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Hydrothermal-synthesized mesoporous nickel oxide nanowall arrays with enhanced electrochromic application



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ABSTRACT

Mesoporous NiO nanowall arrays are prepared by a facile hydrothermal synthesis method in combination with a following annealing process. The as-prepared NiO nanowalls have average diameters of ~10 nm and composed of interconnected nanoparticles ranging from 5 to 30 nm. The NiO nanowall arrays show a hierarchical porous structure from mesopores of 5-20 nm to macropores of 30-200 nm. The electrochromic performances of the NiO nanowall arrays are characterized by means of UV-vis spectroscopy and cyclic voltammetry (CV) measurements. The mesoporous NiO nanowall arrays exhibit much better electrochromic performance with faster switching speed and higher color contrast than the dense NiO film. The mesoporous NiO nanowall arrays show a transmittance variation with 77% at 550 nm, and the coloration and bleaching times are 2 s and 2.5 s, respectively. The improved electrochromic performances are due to the porous morphological characteristics, which provide fast ion and electron transfer resulting in fast reaction kinetics and high color contrast.

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1. Introduction

Electrochromism (EC), defined as a reversible change in optical properties of a material as the result of electrochemical oxidation or reduction, has been intensively studied because of potential applications for smart windows and other information display devices [1,2]. Among numerous electrochromic materials, NiO is an attractive electrochromic material due to its high electrochromic efficiency, large dynamic range, good cyclic reversibility and low material cost [3-6]. However, slow switching speed and low color contrast hinder further advancements in current NiO electrochromic films [1,7]. It is well accepted that electrochromic process is an electrochemical reaction associated with double injection (extraction) of ions and electrons to (from) the electrochromic film. The switching speed is usually not satisfactory because of the slow transport of counter-ions into the active NiO layer to balance charge [8]. All this indicates that the electrochromic process is controlled by the reaction kinetics. Fast switching speed must require fast ion/electron transfer to get fast color change. As for color contrast, it is highly related to the active surface area of the electrochromic materials. Generally, NiO films with large surface area possess much more active reaction area and lead to higher color contrast. Hence, it is highly desirable to design and fabricate high-performance NiO electrochromic films by modifying or

ameliorating their kinetics issue. One effective strategy is to fabricate NiO films with highly porous structure, which can provide short diffusion pathway for the ion/electron and a large active surface, resulting in improved switching speed and optical modulation [7-10].

Up to now, several methods have been developed to fabricate nanoporous electrochromic NiO films, such as template-assisted cathodic electrodeposition [11], anodic electro-deposition process [12], spray pyrolysis [13] and chemical bath deposition (CBD) [7,10]. Previously, Yuan et al. reported a porous bowl-like NiO via monolayer polystyrene sphere template-assisted electrodeposition and enhanced properties [11]. Besides, Tu group [7] and Wu team [12] used chemical bathe deposition and anodic electrodeposition methods to prepare porous net-like NiO films, respectively, and improved electrochromic performances have been demonstrated in these porous NiO films. To the best of our knowledge, there are few reports about electrochromic performance of mesoporous NiO nanowall arrays. Stimulated by the results above, in the present work, we report mesoporous NiO nanowall arrays prepared by a facile hydrothermal synthesis method as well as their electrochromic properties. Impressively, the as-prepared mesoporous NiO nanowall arrays show good electrochromism with high color contrast and fast response speed.

2. Experimental

All solvents and chemicals were of reagent quality and used without further purification. The nickel nitrate (99.9%),

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hexamethylene tetramine (HMT, 99.8%), potassium hydroxide, sodium nitrate (99.9%) were obtained from Sigma–Aldrich Reagent Co. All aqueous solutions were freshly prepared with high purity water ($18 M\Omega \text{ cm}$ resistance).

In a typical preparation, 1 mmol of Ni(NO₃)₂ and 2 mmol HMT (hexamethylene tetramine) in 70 mL of distilled water and then transferred into Teflon-lined stainless steel autoclave liners. Then clean ITO glass with a size of $2.5 \text{ cm} \times 2.5 \text{ cm}$ was immersed into the reaction solution. The top side was protected from solution contamination by uniformly coating with a polytetrafluorethylene tape. Hereafter, the autoclave was sealed and maintained at $105 \,^{\circ}$ C for 1 h, and allowed to cool down to room temperature spontaneously. After the reaction, the substrate was taken out, completely washed with deionized water, and dried in the air. Finally, the sample was annealed at $300 \,^{\circ}$ C in argon for 1.5 h to form mesoporous NiO nanowall arrays.

For the sake of comparison, a dense NiO film was prepared by a cathodic electro-deposition performed in a three-compartment system at 25 °C. The ITO glass was used as the working electrode, saturated calomel electrode (SCE) as the reference electrode and a Pt foil as the counter-electrode. The electrolyte contained 0.5 M Ni(NO₃)₂ and 0.05 M NaNO₃. The electro-deposition was carried out at a constant current of 1 mA cm⁻² for 600 s. Finally, the asprepared sample was dried at 85 °C, and then annealed at 300 °C in argon for 1.5 h. The thickness of the both films was approximately 500 nm determined with an Alpha-step 200 profilometry.

The morphology and microstructure of the annealed films were characterized by a field emission scanning electron microscopy



Fig. 1. XRD patterns of mesoporous NiO nanowall arrays and dense NiO film.

(FESEM, Hitachi S-4700),), transmission electron microscopy (TEM, JEM 200 CX 160 kV), X-ray diffraction (XRD, Philips PC-APD with Cu K α radiation) and X-ray photoelectron spectroscopy (XPS, PHI 5700).

The transmission spectra of NiO films in the fully colored and fully bleached states were measured ex situ over the range from 200 to 900 nm with a SHIMADZU UV-2550 spectrophotometer. The electrochromic performances of NiO films with 2.5 cm \times 2.5 cm in



Fig. 2. SEM images of (a and b) mesoporous NiO nanowall arrays and (c and d) dense NiO film.

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