Materials Science in Semiconductor Processing 27 (2014) 12–18

Contents lists available at ScienceDirect

Materials Science in Semiconductor Processing

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

Facile synthesis and transformation of Te nanorods to CdTe nanoparticles

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ARTICLE INFO

Available online 25 June 2014

keywords:
CdTe nanoparticles
Tellurium nanorods
Thioglycolic acid

ABSTRACT

Luminescent cadmium telluride nanoparticles were synthesized from the reaction intermediate tellurium nanorods as tellurium source at 100 °C through colloidal approach. Oxyanion source of tellurium, (i.e.) potassium tellurite (K_2TeO_3) was used as precursor to synthesize the tellurium nanorods under the low temperature process without any surfactant in various volumes of water. Thioglycolic acid (TGA) was employed as capping agent for the synthesis of CdTe nanoparticles. Absorption and emission spectra of the prepared nanoparticles clearly indicate the size dependent nature of the particles. SEM and EDX analyses of the nanorods reveal the shape of the synthesized nanorods and the presence of elemental tellurium without any impurities. XRD analysis of the prepared samples confirms the existence of trigonal phase of tellurium nanorods (t-Te) and the formation of CdTe particles with cubic zincblende structure. The size distribution of the prepared CdTe nanoparticles was analyzed through TEM analysis. It was found that considerable influence of solvent on morphology of the synthesized tellurium nanorods. The synthesis mechanism of one dimensional structure as seeds for zero dimensional nanoparticles was analyzed.

1. Introduction

Fluorescent semiconductor nanoparticles are widely used in many fields such as optoelectronics, bio-imaging, finger print detection and LED device applications due to their size tunable nature [1,2]. Enhanced light harvesting of semiconductor nanoparticles sensitized photo-electrodes was also widely studied for solar cells [3,4]. The quantum confinement effect makes these nanoparticles towards engineering for such applications. Cadmium telluride (CdTe) because of the direct bandgap has attracted considerable attention in many device applications. Numerous attempts were made to synthesize CdTe nanoparticles with a suitable method. After the successful synthesis of semiconductor nanoparticles in an aqueous system, lot of interest was focussed on the synthesis of CdTe nanoparticles in aqueous medium due to the usage of simple precursors like K_2TeO_3 [5], and simple experimental arrangements. Moreover, the aqueous based approach of synthesizing CdTe nanocrystals was best proven for the active layer formation in hybrid solar cells with the water soluble semiconducting polymers [6]. The optical properties of the aqueous synthesized CdTe nanoparticles have shown mostly similar kind of phenomena as like organometallic based one. The enhancement of the photoluminescence was observed when tellurium nanorods are used instead of bulk tellurium powder as source in the case of CdTe nanoparticles synthesized through a hydrothermal method at higher temperature [7]. The nano-tellurium has the ability to form Te^{2-} reactive species much easier than the bulk Te powder in alkaline solution. Moreover, Tellurium, a p-type narrow bandgap semiconductor (E_g=0.35 eV), is

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http://dx.doi.org/10.1016/j.mssp.2014.06.008
1369-8001/

used for many applications including thermoelectrics, nonlinear infra red optics, photodetectors, photovoltaics, gas-sensing materials, data-storage devices and bio-labels [8]. Further, nano-tellurium is used as attractive template for the synthesis of metal chalcogenide nanoparticles [9–11]. Hence, synthesis of tellurium nanostructures received considerable interest and was reported using many methods including solvothermal, ultrasonic induced growth, and surfactant assisted method, hydrothermal methods with Te powder or TeO_2 as source [12–15]. Mo et al. [16] have reported the insitu synthesis and formation of Te nanotubes and nanobelts through Na_2TeO_3 under alkaline conditions at high temperature. Further, since the alkali metal tellurites (X_2TeO_3 where X=K, Na) are water soluble in nature, the formation of tellurium nanostructures in water with the surfactant has also been widely studied [17–19]. The interesting formation of In_2Te_3 nanosheets from tellurium nanorods in the presence of alkaline solvent was also reported recently [20]. Synthesizing trigonal nanotellurium structures using these methods are the desired one for optoelectronic device applications. Synthesis of one dimensional nanostructure was carried out widely using wet chemical methods. Recently, the aqueous synthesis of ZnSe nanoparticles through the intermediate nanoselenium has been reported [21]. Present work, highlights the aqueous synthesis of luminescent CdTe nanoparticles capped by a short chain thiol ligand (thioglycolic acid) using the tellurium nanorods as tellurium source. The tellurium nanorods were obtained by reducing the oxyanion source of tellurium, (i.e.) potassium tellurite (K_2TeO_3) in aqueous medium at low temperature using a strong reducing agent in the absence of surfactant. The proposed synthesis process is simple and reproducible one compared with other methods.

2. Experimental details

2.1. Synthesis of tellurium nanorods

Precursors in the form of cadmium chloride ($CdCl_2$) SRL (97%), potassium tellurite (K_2TeO_3) CDH (99%), thioglycolic acid ($C_2H_4O_2S$) SPECTROCHEM (95%), sodium borohydride ($NaBH_4$) MERCK (95%), sodium hydroxide (NaOH) SRL (98%), and trisodium citrate dihydrate ($C_6H_9Na_3O_9$) SRL (99%), were used for the synthesis of tellurium nanorods as well as CdTe nanoparticles.

Synthesis of tellurium nanostructures was performed using the method similar to the Gautham and Rao [12] without utilizing any surfactants. Briefly, potassium tellurite (1 mM) was taken in a three necked flask with 100 ml of water, then, excessive amount of the sodium borohydride (ratio1:10) was added into the solution. The mixture was immediately turned to black and the resultant solution was refluxed strongly at the temperature of 60 °C under the nitrogen atmosphere until the solution colour turned into light pink. Then, the solution was kept under dark for two days without any disturbance. The resultant black precipitate was filtered off and washed with water three times. The final precipitate was dried in vacuum. The experiment was repeated for the 50 ml, 25 ml volume of the water by keeping the ratio of potassium tellurite and sodium borohydride as constant.

2.2. Synthesis of CdTe nanoparticles

Cadmium chloride (1 mM) was dissolved in 100 ml water and thioglycolic acid (5 mM) was added in drop wise and the resultant mixture was stirred. The resultant turbidity appearance of the mixture indicates the formation of the cadmium-TGA complex which turned into clear transparent one, when pH was maintained as 10.5 using 1 M NaOH solution. Then tri sodium citrate dihydrate was added with this mixture to avoid the formation of cadmium tellurite ($CdTeO_3$) in the solution [22]. The entire solution was purged with nitrogen in a three necked flask. Then the tellurium nanorods were added simultaneously as tellurium source in the presence of excessive sodium borohydride. The molar ratio of Cd^{2+}: Te^{2-}: TGA for this entire synthesis process is 1:0.5:5. Now, the mixture was strongly heated to 100 °C. The colour of the initial solution was observed as yellow and then converted to orange after prolonged refluxing. Aliquot samples were taken at different time intervals and the size dependency was checked through absorption and emission spectra. The synthesized particles were precipitated by adding the non-solvent iso-propanol and centrifuged. The resultant precipitate was washed twice using ethanol to remove the excess of surface ligand and by-products and then dried in vacuum for further analysis.

2.3. Characterization

Absorption spectra were analyzed using a ELICO SL-159 UV–visible conventional spectrophotometer in the range of 200–800 nm. The samples were taken as liquid in quartz cuvette and diluted using water for the analysis. Fluorescence analyses were carried out for the samples using a JASCO FP-6300 spectrofluorometer at the excitation wavelength 420 nm. The excitation was done using xenon lamp source. The samples were used as liquid form in quartz cuvette. SEM images were recorded using the Carl Zeiss MA15/EVO 18 Scanning Electron Microscope. In order to avoid the charging effect, gold/palladium alloy was coated on samples for SEM analysis. XRD patterns were taken using a powder X-ray diffractometer (SEI FERT)

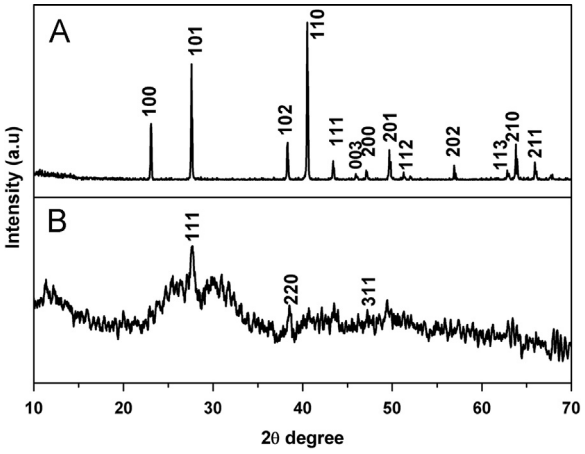

Fig. 1. XRD pattern of (a) Te nanorods and (b) CdTe nanoparticles.