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RAPID COMMUNICATION

Novel method synthesis of CdO nanowires

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Abstract

Large-scale single-crystalline CdO nanowires have been successfully fabricated from the electrochemical deposition mixture film of Cd and Te at 450°C, and characterized by x-ray diffraction (XRD) powder, scanning electron microscopy (SEM) and transmission electron microscopy (TEM). SEM and TEM images show that these nanowires are uniform with diameters of about 40–60 nm, length up to several tens micrometres. XRD and selected area electron diffraction analyses altogether indicate that these CdO nanowires crystallize in a NaCl cubic structure. The growth mechanism of these nanowires is also proposed as vapour–liquid–solid mechanism, in which Te serves as a liquid-forming agent.

Recent progress in the synthesis and characterization of nanowires has been driven by the need to understand the novel physical properties of one-dimensional nanoscale materials and their potential application in constructing nanoscale electronic and opto-electronic devices [1]. Nanowires with different compositions have been explored using various methods including the vapour-phase transport process [2–4], chemical vapour deposition [5], arc discharge [6], laser ablation [7], solution [8, 9] and a template-based method [10]. A large part of this work has been focused on semiconductor systems such as Si [1], Ge [2], GaN [3], GaAs [5, 7] and many studies on oxide systems also exist in the literature. Among them, MgO [11], SiO₂ [12], GeO₂ [4] and Ga₂O₃ [6] nanowires have been reported. Near recently, ZnO, CdO, SnO₂ and In₂O₃ nanobelts also have been fabricated by thermal evaporation method [13].

Binary oxides such as ZnO, CdO, SnO₂ and In₂O₃ have distinctive properties and are now widely used as transparent conducting oxide materials (TCOS) [14] and gas sensors [15]. CdO is one of the promising representatives of these TCOS. This material is an n-type semiconductor, with a direct bandgap at approximately 2.5 eV [16], and an indirect one experimentally found at 1.98 eV [17]. The current studies of TCOS have been focused on thin films and nanoparticles, which can be readily synthesized with various well-established

techniques such as sputtering (for films) and sol–gel (for particles). In contrast, just ZnO [18] and In₂O₃ [19] nanowires have been reported; investigations of others wire-like TCOS nanostructures are cumbersome, because of the unavailability of nanowire structures. In this paper, we report the synthesis and characterization of CdO nanowires fabricated using a novel method at 450°C, which is very lower than the CdO nanobelts prepared at temperature 1000°C [13].

The CdO nanowires were prepared by the following procedure. The mixture film of Cd (70%) and Te (30%) on Al substrate were obtained by direct current electrodeposition from an aqueous (pH ≈ 10) containing 0.1 mol l⁻¹ CdCl₂ and 0.2 mmol l⁻¹ TeO₂ at room temperature with a constant current of 0.2 mA for 6 h. After electrodeposited, the system was dried at 60°C for 2 h, and placed in a ceramic cube and sent into a quartz tube. The quartz tube was heated to 450°C and kept for 2 h at an oxidation atmosphere (10% O₂ and 90% Ar) under flow of about 200 sccm min⁻¹. After cooling down to room temperature, a bright yellow coloured, wool-like product was found on the surface of the Al substrate.

The structures of the products were analysed using x-ray diffraction (XRD) powder (PW 1710 instrument with Cu K α radiation). The morphology and microstructure analysis were conducted using scanning electron microscopy (SEM) (JEOL JSM 6300), transmission electron microscopy (TEM) (JEOL 2010) equipped with an energy-dispersive x-ray fluorescence (EDX). For SEM observation, the products were

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directly mounted on copper stubs. For TEM and high-resolution TEM observations, the as-synthesized products were ultrasonically dispersed in ethanol and a drop was then placed on an amorphous carbon-coated copper grid.

Figure 1(a) shows a typical XRD pattern of the products. The diffraction peaks can be indexed to crystalline CdO and TeO₂ phase, revealing that the product consists mainly of NaCl cubic structure CdO with a cell constant $a = 0.4569$ nm, and trace amount of TeO₂ is consistent well with the JCPDS 9-433. The Al (020) peak came from the Al substrate. Figure 1(b) is the XRD pattern of the as-electrodeposited mixture film, which shows that there exist only peaks of the Al substrate, no other peaks of CdTe, Te, Cd, TeO₂ or CdO were observed. These XRD results indicate that CdO was formed in the progress of the physical vapour deposition. A typical SEM image of CdO nanowires is shown in figure 2. The image shows that uniform CdO nanowires were formed in a high yield. The lengths of the nanowires are up to several tens micrometres.

Further structural characterization and composition of the CdO nanowires were carried out using TEM. TEM studies show that the nanowires have uniform diameters, and typically terminated with a nanoparticle at one end. Figure 3(a), a typical TEM image of such CdO nanowires, shows the

representative morphology of the CdO nanowires capped with nanoparticles (as the arrows marked), with diameters of around 40 nm. The nanoparticles at the wire apexes generally appear dark and have high contrast compared with the stem of the nanowires. EDX analysis (figures 3(b) and (c)) indicates that the nanoparticles are composed of Te (18.31 at%), Cd (34.92 at%) and O (46.77 at%), whereas the stem of the

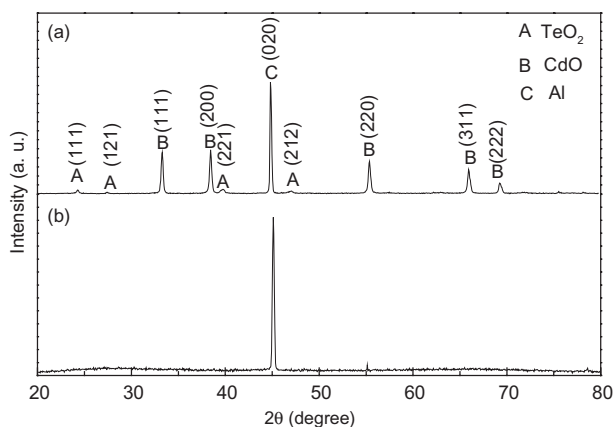


Figure 1. (a) The XRD patterns of the synthesized CdO nanowires. (b) The XRD of the as-electrodeposited mixture film.

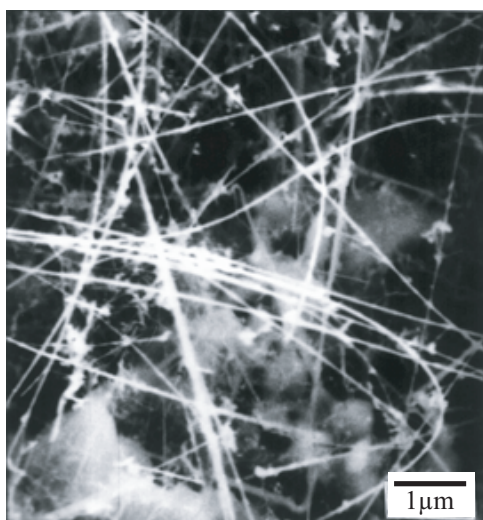


Figure 2. Typical SEM image of the fabricated CdO nanowires.

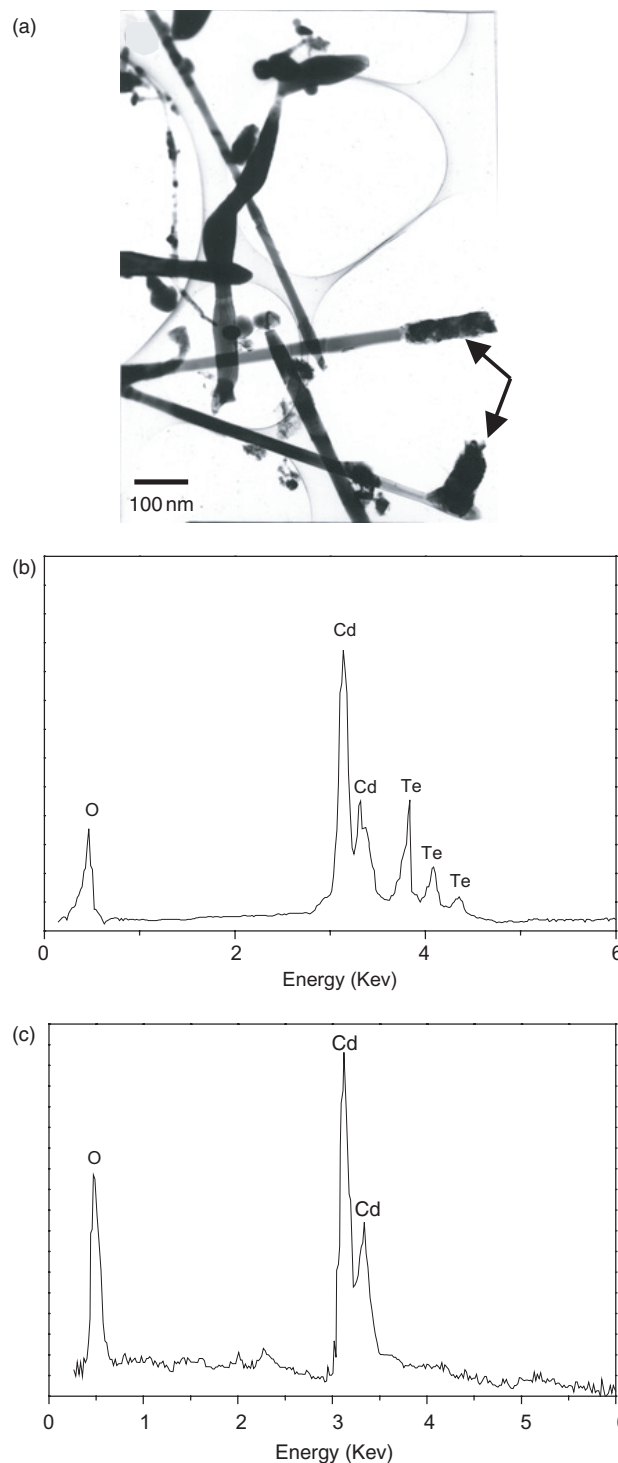


Figure 3. (a) shows a typical TEM image of the CdO nanowires terminated with a nanoparticle at one end. (b) and (c) are the EDX analysis results of the nanoparticles and the stem of the nanowires, respectively.

nanowires consists of Cd (49.3 at%), O (50.17 at%), which is in good agreement with the corresponding ratio of bulk CdO (Cd:O = 1:1). The presence of Te in the nanoparticles at the nanowires end represents strong evidence for a growth process dominated by vapour–liquid–solid (VLS) mechanism. Figure 4(a) shows an individual CdO nanowire, which was further characterized by selected area electron diffraction (SAED) and HRTEM, as illustrated in figure 4(b). The inset of figure 4(b) shows the SAED pattern recorded perpendicular to the axis of this nanowire can be indexed for the $\langle 2\bar{2}0 \rangle$ zone of the cubic CdO, which revealing the single crystalline nature of this nanowire. Subsequently, HRTEM is employed to observe the fine structure of the nanowire. The corresponding HRTEM image, figure 4(b), shows the fine structure of the part of the wire shown in figure 4(a). In this picture, the lattice fringes are spaced 0.235 nm apart. This finding is in agreement with the d value of the (002) planes of the CdO crystal. In addition, the angle between the (002) plane and the long axis of the wire is about 35.3° , which indicates that the wire axis is parallel to the [111] crystalline orientation of the cubic CdO. The SAED pattern has also confirmed that the nanowire preferably grows along [111] in agreement with the HRTEM investigations, which is different from the growth direction of CdO nanobelts along [100] [13]. This difference between CdO nanowires and its nanobelts is probably due to the different synthesized methods.

In order to explain the growth of the CdO nanowires, two important aspects should be considered. First, where

the oxygen atoms come from, and how they combine with cadmium to form the CdO nanowires? Second, what is the mechanism of the growth of the CdO nanowires? In this case, the oxygen atoms come directly from the oxygen gas, and react with the vapour Cd atoms to form CdO molecular.

The mechanism to explain the growth of conventional whisker and nanowires involves the participation of VLS phase in the growth progress. The central idea of the VLS growth concerns the existence of liquid-forming agent. The whole growth process of a VLS mechanism can be divided into two stages: the nucleation and the growth of eutectic alloy droplets and the growth of whiskers (or nanowires) through the liquid droplets due to supersaturating. Typical example of nanowire growth controlled by VLS mechanism is the Si nanowire [1]. The characteristic of a VLS growth is the existence of nanoparticles capped at the end of the nanowires. In the case of CdO nanowires, the typical TEM image (figure 3(a)) shows that there exist nanoparticles (as the arrows marked) at the end of the CdO nanowires. This result is reminiscent and describes that the growth of CdO nanowires was controlled by the VLS mechanism, and Te serves as a liquid-forming agent in the growth of CdO nanowires. This process is similar to the ZnO nanowires prepared from Zn and Se powders, while Se served as a liquid-forming agent in the growth of ZnO nanowires [18].

In summary, first time CdO single-crystalline nanowires were prepared in high yield via a VLS mechanism at 450°C . All the results show that the nanowires are quite straight and uniform with diameters of about 40–60 nm, lengths up to several tens micrometres and a NaCl cubic structure. This technique may be possible application for different other compounds such as ZnO.

Acknowledgment

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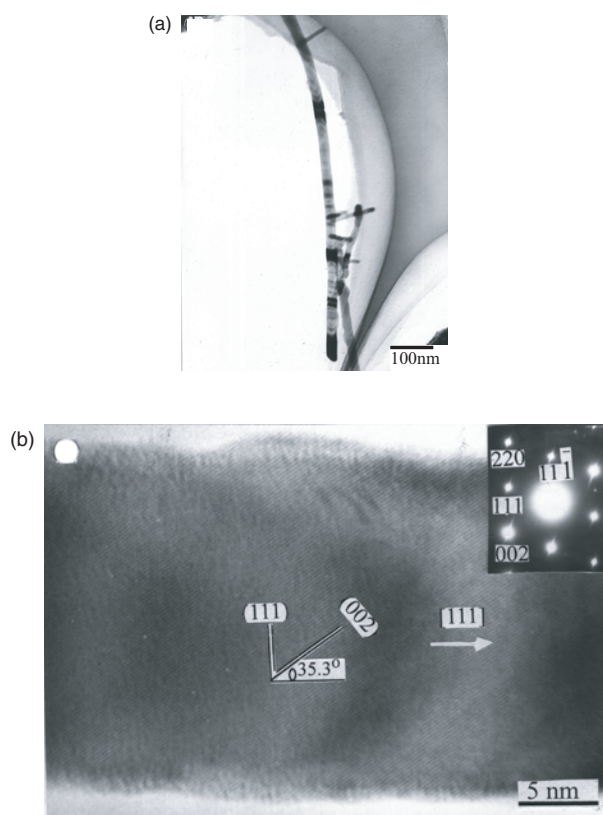


Figure 4. (a) shows an individual CdO nanowire with diameter of about 40 nm. (b) HRTEM shows the fine structure of the part of the wire shown in (a). The inset shows the corresponding SAED pattern recorded along $\langle 2\bar{2}0 \rangle$ zone axis.

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