

## Letter

Fast electrochromic properties of self-supported  $\text{Co}_3\text{O}_4$  nanowire array filmX.H. Xia, J.P. Tu<sup>\*</sup>, J. Zhang, J.Y. Xiang, X.L. Wang, X.B. Zhao

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## ABSTRACT

We report a self-supported  $\text{Co}_3\text{O}_4$  nanowire array film prepared by a facile thermal oxidative decomposition method and its fast electrochromic properties. The electrochromic performances of  $\text{Co}_3\text{O}_4$  nanowire array film are investigated in 0.1 M KOH by means of transmittance, cyclic voltammetry (CV) and chronoamperometry (CA) measurements. The effective area of the  $\text{Co}_3\text{O}_4$  nanowire array film is  $2 \times 2 \text{ cm}^2$ . The  $\text{Co}_3\text{O}_4$  nanowire array film exhibits a transmittance modulation of 32% in the visible region and fast response times with 1.8 s for coloration and 1.4 s for bleaching. The electrochemical stability of the  $\text{Co}_3\text{O}_4$  nanowire array film is characterized by chronoamperometry with potentiostatic cycling. After 4500 cycles, the peak currents of the film maintain 88% of the highest values.

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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 commercial applications for smart windows [1–3]. However, some drawbacks such as slow response time and low color contrast have hindered their applications. An effective way to overcome these problems is to fabricate electrochromic films with a large surface area and short ion diffusion pathway [4]. One-dimensional (1D) nanostructures such as self-supported nanotube and nanowire array films could fulfill these requirements due to their unique structures and intriguing properties, which differ from those of the corresponding bulk-state materials. Their high surface area and high porosity associated with the open nanostructures allows for easy diffusion of ion, resulting in reduced ion-transport resistance and improved response time [5,6].

Of the available electrochromic materials,  $\text{Co}_3\text{O}_4$  is an attractive anodic electrochromic material due to its low power consumption, low fabrication cost and good cyclic reversibility [1]. A variety of methods have been developed to fabricate electrochromic  $\text{Co}_3\text{O}_4$  film, such as chemical vapor deposition (CVD) [7], sputtering [8], spray pyrolysis [9], sol–gel process [10], electrochemical deposition [11] and chemical bath deposition (CBD) [12,13]. Nevertheless, these as-prepared films exhibit compact structures with relatively low electrochromic performance except a CBD randomly porous  $\text{Co}_3\text{O}_4$  film reported in our previous work [13]. On the other hand, several 1D  $\text{Co}_3\text{O}_4$  nanostructures such as nanorods, nanowires and nanotubes have

been prepared by different methods [14–17]; however, most of these researches focused on the 1D  $\text{Co}_3\text{O}_4$  powder form materials and there is little literature into the electrochromic properties of self-supported  $\text{Co}_3\text{O}_4$  nanowire array films. Therefore, the purpose of this work is to report a facile thermal oxidative decomposition method for direct large-area synthesis of self-supported  $\text{Co}_3\text{O}_4$  nanowire array film and its electrochromic properties. The as-prepared  $\text{Co}_3\text{O}_4$  nanowire array film exhibits enhanced electrochromism with fast response time and good cycling stability.

## 2. Experimental

The self-supported  $\text{Co}_3\text{O}_4$  nanowire array film was prepared as follows. Clean indium tin oxide (ITO) with a size of  $2 \times 2 \text{ cm}^2$  were used as substrates. To facilitate the nucleation of  $\text{Co}_3\text{O}_4$  nanowire growth, a 5 nm thick  $\text{Co}_3\text{O}_4$  thin film (seed layer) was first deposited on the ITO substrate by spin coating with a 5 mM ethanolic solution of cobalt acetate, followed by conversion to  $\text{Co}_3\text{O}_4$  at  $350^\circ\text{C}$  for 0.5 h in the flowing argon. In a typical synthesis of  $\text{Co}_3\text{O}_4$  nanowire array film, 5 mmol  $\text{Co}(\text{NO}_3)_2$  was dissolved in 15 mL of 28 wt% ammonia solution and 20 mL of  $\text{H}_2\text{O}$ . The homogeneous solution was magnetically stirred for 0.5 h in air till the original pink color gradually turned into black. Then the solution was transferred to a covered petri dish and heated in an oven at  $90^\circ\text{C}$  for 0.5 h. Afterwards, the substrate was fixed using a Teflon clamp with the seed layer side facing downwards, which was approximately 1–2 mm away from the bottom of the petri dish. Its top side was protected for solution contamination by uniformly coating with a layer of nail polish (its primary ingredients are cellulose nitrate, ethyl acetate and polyester resin). The solution was heated for 5 h at  $90^\circ\text{C}$  for nanowire array growth. Finally, the sample was washed with  $\text{H}_2\text{O}$  and then with

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acetone to remove the nail polish layer on the top and annealed at 200 °C for 1 h in flowing argon. The thickness of the annealed film was approximately 550 nm.

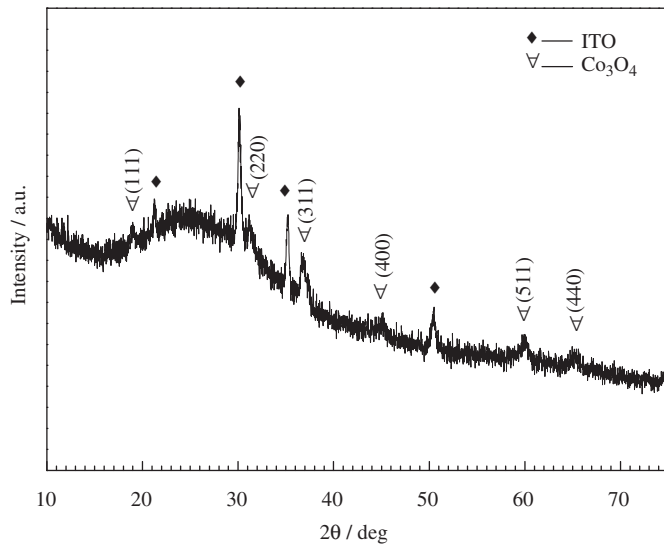


Fig. 1. XRD pattern of  $\text{Co}_3\text{O}_4$  nanowire array film.

The morphology and microstructure of the annealed films were characterized by a field emission scanning electron microscopy (FESEM, Hitachi S-4700) and transmission electron microscopy (TEM, JEM 200 CX 160 kV).

The transmission spectra of  $\text{Co}_3\text{O}_4$  thin films in the fully colored and fully bleached states were measured over the range from 200 to 900 nm with a SHIMADZU UV-240 spectrophotometer. The cyclic voltammetry (CV) and chronoamperometry (CA) measurements were carried out in a three-compartment system containing 0.1 M KOH as electrolyte, Hg/HgO as reference electrode and a Pt foil as counter-electrode. Cyclic voltammetry (CV) measurements were performed using a CHI660c electrochemical workstation with a scanning rate of  $10 \text{ mV s}^{-1}$  between  $-0.1$  and  $0.7 \text{ V}$  at  $25^\circ\text{C}$ .

### 3. Results and discussion

Fig. 1 shows the XRD pattern of film deposited on ITO substrate. The diffraction peaks of the sample can be indexed as spinel  $\text{Co}_3\text{O}_4$  phase (JCPDS 42-1467). Fig. 2a–d show typical SEM images of the as-prepared  $\text{Co}_3\text{O}_4$  nanowire array film by ammonia evaporation induced with a seeded layer method. The  $\text{Co}_3\text{O}_4$  nanowires have a diameter varying from 70 to 100 nm and a length of about 550 nm. They grow relatively vertical to the substrate forming a nanoarray film (Fig. 2d), similar to those reported by Li et al. [18,19]. Their

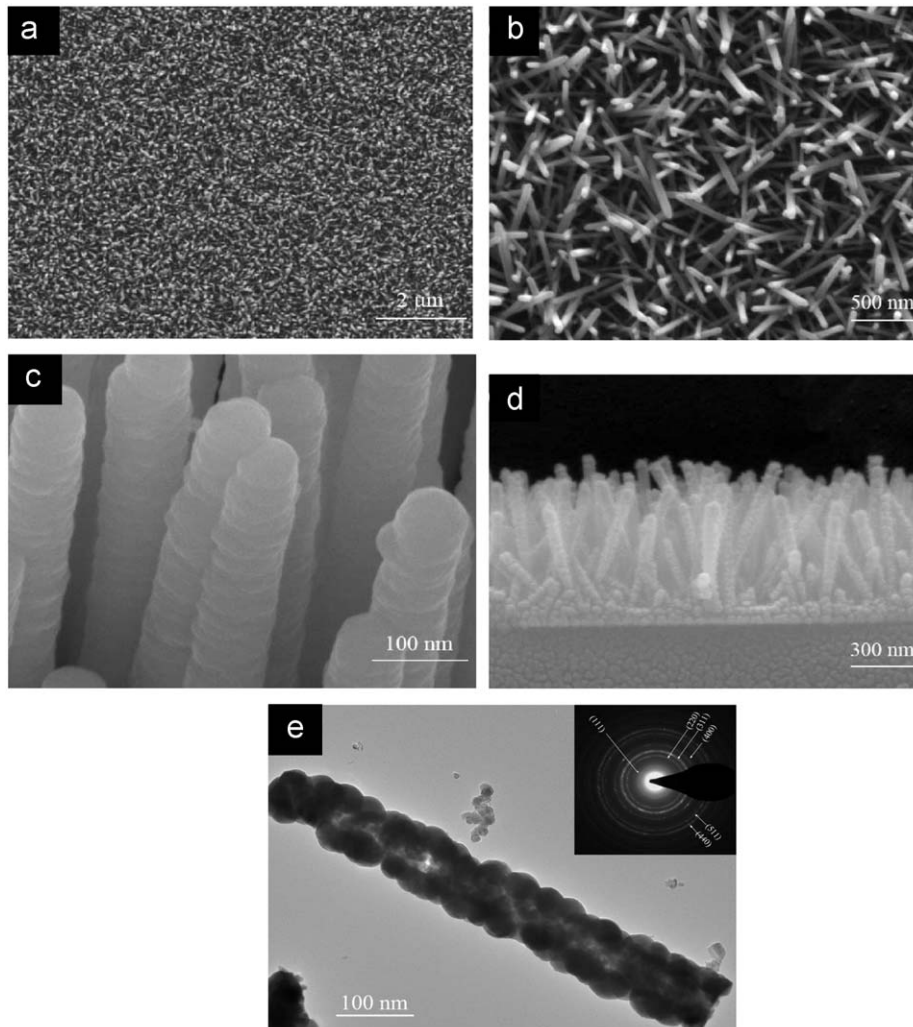


Fig. 2. Morphological and structural characterizations of the  $\text{Co}_3\text{O}_4$  nanowire array film: (a–d) SEM images; (e) TEM image (the SAED pattern in inset).

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