

Annealing synthesis of coralline V_2O_5 nanorod architecture for multicolor energy-efficient electrochromic device

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ABSTRACT

A coralline vanadium pentoxide nanorod architecture on an indium-doped tin oxide substrate for energy-efficient electrochromism has been prepared by a simple annealing treatment from an overfilled amorphous three-dimensionally ordered macroporous vanadia film. The coralline vanadium pentoxide nanorod architecture exhibited multicolor electrochromic performance (yellow, blue-green, and olive), high transmittance modulations (25% and 27% at the typical wavelengths of 460 nm and 1000 nm, respectively), and fast switching speeds (4.8 s for coloration and 7.2 s for bleaching at 890 nm). In addition, the coralline vanadium pentoxide nanorod architecture exhibited desirable cycle stability. After 100 cycles, negligible transmittance modulation decreased in the visible spectrum, and a decrease of only approximately 5.6% was found in the near-infrared spectrum. Cyclic voltammetry measurements indicated that the majority of the current response of the redox reactions of the coralline V_2O_5 nanorod architecture was surface controlled, which resulted in desirable cycling stability and fast switching speeds. A indium-doped tin oxide substrate/vanadia/liquid electrolyte/poly(3,4-ethylenedioxythiophene)/indium-doped tin oxide substrate electrochromic device was assembled, and the device showed multicolor changes with acceptable transmittance modulation and good cycling stability.

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1. Introduction

Nowadays, owing to increasing environmental pollution and growing energy demand, developing eco-friendly energy and reducing energy consumption are two urgent tasks for researchers. Renewable energy technology based on green energy resources such as the sun and wind has been developed and used in some places. However, the majority of energy resources are still based on fossil fuels, which result in further environmental pollution and resource exhaustion [1–4]. Therefore, researchers are focusing on finding energy-efficient technology. As a promising energy-saving and environmental protection technology, electrochromic (EC) materials based smart windows have been attracting enormous attention during the last decades, due to their promising applications in indoor energy saving by controlling the solar light transmission indoors through reversible transmittance modulation. Because of their low operating potential, ~ 1 V, EC materials require very small amounts of energy to change their coloration states. Furthermore, such materials exhibit a memory

effect, which is another advantage in that very little power is needed to maintain their coloration states [5–9]. In addition, EC devices can be also applied to electronic paper-like displays, eyeglasses, antiglare rear-view mirrors in cars, and active camouflage [10,11].

Vanadium pentoxide (V_2O_5) materials with a layered structure [12], as well as the ability of the vanadium ion to change its oxidation state [13], are promising for EC applications. As a widely studied oxide which can show both cathodic coloration and anodic coloration, V_2O_5 materials not only exhibit good transmittance modulation but also show multicolor changes during the reversible Li-ion insertion/removal processes [14–16]. However, the diffusion coefficient of Li ions in V_2O_5 (10^{-13} – 10^{-12} cm²/s) and electric conductivity (10^{-3} – 10^{-2} S/cm) are both low [17–19], and thus the intercalation process is relatively slow. A large surface area and a short diffusion distance are desired in order to achieve faster intercalation and extraction kinetics [19,20]. One effective way to achieve these properties is the use of one-dimensional nanostructures such as nanowires, nanorods, and nanotubes. Various methods have been developed for the formation of nanorods or nanowires [21–24]. However, because these kinds of electrochromic materials are always in powder form, additional processing is needed to attach them to a transparent current

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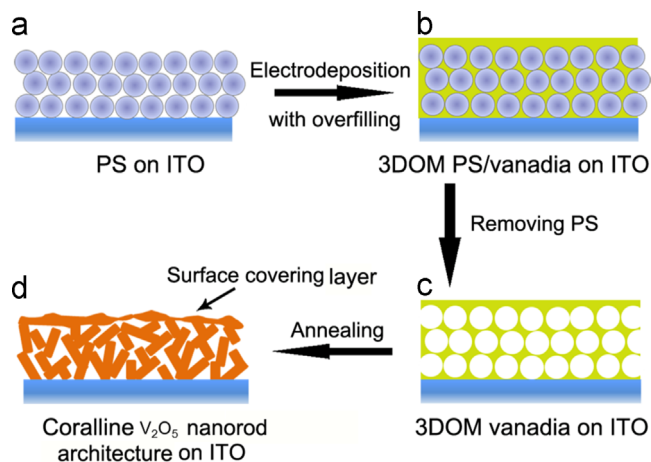


Fig. 1. Schematic of preparation process for coralline V_2O_5 nanorod architecture. (a) Polystyrene (PS) colloidal crystal template on ITO substrate. (b) Template-assisted electrodeposition of vanadia with slight overfilling. (c) Removal of PS template. (d) Annealing treatment for coralline V_2O_5 nanorod architecture.

collector [e.g., indium-doped tin oxide (ITO), fluorine-doped tin oxide (FTO)]. Additionally, weak physical adhesion among individual electrochromic nanomaterials may cause the release of active materials, resulting in the loss of optical modulation in long-term testing [25].

Recently, we reported that a loosely stacked film of one-dimensional V_2O_5 nanorods were generated from polystyrene colloidal crystal template-assisted electrodeposited amorphous three-dimensionally ordered macroporous (3DOM) vanadia by a simple annealing treatment [26]. This method provided a novel binder-free approach for preparing a V_2O_5 nanorod structure directly on ITO substrates. In the present work, we designed a coral-like V_2O_5 nanorod architecture with a thin surface-covering layer to realize multicolor EC performance for energy-efficient EC devices. The purpose of introducing the surface-covering layer is to prevent the structural disintegration and release of the V_2O_5 nanorods. As shown in Fig. 1, the V_2O_5 nanorod architecture was grown by annealing treatment of 3DOM vanadia, whereas the surface-covering layer was formed by slightly overfilling the colloidal template. The obtained V_2O_5 nanostructured film exhibited a coralline nanorod architecture morphology. Owing to the large surface area, short Li-ion diffusion distance, good electrolyte penetration, and strong structural integrity, the V_2O_5 coralline nanorod architecture exhibited a desirable cycling stability with multicolor changes (yellow, blue-green, and olive), high transmittance modulations, and short switching times. Cyclic voltammetry (CV) measurements indicated that the electrochromic redox reactions were surface controlled, further confirming the desirable redox reversibility and stability of the electrochromic performance. In addition, an EC device fabricated using our coralline V_2O_5 architecture and a poly(3,4-ethylenedioxythiophene) (PEDOT) film demonstrated multicolor changes and acceptable transmittance modulation.

2. Experimental

2.1. Preparation of coralline V_2O_5 nanorod architecture

Monodispersed polystyrene (PS) colloidal spheres (diameter of 270 nm) were used to assemble colloidal crystal templates. ITO substrates ($\sim 9 \Omega \text{ cm}^{-2}$) were used as the substrates. The detailed preparation process of the colloidal crystal templates can be found in Ref. [27].

Anodic deposition of vanadium oxide into the PS colloidal crystal templates was performed at a constant voltage of 2 V versus Ag/AgCl from a 1:1 mixture (volume ratio) of distilled water and ethanol containing 0.25 M $VOSO_4 \cdot 5H_2O$ and Pt foil as a counter electrode. An electrodeposition time of 45 s was chosen to slightly overfill the PS templates. The pH of the electrolyte was adjusted to 2.7 using NaOH. After deposition, the samples were immersed in a 1:1 mixture (volume ratio) of dimethylformamide (DMF) and toluene for 24 h to remove the PS templates. Finally, the as-prepared samples were dried at 110 °C for 1 h and then annealed in air at 375 °C for 7 h at a rate of 1 °C min^{-1} to obtain coralline V_2O_5 nanorod architectures.

2.2. Fabrication of electrochromic devices

An aqueous PEDOT solution was purchased from AGFA Company (EL-P 3040). PEDOT films on the clean ITO substrates were prepared by spin coating [2000 rpm (33.3 s^{-1})]. The electrochromic device in the form of ITO/ V_2O_5 /liquid electrolyte/PEDOT/ITO was fabricated by the following process. Two ITO glass-coated active materials were separated by poly(methyl methacrylate) (PMMA) partitions to form an inner cuboid cell for injecting electrolyte. The distance between the electrodes was 1.0 mm. Then the cell was sealed with epoxy glue. A 1 M LiClO_4 /propylene carbonate solution was then injected into the cell through a small hole with a syringe needle and the hole was sealed.

2.3. Characterization and evaluation of electrochromic properties

The morphologies of the as-prepared V_2O_5 films were characterized by scanning electron microscopy (SEM, FEI Helios Nanolab 600i). The crystalline structures of the V_2O_5 films were investigated by using an X'Pert PRO X-ray diffractometer with Cu $K\alpha$ radiation and high-resolution transmission electron microscopy (HRTEM, FEI Tecnai G2F30, 300 kV). Raman spectra were recorded with a spectrophotometer (JY Co.) with an operating wavelength of 613 nm. X-ray photoelectron spectroscopy (XPS) studies were conducted with a PHI 5700 ESCA system using Al $K\alpha$ radiation (1486.6 eV).

In situ visible and near-infrared (NIR) electrochromic measurements were performed using an experimental setup produced in-house (same setup used in Ref. [27]) in combination with a CHI 660C electrochemical workstation (Shanghai Chenhua Instrument Co. Ltd.). The experimental setup was sealed in argon-filled glove box (Vigor Glove Box from Suzhou, China) before testing. One side of the setup was connected to a white lamp (DT-mini-2-GS, Ocean Optics) by an optical fiber; the other side was connected to an optic spectrometer (MAYA 2000-Pro, Ocean Optics). The V_2O_5 film, Pt wire, and Ag/AgCl were used as the working, counter, and reference electrodes, respectively. A 1 M solution of LiClO_4 in propylene carbonate was used as the electrolyte. The transmittance of the ITO glass in the electrolyte was used as a reference for 100% transmittance. Before measuring the electrochromic performance of the V_2O_5 films and device, the film electrodes were subjected to eight cyclic voltammetry (CV) cycles to ensure stability. The transmittance switching response was investigated under alternating potentials. CV measurements were performed at room temperature between +1 and -1 V at different scan rates from 0.2 to 100 mV s^{-1} .

Coloration efficiency is defined by the following equations:

$$OD = \log(T_{bl}/T_{cl}) \quad (1)$$

$$CE = OD/Q_{in} \quad (2)$$

where $T_{b\lambda}$ and $T_{c\lambda}$ represent the transmittance of the bleached and colored samples, respectively. Q_{in} corresponds the injected/ejected

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