Optik 127 (2016) 4621–4624

Contents lists available at ScienceDirect

Optik

journal homepage: www.elsevier.de/ijleo

Morphological evolution of hydrothermally derived ZnO nano and microstructures

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ARTICLE INFO

Article history:
Received 1 January 2016
Accepted 8 February 2016

PACS:
79.60.Jv
81.10.Dn
68.37.Hk
61.10.Nz
78.20.−e

Keywords:
Nanostructures
Growth from solutions
Scanning electron spectroscopy
X-ray diffraction
Optical properties

ABSTRACT

ZnO nanorods with flowerlike morphologies were synthesized via hydrothermal route. The observations depicted that the as-synthesized flowerlike morphology of ZnO consisted of numerous nanorods. The length of nanorod is in the range of 2–3 μm, and the diameter ~ 150–200 nm. The ZnO product was crystallized with hexagonal crystal structure. High resolution transmission microscopy image and electron diffraction pattern revealed a single crystalline nature altogether (0001) direction growth. The photoluminescence spectrum showed a near band edge UV emission peak and defect related blue emission peak in the visible region. Furthermore, the experimental results revealed that the extended reaction period and zinc precursor used in reaction mixture played an inevitable role in the morphological evolution of ZnO nano-/microstructures.

1. Introduction

Zinc oxide is one of the fascinating semiconductor materials that can be used in optoelectronic devices due to its enthralling physicochemical properties that includes a wide band gap (3.36 eV), high exciton binding energy at room temperature (60 meV), high optical transparency in the visible region, and strong piezoelectricity. In addition, among various other metal oxides it is the most cost effective, environmental benign, and efficient semiconductor. It is a promising candidate for a wide variety of applications, such as solar cells [1], laser diode [2], piezoelectricity [3], gas sensors [4], field-emission transistors [5], ultraviolet photodetectors [6], photocatalysis [7], photonic crystals [8], and optoelectronic devices [9] etc. Many researchers are actively engaged to synthesize ZnO with various forms of morphologies. It is mainly due to its size, shape, and dimensionality dependent electro-optical properties. ZnO micro-nanostructures have been grown by various methods that include chemical bath deposition method [10] chemical vapor deposition [11], template-based method [11], polymerization method [12], sol–gel synthesis [13], microwave method [14], laser ablation [15], spray pyrolysis technique [16], hydrothermal method [17], sonochemical method [18], electrodeposition method [19], and thermal evaporation [20]. Among these methods, the hydrothermal method is most practical method with numerous advantages, such as simplicity, inexpensiveness, large scale productivity, low-temperature processing, mild reaction conditions, template and catalyst free, non-toxicity, no requirement of vacuum or carrier gas, maximum yield with high crystallinity and easy control over the preparative parameters [21]. Also, the remarkable benefit of this method is to study the influences of various processing parameters on the size, crystallinity, morphology, and physicochemical properties of the synthesized products [22].

In this work, a simple hydrothermal route has been used to synthesize flowerlike 3D ZnO superstructures consisted of numerous individual as well as grouped nanorods. The method involved direct growth of ZnO using aqueous solution of zinc nitrate and sodium hydroxide. In addition, the influence of various parameters on the growth of ZnO nanostructures was also studied.

2. Experimental procedure

Synthesis of ZnO nanopowder: analytical grade reagents purchased from Showa Chem. (Japan) were used without further

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http://dx.doi.org/10.1016/j.ijleo.2016.02.006
0030-4026/

purification. At the initial stage, an appropriate amount of Zn $(NO_3)_2$. $2H_2O$ was dissolved in 50 ml of deionized water (Milli Q 18.2 MΩ) in a beaker to make the 0.05 M concentration. Then 50 ml (0.05 M) of aqueous solution of sodium hydroxide (NaOH) was added into it under constant stirring. Afterward, the resultant solution was transferred into a Teflon-container that was further sealed in a stainless steel autoclave. The autoclave was kept in a hot air oven and the temperature was gradually raised to 170 °C for 8 h. After stipulated time, the autoclaves were naturally cooled to room temperature. Subsequently, the resulting solid product was collected, washed with distilled water, and dried in air.

The X-ray diffraction (XRD) pattern was recorded using an X-ray diffractometer (Mac Science, MXP18) with CuK_α radiation. The morphological nanostructures of the product was observed by a scanning electron microscope (SEM) using JEOL JSM-5600 model that was equipped with an energy dispersive spectrophotometer (EDS) (Oxford Instruments) for the elemental analysis. The obtained ZnO product was also characterized with a high-resolution transmission electron microscope (HRTEM) images and selected area electron diffraction (SAED). The HRTEM and SAED pattern were obtained from a JEOL JEM-400EX operated at 400 kV. The absorption spectra were recorded with UV–visible spectrophotometer (Hitachi Model-3300, Japan). The photoluminescence (PL) property was studied with Hitachi F-4500 model.

3. Results and discussion

The XRD pattern of hydrothermally derived ZnO product is shown in Fig. 1. The strongest peak in the XRD pattern could be indexed to (1 0 1) at 2θ value of 36.29^0. The other observed peaks can be indexed as (100), (002), (102), (110), (103), (220), (112), (201), and (004) reflections of ZnO at 2θ values of 31.83^0, 34.43^0, 47.55^0, 56.63^0, 62.91^0, 66.43^0, 67.97^0, 69.11^0, and 72.73^0, respectively. It is consistent with standard JCPD file no. 36–1451 that confirms hexagonal (wurtzite) crystal structure. The diffraction peak indexed to (101) is much stronger and sharper than the other peaks with small full width at half-maximum (FWHM: 0.25^0). The peaks were narrow and highly intense, indicating highly crystalline nature of the ZnO nanorods. Fig. 2 shows the EDS pattern of synthesized ZnO product, to depict elemental composition. It shows

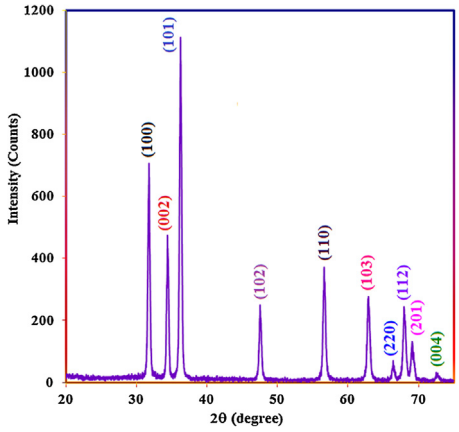

Fig. 1. XRD pattern obtained from synthesized ZnO for 8 h.

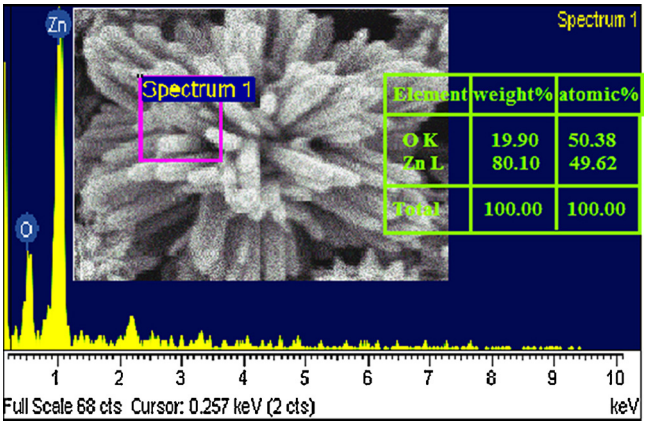

Fig. 2. A typical EDS pattern of synthesized ZnO product for 8 h.

sharp peaks for Zn and O elements. The elemental ratio for Zn:O was found to be 50.38:49.62, which confirms high purity of synthesized ZnO product. Inset in Fig. 2 depict the region selected for the EDS pattern. The peak of Au was observed at 2.2 keV and is

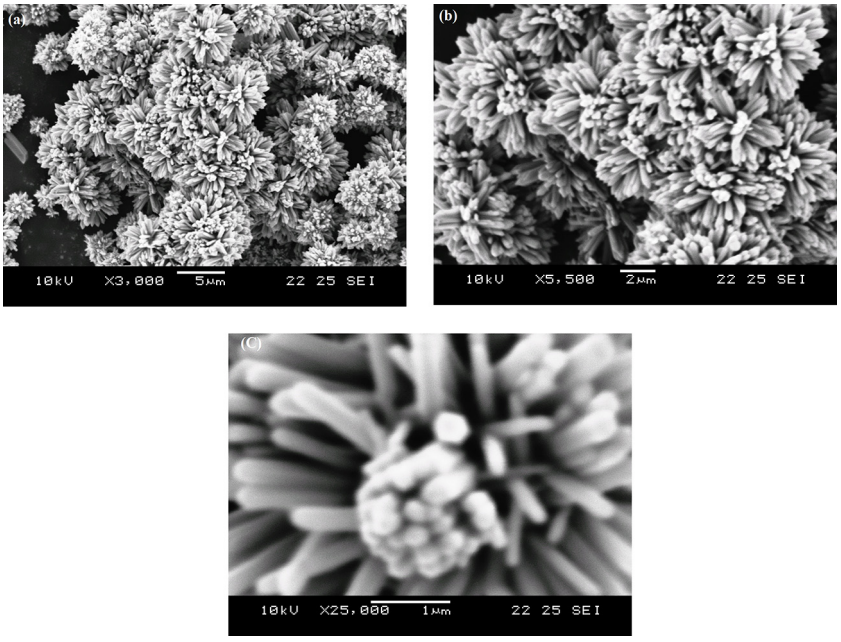

Fig. 3. SEM micrographs of ZnO product grown for 8 h.