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## A fast-switching light-writable and electric-erasable negative photoelectrochromic cell based on Prussian blue films

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### ABSTRACT

We report a fast-switching negative photoelectrochromic cell composed of a dye-sensitized nanocrystalline TiO<sub>2</sub> electrode and Prussian blue counter electrode sandwiching a LiI electrolyte. The cell can be bleached under illumination with shorted TiO<sub>2</sub> and Prussian blue electrodes, and re-colored by applying an appropriate external voltage. The photo-bleaching and electric-coloring processes are fast and reversible. A maximum absorbance modulation of 0.44 recorded at 700 nm is obtained between bleached and colored states for the cell when the PB film's thickness is 452 nm. Illuminated under different levels of light intensity or durations of time, the shorted cell shows adjustable optical absorption from 470 to 840 nm. The *in-situ* transmittance response depicts that the photo-bleaching response is 6.2 s for 70% transmittance change under 100 mW/cm<sup>2</sup> illumination in short circuit configuration, and the re-coloration time is about 600 ms under 2 V bias recorded at 780 nm, with an electrochromic coloration efficiency of 103.4 cm<sup>2</sup>/C. The cell shows a good reversible stability and can be potentially applied in erasable displays.

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### 1. Introduction

In recent years, the growing interest in low-power systems has led to the development of various energy-efficient chromic technologies including electrochromism, photochromism, gaschromism, thermalchromism, and photoelectrochromism [1–16]. Among them, photoelectrochromic devices have attracted significant attention because of their unique function of dynamically controlled solar energy gain and optical properties through switchable glazing in response to illumination, with promising applications in smart windows, sunglasses and light-writable low-information content displays [11,12,17,18]. Although various device configurations and working mechanisms have been reported in recent years [11,19–21], most of the reported photoelectrochromic devices are composed of a light-sensitive photovoltaic layer and an electrochromic layer. A dye-sensitized TiO<sub>2</sub>

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nanoparticle (NP) photoanode has been most commonly adopted as the photovoltaic layer, and various electrochromic materials (inorganic or organic) have been assembled as the electrochromic electrode including WO<sub>3</sub> or polyaniline [11,12,22,23]. Under illumination, photoelectrons generated by the dye molecule inject into the conduction band of TiO<sub>2</sub> and then transport through the external circuit to the counter electrochromic electrode, driving the ions in the electrolyte to intercalate into the electrochromic layer at the same time. Correspondingly, the transmittance of the photoelectrochromic device will be decreased as a result of the darkened electrochromic layer. The bleaching state can be achieved by either short circuit or open circuit configuration with light blocked. The switching response is mainly dependent on the electrochromic material and electrode adopted. Recently, Chen's group improved the bleaching rate of a WO<sub>3</sub>-based photoelectrochromic device using a patterned WO<sub>3</sub>/Pt electrode, providing the charge transfer pathways to accelerate the bleaching process [22]. Under illumination, the cell can be colored in a short circuit configuration with tunable transmittance and bleached more quickly when the circuit is opened (~60 s).

Prussian blue [PB, iron (III) hexacyanoferrate (II)] is a well known synthetic coordination-compounded transition metal

hexacyanometallate, which has attracted considerable attention owing to its various applications in electrochromic [24–26], sensors [27], catalysts [28], and batteries [29]. Different from  $\text{WO}_3$ , PB is an anodically coloring electrochromic material with promising electrochromic properties. It is reported that an electrodeposited PB film exhibits electrochromism between blue and colorless state with a fast response ( $\leq 100$  ms) and a high durability for  $5 \times 10^6$  cycles [30]. The excellent electrochromic performance makes it an ideal candidate for applications in smart windows and electronic paper displays. Although there have been numerous reports regarding electrochromic applications of PB, to our knowledge, such a PB film has not been incorporated in a device to enable fast-switching light-writable and electric-erasable photoelectrochromic operation to date.

In this study, we propose and demonstrate a negative photoelectrochromic cell, composed of an electrodeposited PB electrochromic electrode and a dye-sensitized  $\text{TiO}_2$  NP photoanode. The cell can be bleached in short circuit configuration under illumination and re-coloration can be achieved through applying a proper external voltage with fast switching response and good reversibility, providing a negative mode to the existing photoelectrochromic technology. The bleached cell can also be re-colored in either short circuit under dark state or open circuit under illumination with a slow speed. The photoelectrochromic cell shows obvious color change between colored and bleached state, promising for light-writable and electric-erasable information display applications.

## 2. Experimental details

### 2.1. Electrodeposition and characterization of PB films

Fluorine doped tin oxide ( $\text{SnO}_2:\text{F}$ , FTO) glasses (NSG Group,  $R_s = 14 \Omega/\square$  and 2.2 mm thick) washed by acetone, isopropanol, and de-ionized water in sequence were used as substrates. The electrodeposition of PB films was carried out in a standard three-electrode system (VersaSTAT 3F Potentiostat/Galvanostat) with a clean FTO glass (2 cm  $\times$  3 cm) as the working electrode, a platinum sheet as the counter electrode, and a  $\text{Ag}/\text{AgCl}/\text{sat'd KCl}$  as the reference electrode. The electrodeposition bath of PB contained 10 mM  $\text{K}_3\text{Fe}(\text{CN})_6$  (Aldrich), 10 mM  $\text{FeCl}_3$  (Aldrich), and 0.1 M KCl (Aldrich). The PB films were prepared by applying a constant cathodic current density of  $50 \mu\text{A}/\text{cm}^2$  for 100, 300, 500 and 700 s. Then the as-deposited blue PB films was rinsed by de-ionized water gently and dried at room temperature in atmosphere.

### 2.2. Preparation of dye-sensitized $\text{TiO}_2$ nanocrystalline photoanode

To fabricate the photoanode,  $\text{TiO}_2$  NP paint (Solaronix, Ti-Nanoxide T20/SP) was first screen-printed on a piece of FTO glass, which was ultrasonically cleaned sequentially by acetone, isopropanol, and de-ionized water. After calcination at  $450^\circ\text{C}$  for 1 h, a transparent and well adhesive  $\text{TiO}_2$  film with a thickness of  $\sim 4 \mu\text{m}$  was obtained. Dye absorption was carried out by immersing the  $\text{TiO}_2$  electrode into a Z907 solution [(*cis*-bis(isothiocyanato)(2,2'-bipyridyl-4,4'-dicarboxylato)(4,4'-di-nonyl-2'-bipyridyl)ruthenium(II)) in dilute (8  $\mu\text{M}$ ) ethanol solution containing 60 mM chenodeoxycholic acid] for 110 min. The electrode was then washed with acetonitrile for 3 times to remove the unanchored dye molecules. The dye coverage was kept low to make the cells semi-transparent in the "off" state.

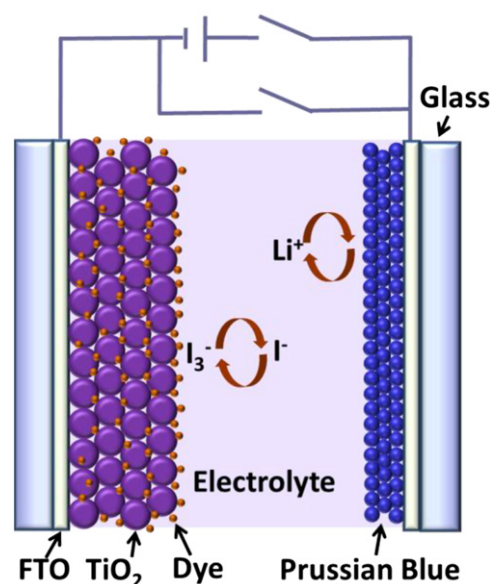


Fig. 1. Schematic of the proposed hybrid photoelectrochromic cell.

### 2.3. Assembly of the photoelectrochromic cell

The dye-sensitized  $\text{TiO}_2$  nanocrystalline photoanode and PB electrode were bonded together with hot-melt Surlyn spacers. Then a liquid electrolyte solution composed of 0.1 M  $\text{LiI}$  and 0.01 M 4-*t*-butylpyridine in  $\gamma$ -butyrolactone was introduced between the two electrodes by capillary action. Finally the cell was sealed with epoxy. The final cell has a configuration of

$\text{FTO}|\text{TiO}_2(\text{dye})|\text{LiI}+4\text{-}t\text{-butylpyridine}|\text{PB}|\text{FTO}$

which is also schematically shown in Fig. 1.

### 2.4. Characterization

The morphologies of electrodeposited PB films were characterized by field emission scanning electron microscopy (FESEM JSM 6340F). Cyclic voltammogram (CV) of the PB film was performed with 0.2 M  $\text{LiClO}_4$  in  $\gamma$ -butyrolactone as the electrolyte, a platinum sheet as the counter electrode and  $\text{Ag}/\text{AgCl}/\text{sat. KCl}$  as the reference electrode. Thicknesses of the PB films were measured by a Tencor P-10 surface profiler. The transmittance was measured by a JASCO V670 UV-vis-NIR spectrophotometer. A 150 W Xe lamp with optical filter ( $> 300$  nm) was used as the light source and the light intensity was calibrated by a pyranometer (PMA 2144 from Solar Light)

## 3. Results and discussion

### 3.1. Characterization of electrodeposited PB films

The morphologies of electrodeposited PB films were investigated (see Fig. 2a). It can be clearly seen from Fig. 2(a) that the film is composed of accumulated nanoparticles with sizes of 20–50 nm. There are a lot of cracks with an average gap dimension of  $\sim 20$  nm among the film, formed due to stress. The cyclic voltammetry (CV) curve of the PB film was measured with 0.2 M  $\text{LiClO}_4$  in  $\gamma$ -butyrolactone as electrolyte at a scan rate of 100 mV/s (Fig. 2b). The broad oxidation and reduction peaks appear at 0.84 and  $-0.8$  V, respectively, which are similar to those reported for

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