



Preparation of novel layer-stack hexagonal CdO micro-rods by a pre-oxidation and subsequent evaporation process



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ABSTRACT

Novel layer-stack hexagonal cadmium oxide (CdO) micro-rods were prepared by pre-oxidizing Cd granules and subsequent thermal oxidation under normal atmospheric pressure. X-ray diffraction (XRD) and scanning electron microscopy (SEM) were performed to characterize the phase structure and microstructure. The pre-oxidation process, vapor pressure and substrate nature were the key factors for the formation of CdO micro-rods. The diameter of micro-rod and surface rough increased with increasing of thermal evaporation temperature, the length of micro-rod increased with the increasing of evaporation time. The formation of hexagonal layer-stack structure was explained by a vapor–solid mechanism.

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1. Introduction

One-dimensional (1D) or quasi-1D semiconductor nanostructural materials have attracted considerable attention in recent years for their unique properties and numerous applications in opto-electronic nanoscale devices and functional materials. Among these materials, Cadmium oxide (CdO) is a II–VI semiconductor with a direct band gap of 2.5 eV and an indirect band gap of 1.98 eV, promising applications in catalysts, sensors, solar cells and optoelectronic devices [1,2]. So the controllable synthesis of CdO nanostructures have attracted much attentions, and a number of CdO nanostructures have been synthesized in different morphologies including nanowires [3,4], nanotubes [5,6], nanofibers [7], nanorods [8–11] and nanoparticles by different methods, including the template hydrothermal process [12], thermal evaporation method [6,13,14], chemical bath deposition method [15] and solvothermal condition [11,16]. The thermal evaporation process was an effective method to synthesis CdO nanostructures in various morphologies such as nanoneedles [13], nanorods and nanoparticles [14] nanowire and nanotubes [6] by control of the evaporation process. And catalyst were often used to prepare 1D nanostructure during the evaporation process,

however, the catalyst in the product were detrimental for its application in device. Therefore, it was necessary to obtain an effective method for the preparation of 1D material without catalyst. Although many 1D nanostructures have been prepared by different methods, these 1D nanostructures were all in square or circular cross section and with smooth surface. Novel hexagonal micro-rods with a layer-stack structure were obtained by a pre-oxidation and thermal evaporation technique, and the growth mechanism was studied in this paper.

2. Experimental

CdO microstructures were synthesized by a pre-oxidization and subsequent thermal evaporation method. A certain amount of pure Cd granules with several millimeters in dimension were mixed with excessive hydrogen peroxide solution (30% concentration) in a beaker for 8 h at room temperature, the granules were cleaned out with distilled water and dried, then the pre-oxidization Cd granules can be obtained and were put into a crucible. The muffle furnace was heated up to high temperature (800 °C, 810 °C, 820 °C or 860 °C) and then the crucible was rapidly put inside the muffle furnace. The pretreated Cd granules were maintained at high temperature for a period of evaporation duration time range from 5 min to 30 min under normal atmospheric pressure, and then the crucible was removed from the furnace. During the thermal evaporation process, Cd granules were vaporized and the oxidization of Cd vapors lead to the nucleation and growth of CdO products on the sidewall of crucible.

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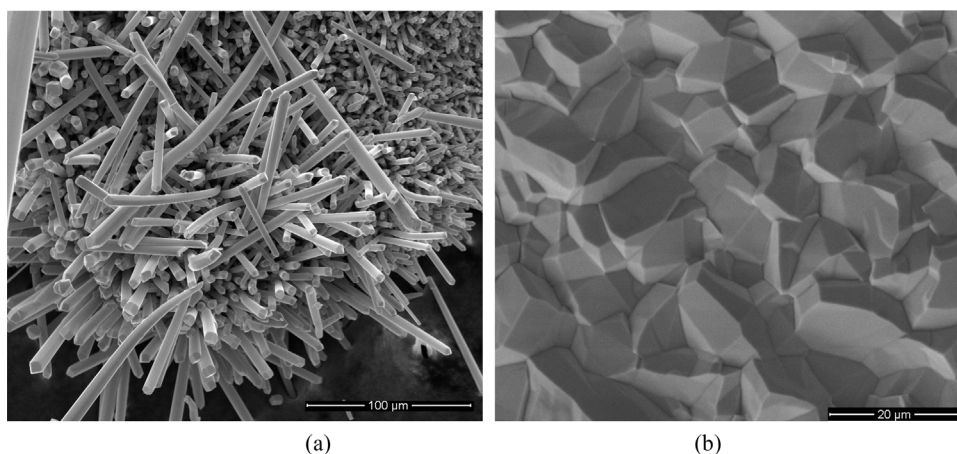


Fig. 1. CdO products obtained at different distance to the evaporation source (a) 1.2 cm (b) 2.2 cm.

The crystal structure of the products was analyzed by X-rays diffraction (XRD) with using a Siemens D5000. Scanning electron microscopy (FEI-Quanta200 SEM) was used for microstructure observation.

3. Results and discussion

The pre-oxidized Cd granules were thermal evaporated at 810 °C for about 30 min. It can be seen clearly by eyes that there were many rods with several millimeter in length at the lower part of sidewall and the morphology was different from the products on the upside of sidewall. Fig. 1 shows the SEM images of the products. Fig. 1(a) shows the SEM image of the product obtained at the lower side of the crucible sidewall, whose distance to the evaporation source was about 1.2 cm, and Fig. 1(b) shows the SEM images of the products obtained on the upside of crucible sidewall, whose distance to the evaporation source was about 2.2 cm. It can be known that the morphology of CdO was depended on the distance to the evaporation source according to these results. At a lower distance many micro-rods were formed, however, only a coating composed of particles can be found at higher distance. The phase structures of these samples were characterized by XRD analysis and the results were shown in Fig. 2, curve (a) and (b) corresponding to the diffraction spectra of the sample obtained at lower and higher distance, respectively. It can be seen that all the diffraction peaks can be indexed as a cubic CdO structures (JCPD

card No 05-0640), and no peak from Cd and other phases can be found in the XRD spectrum, which indicated that the prepared product was pure CdO. And the XRD powder JCPDS file of CdO states the standard values correspond to the cubic CdO planes, their the relative peak intensity (I) of (111), (200), (220), (311) and (222) are 100, 88, 43, 28 and 13, respectively. These XRD spectra shows a strong (111) diffraction peak and weak (200) diffraction peak, indicating the product growth with preferred orientation.

The changes of product morphologies with the distance to the evaporation source can be explained vapor–solid mechanism and the vapor pressure. In general, the growth mechanism of one-dimensional structures can be explained by the anisotropic growth via a vapor–solid mechanism. The nucleation probability (p) on the surface of a nanostructure was given by the following equation [17,18]:

$$P = B \exp \left(\frac{-\pi \sigma^2}{K_B^2 T^2 \ln \alpha} \right) \quad (1)$$

where B was a parameter constant, σ was the surface energy of the solid, K_B was Boltzmann's constant, T was the absolute temperature, and α was the supersaturation ratio between the actual vapor pressure and the equilibrium vapor pressure corresponding to temperature T . For the same supersaturation ratio, the growth direction of one-dimensional structure was usually along a low crystal index, which was due to this direction was corresponding to the lowest surface energy, and $\sigma_{\min}$ suggesting the highest nucleation probability. And it can be known that the actual pressure decreased with the increase of the distance to evaporation source, it means α decrease with increasing of the distance, which results in a low nucleation probability. And according to Eq. (1), the influence of surface energy of different direction can be ignored when α reach to a small value, it means the growth probability was almost approximate. Therefore, the rod-structure can be obtained near to the raw materials and a coating with larger grain size can be obtained for the conditions far from the evaporation source.

The same experimental have also been done for the samples without pre-treatment, however, no evaporation phenomena can be observed, the rudimental products kept in the same morphology as the raw granules. A thick CdO layer was formed on the Cd granules and the core of the rudimental product were kept in the state of Cd. According to the above results, it can be known the pre-treatment by H_2O_2 solution played an important role on the formation of CdO rods. In order to explain these phenomena, the influences of pre-treatment on the transformation of Cd were

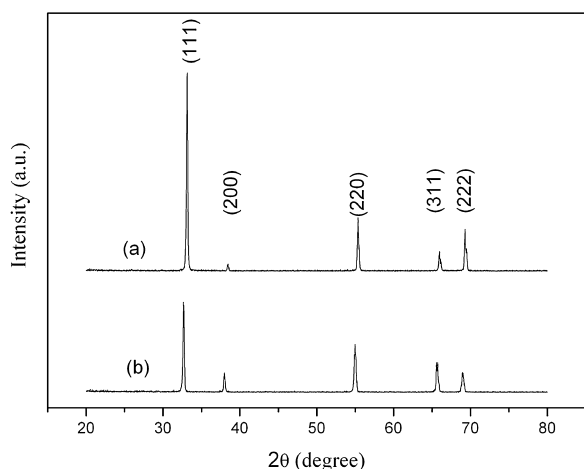


Fig. 2. XRD spectra of CdO products.

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