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Investigation and characterization of ZnO Nanostructures synthesized by electrochemical deposition

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Abstract

ZnO with cube-like nanostructures have been fabricated on tin-doped indium oxide (ITO) coated glass substrates via electrochemical deposition method. The electrolytes used containing equimolar of zinc chloride (ZnCl₂) and potassium chloride (KCl) ranging from 0.05 M to 0.2 M. The samples produced subsequently were examined by field emission scanning electron microscope (FESEM), x-ray diffractometer (XRD), energy dispersive X-ray (EDX) spectroscopy and current transient profile. The average size of the cube-like nano ZnO materials, as observed from FESEM images is decreased from 207 nm to 102 nm with increasing of electrolytes concentration. The XRD measurement indicated that the as-deposited nanostructures are converted from amorphous to hexagonal wurtzite ZnO phase after heat treatment at 600 °C for 3 hours. Photoresponse measurement revealed that the grown sample is sensitive to the 372 nm ultraviolet (UV) light.

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Nomenclature

CVD	chemical vapor deposition
DI	de-ionized
ECD	electrochemical deposition
EDX	energy dispersive X-ray
FESEM	field emission scanning electron microscope
ITO	tin doped-indium oxide

MBE	molecular beam epitaxy
SMU	source measure unit
UV	ultraviolet
XRD	x-ray diffractometer

1. Introduction

ZnO is a II-VI semiconductor with wide direct band gap of 3.37 eV at room temperature^{1,2}. It received great interest in fabricating high-performance optoelectronic devices such as light emitting diodes, laser diodes, ultraviolet range detecting devices, chemical sensors, solar cells³⁻⁶ and etc.

Various methods have been reported to grow ZnO such as chemical vapor deposition (CVD), molecular beam epitaxy (MBE), sputtering, thermal oxidation, electrochemical deposition (ECD)⁶⁻¹⁰ and etc. Among the aforementioned, ECD is found to be the most interesting as it offers simplicity, fast growth rate and cost effectiveness^{8,9}.

There are two issues need to be heeded when ECD is used to grow high-quality ZnO nanostructures, a conducting solution and post-annealing treatment¹¹. In most of the reported works, KCl was used as a supporting electrolyte to ensure good conductivity in aqueous solution^{11,12}. However, chlorine will be incorporated together with the ZnO nanostructures, which affects its structural and electrical properties. Thus, post-annealing has to be done after the electrodeposition to remove the incorporated chlorine and crystallized the electrodeposited ZnO nanostructures^{8,11}.

The deposition reaction in aqueous ZnCl₂ solution and the formation of hydroxide ions from oxygen and water can be described as follows¹²:



In this work, we report the fabrication of ZnO nanostructures under different concentrations of electrolyte and its structural properties and photosensitivity will be determined subsequently.

2. Material and methods

ZnO nanostructures were grown by ECD technique in an electrolyte containing ZnCl₂ and KCl ranging from 0.05 M – 0.2 M with increment of 0.05 M per experiment. The molar ratio of ZnCl₂ and KCl was kept at 1 throughout the experiment.

The electrodeposition of cube-like ZnO nanostructures were performed on the conductive side of the ITO coated glass slides, which acted as the working electrode while copper wire as counter electrode. Prior to electrodeposition, the ITO substrates were cleaned in acetone, methanol and de-ionized (DI) water for 5 minutes each in an ultrasonic bath and dried under air flux. The copper wire was sanded by abrasive paper to remove the native oxide layer and then rinsed with isopropanol.

A beaker filled with electrolyte was immersed in a water bath where the whole setup was heated on a hotplate at 90°C. Then the electrodeposition was carried out with voltage of 8V using a Keithley 2400 source measure unit (SMU) that interfaced to a computer.

The surface morphology and elemental composition of the samples were characterized using FESEM and EDX (Nova NanoSEM 450). Meanwhile, XRD patterns (PANalytical X'Pert PRO MRD PW3040, CuKα1 with wavelength 0.154 nm, settings at 40 kV, 30 mA) were used to determine the structural properties of the samples. The photoelectric transient profile was measured by Keithley 2400 source measure unit (SMU) with constant potential of 15 V. The peak wavelength and power of the UV source are 372 nm and 36 W, respectively. The distance in between the sample and light source was about 2 cm.

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