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To cite this article: Sanjeev Kumar and Satinder K Sharma 2010 *Phys. Scr.* **82** 025801

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Low-temperature synthesis and characterization of cadmium sulfide nanowires grown using simple chemical reaction through the pores in an alumina template

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Received 21 January 2010

Accepted for publication 17 June 2010

Published 13 July 2010

Online at stacks.iop.org/PhysScr/82/025801

Abstract

The synthesis of cadmium sulfide (CdS) nanowires using a simple chemical reaction (ion exchange) method through the pores in an alumina template is reported. The chemical reaction takes place inside the pores and the reaction yield is deposited in the pores. The morphological, structural and optical characterization of CdS nanowires is also reported. The Raman spectrum shows that CdS nanowires have good crystalline nature. The value of the optical bandgap observed is 2.7 eV for CdS nanowires. Room temperature photoluminescence measurements exhibit an emission peak at about 507 nm.

PACS numbers: 81.07.Gf, 78.67.Uh, 63.22.Gh

(Some figures in this article are in colour only in the electronic version.)

1. Introduction

Nanostructures, which exhibit unique properties that are very different from those of bulk, have been the subject of extensive investigations over the past two decades. One-dimensional nanomaterials, such as nanotubes, nanorods and nanowires, are attractive not only for fundamental physics but also for their wide-ranging potential applications in nanodevices [1–4]. Cadmium sulfide (CdS) has received great attention as a potential semiconductor due to its direct bandgap in the visible region [5, 6]. Different kinds of CdS nanostructures such as nanoparticles, nanotubes, nanowires, etc have been widely investigated, such as nonlinear optical devices, photovoltaic cells, etc [7–11]. Various techniques such as chemical vapor deposition, molecular beam epitaxy, template synthesis, the chemical solution route, etc have been used for the generation of CdS nanostructures [12–22].

In this paper, we report on the generation of CdS nanowires using a simple chemical reaction (ion exchange)

method through the pores in an alumina template. The chemical reaction takes place inside the pores and the reaction yield is deposited in the pores. The morphological, structural and optical characterization of CdS nanowires is also reported.

2. Experimental details

An alumina membrane (Whatman) was used for the fabrication of CdS nanowires. The pore size was 100 nm. All the reagents we used for the chemical solution were of pure analytical grade. Triply distilled water was used for the preparation of solutions.

A two-compartment cell was used for the generation of CdS nanowires. An alumina template was fixed between the two compartments to act as a semi-permeable membrane. In the first compartment, an aqueous cadmium acetate solution (0.2 M with some drops of NH₄OH) was added and allowed to diffuse through the membrane for 30 s prior to the introduction

of the second reagent, sodium sulfide (0.2 M), in the second compartment. Both solutions diffused toward each other through the pores of the membrane and reacted to yield the CdS. The synthesis process was continued for 60 min at room temperature.

For the atomic force microscopy (AFM) characterization, a drop of NaOH solution was put on the sample and remained on it for 10 min. A thin layer of anodic alumina was dissolved. Then the sample was washed with distilled water and ethylene alcohol several times. The NDMTT basic model (AFM) was used for surface characterization.

For scanning electron microscopy (SEM) characterization, firstly the prepared sample of CdS nanowires embedded in the alumina membranes was put in an NaOH solution to dissolve the alumina membrane. The concentration of the NaOH solution was taken as very low (0.5 M) so that it should not damage the CdS nanowires. It takes a long time to completely remove the host membrane. After that, the nanowires were washed with distilled water and ethylene alcohol several times. The dried sample of CdS nanowires was coated with gold–palladium alloy using a Jeol JFC sputter coater and then examined under a Jeol JSM 6100 scanning electron microscope. The crystallographic studies of CdS nanowires embedded in an alumina template were carried out using a Panalytical X'pert x-ray diffractometer in the 2θ range of $20\text{--}60^\circ$ using $\text{CuK}\alpha$ radiation. Optical absorption spectra were recorded within the range $200\text{--}800\text{ nm}$ using a Shimadzu 2500 UV–Vis spectrophotometer. Photoluminescence (PL) measurements were recorded on a FluoroMax-3 (Jobin-Yvon, Edison, NJ, USA) equipped with a photomultiplier tube and a xenon lamp.

The measurement of disorderliness of CdS nanowires was carried out by a WiTec CRM 2000 Raman spectrometer coupled with a high-resolution confocal optical microscope. With this combination, it is possible not only to obtain a Raman spectrum of a sample but also to combine its chemical information with a lateral resolution in the sub-micrometer regime. In Raman studies, excitation was provided by the 514.5 nm line of an Ar^+ laser. The microscope objective we used was Nikon 100X, with a working distance of 0.26 unit. Thus, the laser beam diameter at the focus is approximately in the submicron regime.

3. Results and discussion

Figure 1(a) shows a three-dimensional view of the AFM topography image of CdS nanowires. It reveals the surface morphology of CdS nanowires grown within an anodic alumina membrane template. The diameters of the tips of the nanowires were found to be similar to the pore size. This is attributed to the overfilling of alumina pores. The SEM micrograph (figure 1(b)) presents the surface morphology of complete CdS nanowires. It is clearly seen from the micrograph that the diameter of the wires (100 nm) is similar to the pore size.

The x-ray diffractogram (figure 2) depicts the zinc blende nature of CdS nanowires (JCPDS file 10-454). The dominant peaks were observed at 26.6° , 44.3° and 52.3° in the x-ray diffractogram [15]. The diffraction peaks mainly correspond to the (100), (220) and (311) planes of cubic CdS. Due to

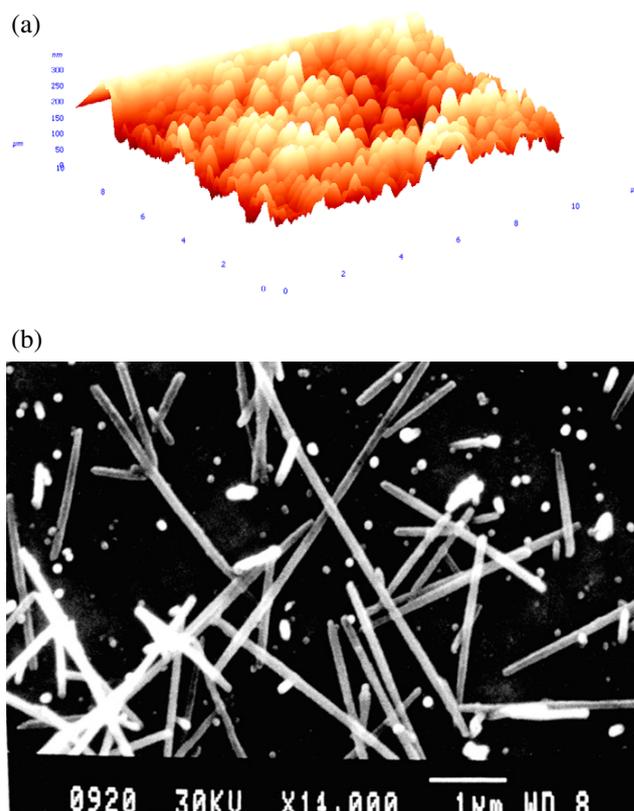


Figure 1. (a) Three-dimensional view of AFM topographic image of CdS nanowires. (b) SEM micrograph of CdS nanowires.

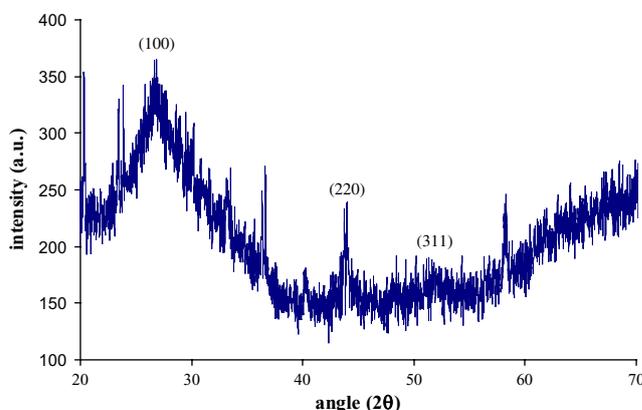


Figure 2. X-ray diffractogram of CdS nanowires.

noise, some very sharp shooting peaks are also observed, having no relevance. The broadening of x-ray diffraction (XRD) peaks implies that the CdS nanowires thus synthesized are possibly not single crystalline but are assembled from nanocrystallites.

Crystallite size can be estimated by using the Debye–Scherrer formula

$$D = 0.93\lambda / \beta \cos \theta,$$

where D is the diameter of the crystallite, $\lambda = 1.518\text{ \AA}$ ($\text{CuK}\alpha$ radiation wavelength), β is full-width at half-maxima and θ is Bragg's angle. By substituting these values, the size of the nanocrystallite was found to be about 3.4 nm.

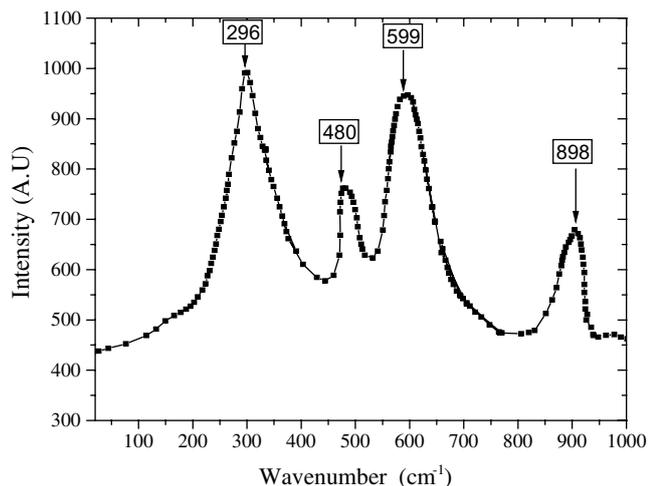


Figure 3. Raman spectrum of CdS nanowires embedded in an alumina membrane.

Raman scattering is very sensitive to the microstructure of nanocrystalline materials and can be used to probe the CdS nanowires. The Raman spectrum of CdS nanowires was recorded at 25 °C using a 514.5 nm Ar⁺ laser beam for excitation. Figure 3 shows the Raman spectra for the CdS nanowires with evidence of typical Raman peaks longitudinal mode in the spectrum. However, the low-frequency E_2 mode at 43 cm⁻¹ is not observed in this spectrum at 514.5 nm excitation, which supports our previous XRD results that the grown CdS nanowires are zincblende in nature. The fundamental frequency modes of Raman peaks assigned at about the longitudinal optical (LO) mode at 296 cm⁻¹ and the first overtone at 599 cm⁻¹ (2LO) were observed. The third harmonic at 898 cm⁻¹ corresponds to the first-, second- and third-order LO phonon modes of CdS, respectively [23]. These findings are in agreement with those of Shiang *et al* [24] but are in contradiction to an earlier study on CdS nanowires reporting a large red shift of the frequency with decrease in size [26]. However, the Raman shifts in the CdS spectrum reported in figure 3 are similar to that of the bulk CdS spectrum [25–27].

Along with these significant peaks in the CdS nanowires spectrum, an additional weaker peak of approximately 147 CCD counts, relative to other peaks of the CdS nanowires spectrum at 480 cm⁻¹, was also detected with 514.5 nm excitation wavelength. This weaker peak probably originated from surface defects, impurities present in the CdS nanowires sample, specifically the unpassivated nature of the surface as well as bulk crystallite defects, originated during the growth process [25, 28]. Additionally, this peak could be because of the CdS nanowires being not in resonance with the laser line (514.5 nm). These results show that the CdS nanowires grown within the anodic alumina membrane have good crystallinity.

The UV–Vis absorption spectrum of CdS nanowires is shown in figure 4. The absorption peak is located around 460 nm. The value of the bandgap evaluated using the UV absorption spectrum is 2.7 eV (inset, figure 4), which is higher than that of bulk CdS (2.42 eV). Blue shift of the order of 0.28 eV is observed in 100 nm CdS nanowires. Figure 5 presents the PL spectrum of CdS nanowires observed at room temperature with 325 nm excitation wavelength.

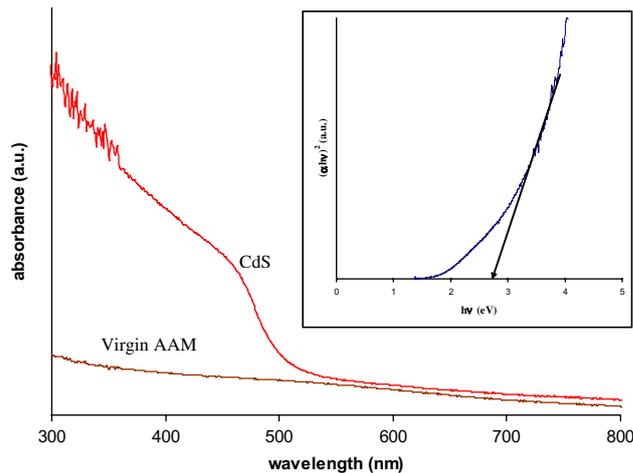


Figure 4. UV–Vis absorption spectrum of CdS nanowires. The inset shows the plot to determine the direct bandgap of CdS nanowires.

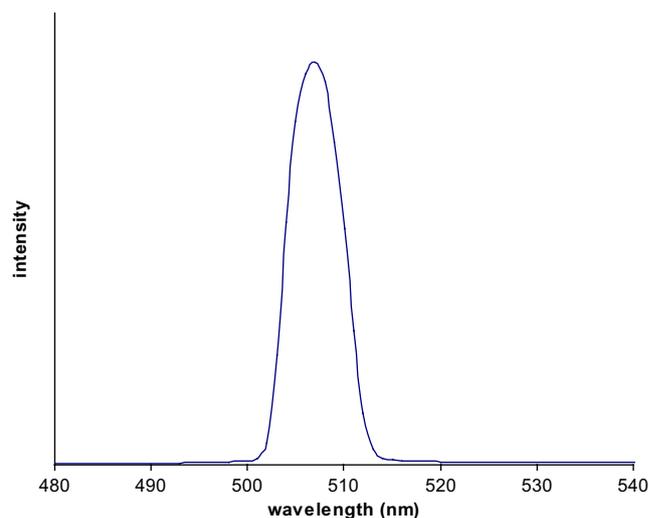


Figure 5. PL spectrum of CdS nanowires.

The PL spectrum reveals one emission peak, located at about 507 nm. As the PL peak energy observed (2.45 eV) at room temperature is less than the bandgap energy calculated (2.7 eV), the luminescence may be due to transitions involving defect states [13]. The source of defect states may be associated with sulfur vacancies, intrinsic defects or impurities [15]. These defect states may originate magnetism in CdS nanowires. CdS acts as a diamagnetic in bulk form but in the form of nanomaterials it acts as a ferromagnetic. Hence, the CdS nanowires can be used as dilute magnetic semiconductors for spintronics applications [29].

4. Conclusions

The simple chemical reaction technique is a useful technique for the preparation of CdS nanowires. XRD shows the zincblende (cubic) nature of CdS nanowires. For the observation of quantum size effect, the diameter of nanowires should be very small (comparable to the Bohr exciton radius). Nanowires with 100 nm diameter show the bulk CdS behavior. However, a blue shift of the order of 0.28 eV is observed

in 100 nm CdS nanowires. The PL spectrum shows a strong emission peak at about 507 nm due to defect states in nanowires. The Raman spectrum shows that the CdS nanowires have good crystalline nature.

Acknowledgments

We thank Dr Shyam Kumar and Dr Sanjeev Aggarwal, Physics Department, Kurukshetra University, Kurukshetra for providing the facilities of UV–Vis and photoluminescence spectrophotometers and Dr Mukesh Kumar, Electronic Science Department, Kurukshetra University, Kurukshetra for providing the AFM facility. We also thank Dr D Mehta, Physics Department, Punjab University, Chandigarh for providing the EDXRF facility and Mr Jagtar Singh, CIL, Punjab University, Chandigarh for providing the XRD facility.

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