

# Preparation and characterization of high uniformity zinc oxide nanosheets

Xiaoyan LI<sup>1,2</sup>, Pei LIANG (✉)<sup>2</sup>, Le WANG<sup>2</sup>, Feihong YU<sup>1</sup>

<sup>1</sup> State Key Laboratory of Modern Optical Instrumentation, Department of Optical Engineering, Zhejiang University, Hangzhou 310027, China  
<sup>2</sup> College of Optical and Electronic Technology, China Jiliang University, Hangzhou 310018, China

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**Abstract** This paper reports a synthesis of zinc oxide (ZnO) nanosheets by hydrothermal method. ZnO nanosheets on Al substrate were generated by hydrothermal synthesis with zinc nitrate hexahydrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) and hexamethylenetetramine (HMT) as a research system, which were controlled conditions of the reaction. The energy dispersive spectroscopy (EDS), scanning electron microscope (SEM) and transmission electron microscopy (TEM) images were achieved to determine the characterization of ZnO nanosheets. The diameter of ZnO nanofilm was from 0.5 to 1  $\mu\text{m}$ , and its thickness ranged from 30 to 50 nm.

**Keywords** hydrothermal method, zinc oxide (ZnO), nanosheets

## 1 Introduction

Zinc oxide (ZnO) is a semiconductor material with a wide direct band-gap (3.37 eV), large exciton binding energy (60 meV) [1,2]. Moreover, ZnO is a cheap, abundant, chemically stable and non-toxic material. Nanostructured ZnO attracted more and more attention over the past few years due to its potential applications in solar cells [3], photocatalysis [4], optoelectronics [5], and field emission [6]. Nanosized inorganic semiconductors exhibit a wide range of electrical and optical properties, which depend sensitively on both size and shape [7,8]. For one-dimensional ZnO nanostructures, different shape structures such as nanorods [9], nanowires [10], and nanobelts [11] have been reported. Two-dimensional ZnO nanostructures, for instance sheet-like structures or platelets, have great importance for constructing functional nanodevices due to their high surface to volume ratios. So two-dimensional

ZnO nanostructures, such as nanosheets, have received increasing attention [12,13].

For the excellent crystallization of ZnO, the techniques for deposition ZnO thin film can be used to fabricate ZnO nanosheets. Thin films of ZnO have been deposited by using several deposition techniques, such as chemical vapor deposition [14], magnetron sputtering [15], pulsed laser deposition [16] and hydrothermal technique [17].

Hydrothermal technique is a method of synthesis of single crystals that depends on the solubility of minerals in hot water under high pressure. The crystal growth is performed in an apparatus consisting of a steel pressure vessel called autoclave, in which a solvent is water. Hydrothermal technique is a promising synthetic method because of low process temperature and very easy to control the crystal size. The hydrothermal process have several advantage over other growth processes, such as simple equipment, catalyst-free growth, low cost, large area uniform production, environmental friendliness and less hazardous. The particle properties, such as morphology and size, can be controlled during the hydrothermal process by adjusting the reaction temperature, time and concentration of precursors.

Wang et al. [18] synthesized ZnO nanosheets with thickness about 50 nm by hydrothermal synthesis approach under the condition of sodium tripolyphosphate (STTP) added into hydrothermal solution. Chin et al. [19] demonstrated an improved and rapid method to synthesize uniform two-dimensional ZnO nanosheets at low temperature. Yang et al. [20] synthesized flower-shaped ZnO nanosheets by a simple hydrothermal method at low temperature without any catalysts or templates.

In this paper, we reported a facile, low-temperature solution synthesis of ZnO nanosheets. The synthesized ZnO structures were examined in detail with morphology and structure by scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) and transmission electron microscopy (TEM).

## 2 Experimental procedures

All the reagents used in the experiments were analytical grade, and they were used without further purification. Zinc nitrate hexahydrate ( $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ) was used as a starting material. The ZnO nanosheets were prepared from zinc nitrate in a neutral aqueous solution under hydrothermal conditions. Al substrates were cleaned and degreased ultrasonically initially firstly by HCl and soap water and then by hot ethanol and acetone for 10 min each.

Firstly,  $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  was dissolved by deionized water to form 0.02 mol/L zinc nitrate aqueous solution, then hexamethylenetetramine (HMT) was added to the above solution, the molar ratio of  $\text{Zn}^{2+}$  and HMT were maintained at 1:1. The resulting solution was stirred by using a magnetic stirrer for 1 h, and then was transferred into a 200 mL stainless steel autoclave.

All cleaned Al substrate were put into the autoclave, and hydrothermal treatments were carried out at  $95^\circ\text{C}$  for 2.5 h. After that, the autoclave was allowed to cool down naturally. The Al substrates were taken out of autoclave and washed with ethanol and deionized water for several times and dried in air at  $40^\circ\text{C}$ .

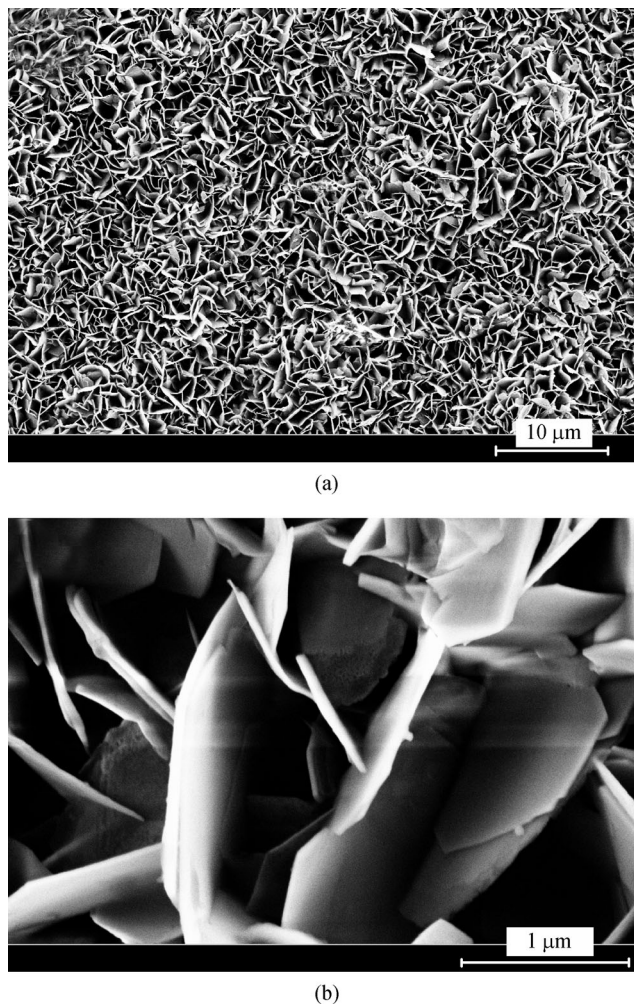
The surface morphology was investigated by using a SEM (ZEISS ULTRA55). The size distribution and morphology of the samples were analyzed by an H-800 TEM operated at 200 kV. The domains of the polymers were identified by an Oxford INCA PentaFET X3 EDS system.

## 3 Result and discussion

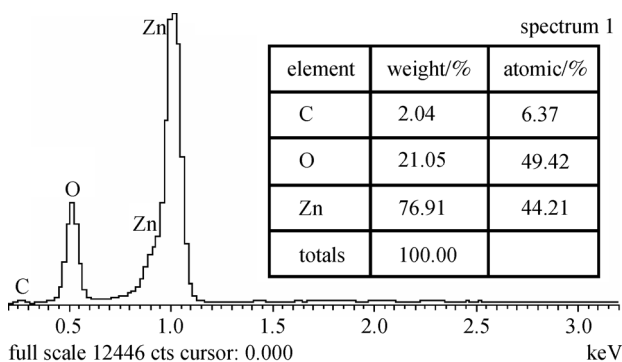
The general morphologies of synthesized ZnO nanosheet were examined by SEM and shown in Fig. 1. The low-magnification image shows that the synthesized products are “S” sheet-like morphology in high density (Fig. 1(a)). The high magnification SEM image shows that the nanosheets are aggregated and mingled in each other (Fig. 1(b)), which is due to the long range electrostatic interactions among the polar charges of the  $\{0001\}$  planes [21]. The average dimensions of the observed nanosheets ranges from 0.5 to 1  $\mu\text{m}$  with the typical thickness varying from 30 to 50 nm.

To identify the composition of the synthesized ZnO nanosheets, EDS attached with field emission scanning electron microscopy (FESEM) was used. The results of EDS spectroscopy in Fig. 2 indicates that the ZnO nanosheets are mostly composed of Zn and oxygen (O), whereas the peak of carbon (C) should come from impurity of Al substrate. Figure 2 shows the atomic percent of O element is 49.42%, and Zn element is 44.21%. The atom percentage ratio of Zn to O is almost 1:1.

To obtain structural characterization of synthesized ZnO nanosheets, TEM analysis was done, and results were

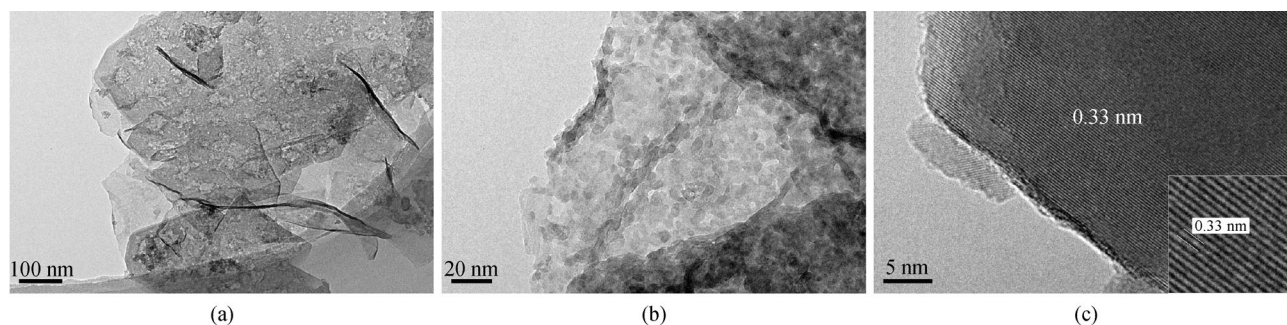


**Fig. 1** SEM images of nanostructured ZnO nanosheets. (a) Magnification: 2000; (b) magnification: 30000



**Fig. 2** EDS spectrum of synthesized ZnO nanosheets by simple hydrothermal process

demonstrated in Fig. 3. As shown in Fig. 3(a), ZnO nanosheets has high transparent characteristic. There are some black textures in the film, which are the overlapping



**Fig. 3** Typical (a), (b) low-magnification and (c) high-resolution TEM images of synthesized ZnO nanosheets by simple hydrothermal process

section of nanosheets. High-resolution TEM image in Fig. 3(c) shows that the ZnO nanosheets in the filled region are well crystallized and polycrystalline in nature.

## 4 Conclusions

A successful synthesis of ZnO nanosheets was done by simple hydrothermal process at low-temperature. The samples were investigated by the techniques of EDS, SEM and TEM. The results showed that the obtained ZnO nanosheets had good crystallization. The diameter of ZnO nanofilm was from 0.5 to 1  $\mu\text{m}$ , and its thickness ranged from 30 to 50 nm.

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