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Rapid synthesis of ZnO nanostructures through microwave heating process

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Abstract

In this paper, polycrystalline zinc oxide (ZnO) nanostructures have been prepared by a hydrothermal synthesis through rapid microwave heating (180 s). The structure, composition and optical properties of the products were examined by scanning electron microscopy (SEM), energy dispersive x-ray spectrum (EDS), ultraviolet–visible spectroscopy (UV–vis), x-ray diffraction (XRD), photoluminescence spectroscopy (PL) and Raman spectroscopy. Typically, the synthesized nanostructures were zinc-rich with diameter ranging from 20 nm to 200 nm in length. From the Raman spectroscopy and PL measurements, it was found that the as-deposited films contain vacancy defects that originated from the rapid synthesis process.

Keywords: A. Microwave processing; B. Nanocomposites; C. Optical properties; D. ZnO

1. Introduction

Zinc oxide (ZnO) is a wide bandgap (3.37 eV) semiconductor with large exciton binding energy that has been used in wide range of applications such as photovoltaic [1,2], sensors [3–5], thin film transistors [6,7], humidity sensing [8] and nano-generators [9]. One of the attractive features of ZnO is the wide range of nanostructure it can adapt through control of process parameters. Previous studies have successfully synthesized nanowires [10–13], nanorods [14–17], nanopetals [18,19], and nanotripods [20].

Several methods have been adapted to prepare ZnO nanostructures such as thermal chemical vapor deposition (CVD) over catalyst coated substrates [21,22], electrochemical process on anodized alumina membrane [23] and hydrothermal growth on pre-seeded layer [24,25]. Although high quality ZnO nanomaterials can be obtained through thermal CVD process, excessive process temperature and complicated vacuum equipment presents major up-scaling and cost related issues. Alternatively, an anodized alumina membrane process presents versatile template process for manufacturing ZnO and can be adapted to wide range of nanomaterials (cadmium telluride [26], ZnO [27], copper indium gallium selenide [28] etc.). However, high-quality anodized alumina membrane is expensive and suffers from scaling issues. On the other hand, hydrothermal process is an attractive process due to its low technological requirements and low-process temperature ($<100\,^{\circ}C$) that enables depositions on low-cost, flexible plastic substrates. Generally, hydrothermal process consists of two steps; namely, seed layer deposition and ZnO growth. Firstly, the seed layer is prepared by refluxing ZnO containing compounds (e.g. zinc acetate) in alcoholic solvent. Subsequently, the prepared solution is spin coated onto the substrate and annealed to enable conformal seed layer formation. The second step, consists of transferring the precoated substrate into heated chemical bath which consists of zinc nitrate and ammonia-based nutrient solution (hexamine). After the nutrient solution is replenished, the growth rate of ZnO nanostructures slows down and eventually terminates. In order to produce ZnO nanowires with practical length (5 μm) typical growth time lasts for 2–12 h [25]. Such slow deposition rate is impractical for industrial production.

Consequently, it would be attractive to find the growth of ZnO nanomaterials accelerate through alternative deposition techniques. Microwave assisted process has been of interest ever since the first reports [29,30]. Noteworthy features include rapid volumetric heating and

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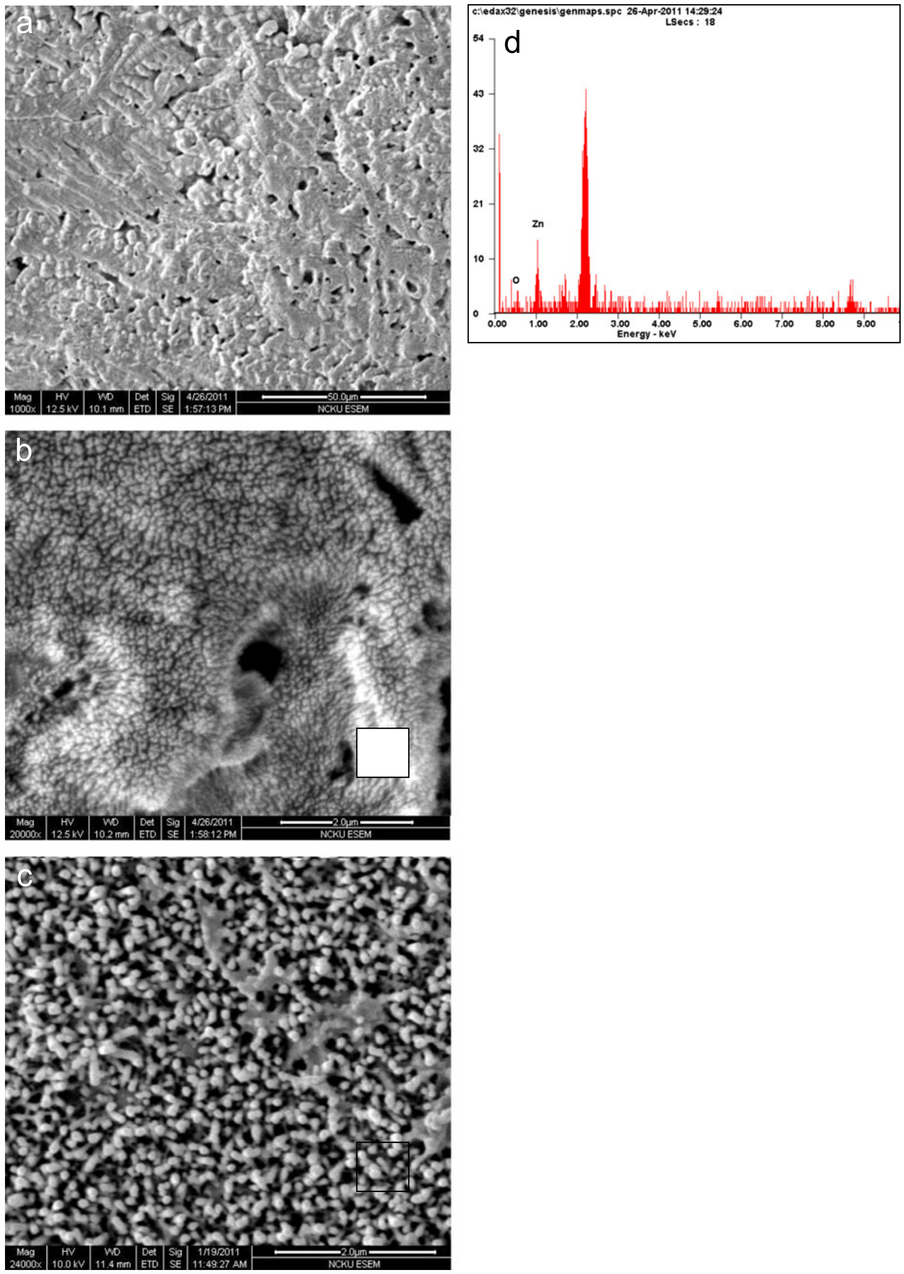

Fig. 1. (a) Top view SEM image of the as deposited ZnO nanostructure at lower resolution. (b) Top view SEM image of the as-deposited ZnO nanostructure at higher resolution. (c) Typical top view SEM image of the hydrothermally grown ZnO nanowire by hot-plate heating. (d) Typical EDS composition analysis of the microwave synthesized ZnO.

energy saving as compared to conventional heating methods. Consequently, microwave assisted heating has been adapted to synthesis various nanocrystalline ZnO oxide. In this study, a one pot, fast synthesis method of zinc oxide nanomaterials by microwave irradiation has been developed and thoroughly characterized through SEM, EDS, UV–vis, XRD, PL and Raman spectroscopy.

2. Experimental

All chemicals were of analytical grade, purchased from Sigma Aldrich and used without further purification. In a typical procedure, soda lime glasses were sequentially cleaned by ultrasonication in acetone, methanol and deionized water (DI). The cleaned soda lime glass substrates were blown dry by N_2 flow and coated with ZnO seed layer by spinner. The ZnO seed layer solution was prepared by dissolving 1 mM of zinc acetate into IPA using a magnetic stirrer without external heating. Then, the spin-coated seed layer is heated to 300 °C for 10 min to ensure the formation of nano-sized ZnO seed. Such coating and annealing of seed layer were repeated three cycles to enhance adhesion to the substrates.

The seed-coated glass substrates were transferred into the growth solution (equimolar aqueous solution of zinc