Research Article

Synthesis and Characterization of Selenium Nanowires

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Selenium nanowires have been grown by chemical method. The effect of annealing on the properties of the prepared selenium nanowires has been studied. The X-ray diffraction studies indicated the formation of selenium with trigonal phase, and no secondary phase was observed. The composition analysis results show that selenium is present in the sample. HRTEM images reveal that selenium nanowires have diameter ranging from 30–50 nm and length of 1–2 μm . The diameter and length of nanowires have been found to increase with increase in annealing temperature.

1. Introduction

One-dimensional (1D) nanostructural materials such as nanotubes, nanowires, and nanorods have recently received much attention due to their interesting properties and are potential candidates for application in different fields. The one-dimensional nanostructured selenium nanowires have been widely studied because of their functional properties in superconductivity [1], high photoconductivity, and catalytic activity. Selenium nanowires find application in the areas of photoelectric cells, xerography, light-measuring devices, and solar batteries [2]. Fan et al. [3] have synthesized Se microrods, microtubes, and dendrite type microstructure by using Se powder and NaOH as starting materials under hydrothermal condition. t-Se in the form of nanowires and nanoribbons or nanobelts have been synthesized using β -carotene [4] and cellulose [5] as reducing agent by same method at 160°C. Zhang et al. have prepared t-Se nanowire arrays by electrodeposition using anodic porous alumina templates [6]. Li and Yam's group has employed ascorbic acid as the reducing agent under the assistance of β -cyclodextrin to make selenium nanowires [7]. Chen et al. have prepared t-Se nanorods through a hydrothermal route [8]. Selenium

has a high reactivity towards a variety of chemicals, which can be explored to transform selenium element into many other important functional materials, such as PbSe, ZnSe, and CdSe [9–11]. Selenium is extensively known as an important elemental semiconductor as well as an essential element in biology and chemistry. Selenium is also one of the essential trace elements for human beings. It has been confirmed that selenium can improve the activity of the selenoenzyme, glutathione peroxidase and prevent free radicals from damaging cells and tissues in vivo. Se nanomaterials have been prepared by different workers using various techniques such as hydrothermal route, chemical vapor deposition, sonochemical approaches, refluxing processes, and cellulose-directed growth [12–15].

The size and shape of nanomaterials have a major influence on their physical and chemical properties, and the ability to control these parameters remains a great technological challenge with important implications for nanoscale science. Special attention has been directed towards the control of size and morphology of chalcogenide nanostructures. Recently, synthesis of nanowires with controlled alignment, size, shape, and compositions based on one-dimensional structure has triggered great interest due to the fact that

FIGURE 1: X-ray diffraction pattern of Se (a) as prepared, (b) 100°C, and (c) 200°C annealed samples.

these nanowires may provide opportunities to exploit new phenomena and novel properties. A large number of work about the preparation of nanowire structures have been reported. Notable examples including the nanowires of Se via a chemical precipitation method [16], Se nanowires by a hydrothermal technique [17] have also been reported. These methods, however, have several major drawbacks, including the use of toxic chemicals, extreme synthesis conditions involving high-operating temperatures and/or pressures, and highly acidic or alkaline reaction conditions. The method used in the present study is simple and is of low cost and scale-up route and less toxic precursors and solvents have been used. In this paper, we report about the preparation and characterization of selenium nanowires using chemical method.

2. Experimental

Selenium nanowires have been prepared using the required precursors by chemical method. An aqueous solution of 0.05 M of SeO_2 and starch (50 mg) were dissolved in water and stirred for about one hour at room temperature. Ascorbic acid (0.025 M) was added dropwise to the above mentioned solution. The colour of the solution changed into brick-red, indicating the formation of Se nanoparticles in the solution. The solution was stirred for 5 hours at room temperature. After 5 hours, the suspension was centrifuged and washed with water and ethanol several times. The samples were then suspended in ethanol and allowed to age for 2 hours without stirring. After centrifugation, the samples were dried at room temperature. The Se samples have been annealed at 100°C and 200°C for 1 h using a heating rate of 2°C/min.

X-ray diffraction studies have been carried out using PANalytical X-ray diffractometer, and surface morphology of the samples has been studied using scanning electron microscope (JEOL JSMS 800-V). Transmission electron

FIGURE 2: SEM image of Se (a) as prepared, (b) 100°C annealed, and (c) 200°C annealed.

microscope (HRTEM) images of the prepared Se have been recorded using a JEOL JEM2100 microscope. Compositional analysis of the samples has been studied using energy dispersive analysis of X-rays (JEOL Model JED-2300).

3. Results and Discussion

Figure 1(a) shows the X-ray diffraction patterns of as prepared Se. The diffraction peaks at 2θ (degrees) of 23.57°, 29.73°, 41.28°, 43.68°, 45.43°, 51.72°, 56.07°, 61.62°, 65.24° and 71.60° are, respectively, indexed as the (100), (101), (110), (102), (111), (201), (112), (103), (210), and (113) planes of Se. All the diffraction peaks in the 2θ range

FIGURE 3: EDAX spectra of as-prepared Se.

measured correspond to the trigonal structure of Se with lattice constants $a = 4.35 \text{ \AA}$ and $c = 4.93 \text{ \AA}$ and are in good agreement with those on the standard data card (JCPDS card No. 06-0362). Figures 1(b) and 1(c) show that the X-ray diffraction patterns of 100°C and 200°C annealed Se. The (100), (101), (110), (102), (111), (201), (112), (210), and (113) peaks of the 100°C and 200°C annealed selenium are slightly shifted when compared to the as-prepared selenium because annealed samples are under a tensile strain, and the amount of strain increases with increase in annealing temperature. The sharpness of the diffraction peaks suggests that the product is well crystallized. The crystallite size of selenium is calculated using Scherrer's equation,

$$D = \frac{K\lambda}{\beta \cos \theta}, \quad (1)$$

where D is the grain size, K is a constant taken to be 0.94, λ is the wavelength of the X-ray radiation, β is the full width at half maximum, and θ is the angle of diffraction. The crystallite size has been calculated and is found to be in the range 30–50 nm for as-prepared, 100°C , and 200°C annealed selenium, respectively.

Figure 2 shows the scanning electron microscope (SEM) image of the as-prepared Se, 100°C and 200°C annealed selenium sample. Figure 2(a) shows the initial formation of as-prepared selenium nanowire. Figure 2(b) shows the SEM image of the 100°C annealed Se nanowires. The SEM image reveals that the Se nanowires are of uniform size with a mean diameter of 30 nm. SEM image of 200°C annealed Se is shown in Figure 2(c). It can be seen that nanowires have a length of several micrometers and diameter ranging from 30 to 50 nm. It can be clearly seen that the lengths of the Se nanowires increase rapidly with increasing the annealing temperature; however, the diameters increase slowly. Therefore, it is understood that the aspect ratios and lengths of the Se nanowires can also be controlled by annealing temperature. Figure 3 shows the energy dispersive X-ray analysis (EDAX) spectra of the Se sample. In the EDAX, Se

(a)

(b)

FIGURE 4: (a) Transmission electron microscope and (b) selected area electron diffraction images of the as-prepared Se nanowires.

is the only element detected, indicating that the sample is highly pure.

Figure 4(a) shows the transmission electron microscope (TEM) image of as-prepared Se nanowires. The image shows that the nanowires are straight and uniform with an average diameter of about 30 nm. Figure 4(b) shows the selected area electron diffraction (SAED) pattern of as-prepared Se nanowires. Selected area electron diffraction image (Figure 4(b)) exhibits diffraction rings corresponding to the (100), (101), (110), (102), (111), and (201) directions of the trigonal phase of Se. The d -spacing values obtained for all the diffraction rings from the SAED pattern match very well with that of trigonal phase Se. Figure 5(a) shows the TEM image of 200°C annealed Se nanowires. The TEM image (Figure 5(a)) confirms the formation of Se nanowires with several micrometers of length and 50 nm diameter.

FIGURE 5: (a) Transmission electron microscope and (b) selected area electron diffraction images of the Se nanowires annealed at 200°C.

Figure 5(b) shows the SAED pattern of 200°C annealed Se nanowires. SAED image (Figure 5(b)) exhibits diffraction rings corresponding to the (100), (101), (110), (102), (111), (201), and (103) directions of the trigonal phase of Se. The d -spacing values corresponding to plane (100) for as-prepared and annealed (200°C) samples are found to be 3.76 Å and 3.73 Å. The d -spacing values calculated from these Figures (4(b) and 5(b)) are in close agreement with the values obtained from X-ray diffraction studies.

4. Conclusion

Selenium nanowires have been prepared by chemical method. X-ray diffraction analysis reveals that the as-prepared and annealed selenium samples are of trigonal structure. The composition analysis results show that Se is present in the sample. TEM images reveal that Se nanowires have diameter ranging from 30–50 nm and length 1–2 μm .

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