

Preparation of all-solid-state supercapacitor integrated with energy level indicating functionality



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ABSTRACT

All-solid-state supercapacitor with the function of displaying energy level is a hot research topic at present. In this research, with Polyaniline (PANI) as the anode, WO₃ as the cathode and Polyvinylpyrrolidone (PVP)-LiClO₄ as the solid electrolyte, an all-solid supercapacitor had successfully obtained with electrochromic function. The results show that the supercapacitor possesses good electrochromic and electrochemical performance, which can thus result in rapid and reversible color changes in different operating voltages (−1 V ~ 1 V). As for the changing color during the discharge process, it indicates the changes of energy level. Moreover, as indicated by the research mechanism, the supercapacitor provided with the function of color changing has exploited a new path for the development of intelligent all-solid-state supercapacitor in the future.

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1. Introduction

Clean energy storage devices are endowed with high power density, long cycling stability and high charge density, supercapacitor has received great attention and been applied to electric vehicles, power transmission, air crafts, etc. [1–6]. In recent years, developing new features and functions in the process of fabricating high-performance electrochemical supercapacitor is crucial to the intellectualization of products [7–12]. For example, in daily production, the exhausting of the device's energy cannot be recognized until it stops working. With the presence of some kinds of supercapacitors that changes its outlook according to charge and discharge status, we can know that the energy has been exhausted before the device stops working based on the visible change of energy storage, which would thus largely improve the working efficiency and convenience [13–16]. One way to realize this goal is to create a supercapacitor capable of changing its own color. As for the raw material, on one hand, it should have good energy storage capability; on the other hand, it can change its color reversibly in different voltages by a redox reaction. At present, researchers have conducted extensive studies on two kinds of materials, one of which is inorganic transition metal oxides such as WO₃ [17–22], and the other is conductive polymers, such as PANI [23–30]. The

electrochromic material in a supercapacitor could display different colors during working time in a common and effective way.

Besides, solid polymer electrolyte has become very important in electrochemical devices, especially in the transparent electrochemical storage devices functioning with an increasing number of transparent solid polymer electrolytes [31–33]. Among various polymer electrolytes, PVP has attracted great attention due to its good environmental stability, ease of processing and good transparency [34–36]. Therefore, transparent solid electrolyte with PVP and LiClO₄ is essential to the application of electrochromic supercapacitors.

In this study, it has been pointed out that the electrochromic all-solid supercapacitors with PANI as the anode, WO₃ as the cathode and PVP-LiClO₄ as the solid electrolyte, can present rapid and reversible color change in different voltages (−1 V ~ 1 V), with the changing color displaying changes of energy level. We believe that this supercapacitor equipped with newly developed function has opened a door for the future development of portable and highly intelligent supercapacitors.

2. Experimental

2.1. Preparation of PANI electrode

The PANI electrode was prepared via an electrochemical method. The conductive glass (FTO) was ultrasonically washed in acetone, ethanol and deionized, respectively. After that, the sample was dried at 60 °C. Aniline was purified by vacuum

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distillation before being dissolved in H_2SO_4 with a concentration of 0.5 mol L^{-1} . Moreover, carbon was adopted as the working electrode, platinum as the auxiliary electrode and Ag/AgCl as the reference electrode, the PANI film was prepared under the ambient temperature.

2.2. Preparation of WO_3 electrode

Under the protection of nitrogen at 200°C , WO_3 in nano-linear form was deposited on the surface of FTO through the solvothermal deposition approach. Then, it was washed with deionized water and dried at 60°C . Besides, a layer of photosensitizer was coated on its surface through the spin-coating method. After that, a standard UV photolithography technique was employed to engrave a pattern, “XATU”, shorthand for our University, Xi'an Technological University. At last, the remaining photosensitizer was washed off by acetone. With the above steps, a WO_3 electrode with “XATU” mask was successfully prepared.

2.3. Preparation of electrolyte and device package

The preparation of transparent solid electrolyte with PVP and LiClO_4 as the raw material is based on literature review [34]. In brief, a certain amount of PVP (the average molecular weight is 1.3×10^6) and LiClO_4 were dissolved in ethanol at the room temperature for 30 min. With PANI electrode as the base material, solid electrolyte films were prepared through the dipping process. The soaking extraction rate is 180 mm/min , with the films being dried for 10 min by halogen lamp at 50°C in between each dipping. At last, the freshly prepared sample was fastened to the WO_3 electrode under the simple hand pressure. Several glue fixing points were applied to the interface so as to ensure stability. The preparation process is shown in Fig. 1.

2.4. Characterization

The structure of material was characterized by scanning electron microscopy (SEM; JEOLJSM-6360LV), X-ray diffraction (XRD; Ultima IV, Rigaku) and Fourier transform infrared spectroscopy (FTIR; WQF-31). Regarding the optical performance, it was tested by ultraviolet- visible spectrophotometer (UV-vis; UV-2012) through the transmission spectra under $300\text{--}800 \text{ nm}$ wavelength range. As for the electrochemical characteristics, they were evaluated by cyclic voltammetry (CV), galvanostatic Constant-current charge-discharge and cycle stability in a CHI660E electrochemical workstation (Shanghai CH Instruments Co., China).

3. Results and discussion

3.1. Structural and morphological analysis

Fig. 2(a and b) are the images of SEM about WO_3 . As shown in the figure, WO_3 is in nanostructure composed of intertwined nanowires of which the diameter is about $5\text{--}10 \text{ nm}$ and the length is about $1 \mu\text{m}$. Fig. 2(c and d) are the images of PANI film, from which it can be seen that PANI is in nanoparticle structure, and the nanoparticles are relatively dense and their diameter ranges are around 100 nm . Besides, Fig. 2(e) is the image of the device's cross-section, which shows that the upper layer in darker color is WO_3 with a thickness of about $0.5\text{--}1 \mu\text{m}$, the lower layer is PANI with a thickness of about $1 \mu\text{m}$ and the middle layer is PVP- LiClO_4 with a thickness of about $30 \mu\text{m}$. On the whole, the PANI and WO_3 electrode layers have not only effectively coated on the surface of electrolyte but also closely contacted with the electrolyte layer.

The infrared spectroscopy of PANI and WO_3 is shown in Fig. 2(f). As shown in this figure, the peak at 611 cm^{-1} is the para benzene N-H bending vibration. The peak at 835 cm^{-1} is para-disubstituted benzene C-H out-plane bending vibration. The peak at 1106 cm^{-1} is quinone N stretching vibration. The peak at 1295 cm^{-1} is the stretching vibration peak of $-\text{SO}_3\text{H}$, and the peak at 1507 cm^{-1} is the stretching vibration peak of C-N. Based on the infrared spectroscopy of these chemical characteristic groups, there illustrate that the PANI exists in the form of conductive Emeraldine Salt. In Fig. 2(f), the peak at 801 cm^{-1} is the bending vibration peak of C-H. 1114 cm^{-1} while 1463 cm^{-1} are absorption peaks of C-OH. Besides, 2338 cm^{-1} is the stretching peak of C-H. It proves that there are no relatively strong chemical functional groups in WO_3 and the weak peaks result from the residual alcohol solvent.

Fig. 2(g) is the typical XRD diffraction pattern of PANI and WO_3 . In Fig. 2(a), we can see that WO_3 has strong diffraction peaks. The diffraction peaks occurring at 23.2° , 24.5° , 26.7° , 33.4° , 35.7° , 42.2° , 47.5° , 50.8° and 56.2° correspond to crystal face diffraction (001), (200) (120) (111) (220) (020) (222), (040), (140) and (420), respectively. It suggests that in high-temperature solvent, the WO_3 monoclinic phase would experience gradual decrease, with the main crystalline phase being the orthorhombic phase. Furthermore, maintaining in preferred orientation trend, the plane is in line with diffraction card (PDF20-1324) in which the peak is relatively strong and the degree of WO_3 crystallization is high. In comparison, the crystalline region of PANI is smaller, thus presenting relatively significant and sharp diffraction peaks at 25.2° and 28.6° . This result indicates that proton dopant in PANI prepared in acidic electrolyte solution would enhance the

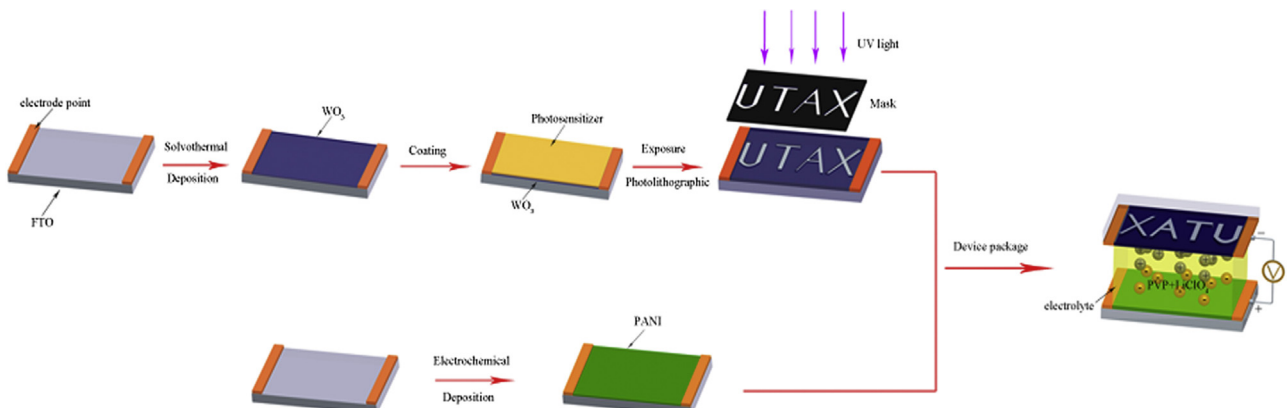


Fig. 1. Schematic illustration showing the procedure for preparation of All-solid-state electrochromic-supercapacitor of PANI- WO_3 .

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