

Short communication

Continuous micro flow synthesis of ZnO nanorods with UV emissions

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Abstract

In this paper, a new process was presented to synthesize ZnO nanorods with diameter less than 10 nm. By using polytetrafluoroethylene capillary tube as micro flow reactor, ZnO nanorods could be controllably synthesized by changing the flow rates of pumped stock solutions. The effects of flow rates on the structures of as-synthesized ZnO were investigated. Transmission electron microscope (TEM) images demonstrated that longer and thicker ZnO nanorods could be synthesized with slow flow rate. Photoluminescence (PL) spectra with UV emissions were also observed. For ZnO nanorods synthesized with slow flow rate, its corresponding UV emission would shift to low energy.

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1. Introduction

Zinc oxide is one of the most promising electronic and photonic materials because of its wide direct band gap and relatively large exciton binding energy. ZnO nanomaterials with one-dimensional (1D) structures, such as nanorods or nanowires are especially attractive due to their tunable electronic and optoelectronic properties, and potential application in devices at nano-scale. ZnO nanorods or nanowires are expected to further lower the lasing threshold because of quantum effects that result in the enhancement of density of states near the band edges and radiative recombination due to carrier confinement [1–5]. Recently, a variety of methods, including radio frequency sputtering, metal-organic chemical vapor deposition, and catalyst-assisted vapor phase transport techniques, have been employed to fabricate ZnO nanorods (or nanowires) on various substrates [6–8]. On the other hand, wet-chemical approaches have also been widely used for the fabrication of ZnO nanorods because of their simplicity and its promising option for large-scale production [9–11].

In recent years, micro-reactor technology has gained a great deal of attention [12–16]. In micro reactors, the reaction is confined into a very small area (down to micro-scale); heat-transfer and mass transport are much more efficient. Such high heat and

mass exchanging efficiency allows rapid heating and cooling with precisely defined residence times, which make it possible to decrease the mixing and diffusion time with reduced influence of mass transport on the speed of a reaction [14]. Thereby, the reaction is precisely controllable using a micro-reactor procedure, for example, by using micro flow reactor, it is simple to precisely control synthesis time just by changing the flow rates of pumped solution. In addition, continuous synthesis is a fundamental advantage of micro reactor technology.

In this paper, using polytetrafluoroethylene (PTFE) capillary tube as micro flow reactor, a micro fluidic technology was presented to synthesize ZnO nanorods continuously. At the beginning of this paper, a modified chemical method was adopted to controllably synthesize ZnO nanorods in the PTFE micro flow reactor by adjusting the flow rates of the pumped stock solutions; then the characteristics of as-synthesized ZnO nanorods were discussed.

2. Experimental section

In this paper, two chemical methods [17,18] were modified to make it possible to synthesize ZnO nanorods with a micro reactor technology. Typically, as shown at the bottom of Fig. 1, 50 ml 0.5 M 1-octanol solution of tetramethylammonium hydroxide mixed with 2 ml ethylenediamine (EDA) and 50 ml 0.01 M dimethyl sulfoxide (DMSO) solution of Zn(OAc)₂·2H₂O dissolved with 0.4 g ethyltrimethylammonium bromide (CTAB)

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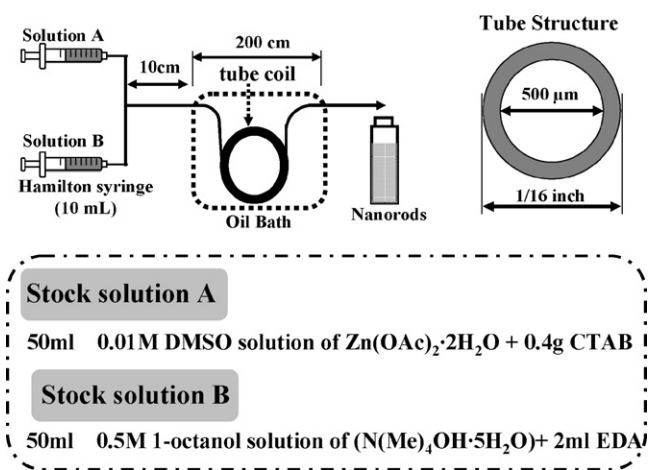


Fig. 1. Schematic diagram of the experiment set-up for the synthesis of ZnO nanorods in a PTFE capillary micro flow reactor.

were prepared as stock solutions. ZnO nanorods were synthesized by injecting equal volume of two stock solutions into capillary tubes by syringe pumps at the same time. The capillary tube was coiled and immersed into oil bath with different temperatures, as shown as the experiment set-up in Fig. 1. In this experiment, PTFE capillary tube with interior diameter of $500 \mu\text{m}$ was used, the length in heating zone was kept as 2 m. By changing the flow rate of pumped solutions, the reaction time in the heating zone can be adjusted. For example, when stock solutions were injected by two 10 ml syringes with flow rates

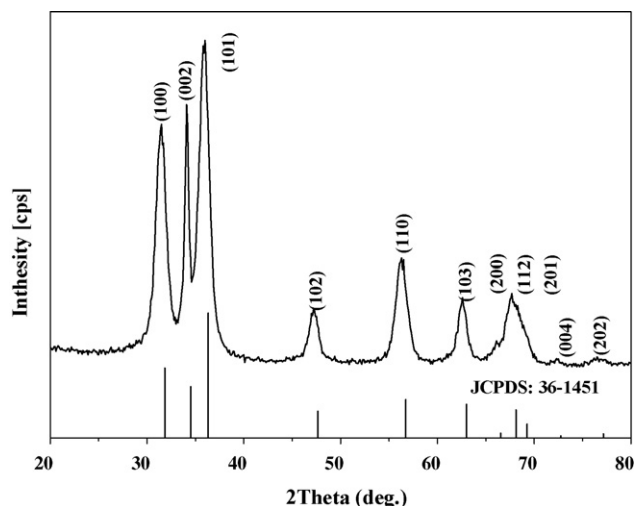


Fig. 2. The X-ray diffraction patterns of the ZnO nanorods synthesized in the micro capillary tube with a flow rate of $19.63 \mu\text{l}/\text{min}$ at 120°C .

of 196.3, 39.26 and $19.63 \mu\text{l}/\text{min}$, their corresponding reaction times were 1, 5 and 10 min, respectively. In this experiment, EDA was used to enhance the heterogeneous growth of ZnO nanorods; CTAB was used to passivate the surface and make ZnO nanorods more dispersed. The PTFE capillary tubes were purchased from GL Sciences Inc. With 10 ml Hamilton gastight syringes and IC 3210 micro syringe pump from KD Scientific, stock solutions were pumped into PTFE capillary tubes. All the

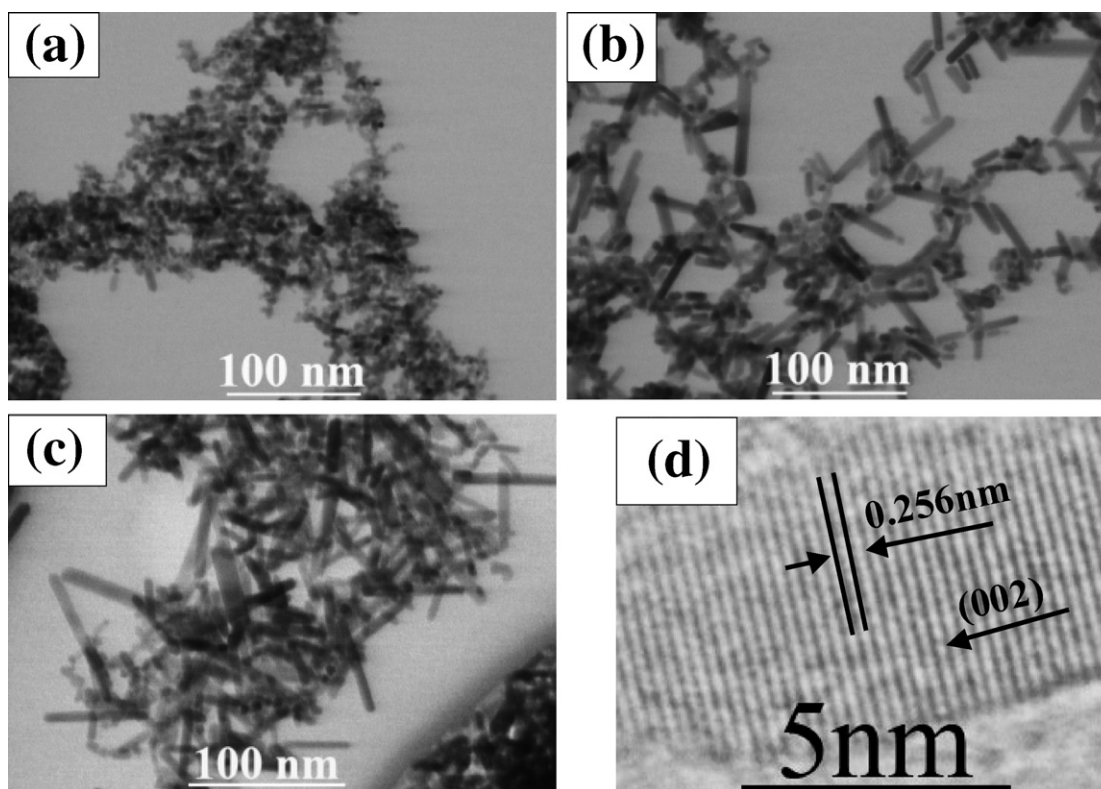


Fig. 3. TEM images of the ZnO nanorods synthesized in micro fluidic reactor with different flow rates: (a) $196.3 \mu\text{l}/\text{min}$, (b) $39.26 \mu\text{l}/\text{min}$ and (c) $19.63 \mu\text{l}/\text{min}$ at 120°C , and (d) the HRTEM image of the sample shown in (c).

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