**Fabrication of Single Crystalline Cadmium Nanowires by a Facile Low Temperature Vapor Phase Method**

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Single-crystalline cadmium nanowires were successfully fabricated by vaporization of cadmium metal powders in a horizontal quartz tube furnace at 250 °C. The vaporization was carried out for 30 minutes and yielded nanowires of diameters of 80 to 250 nm and lengths up to several tens of microns. The nanowires were deposited on a Si (111) substrate kept at the lower temperature zone (150–175 °C) of the furnace. When the deposition temperature was lower than this, hexagonal nanodisks were produced. The possible mechanism for the formation of the obtained nanostructures is discussed.

**Keywords:** Single Crystalline Metal Nanowires, Cadmium, Vapor-Phase Method.

1. INTRODUCTION

Single crystalline, one-dimensional (1D) nanostructures have attracted much interest because of their possible novel applications in various fields ranging from biosensors to the fabrication of nanodevices.¹⁻⁶ Several methods have been reported for the fabrication of these 1D nanostructures. Vapour-phase synthesis is probably one of the most widely used among them.¹⁻⁶ Cadmium (Cd) is an important heavy metal used in the nuclear reactors because of their tendency to absorb low energy neutrons. The rods of Cd or its alloys are used for controlling the fission process in nuclear reactors.³ It is used in the nickel-cadmium storage battery. Cadmium pigments are widely used because of their insolubility in water as well as in organic solvents, and stability against heat and ultraviolet light. The 1D nanostructures of several cadmium compounds, such as CdS, CdSe, and CdTe have been synthesized and studied extensively.⁶⁻¹⁰ Zuo et al. demonstrated the surface enhanced Raman scattering (SERS) of pyridine using cadmium metal film.¹¹ The fabrication of Cd nanowires (NWs) might potentially improve its efficiency in the above applications.

Rao and coworkers⁵ demonstrated the synthesis of Cd NWs by a nebulized spray pyrolysis process at a temperature of about 800 °C using cadmium acetate as a precursor. Cadmium nanotubes were also synthesized by the pyrolysis of CdS powder at a temperature range of 700 to 1000 °C.¹² Recently, Jiang et al. demonstrated the synthesis of Cd NWs by ion irradiation of single crystal wafer of Cd₃Nb₂O₉.¹³ In this article, we report the fabrication of single crystalline Cd NWs by a facile low temperature (250 °C) vaporization of Cd metal. It neither involves a catalyst nor a template, and only Cd-metal powder is used as the initial material.

2. EXPERIMENTAL DETAILS

Typically, 0.5 g of bulk cadmium powder (99.99%, Aldrich) kept in an alumina boat was placed at the centre of a horizontal quartz tube furnace. The system was pre-evacuated to 0.3 torr and then was purged with argon gas at a flow rate of 500 standard cubic centimeters per minute (scm) for 30 minutes. The products were obtained by controlling several parameters, such as temperature (T), pressure (P), reaction time (t), and the argon gas flow rate (k). The nanowires were deposited on a Si (111) substrate at the temperature of 150–175 °C, which was placed downstream (3 to 4 inches from the boat) of the flowing gas at an optimum condition of P ~ 5 torr, t ~ 30 min, and k ~ 500 sccm. Cadmium powder was kept at 250 °C.

X-ray diffraction (XRD) pattern of the specimen was recorded on a Rigaku D/max-rc (12kW) diffractometer operated at 40 kV voltage and 80 mA current with filtered 0.15405 nm CuKα radiation. X-ray photoelectron spectroscopy (XPS) measurements of the specimen were carried out by an ESCA 2000, V. G. Microtech UK, at a pressure...
of 1 × 10⁻⁹ torr using the Mg Kα line with a photon-energy of 1253.6 eV. Field emission scanning electron microscope (FESEM) images of the Cd NWs were taken by a Philips XL30S. Transmission electron microscope (TEM), high-resolution TEM (HRTEM) images, selected area electron diffraction (SAED) pattern, and energy-dispersive X-ray spectrometry (EDS) were taken by Jeol JEM-2100F transmission electron microscope operated at 200 kV.

3. RESULTS AND DISCUSSION

Figure 1 shows the FESEM image of the obtained Cd NWs deposited on the Si substrate kept at a temperature of 150–175 °C. (Evaporation temperature was 250 °C.) Large amount of nanowires were deposited on the substrate. Their diameters vary from 80 to 250 nm and lengths up to several tens of micrometers. The high magnification image of an individual nanowire given in the inset of Figure 1 shows that the surfaces of these nanowires are clean and smooth. The nanowire diameters are highly dependent on the vaporization temperature of the cadmium powder and also on the reaction time. On raising the evaporation temperature to 350 °C or higher, Cd NWs were formed with diameters larger than 700 nm. Similar results were also observed when the evaporation time was increased from 30 min to 90 min or longer. While, pressure and the argon flow rate had little effect on the morphology of the obtained nanowires, the diameters of the nanowires were increased to 400–500 nm either by raising the pressure from 5 to 15 Torr or by reducing the flow rate from 500 to 10 sccm.

The analysis of the crystal structure of the specimen was carried out using XRD. Figure 2(a) displays the XRD pattern of the obtained Cd NWs. All the observed peaks in the diffractogram were unambiguously indexed to the hexagonal crystal structure of cadmium (JCPDS No: 05-0674) with lattice parameters \( a = 0.297 \text{ nm} \) and \( c = 0.560 \text{ nm} \) of space group \( \text{P6}_3/\text{mmc} \). The peak positions and the intensities in the diffractogram match well with the standard values. The obtained nanowires were highly pure because only Cd metal powder was used as the precursor. This is studied with the EDS shown in Figure 2(b), which shows only the peaks of cadmium. The carbon and copper peaks in the EDS spectrum originated from the carbon coated copper grid used for the sample preparation.

The elemental composition of the cadmium nanowires was also analyzed with the XPS shown in Figure 3(a) and (b). The peak at 284.8 eV is due to the C1s line carbon contamination and used as a reference. The wide scan spectrum shows that the nanowires are composed of only cadmium. The peaks at 10.8, 405.2, 411.9, 618, and 652 eV correspond to the 4d, \( 3d^{10}_{2} \), \( 3d^{9}_{2} \), \( 3p_{1} \), and \( 3p_{1} \) binding energy of elemental cadmium, respectively. No oxygen peak was detected in the spectrum demonstrating that the nanowires are free of oxide coating. This observation supports the results from the EDS analysis and will be further investigated with the HRTEM image. The high resolution XPS spectrum shown in Figure 3(b) shows more...
Mohanty et al. Fabrication of Single Crystalline Cadmium Nanowires by a Facile Low Temperature Vapor Phase Method

RESEARCH ARTICLE

Fig. 3. (a) XPS wide scan spectrum and (b) the high resolution spectrum of the Cd NWs.

details of the $3d_{3/2}$, $3d_{5/2}$ peaks. They were observed at 405.2 and 411.9 eV, respectively, and the peak positions compared well with the standard value of the element.14

The morphology, composition, and size of the NWs were further studied with TEM. HRTEM micrograph and the SAED pattern were used in order to confirm the structure and the growth direction of the obtained Cd nanowires. Figure 4(a) and 4(b) display TEM and HRTEM images of a representative nanowire. The nanowire has a diameter of $\sim 175$ nm with very clean surface. The HRTEM image taken from a portion of this nanowire shown in Figure 4(b) confirms the structural uniformity. The clear lattice fringes in this image together with a sharp SAED pattern (inset, Fig. 4(a)) confirm that the nanowire is single crystalline. A lattice spacing of 0.258 nm as observed agrees well with the separation between (100) lattice plane of hexagonal cadmium. Thus, the nanowire grew along the same [101] direction as reported by Rao and coworkers.5

It has been reported that in a vapor-phase synthesis, the degree of the supersaturation at a particular experimental condition is the most important factor to control the growth morphology of the obtained nanostructures.4 A low degree of supersaturation facilitates the formation of the nanowires, and medium or high supersaturation supports growth of bulk crystal or powder, respectively.4 The supersaturation factor, nucleation size, and growth time were controlled by adjusting the experimental conditions such as temperature, pressure, and the proper dilution of Ar mixture, in order to manipulate the lateral dimension of the nanowires. In the present case, we believe that the nanowires were formed through a vapor-solid (VS) mechanism due to the low degree of supersaturation of the cadmium vapor at the temperature region of 150–175 °C, which is lower than the melting point (321 °C) of cadmium metal.4

When the deposition temperature is lower than 150 °C, the degree of supersaturation becomes medium and hexagonal nanodisks were formed supporting the suggested mechanism. Figure 5 shows the FESEM image of thus

Fig. 4. (a) TEM image and (b) HRTEM image of a representative Cd nanowire. The SAED pattern is given in the inset to (a). As shown with the HRTEM image and the SAED pattern, the nanowire is single crystalline with (101) growth direction.

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obtained cadmium nanodisks. The disks are 1–2 μm in diameter and 50–250 nm thick, which are stacked one over another. Similar hexagonal nanodisks are reported in the ZnO,15 other metal oxides,16 and metals.17 Further details of the formation mechanism of the nanowires and the hexagonal nanodisks are under investigation.

4. CONCLUSION

In summary, single crystalline metallic cadmium nanowires were synthesized by a low temperature vapor-phase method. This method does not involve a catalyst or templates, and only pure cadmium metal is used as initial material. In addition to the nanowires, hexagonal nanodisks were also formed at a lower temperature zone. Although the properties and applications of Cd nanowires have not been explored yet, the obtained nanostructures can be potentially used for studying the surface enhanced Raman scattering and also can be used as a template for the fabrication of other functional nanomaterials. The method can be further extended for the fabrication of one-dimensional nanostructures of low melting point metals, such as bismuth, tellurium, etc.

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References and Notes


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