

# Electrochemical actuator based on single-layer polypyrrole film

Gaoyi Han<sup>a,b</sup>, Gaoquan Shi<sup>a,\*</sup>

<sup>a</sup> Department of Chemistry and Key Lab of Bio-organic Phosphorus, Tsinghua University, Beijing 100084, PR China

<sup>b</sup> Institute of Molecular Science, Chem Biol and Mol Engn Lab, Educ Minist, Shanxi University, Taiyuan 030006, PR China

Received 2 November 2004; received in revised form 8 January 2005; accepted 28 February 2005

Available online 25 March 2005

## Abstract

Polypyrrole film with porous and compact zones in its vertical direction has been synthesized by direct oxidation of pyrrole in the mixed solvent of acetonitrile and dichloromethane contained a certain amount of anion surfactant of sodium 1,4-bis(2-ethylhexyl)sulfosuccinate (AOT). The actuator based on this single-layer film bends uniformly with the maximum bending angle much larger than 90° and has a fast response rate ( $\sim 60^\circ \text{ s}^{-1}$  at 1.0 V versus SCE) and a long lifetime ( $>20,000$  cycles). The high performance of this actuator is mainly due to that the PPy film has a high conductivity and the actuator does not have delamination problem.

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**Keywords:** Polypyrrole; Anisotropy film; Actuator; High-performance

## 1. Introduction

In the last few decades, several soft actuators with volume changes of intrinsically conducting polymers (CP) during redox processes have been studied extensively [1,2]. This volume change can be expressed as electrochemomechanical deformation. Most of the actuators have a bilayer or a trilayer configuration [3–5]. In the bilayer actuators, a single actuating film is affixed to a second, electromechanically inert layer (e.g., a non-conducting adhesive tape). The trilayer actuators usually consist of two conducting polymer films attached to either sides of a passive film. In these cases, one of the conducting polymer films acted as working electrode and the other acted as counter and reference electrodes [4]. The electrochemically inert layer in either bilayer or trilayer actuators can increase the strength of the layered composite films, while it reduces the efficiency of these devices because of its weight contribution to the devices. Moreover, the physical adhesion between the inert layer and the CP layers cannot sustain repeated volume alteration at the interface [6,7]. Recently, we used high-strength polythiophene film to replace the elec-

trochemically inert layer, and improved the performance of the actuator [8]. Takashima et al. reported a bi-ionic actuator (BIA) consisting of only two layers of polypyrrole (PPy) films, which was driven by a combination of expansion of one PPy layer and contraction of the other layer during redox process [9,10]. Furthermore, the response rates of the actuators based on conducting polymer films have been improved by integrating a gold layer between the two layers of PPy films [11] or spiral-wound metal wire into the conducting polymer tube device that do linear movement [12]. Tada et al. fabricated an actuator by using a polypyrrole sheet with vertical anisotropy. The sheet was electrosynthesized in a thin slab vessel consisting poly(tetrafluoro ethylene) (PTFE) films. In this case, the size of the polypyrrole film was limited by the size of needle working electrode and the space of the vessel [13]. Recently, Wang et al. fabricated monolithic polyaniline electrochemical actuators and the actuators have a long lifetime ( $>3000$  cycles with 45° bending angles at 0.1 Hz) [14,15]. However, the asymmetric porous polyaniline films were synthesized through multi-step procedures. Furthermore, the actuators were operated at a high driving potential ( $\pm 2$  V) in an acid medium ( $1 \text{ mol l}^{-1}$  HCl) and could not do uniform bending with a maximum bending angle of only  $\pm 90^\circ$ . This is mainly due to the polyaniline films

\* Corresponding author. Tel.: +86 10 6277 3743; fax: +86 10 6277 1149.  
E-mail address: [gshi@tsinghua.edu.cn](mailto:gshi@tsinghua.edu.cn) (G. Shi).

has a low conductivity ( $\sim 0.1 \text{ S cm}^{-1}$ ). Thus, the fabrication of single-layer CP actuator with high performance in a neutral medium remains as a challenge. In this paper, we desire to report a high-performance actuator based on single-layer anisotropy polypyrrole film.

## 2. Experimental

### 2.1. Materials

Pyrrole (Chinese Army Medical Institute) was distilled under reduced pressure before use. AOT was purchased from ACROS and used directly. Anhydrous lithium perchlorate (99%), dichloromethane (99%) and HPLC-grade acetonitrile with a purity higher than 99.9% (Tianjing Shiyou Biological and Medical Technology Co., Tianjing, China) were also used as received.

### 2.2. Electrosyntheses and examinations

Electrochemical syntheses and examinations were performed at room temperature in a one-compartment cell with the use of a Model 283 potentiostat–galvanostat (EG&G Princeton Applied Research) under computer control. The working and counter electrodes were stainless steel (AISI 304) sheets ( $2.0 \text{ cm} \times 0.5 \text{ cm}$  and  $3.0 \text{ cm} \times 2.0 \text{ cm}$ , respectively) and placed  $1.5 \text{ cm}$  apart. All potentials were referred to a saturated calomel electrode (SCE).

PPy films were deposited from the mixed solvent of acetonitrile and dichloromethane (volume ratio = 5/1) containing  $0.3 \text{ mol l}^{-1}$  pyrrole and  $0.05 \text{ mol l}^{-1}$  AOT at a constant potential of  $1.1 \text{ V}$  (versus SCE). All solutions were de-aerated by bubbling dry nitrogen, and maintained a light nitrogen overpressure during the experiments. The thickness of the films was controlled by the total charges passed through the cell and measured by the use of a thickness monitor (CH-1-ST, Shanghai China).

The movements of the single-layer PPy films (size =  $15 \text{ mm} \times 1 \text{ mm} \times 0.025 \text{ mm}$ ) were investigated in a one-compartment cell by the use of  $1 \text{ mol l}^{-1}$   $\text{LiClO}_4$  aqueous solution as the electrolyte. The working electrode was a freestanding PPy film, the counter electrode was a stainless steel (AISI 304) sheet and the potential were referred to a saturated calomel electrode (SCE).

### 2.3. Characterizations

The morphology of the films was studied by using a KYKY2800 scanning electron microscope (Beijing Scientific Instrument Company, Beijing, China) after sputtering with a thin layer of gold. Raman spectra were recorded on an RM 2000 microscopic confocal Raman spectrometer (Renishaw PLC, England) employing a  $633 \text{ nm}$  laser beam and a charge coupled device detector (CCD) with  $4 \text{ cm}^{-1}$  res-

olution. The spectra were recorded using a  $20\times$  objective and accumulated for  $30 \text{ s}$ . The power was always kept very low ( $\sim 0.5 \text{ mW}$ ) to void destroying the samples. The tensile strengths of the films were measured by the use of a WD-1 electronic stretching machine (Changchun, China). The *dc* conductivity was measured by conventional four-probe technique.

## 3. Results and discussion

### 3.1. Electrosynthesis and electrochemistry of PPy film

Acetonitrile was found to be a poor solvent for AOT, thus a certain amount of dichloromethane was added to increase the solubility of AOT in the electrolyte. Fig. 1 shows the successive cyclic voltammograms (CVs) of  $0.3 \text{ mol l}^{-1}$  pyrrole in a mixed solvent of acetonitrile and dichloromethane (the volume ratio of acetonitrile to dichloromethane is 5/1) containing  $0.05 \text{ mol l}^{-1}$  AOT at a potential scan rate of  $40 \text{ mV s}^{-1}$ . As can be seen from this figure, each CV has a couple of broad redox waves of the polymer in the potential range of  $-1$  to  $1.0 \text{ V}$  (versus SCE). The strong oxidation current wall appeared at the potentials higher than ca.  $0.8 \text{ V}$  led to the formation of a black film on the working electrode surface.

The electroactivity of an as-grown PPy film (grown at  $1.1 \text{ V}$  (versus SCE) for  $240 \text{ mC cm}^{-2}$ ) coated on the working electrode surface and a freestanding film (size =  $15 \text{ mm} \times 1 \text{ mm} \times 0.025 \text{ mm}$ ) was studied in the monomer free  $1.0 \text{ mol l}^{-1}$  aqueous solution of  $\text{LiClO}_4$ . As shown in Fig. 2, the cyclic voltammograms of the PPy films have a couple of broad oxidation and reduction waves and the films have good electrochemical stability (Fig. 2a and c). The wave currents have linear relationships with potential scan rates in the range of  $20$ – $120 \text{ mV s}^{-1}$ , indicating a redox couple fixing on the electrode surface (Fig. 2b). Furthermore,

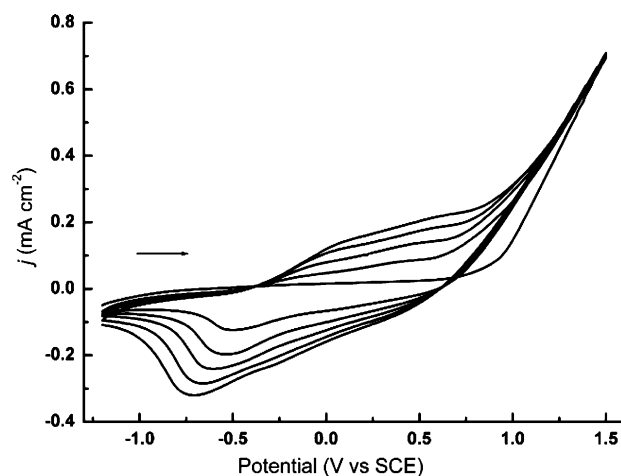


Fig. 1. Cyclic voltammograms of  $0.3 \text{ mol l}^{-1}$  pyrrole in a mixed solvent of acetonitrile and dichloromethane (the volume ratio of acetonitrile to dichloromethane is 5/1) containing  $0.05 \text{ mol l}^{-1}$  AOT at a potential scan rate of  $40 \text{ mV s}^{-1}$ .

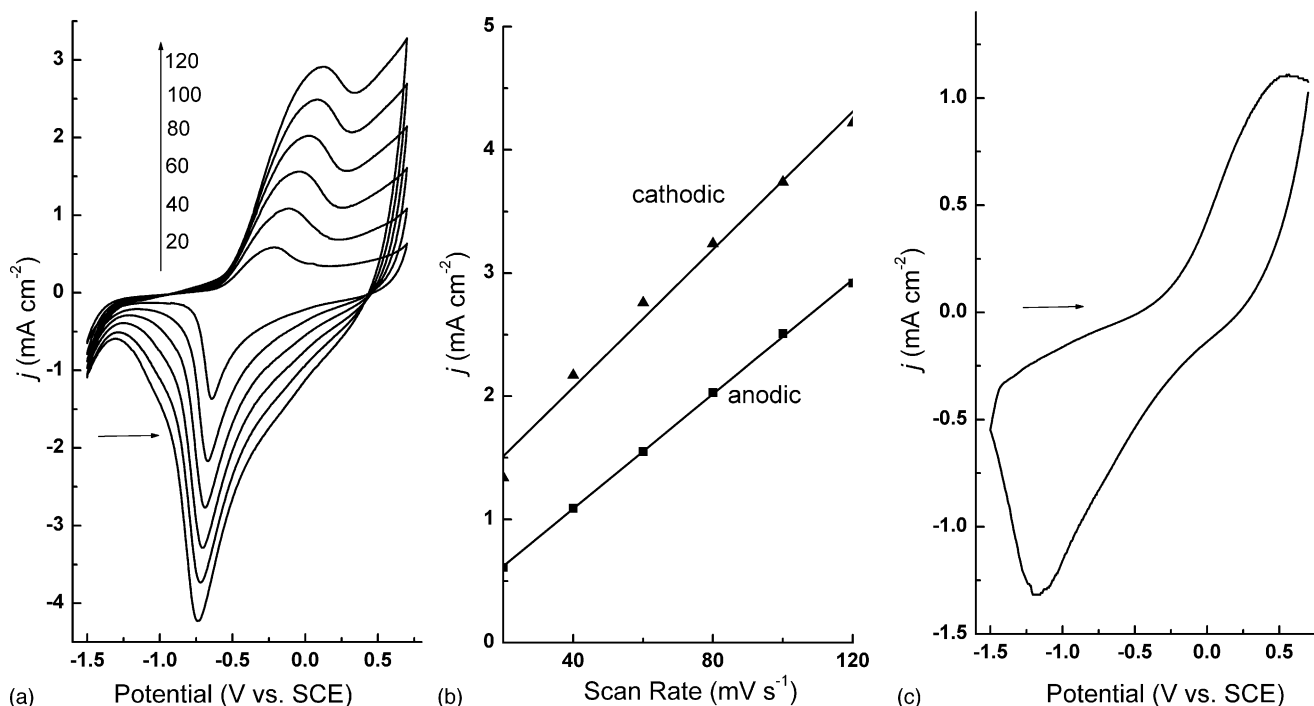


Fig. 2. (a) Cyclic voltammograms of a PPy film grown at 1.1 V (vs. SCE) for  $240 \text{ mC cm}^{-2}$  on a stainless steel electrode at various potential scan rates (20–120  $\text{mV s}^{-1}$ ) in  $1.0 \text{ mol l}^{-1}$  aqueous solution of  $\text{LiClO}_4$ ; (b) plots of wave current density vs. potential scan rate for Fig. 2a; (c) cyclic voltammogram of a free-standing PPy film with size of  $15 \text{ mm} \times 1 \text{ mm} \times 0.025 \text{ mm}$  at a scan rate of  $40 \text{ mV s}^{-1}$  in  $1.0 \text{ mol l}^{-1}$  aqueous solution of  $\text{LiClO}_4$ .

the films can be cycled repeatedly between the conducting (oxidized) and insulating (neutral) state without significant decomposition. The PPy film coated on a metallic electrode showed a reduction wave at approximately  $-0.67 \text{ V}$  and an oxidation wave at approximately  $-0.11 \text{ V}$  (Fig. 2a). The potential difference between the oxidation and reduction current peaks ( $\Delta E_p$ ) was as large as  $0.56 \text{ V}$  (at a scan rate of  $40 \text{ mV s}^{-1}$ ). On the other hand, at the same potential scan rate, the free-standing PPy film showed a broader oxidation wave at approximately  $0.57 \text{ V}$  and a reduction wave at approximately  $-1.1 \text{ V}$  (Fig. 2c). The  $\Delta E_p$  ( $\sim 1.7 \text{ V}$ ) is much larger than that of the film coated on a metallic electrode ( $0.56 \text{ V}$ ). This is mainly due to that the free-standing film has

much lower conductivity than that of the metallic electrode, which resulted in larger IR drops.

### 3.2. Morphology, conductivity and mechanical property of PPy film

The typical scanning electron micrographs (SEM) of an as-grown PPy film are shown in Fig. 3. It is interesting to find that the film have different morphologies in its two side surfaces. The side in contact with the electrolyte was rough and compact and similar to that of the electrosynthesized PPy films reported previously (Fig. 3A) [16–18]. The side in contact with the electrode is porous (Fig. 3B) and its morphology

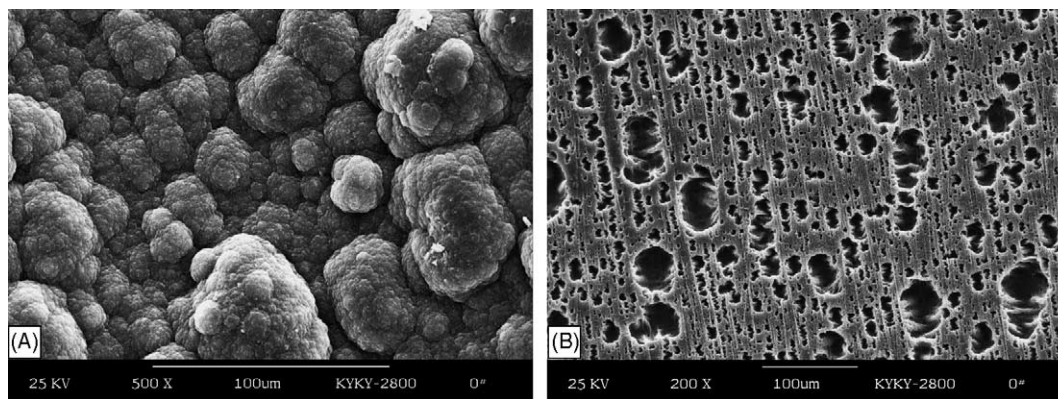


Fig. 3. SEM images of a PPy film grown at 1.1 V with a thickness of  $0.025 \text{ mm}$ : (A) the surface in contact with the electrolyte and (B) the surface in contact with the electrode.



Fig. 4. Snapshots of a single-layer PPy film during redox cycle: a transition state of the film reduced at  $-1.0$  V (vs. SCE, A) or oxidized at  $0.8$  V (B, vs. SCE) and the final state of the film (C). The film was held as the surface in contact with electrolyte in right.

is quite different from that of the PPy films deposited from the other systems (usually flat and compact). The sizes of the pores are ranged from several micrometers to  $50\ \mu\text{m}$ . The unique microstructures of the PPy films most possibly result from an emulsion drop template polymerization process. AOT is a widely used anion surfactant for positive or inverse emulsions. Acetonitrile has relative higher polarity than that of dichloromethane and it is a poor solvent for AOT. Thus, the emulsion dichloromethane solution of AOT was enwrapped by acetonitrile [19]. As a positive potential was applied, the emulsion drops with negative charges close to the anode were assembled on the working electrode surface under the function of electric field. Pyrrole dissolved in acetonitrile was polymerized around the surfaces of the drops to generate a porous film. Recently, Takashima et al. also reported that porous polypyrrole films with pore size in the range of  $0.1\text{--}1.0\ \mu\text{m}$  could be synthesized by oxidation of pyrrole in the mixed electrolyte of water and ethyl acetate [20]. In comparison, the PPy films synthesized in our system have much larger (up to  $50\ \mu\text{m}$ ) and deeper pores, and better vertically anisotropy structures.

The conductivity of the as-grown PPy film was measured to be about  $15\ \text{S cm}^{-1}$  by using the convenient four-probe technique. This value is much higher than that of porous polyaniline films ( $\sim 0.1\ \text{S cm}^{-1}$ ) [14,15]. It has a tensile strength of  $36 \pm 5\ \text{MPa}$  and an elongation at break of  $4.5 \pm 0.5\%$ . These films are shiny and black in color with good flexibility and can be folded or mechanically shaped into desired structures such as fibers, cycles and triangles.

### 3.3. Bending movements of the single-layer PPy film

The bending movements of the PPy film were studied electrochemically in  $1.0\ \text{mol l}^{-1}$   $\text{LiClO}_4$  aqueous solution. As demonstrated in Fig. 4, a film with size of  $15\ \text{mm} \times 1\ \text{mm} \times 0.025\ \text{mm}$  can uniformly bend from the position of  $0^\circ$  (vertical) to  $-90^\circ$  (Fig. 4A, reduction) at  $-1.0$  V (versus SCE) and to  $+90^\circ$  (Fig. 4B, oxidation) at  $0.8$  V (versus SCE) initially and subsequently the film was curled for several cycles (Fig. 4C, the bending direction of the film was referred to the film surface in contact with the electrolyte), indicating the maximum bending angle is much larger than  $90^\circ$ . The actuator can bend to  $\pm 90^\circ$  at a low driving potential of  $-0.4$  or  $0.1$  V. The movement rate of the PPy film was measured to be  $\sim 60^\circ\ \text{s}^{-1}$  at a potential of  $1.0$  V (Fig. 5a), which was much higher than those of the single-layer polyaniline film ( $\sim 20^\circ\ \text{s}^{-1}$  at  $1.0$  V) [14,15] and PPy bi-ionic actuators

( $\sim 30^\circ\ \text{s}^{-1}$  at  $1.0$  V) [11]. The PPy film was subjected to multiple potential steps of reduction at  $-0.8$  V and oxidation at  $0.8$  V at  $0.5$  Hz (bending angle  $> 45^\circ$ ). After 20,000 cycles, the film still can bend to  $90^\circ$  at a potential of  $0.8$  V. However, its movement rate was decreased from  $40$  to  $3^\circ\ \text{s}^{-1}$  (Fig. 5b). Accordingly, the lifetime of this single-layer PPy actuator is much higher than those of PPy/Au bending actuators ( $\sim 2000$  cycles) and the monolithic polyaniline actuators ( $\sim 3000$  cycles) [14,15,21]. The high performance of this actuator is mainly due to the PPy film has a high conductivity and can do uniform bending at a low driving potential and without delamination problem.

### 3.4. Mechanism study

The as-grown PPy film is in oxidized state and doped by AOT anions. When the film was reduced, AOT anions could not be ejected from the film because of their large sizes. In order to maintain the overall charge neutrality, the counter cations ( $\text{Li}^+$  ions) and water molecules from the electrolyte were incorporated into the film, and the film swelled [22,23]. The porous zone of the film has smaller amount of the polymer in a given volume and can provide smaller deformation than that of the compact zone. As a result, the film was bended to the porous surface direction. Otherwise, when the film was oxidized, the film was bended to the compact surface direction.

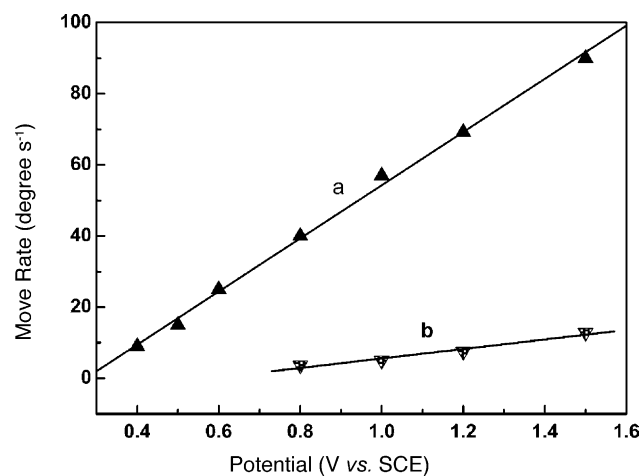


Fig. 5. Movement rates of single-layer PPy film in freestanding state in  $1\ \text{mol l}^{-1}$   $\text{LiClO}_4$  aqueous solution at different galvanostat conditions: (a) first cycle; (b) after 20,000 cycles of multiple potential steps of reduction at  $-0.8$  V and oxidation at  $0.8$  V at  $0.5$  Hz.



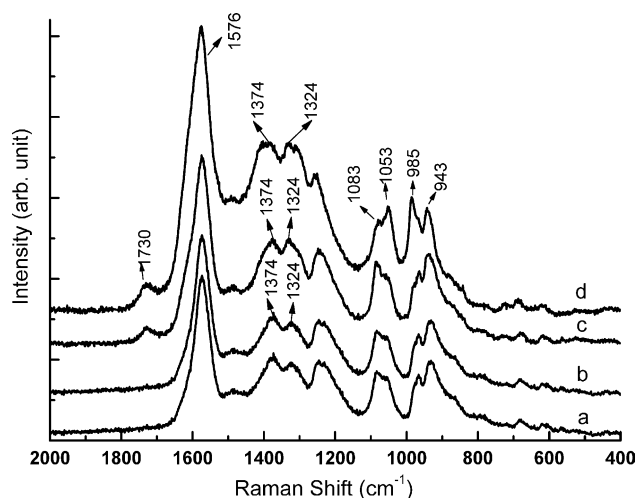


Fig. 6. Raman spectra of a PPy film before (a and b) and after (c and d) doing anodic movement. (a and c) The surface in contact with the electrolyte; (b and d) the surface in contact with the electrode.

Fig. 6 illustrates the Raman spectra of a PPy film recorded before and after doing anodic (oxidation) bending motion in the aqueous solution of  $\text{LiClO}_4$ . As can be seen from this figure, the Raman spectra of the two surfaces of the as-grown film have similar spectral features (Fig. 6a and b). The strong band located at approximately  $1580\text{ cm}^{-1}$  represents the C=C backbone stretching of PPy. The double peaks at about  $1050$  and  $1080\text{ cm}^{-1}$  are assigned to the C–H in plane deformation [24]. The other double peaks at approximately  $1320$  and  $1370\text{ cm}^{-1}$  are attributed to the ring-stretching mode of PPy [25]. The bands located at about  $940$  and  $990\text{ cm}^{-1}$  are assigned to the ring deformation associated with dication (bipolaron) and radical cation (polaron), respectively. The bands at  $1080$  and  $1370\text{ cm}^{-1}$ , belonged to the C–H in-plane and the ring stretching, respectively, which are assigned to the oxidized species of PPy according to the literatures [26,27]. The conductivity of the PPy is strongly related to Raman peak intensity of oxidized PPy. As shown in Fig. 6a and b, the band related to the oxidized species at approximately  $1080\text{ cm}^{-1}$  is stronger than the band associated with the neutral species at  $1050\text{ cm}^{-1}$ . However, after doing the anodic bending motion, the Raman spectra of the two surfaces of the PPy film are different (Fig. 6c and d). The spectrum recorded on the surface in contact with electrolyte (the compact side) has spectral features of highly oxidized PPy film (Fig. 6c). Furthermore, in comparison with those in the spectra of Fig. 6a and b the  $1080\text{ cm}^{-1}$  band in Fig. 6c is also enhanced. On the other hand, in the spectrum recorded on the surface in contact with electrode (the porous side), the  $1080\text{ cm}^{-1}$  band is weaker than the  $1050\text{ cm}^{-1}$  band (Fig. 6d), indicating a low doping level. These results demonstrated that the compact side of the film could be oxidized more efficiently than the porous side. These observations also support that the compact zone of the film can provide a relatively larger electrochemical deformation than that of the porous zone of the film during the redox process, which makes the film bending.

## 4. Conclusions

In conclusion, polypyrrole film with porous and compact zones in its vertical direction can be synthesized by direct oxidation of pyrrole in the mixed solvent of acetonitrile and dichloromethane containing a certain amount of anion surfactant of AOT. The emulsion drops assembled on the working electrode surfaces acted as the templates for the formation of the micro-pores. Single-layer porous PPy film possesses bending motion ability based on its vertical anisotropy structure. The actuator bends uniformly at a low driving potential and has a fast response rate and a long lifetime.

## Acknowledgements

This work was supported by the National Natural Science Foundation of China with grant nos. 50225311, 20374034, 90401011 and 973 project with grant no. 2003CB615700.

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## Biographies

**Gaoyi Han** received his bachelor and PhD degrees from Shanxi University and Nanjing University, respectively. He then joined Prof. Gaoquan Shi's research group in Tsinghua University as a postdoctoral fellow (2002–2004). He is currently an associate professor at Shanxi University, China.

**Gaoquan Shi** was born in Rugao, Jiangsu Province, China, in 1963. He received his BS (1985) and PhD (1992) degrees from Nanjing University. He joined Nanjing University as an assistant professor in 1992, and then promoted to associate professor in 1993 and professor in 1995. He moved to Department of Chemistry, Tsinghua University as a professor (2000 to date). His interests focus on syntheses and applications of conducting polymers.