

SnO₂ nanobelts and nanocrystals: Synthesis, characterization and optical properties

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ABSTRACT

SnO₂ nanobelts and nanocrystals have been synthesized in large quantity using the thermal evaporation and oxidation method via careful control of experimental parameters. The products were characterized with scanning electron microscopy, X-ray powder diffraction, transmission electron microscopy, and ultraviolet absorption spectroscopy. The as-synthesized SnO₂ nanobelts primarily consist of tetragonal rutile structure and orthorhombic phases, whereas the composition of the SnO₂ nanocrystals is a relative pure rutile structure. Detailed microstructure characterization reveals that stacking faults and a layered structure exist in the nanobelts and a twin structure was frequently observed in the nanocrystals. The ultraviolet absorption spectra of these two nanostructures were measured. Based on the above microstructural and optical results, the possible growth mechanism, and the structure-properties relationship were discussed for the nanobelts and nanocrystals.

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1. Introduction

During recent decades, nanostructured materials have received considerable attention from the scientific and engineering communities [1,2]. These structures exhibit distinct properties from those of bulk materials due to their small size and large surface-to-volume ratios, and accordingly, nanostructured materials have emerged as promising candidates for realizing nanoscale electronic, optical, and mechanical devices with enhanced performance. Among them, one-dimensional (1D) nanomaterials, such as nanowires, nanotubes, and nanobelts can function as both nanoscale device elements and interconnects while keeping unique properties due to size confinement in the radial direction [3,4]. Therefore, 1D nanostructures, in particular semiconductor 1D nanostructures, have been successfully synthesized by various methods such as thermal evaporation [5–7], chemical vapor deposition (CVD) [8,9], laser ablation [10], template [11], and sol–gel [12] techniques. Their optical, electronic, and magnetic properties

as well as their potential use in various applications have also been extensively investigated.

Nanocrystals emerged earlier than 1D nanostructures, partly due to the fact that they are readily generated via various experimental methods. These manufacturing methods include: melt crystallization, CVD, laser, self-propagating high-temperature synthesis (SHS), hydrothermal processes and others [13–15]. Nanocrystals have wide application in the electronic, optical, mechanical, magnetic, and sensing materials fields [16,17].

SnO₂, an n-type wide band gap ($E_g = 3.6$ eV, at 300 K) semiconductor with appreciable degree of iconicity, is attractive for potential applications in gas sensors [18,19], catalyst supports [20], and transparent conducting electrodes [21]. Recently, a series of SnO₂ nanobelts, nanowires and nanocrystals have been synthesized and investigated [22–25]. However, systematic comparisons and investigations of the relationship between microstructures and properties of the above different morphologies are still lacking. In this contribution, SnO₂ nanobelts and nanocrystals are synthesized via careful control of the air flow rates of thermal evaporation and oxidation. In addition, their microstructures and optical properties are determined and analyzed. Via comparisons of the microstructures and optical

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properties of these two morphologies, our objective is to shed light on the microstructure–properties relationship in nanomaterials.

2. Experiments

A horizontal alumina tube (outer diameter: 8.1 cm; inner diameter: 7.1 cm; length: 98 cm) was mounted inside a high-temperature tube furnace. Powders of metal tin were placed in an alumina crucible. After transferring the crucible to the center of the alumina tube, the tube was evacuated by a mechanical rotary pump to pressure of 10 Pa. During the experiment, a constant flow of Ar mixed with air was maintained at a stable flow rate and the pump continually evacuated the system so that the pressure inside the tube was kept at 5×10^4 Pa. The flow rates used were 100 sccm Ar and 20 sccm air for the preparation of nanobelts, and 100 sccm Ar and 200 sccm air for nanocrystals. Silicon substrates were placed in a position downstream from the air flow in the furnace to collect the reaction products. The temperature of the alumina tube was slowly increased from room temperature to 1150 °C and held at this temperature for 1 h before furnace cooling back to room temperature. After the furnace was cooled down, it was found that a white, wool-like product was suspended from the inner wall of the tube in the case of the nanobelt experiments, and a bright white layer formed on the silicon substrate in the case of the nanocrystal experiments.

Powder X-ray diffraction (XRD) patterns of the products were obtained with an X-ray diffractometer (Bruker D8 with CuK α radiation). Scanning electron microscope (SEM) images were taken on a Philips XL 30 FEG SEM. Transmission electron microscopy (TEM) samples were prepared by dispersing the powder products in alcohol by ultrasonic treatment, dropping them onto a porous carbon film supported on a copper grid, and then drying them in air. Low-magnification images and selected area electron diffraction (SAED) of the as-deposited products were taken on a JEOL-2010 transmission electron microscope. A Tecnai G² F30 transmission electron microscope, equipped with high-angle-angular-dark-field (HAADF) detector, Gatan imaging-filter (GIF) energy dispersive X-ray spectroscopy (EDX) systems, was used for Z-contrast imaging and composition analysis. A UV–vis spectrophotometer (JASO-V550) was used to obtain the optical absorbance spectra of the samples.

3. Results and discussion

3.1. Microstructures

Fig. 1(a) and (b) is the SEM image of the as-synthesized products, the morphology and scale of the nanobelts and nanocrystals are clearly evident. It is confirmed from a number of SEM images that the length of the nanobelts ranges from several tens of micrometers to the order of millimeters. The width of the nanobelts is about 100–200 nm, while the diameter of nanocrystals is about 30–50 nm. Moreover, it is discernable that both of the products are present in large quantities.

XRD patterns reveal the composition and crystal structure of the as-synthesized products, as shown in Fig. 2(a). The majority of the diffraction peaks for nanobelts can be indexed to the tetragonal rutile SnO₂ (space group number 136) with lattice parameter of $a = 4.72$ Å and $c = 3.19$ Å (JCPDS 41-1445). Some of the peaks are indexed to the orthorhombic phase SnO₂, which implies that some nanobelts consist of orthorhombic SnO₂ (space group number 19) with lattice parameter of $a = 4.714$ Å, $b = 5.727$ Å, and $c = 5.214$ Å (JCPDS 29-1484). In addition, some

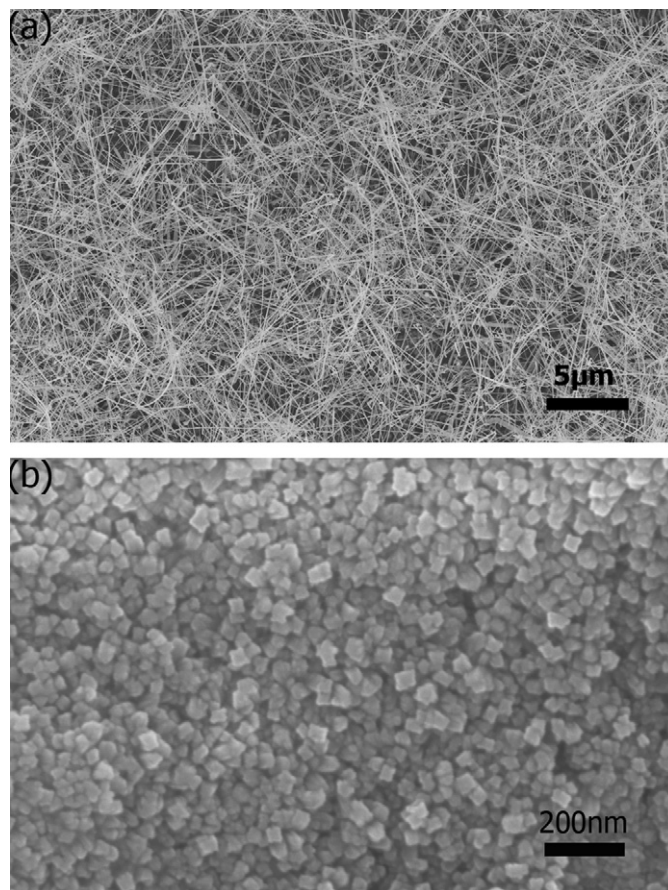


Fig. 1. (a) SEM image showing the typical morphologies of the as-synthesized SnO₂ nanobelts and (b) SEM image showing the typical morphologies of nanocrystals.

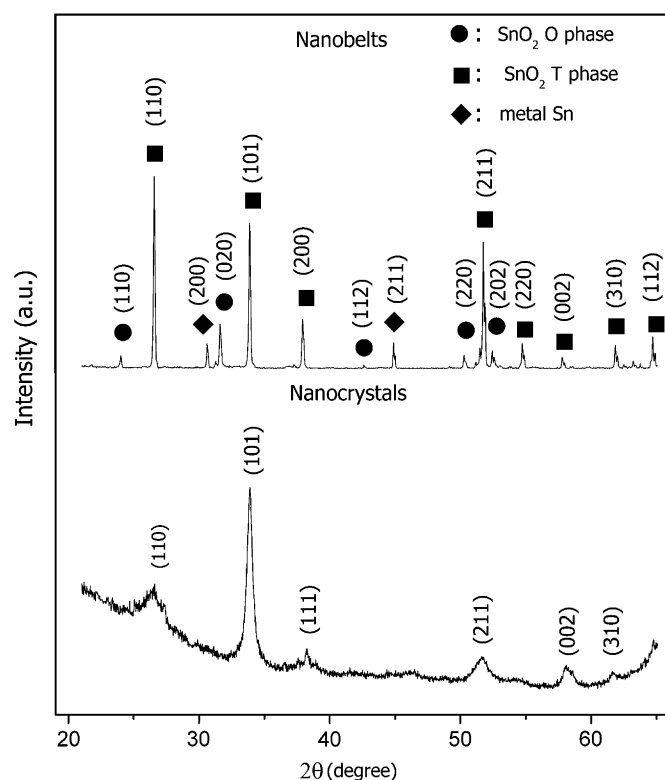


Fig. 2. XRD pattern of the as-synthesized nanobelts and nanocrystals.

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