



Fabrication of one-dimensional ZnO nanotube and nanowire arrays with an anodic alumina oxide template via electrochemical deposition



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ABSTRACT

In this work, two kinds of one-dimensional ZnO nanowires (NWs) and nanotubes (NTs) were synthesized by using electrochemical deposition with the aid of a high aspect ratio anodic alumina oxide (AAO) template. ZnO NWs and NTs were characterized by using X-ray diffraction, field emission scanning microscopy, transmission electron microscopy, and energy-dispersive X-ray spectroscopy. An AAO template was fabricated by two-step anodization in 0.3 M oxalic acid with a voltage of 80 V. The thickness and channel diameter of the AAO template were about 150 μm and 120–150 nm, respectively. The morphologies of the ZnO nanostructures synthesized under 20 vol.% H₂O₂ with various electrolyte concentrations of 0.1 M and 0.5 M ZnSO₄, were NTs and NWs, respectively. Both NTs and NWs were uniform in size, which corresponded to the sizes of AAO pores. The thickness of the NTs walls can be controlled based on the deposition time and current density. The crystallinity of the ZnO NTs and NWs annealing in the air were restricted by AAO pore. The growth of the ZnO NTs and NWs was caused by heterogeneous nucleation, and different growth rates through the wall of the AAO will result in different nanostructures, with the growth of the NTs being slower than that of the NWs.

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1. Introduction

In order to reduce carbon emissions, many products need to be lighter and more environmentally friendly. Nanostructure materials have both these qualities, and thus have been extensively studied in the last decade, with significant attention being paid to low-dimensional (i.e., zero- and one-dimensional) nanomaterials [1–8]. Research shows that when materials are miniaturized to the nanoscale, the bandgap will change along with the size of the nanowires (NWs) [9]. Due to the quantum confinement effect, electrons can only move along the axis of NWs, and thus controlling the size and shape of such materials will play an important role in the fabrication and application of nano-electronics.

Two procedures are often used to grow one-dimensional nano-materials. The first is chemical vapor deposition, which uses the vapor–liquid–solid mechanism. In this approach, nanostructures are mainly grown with the aid of a catalyst. However, the particle size, shape and uniformity of the catalyst used are difficult to control, causing

a nonuniform morphology and density in the resulting NWs arrays [10]. The other procedure is a template-assisted method, in which the morphology of the NWs is limited by the templates used, and thus the size and shape of the resulting nanostructures can be consistent. The template-assisted method is an effective way for the preparation of nanostructure arrays with different diameters and aspect ratios. Over the last twenty years, anodic aluminum oxide (AAO) has been one of the most widely-used materials in such templates, which are usually composed of six square nest chambers, with each cell having a hole in the center. The hole size can be controlled by using different anodizing voltages and electrolytes, and range from a few nanometers to several hundred nanometers [11], with the AAO film thickening as the anodization time increases. Masuda and Fukuda [12] used a two-step anodization process to produce an anodic aluminum oxide film with high uniformly holes of around 3–100 nm. Since AAO has both high chemical and thermal stability, there is considerable interest in the use of AAO templates to produce highly uniform one-dimensional nanostructures with a high aspect ratio.

ZnO is a well-known member of the II–VI group of n-type semiconductors, having a wurtzite hexagonal structure. Because of the wide bandgap (3.37 eV) of ZnO, it is transparent to the visible light region, so it is suitable for use in transparent conductive films [13]. In addition, the high exciton binding energy (60 meV) means that it has an excellent luminous efficiency at room temperature, and thus has been utilized

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Table 1

Experimental details of electrochemical deposition of ZnO nanostructure into AAO template.

ZnO morphology	Electrolyte	Current density	Deposition time	Annealing condition	Sample number
ZnO NTs	ZnSO ₄ 0.1 M H ₂ O ₂ 20 vol.% 80 °C	6 mA/cm ²	40 min	500 °C	A
				10 h (air)	
		10 mA/cm ²	20 min	500 °C	B
				10 h (air)	
ZnO NWs	ZnSO ₄ 0.5 M H ₂ O ₂ 20 vol.% 80 °C	10 mA/cm ²	40 min	X	C
				500 °C	D
				10 h (air)	
				300 °C	E
				10 h (air)	F
				400 °C	G
				10 h (air)	
				500 °C	G
				10 h (air)	

as a short-wavelength light-emitting material and in ultraviolet lasers [14,15]. ZnO not only has good optical properties, but also excellent semiconducting properties, without doping these are caused by the non-stoichiometric zinc and oxygen atoms [16]. When oxygen vacancies or zinc interstitials exist, they will introduce a donor level in the

energy gap and produce free electrons. The high mechanical strength, thermal stability and chemical stability [17] of ZnO mean that it can be used as a field emission material [18] or in vacuum electronic applications. In addition, gas can easily be adsorbed on the ZnO, thus changing its electrical conductivity, which is why this material can be used in gas sensors [19,20].

Elias et al. [21] used ZnCl₂, ZnSO₄ and Zn(CH₃COO)₂ as the electrolytes to fabricate NWs with aspect ratios of 9, 20, and 60 without template, although they were not able to produce high aspect ratio nanostructures because of template-free. Some researchers have been fabricating ZnO nanowires by electrodeposition method in template. For example, Zheng et al. [22] used one-step electrodeposition technique base on AAO template. They utilized Raman spectroscopy and reported that the ZnO NWs they produced were formed by many single crystals and the NW aspect ratio is about 12. They also reported that microtwins, stacking faults and the low-angle boundaries of the AAO template will induce different orientations in the ZnO. Wang et al. [23] also used electrodeposition method with non-aqueous solvent (dimethyl sulfoxide, DMSO) to dissolve the membrane vesicles in the AAO template and help the growth of ZnO NWs with aspect ratio about 33. Li et al. [24] first used electrochemical deposition to synthesize zinc NWs in AAO template, and then annealed them for various times in order to obtain ZnO NWs. Lai and Riley [25] used H₂O₂ and Zn(NO₃)₂

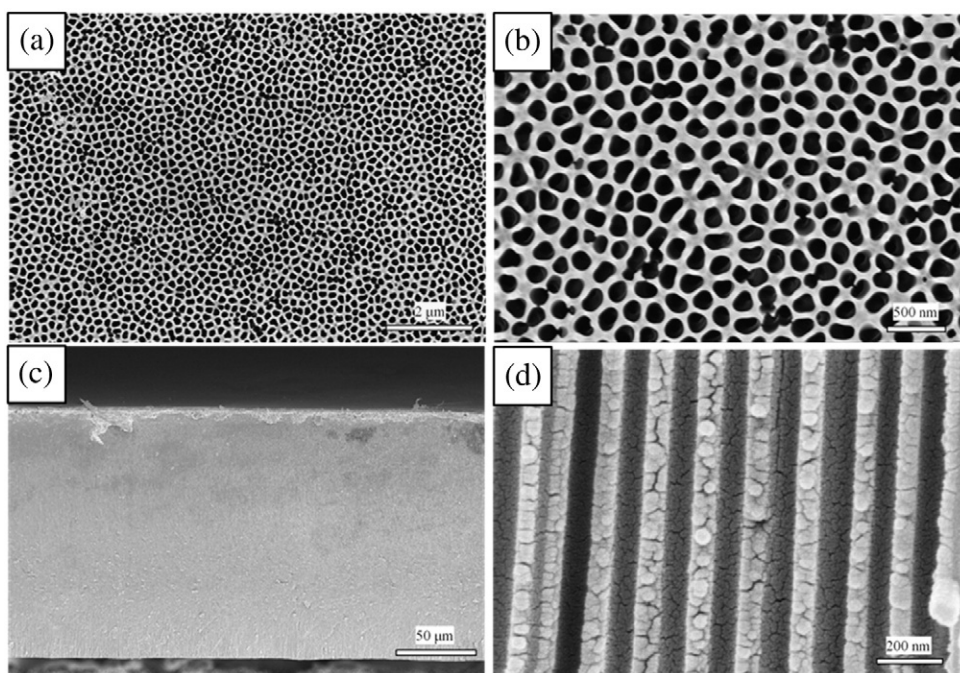


Fig. 1. SEM images of AAO template (a, b) plane-view (c, d) cross-section.

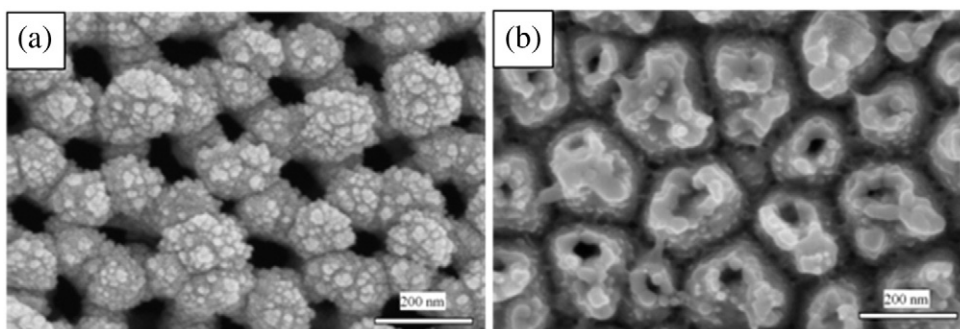


Fig. 2. SEM images of Pt working electrode (a) downward side and (b) upward side after removing the AAO template.

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