Solid State Sciences 11 (2009) 439–443

Contents lists available at ScienceDirect

Solid State Sciences

journal homepage: www.elsevier.com/locate/ssscie

Low temperature synthesis and characterization of rosette-like nanostructures of ZnO using solution process

Rizwan Wahab [a], S.G. Ansari [a], Hyung-Kee Seo [a,b], Young Soon Kim [a], Eun-Kyung Suh [b], Hyung-Shik Shin [a,*]

[a] Thin Film Technology Laboratory, School of Chemical Engineering, Chonbuk National University, 664-14, Duckjin-dong 1ga, Chonju-561756, Republic of Korea
[b] Department of Semiconductor Science and Technology, Semiconductor Physics Research Center, Chonbuk National University, Chonju-561756, Republic of Korea

ARTICLE INFO

Article history:
Received 25 March 2008
Received in revised form 26 June 2008
Accepted 9 July 2008
Available online 25 July 2008

Keywords:
ZnO
Rosette-like structures
Solution process
Low temperature

ABSTRACT

Rosette-like structures of ZnO were synthesized at low temperature (60 °C) using solution process over 20 min of time. Hydroxylamine hydrochloride was used as capping agent with zinc nitrate hexahydrate and sodium hydroxide. Transition from triangular shaped plate like particles to rosette-like structure and to individual nanorods is observed with increasing refluxing temperature. Single-crystalline nature with wurtzite hexagonal phase is confirmed from transmission electron microscopic observations. Photoelectron spectroscopic measurement presented spectra close to the standard bulk ZnO, with an O 1s peak composed of surface adsorbed O–H group, O^{2-} in the oxygen vacancies on ZnO structure and ZnO.
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1. Introduction

Controlling the shape and size of the synthesized materials according to ones' desire is a key challenge in current nanoscience research. Intensified efforts are being put to control the size, shape and dimensions especially of nanocrystalline materials to explore the novel properties and applications [1–3].

Variety of ZnO nanostructures such as nanowires, nanobelts, nanobridges, nanonails, nanoribbons, nanorods, nanotubes, and whiskers are reported in the literature [4–8]. It is noticed that morphology of the synthesized nanostructures strongly depends on the synthesis route and structure directing (capping) agent. Literature survey indicates the need to investigate the effect of directing agents on the structure and morphology of these nanostructures [9,10].

In addition to the reported morphologies, reports on rosette/sisal-like morphologies are few. For example, Geng et al. synthesized sisal-like zinc oxide nanostructures on silicon substrate in a quartz tube furnace using Ar gas and zinc acetylacetonate as a source material for zinc in 2 h at 400–500 °C [11]. In another report, Xu et al. synthesized sisal-like zinc oxide nanocone assemblies on zinc foil using zinc sulfate, NaOH and ammonium fluoride by the hydrothermal method at 60 °C in 12–15 h [12]. Similarly, Yan and Xue et al. presented the synthesis of interesting hexagonal ZnO tapered-tubes, hallow $ZnCO_3$ microspheres and aligned zinc oxide nanorods on the Zn foil by the electrodeposition, solution and hydrothermal route between 45 and 120 °C in 16 h. Including these they also showed the use of simple spontaneous ion-replacement reactions for the various types of metal oxides (ZnO, CuO, CdO, CaO and Al_2O_3) at micro- to nanoscale level [13–16].

Here we report synthesis and characterization of ZnO, with morphology similar to rosette-like structure composed of hexagonal nanorods, using zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$) and NaOH. Hydroxylamine hydrochloride is used as a capping agent. To the best of our knowledge, use of hydroxylamine hydrochloride as a capping molecule for the synthesis of rosette-like structures is not reported yet. A possible growth mechanism is proposed for the formation of such structure.

2. Experimental

All the chemicals were purchased from Aldrich Chemical Corporation and used without further purification. In a typical synthesis process, ~4.46 g of zinc nitrate hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$) and 0.347 g of hydroxylamine hydrochloride (0.1 M) were dissolved in 50 ml of de-ionized (DI) water, and stirred. No precipitation was observed when these two compounds were mixed. Sodium hydroxide solution (3 M, 6 g NaOH in 50 ml DI water) was then added to the stirring solution. White precipitates were observed in few seconds but stirring was continued for 20 min. The resultant white precipitates were neutralized by washing several

* Corresponding author. Fax: +82 63 270 4465.
E-mail address: hsshin@chonbuk.ac.kr (H.-S. Shin).

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doi: 10.1016/j.solidstatesciences.2008.07.009