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Effect of Annealing on the Crystal Structure of Template Synthesised CdS Nanowires

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ABSTRACT: Design, fabrication and applications of nanostructures and nanomaterials fall under the realm of nanotechnology and it is strong future requirement of the society. Synthesis and characterization of one-dimensional nanostructures have attracted attention primarily due to their potential myriad applications in different fields including electronics, magnetic, and optical devices. Among the various techniques used in the fabrication of one-dimensional structure, the template synthesis is an elegant, versatile and economic method. Highly ordered CdS nanowires each having 200 nm diameters were synthesised into the pores of anodic alumina membrane (AAM) template by using direct chemical deposition method. The nanowires were deposited using cadmium sulphate as Cd^{2+} ion source and sodium sulphide S^{2-} ion source . The nanowires were characterized by Scanning Electron Microscope (SEM) and x-ray diffraction (XRD) to see surface morphology, and crystal structure respectively. The as- deposited CdS nanowires embedded in anodic alumina membrane (AAM) are having cubic structure. The embedded nanowires were annealed in air at 673 K for 5 h and effect of annealing on crystal structure studied. It has been found that the crystal structure gets changed from cubic phase to hexagonal phase on annealing.

Keywords*:* CdS Nanowires, Anodic Alumina Membrane, X-ray diffraction, Scanning Electron Microscope

INTRODUCTION

One of the critical challenges faced currently by researchers in the study of nanostructures and nanomaterials is the availability of experimental skills, equipment and instrumentation systems for handling, measurement and manipulation of materials at the nanometer scale. Characterization and manipulation of individual nanostructure require atomic level resolution in addition to extreme sensitivity and accuracy of the instrument used. The new phenomenon, physical properties and short range forces play significant role on the nanometer scale, which is not otherwise noticeable at macroscopic level [1–6]. Among the various techniques used in the fabrication of one-dimensional structures, the template synthesis is an elegant, versatile and economic method [5–16]. CdS is an attractive II–IV group semiconductor materials. Because of a wide band gap, CdS is used in various applications such as optoelectronics devices [17], solar cells [18], thin film field effect transistors [19] and so on. Template synthesis of CdS has been carried out by alternating current as well as direct current elecro-depopsition into the pores of anodic alumina membrane (AAM), but high temperature and expensive organic reagent (dimethyl sulfoxide) requirements are necessary to dissolve S and Se in these methods [20, 21]. Room temperature electrodeposition, without using expensive organic reagent, also has been carried out for CdS nanowires [22], but highly precise conditions like concentration, pH and cathodic potential are also required in order to have good stochiometry ratios. We report here a non-galvanic method (chemical method) at room temperature (25 °C) for the preparation of an ordered array of CdS nanowires using AAM as template which is sandwiched in a two-compartment cell. CdS nanowires are prepared by using $CdSO_4$ solution (complexed with ammonia) is used as Cd^{2+} source and Na_2S is used as S^{2-} source. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) studies were carried for morphological and structural characterizations, respectively.

EXPERIMENTAL PROCEDURE

All the chemical reagents used were RA grade and without further purification. $CdSO_4$, Na_2S and ammonia solution were from s.d.fine-Chem Ltd. Mumbai, India, and all solutions were prepared in de-ionized water. Anodic alumina membrane (AAM), anodisc-21 (Whatman, UK) with pore diameter 200 nm were used as templates. For the deposition of CdS nanowires, 200 nm AAM was sandwiched in a two-compartment cell (Figure. 1). A 50 mM solution of $CdSO_4$ complexed with ammonia was filled in one compartment and a 50 mM solution of Na_2S was filled in other compartment, and the cell was left for 12 hr so that CdS nanowires were formed inside the pores of AAM via precipitation reaction.

The CdS nanowires embedded in AAM template were placed on glass plate and crystallographic studies were carried out using a Philips PW 1710 X-ray diffractometer in 2θ range from 10° to 70° using Cu Kα radiation. Thereafter, the annealing of as deposited CdS nanowires were carried out at 400 ºC in vacuum for 5 hr and again the crystalographic studies were carried out. The morphology of CdS nanowires was examined by scanning electron microscope, by first liberating them from the host matrix by dissolving AAM template in a 1M NaOH solution at 25 °C for 1 hr, followed by subsequent washing. The sample was cleaned and dried and mounted on specially designed aluminium stubs with the help of the adhesive tape. The thin gold coating of sample containing nanowires was done w using JEOL, FINE SPUTTER JFC-1100 sputter coater and viewed under JEOL, JSM 6100 scanning electrom microscope.

RESULTS AND DISCUSSIONS

Growth mechanism of CdS nanowires can be explained as follow,

In one of the compartments of the cell, the cationic precursor solution of $[Cd(NH_3)_4]^{2+}$ complex release Cd^{2+} ions as:

$$[Cd(NH_3)_4]^{2+} \rightarrow Cd^{2+} + 4\,NH_3$$

In the other compartment of the cell, the anionic precursor solution of Na_2S undergoes hydrolysis to give S^{2-} ions as:

$$Na_2S \rightarrow 2\,Na^{+} + S^{2-}$$

In the pores of AAM, Cd^{2+} combine with S^{2-} to give CdS precipitate as:

$$Cd^{2+} + S^{2-} \rightarrow CdS$$

When the cell is left for 12 hr, the above process continues till the pores are completely filled with the CdS. This results into the formation of CdS nanowires.

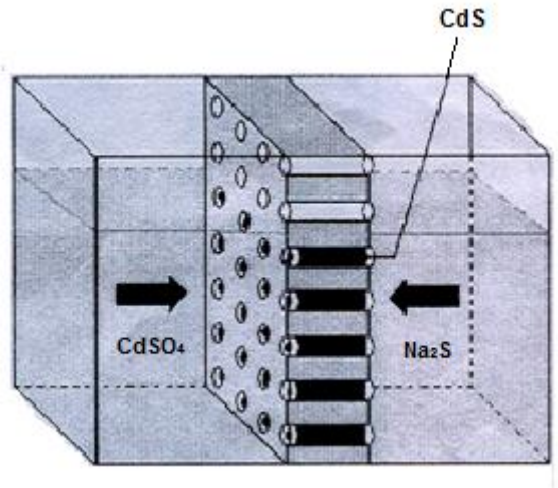

Figure 1. A two-compartment cell with AAM sandwiched between the two compartments.

Figure 2, showing the XRD of CdS with broadened peaks, indicates that nanowires are composite of nanocrysallites/nanoparticles with size less than 3 nm [23,24]. The diffraction peaks of CdS were observed at 2θ = 25.15° and 2θ = 42.45° corresponding to (111) and (220) planes of cubic CdS crystalline structure [25, 26]. Figure 3 shows the XRD of annealed CdS nanowirs. It can be noted that the peaks in XRD pattern have become sharp and also the crystal structure get changed from cubic phase to hexagonal phase due to annealing effect[27]. Also the size of nano-crystallites has been increased due annealing [28].

Figure 4 shows SEM image of CdS nanowires. It can be seen that diameter of nanowires is about 200 nm that closely corresponds to the diameter of pores of the template used and also all the CdS nanowires have uniform parallel orientation, diameter and direction of growth which is due to the ordered pores in template

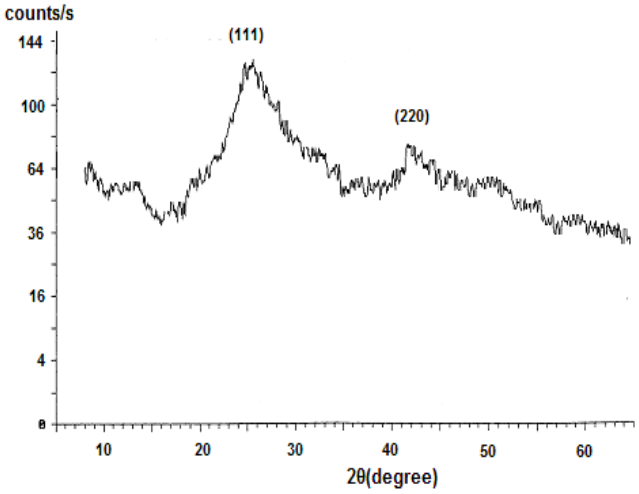

Figure 2. XRD spectrum of as-deposited CdS nanowires.

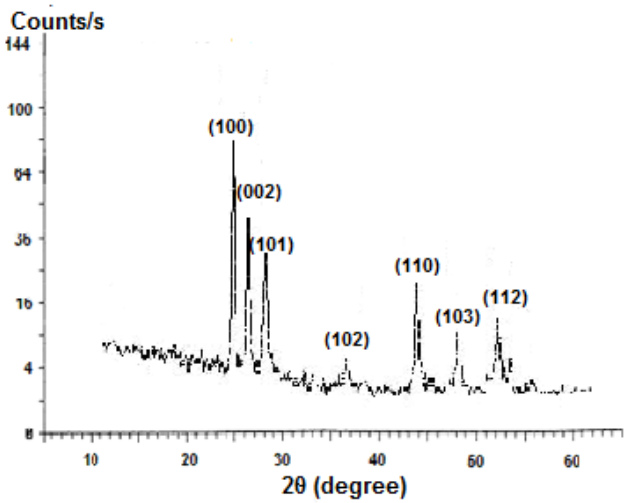

Figure 3. XRD spectrum of annealed CdS nanowire array.

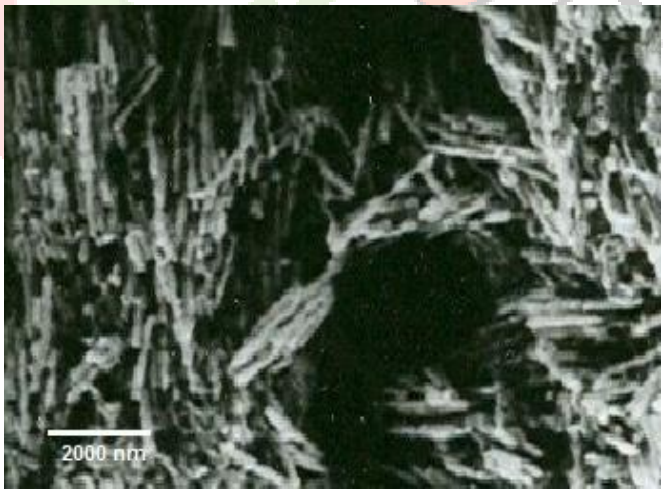

Figure 4. SEM image of CdS nanowires synthesized into AAM with pore diameter 200 nm (length of bar is 2μm).

CONCLUSION

Highly ordered CdS nanowires composed of nanocrystallites are template synthesized into the pores of AAM using non-galvanic (chemical) method at room temperature. The size of nano-crystallites get increased due annealing of CdS nanowires. The crystal structure get changed from cubic phase to hexagonal phase due to annealing effect. The SEM analysis shows that nanowires are highly ordered and uniform in diameter. The mechanism of the grown nanowires is also demonstrated. This method can be possibly used in the synthesis of other chalcogenides also.

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