

Electrochromic properties of nanochromic windows assembled by the layer-by-layer self-assembly technique

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Abstract

Nanostructure controlled electrochromic (EC) multilayer films were assembled by the layer-by-layer (LBL) technique using poly(aniline-*N*-butylsulfonate)s (PANBUS) as polyanion, and vinyl benzyl dimethyl alkyl ammonium-chloride (VBDA) and polyaniline as the counter cation. The building up of such multilayer films on an indium-tin oxide (ITO) substrate was characterized by the increment of the absorbance through UV–Vis spectroscopy and layer thickness. Electrochromic response was highly dependent on the alkylchain length of VBDA. Higher color contrast was observed when the EC film was prepared from the VBDA having a shorter alkylchain length. An all-solid-state EC display showed EC response at 3 V within 5 s with a stable memory effect.

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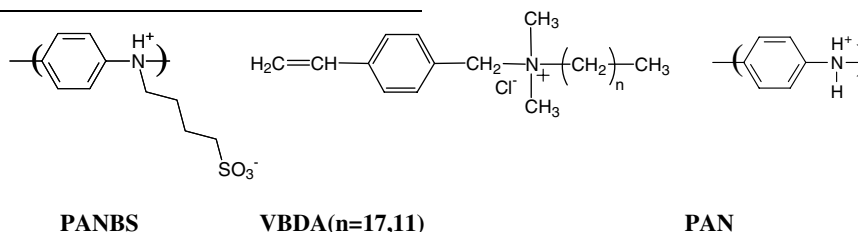
Keywords: Layer-by-layer self-assembly; Electrochromic; Multilayer films

1. Introduction

Electrochromic (EC) devices have attracted much interest due to their low power consumption as well as their memory effect [1]. Much progress has been made in developing new materials that exhibit long-term stability and various colors at different potentials, and in fabricating through nanotechnology [2]. In particular, the electrostatic layer-by-layer (LBL) assembly technique allows precise control of EC layers deposition [3].

In previous study, we reported the effect of diverse layer composition and sequence of nanostructured EC films to

electrochromic properties [4]. Herein we report LBL assembled conducting polymer films for an all-solid-state EC device. As an alkylsulfonated polyaniline offers high redox stability as well as electrochromic and self-assembling properties [5–7], poly(aniline-*N*-butylsulfonate)s (PANBS) were adopted as the electroactive anionic polymers for the layering. The counterions for the PANBS layers were polyaniline (H⁺-doped) as the conductive cation, and vinyl benzyl dimethyl alkyl ammonium-chloride (VBDA) as the resistive polycation. The structures are given below:



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2. Experiment

2.1. Materials

Methoxy poly(ethylene glycol) 1000 monomethacrylate (MPEGM) was purchased from Polyscience Inc. Triallyl-1,3,5-triazine-2,4,6-(1*H*,3*H*,5*H*)-trione (TATT, Aldrich) was used as a crosslinker. PEGDME ($M_w = 330$) was obtained from Aldrich and was purified by distillation before use. Lithium trifluoromethanesulfonate (LiCF_3SO_3) was purchased from Merck. Darocure 1173 (Ciba Specialty Chemicals) was used as a photocuring agent. TiO_2 (Degussa P25, diameter = 21 nm) was dried in a vacuum oven at 150 °C for 15 h before it was used. Sodium persulfate was obtained from Aldrich. VBDA was synthesized according to Ref. [8]. Aniline *N*-butanesulfonic acid was prepared as previously reported [7]. Chemical polymerization was carried out in a reaction vessel at 0 °C using sodium persulfate as an oxidant [9,10].

2.2. Preparation of polymer-salt membrane

MPEGM (2.5 g), TATT (0.3 g), PEGDME (2.5 g), and Darocure 1173 (0.25 g) were mixed in a dark glove box using a magnetic stirrer. LiCF_3SO_3 (0.25 g) was dissolved in the aforementioned mixture ($[\text{EO}]/[\text{Li}] = 12.5$), and the resulting mixture was mixed with nanosized TiO_2 . The mixture was stirred vigorously for 1 day to yield a radical, curable electrolyte solution.

2.3. Nanochromic multilayer film deposition

ITO glass was pretreated with *N*-(2-aminoethyl)-3-aminopropyl-trimethoxysilane to make the substrate surface hydrophilic. A multilayered film was obtained by layer-by-layer self-assembly, alternatively dipping the ITO glass into a 0.2-wt.% PANBS aqueous solution and a polycation solution (PAN or VBDA, 2 mg/ml) for 10 min. The electrochromic PANBS layer was deposited first as an anionic layer and then PAN or VBDA as a counteranion layer of to produce a bilayer, which was built in sequence.

The LBL film was dried with nitrogen gas after washing with distilled water. The drying procedure between the adsorption of either cationic or anionic layers was necessary for building uniform, self-assembled layers.

$|\text{PAN}|\text{VBDA}|\text{PAN}|\text{VBDA}|\text{PAN}|\text{VBDA}|\text{PAN}|$

(| = PANBS, VBDA of L1; $n = 17$, L2; $n = 11$)

The total number of layers in each film was the same (15 layers), and the first layer of each film was PANBS, so that the interfacial barrier ITO/PANBS for the electron transfer would be the same for all the films. To reduce interlayer barrier effect, VBDA which have shorter alkyl chain than that of L1 VBDA were introduced to L2.

2.4. Instrumentation

The in situ spectroelectrochemical system consisted of a computer-controlled reflectance spectrophotometer using a Avaspec-2048 fiber optic spectrometer and an electrochemical subsystem (BAS-epsilon). The maximum difference in absorbance between the colored and bleached states was observed for the PANBS-based electrochromic films. Layer thickness was determined by an alpha step (TENCOR INSTRUMENTS, Alpha-step IQ).

3. Results and discussion

3.1. Film growth by LBL assembly

Electrochromic films were fabricated using the LBL method, which involves the dipping of a clean ITO glass substrate to dilute aqueous solutions of polycation and polyanion in an alternating manner, followed by rinsing between each deposition step [3]. With this technique the nanostructure of the multilayer could be controlled as reported before. The layer thickness of the film increased as the layer number was increased, as shown in Fig. 1. UV-Vis spectroscopy was used to monitor the growth of the LBL films on the ITO glass. The absorbance at the selected wavelength increased as the number of layers increased. visible spectral change for film upon layer

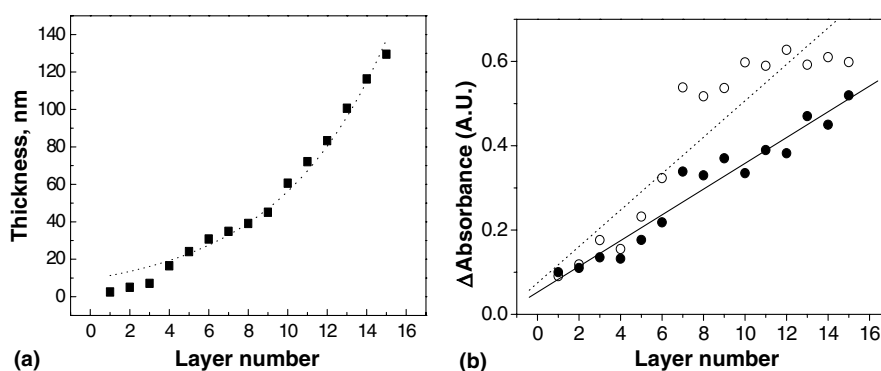


Fig. 1. (a) Growth of the layer thickness as a function of the number of layers in the EC film (L1). (b) Plot of the layer thickness growth in L1 against absorbance increase at 326 nm (○) and 440 nm (●).

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