension up to 12.5 mm (50% deformation) in each cycle. The conductivity change of the sensing fabrics in both stretched and relaxed states was recorded using a digital multi-meter (Keithley Model 2010) to investigate its strain sensitivity. SEM images of the pristine and PPy-coated fabrics were obtained using a scanning electron microscope (Leica stereoscan 440). The morphology and thickness of PPy film on the surface of the fibers were examined by a scanning probe microscope system (SPI4000/SPA300HV from Seiko Instruments) under ambient conditions. SPM images were obtained from cross-sections and the surface of a composite fiber, respectively. Both thermal and humidity aging tests were carried out by recording the conductivity change of the PPy-coated fabrics put in a climatic chamber (Hotpack Series 922), where both the temperature and

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humidity can be controlled. For the thermal aging, the humidity is always kept at 65%RH. The temperatures investigated include 20, 35, 50 and 60°C and the temperature changes every two hours. The humidity aging was carried out at 30°C. The humidities investigated include 40, 55, 70 and 90%RH and the humidity changes every two hours.

III. Results and Discussion

With respect to low sensitivity of PPy-coated fabric sensor prepared by the solution polymerization in [2, 5], we propose the low sensitivity is related to the thick coatings composed of multilayers of PPy deposited on fabric substrate. The multilayers of PPy may not change in the same way as the substrate when the fabrics are elongated. To obtain a flexible strain sensor with both high sensitivity and good stability from PPy coated fabrics, we employ the method of CVD at low temperature to obtain very thin and dense coatings of PPy on the surface of a Tactal (83%)/Lycra (17%) fabrics.

To understand the mechanism well, morphology and thickness of PPy film on the surface of the fibers were examined by SPM. In Fig. 1(a), the insulating region is shown in the darker region and the electrically conductive region in the lighter region. PPy displayed a continuous pathway of conductive domain with 200nm to 300nm in thickness on Tactal fibre base. Fig. 1(b), (c), and (d) gives the phase images of Tactal fibre and topographical views from different viewpoints, respectively. Fig.1 (b) and (c) show that the PPy coating film is composed of well-connected PPy granules, covering up whole fibre surface with finer, denser, and more uniform. Fig.1 (d) shows the height of the rugged PPy coating layer on the Tactal fibre. As shown in Fig. 1, the PPy tended to form a connected layer on the substrate when polymerization was carried out. Before the well-connected first layer formed, PPy oligomer nucleated on the substrate in numerous places. Due to the strong interaction between the substrate and PPy, the nucleation rate is fast and the distance between nuclei is relatively small. The nuclei grows quickly in the lateral directions and spread throughout the surface of the fibres. The initially connected layer comes from multiple nucleation sites with either isotropic or anisotropic growth rates on the substrate. Further work is required to investigate the interaction between the PPy and substrate polymers.

Fig. 1 (a) Conductive layer of PPy-coated fibre. The scanned area is 6 X 6um

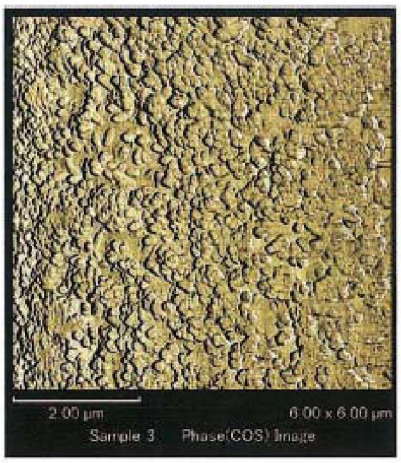

Fig. 1 (b) Morphology of the PPy coating layer from different viewpoint.

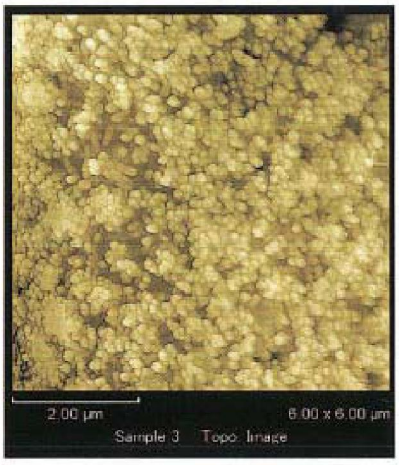

Fig. 1 (c) Morphology of the PPy coating layer from different viewpoint.

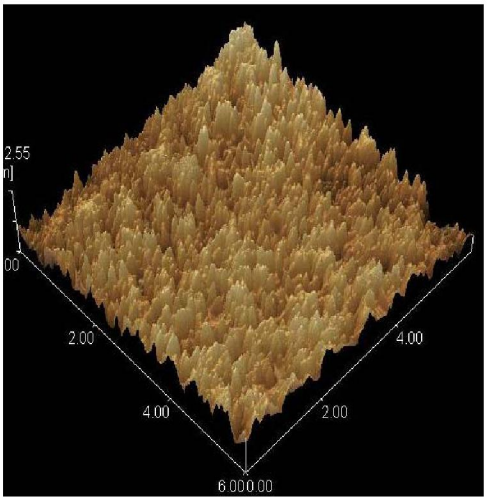

Fig. 1 (d) Height of PPy-coated layer. The scanned area is 6 X 6um

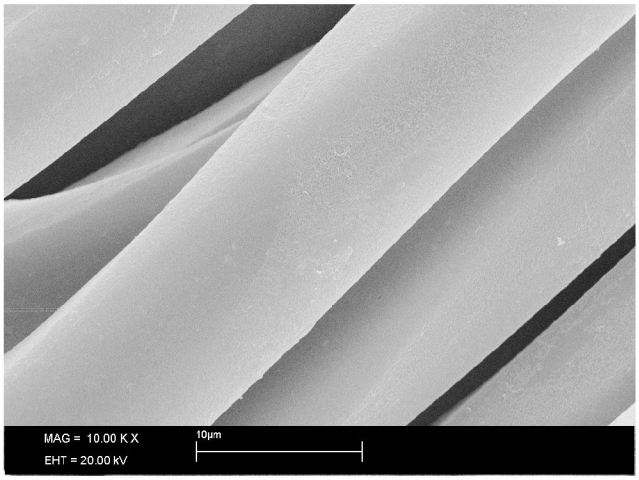

Fig. 2 (a) SEM images of pristine fabrics.

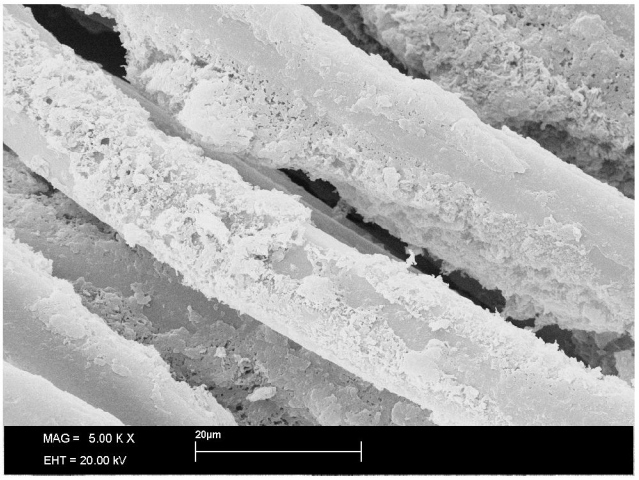

Fig. 2 (c) SEM images of PPy-coated fabrics prepared by solution polymerization.

The low temperature polymerization of pyrrole on surface of fabrics was helpful for obtaining thinner coatings resulting in more adherent film and more ordered structure of PPy with higher conductivity. Fig. 2(a) , (b) and (c) show SEM photography of the film of pristine fabrics, PPy-coated fabrics prepared by CVD at low temperature and by solution polymerization, respectively. It reveals that the fabrics coated with PPy formed by CVD exhibits a very smooth surface as the pristine one, while that prepared by the solution polymerization is featured with a rough surface. The thin PPy coatings are helpful to improve strain sensitivity; however they are unbeneficial for environmental stability. A smooth and dense layer of PPy film is more resistant to the attack of oxygen and water molecules, thus enhances its stability.

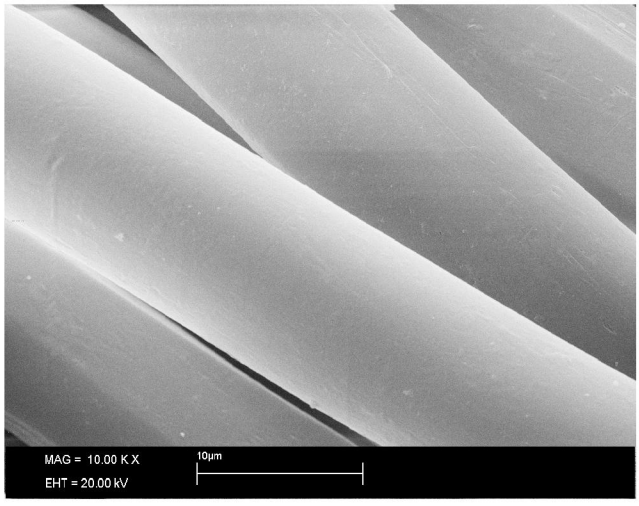

Fig. 2 (b) SEM images of PPy-coated fabrics prepared by CVD at low temperature

The typical conductivity change-strain curves of the PPy-coated fabrics prepared by CVD at low temperature during ten cycles of elongation and relaxation measurements are illustrated in Fig. 3. The resistance of the sensor increased sharply with the extension up to a large deformation of 50%, and the sensing curves are almost the same during ten cycles of the test, suggesting good repeatability of the sensor. Normalized resistance $\left[\frac{\Delta R}{Ro}\right]$ is about 80, and the strain sensitivity $\left[\frac{\Delta R}{\varepsilon Ro}\right]$ is as high as about 160, where ΔR are the resistance change of the fabric under extension and R_0 is the original resistance, i.e. the resistance of the fabric in relaxed state, ε is the extension, respectively. To our best knowledge, this is the highest sensitivity reported up to date.

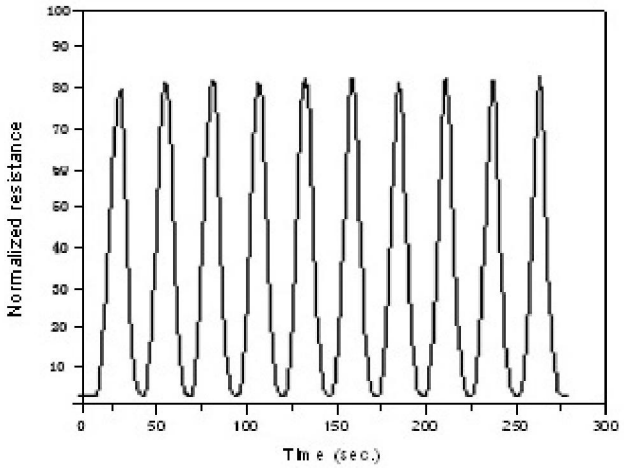

Fig. 3. The conductivity changes with the elongation of PPy-coated fabrics (deformation=50%).

In addition to high sensitivity, good environmental stability is especially important for the practical application. The conductivity degradation of PPy is closely related to its reaction with oxygen, especially under elevated temperatures, and moisture. Therefore, the stability of the strain sensors was first investigated by the thermal aging and humidity aging tests. The thermal aging test is composted of six cycles of heating and cooling tests. The temperature ranged from 20 to 60°C, and the conductivity change with temperature of the sensors during the thermal aging process is presented in Fig. 4. It can be seen that the difference in resistance between the heating and cooling cycle is quite small. In addition, only a slight increase in resistance is observed after each heating-cooling cycle, which may be related to the stability of the dopant ion and the rearrangement of the PPy chain. It is found that the conductivity drop at 20°C is as small as 16% after the thermal aging of six cycles, which is indicative of the good stability of the sensor. Furthermore, we find that the conductivity of the sensor shows only a slight dependence on the temperature, and the temperature coefficient is calculated to be only -0.6%/°C.

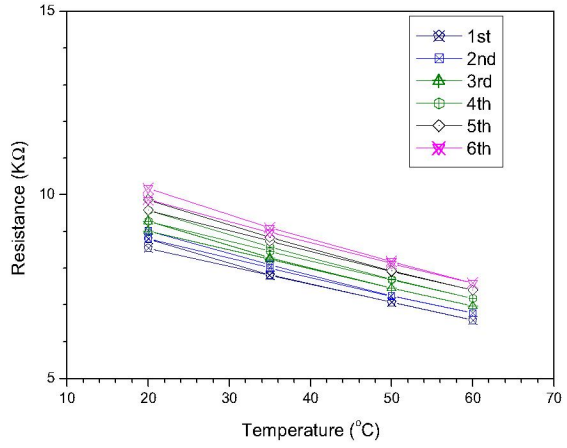

Fig. 4. Resistance change versus temperature during the thermal aging tests

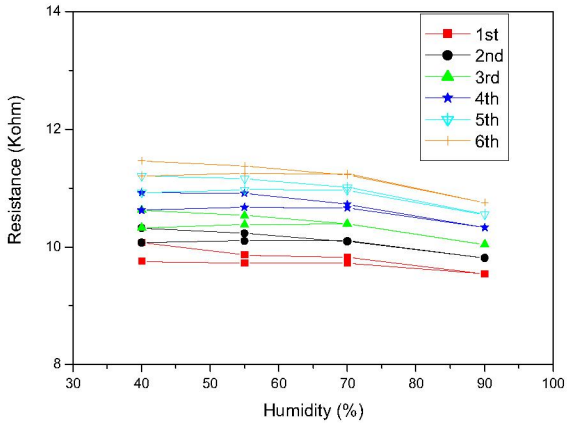

Fig. 5. Resistance change versus humidity during the humidity aging tests

Humidity can also exert an influence on the conductivity of PPy. The stability of the sensor was further investigated by the humidity aging. The conductivity change with humidity of the sensor during six cycles of humidifying and desiccating tests was plotted in Fig. 5. It can be seen that the difference in conductivity for the humidifying and desiccating process is very small, suggesting little hysteresis. The largest conductivity loss was observed at 40%RH, which is only 15 % after humidity aging of six cycles, suggesting the good stability of the sensor. It was also found that the humidity increase can only slightly decrease the resistance of the sensor, and the conductivity change when the humidity is increased from 40 to 90%RH is only 0.66 %.

The thermal and humidity aging tests show that the conductivity of the strain sensor from PPy-coated fabrics is only slightly dependent on the humidity and temperature, while the sensor exhibits very high strain sensitivity. Therefore, it can be concluded that the effect of the temperature and humidity on the conductivity of the strain sensor can be ignored provided they fall in the range already investigated during the aging tests, which is an advantage for its potential application.

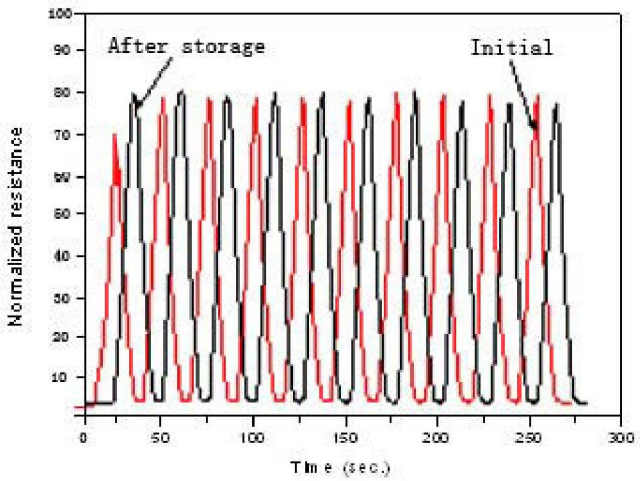

Fig. 6 (a) Conductivity changes versus strain of the sensors prepared at low temperature before and after the storage

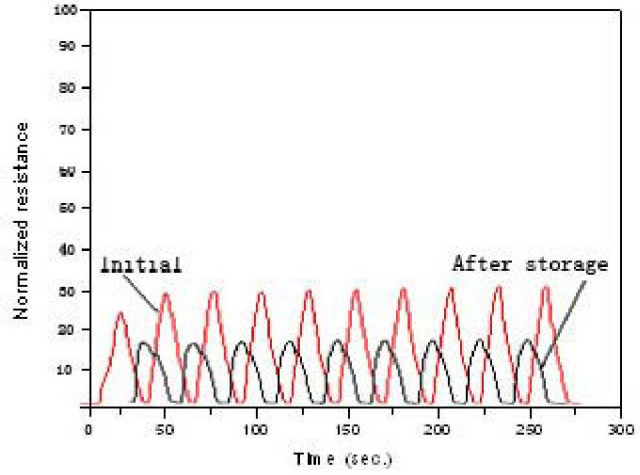

Fig. 6 (b) Conductivity changes versus strain of the sensors prepared at room temperature before and after the storage

TABLE I. THE RESISTANCE CHANGES DURING THE STORAGE OF THE SENSORS PREPARED AT DIFFERENT TEMPERATURES

Preparation temperature	Initial resistance (k Ohm)	Resistance after storage (k Ohm)
Low temperature	11.2	13.2
Room temperature	70.1	312

A more direct proof of the stability of the PPy-coated fabrics is the variation of the conductivity and sensing properties of the sensor during storage of a long time. Table I presents the resistance of the sensor prepared at both low and room temperatures before and after the storage of about eighteen months. And their strain sensing properties before and after the storage is illustrated in Fig. 6. It can be seen that the sensor prepared at low temperature exhibits a conductivity loss of only ~15% after eighteen months, and its strain sensing properties is almost unchanged. In contrast, the one prepared at room temperature shows a conductivity loss of ~ 80%. In addition, the strain sensitivity also decreased by a half. Therefore, it can be concluded that the flexible strain sensor from the PPy-coated fabrics so prepared shows good environmental stability and the low temperature preparation is beneficial for the improving the stability of the sensor

IV. CONCLUSION

In summary, a polypyrrole-coated fabric strain sensor featured with high strain sensitivity, good stability and large deformation has been developed. It is proposed that the chemical vapor deposition of thin coatings of polypyrrole on fabrics surface leads the high strain sensitivity of the sensor, while the low temperature polymerization of pyrrole helps improve its stability. Morphology and thickness of PPy film on the surface of the fibers examined by SPM and SEM help understand the mechanism well. The PPy-coated fabric strain sensor so prepared is expected to find applications in sensing garment, wearable hardware and rehabilitation, etc.

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