Manipulation of the Morphology of ZnSe Sub-Micron Structures Using CdSe Nanocrystals as the Seeds

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ZnSe sub-microstructures with different morphologies, sizes, and microstructures were synthesized by a modified vapor deposition method using CdSe nanocrystals (NCs) as the seeds. On the basis of the appearance of the as-synthesized products, we show that the substrate temperature and CdSe NCs are the critical factors for controlling the size and the structure of various ZnSe sub-microstructures. By manipulating the growth driving force (i.e., the degree of supersaturation), the morphology of the ZnSe crystals can be varied sequentially from nanobelts to meso-scale rods to wires/branched wires. Their morphologies and structures were characterized by X-ray spectroscopy, Raman spectroscopy, scanning electron microscopy, and high-resolution transmission electron microscopy. The nanobelts grew along the [01−10] direction, whereas the meso-scale rods and wires grew along [0001]. Cathodoluminescence properties and growth mechanisms of these ZnSe structures are also discussed. The present results suggest that the simple method might be useful for the synthesis of other semiconductor nanostructures with manipulated morphologies to meet the growing demands of nanoscale science and technology.

Introduction

Semiconductor nanocrystals, especially 1-D nanostructures, have drawn much attention in recent years because of their great potential for fundamental research on the effect of dimensionality and size on physical properties, as well as for electronic and optoelectronic nanodevices application.1–3 As an important II–VI compound, ZnSe (with band gap energy of 2.70 eV) has been considered as a perspective material for optoelectronic devices, including blue laser diodes (LDs), light-emitting diodes (LEDs), and photo detectors.4–6 Recently, ZnSe-based microstructures have been widely investigated for their potential applications in high-density optical storage, full-color displays, etc.7 Moreover, ZnSe exhibits significantly larger exciton binding energy (21 meV) than that of GaAs (4.2 meV), which makes it an ideal candidate for efficient room-temperature exciton devices and devices with improved temperature characteristic.8 Like many other systems, shape control has received considerable attention for ZnSe, because in many cases it allows one to tune the properties for various applications with a great versatility that can be achieved otherwise. Recently, various ZnSe nanostructures have been prepared and widely studied. For example, ZnSe nanowires were synthesized by laser ablation of ZnSe powder,9 catalyst-assisted Molecular-beam epitaxy (MBE),10 metal-organic chemical vapor deposition (MOCVD),11 thermal evaporation,12 laser-assisted catalytic growth (LCG),13 atomic layer deposition (ALD)14 etc. ZnSe nanorods were fabricated by Sn-catalyzed thermal evaporation15 and solvothermal route.10 Although several research groups have developed diverse methods to generate ZnSe nanostructures, the ability to control the shape of ZnSe has been modestly successful. It still remains a grand challenge to synthesize high-quality ZnSe structures with manipulable morphology with high yield via a facile method.

More recently, the vapor deposition (VD) method has been proved to be very effective for synthesizing inorganic nanostructures. Specifically, comparatively little work has been performed on the morphological manipulation during vapor-phase growth.16 Herein, we demonstrate that the shape of ZnSe crystals can be manipulated sequentially from nanobelts to sub-microrods to wires by controlling the growth kinetics. Even branched ZnSe meso-scale wires can be achieved. To the best of our knowledge, this is the first synthesis of several ZnSe sub-microstructures, simultaneously and in bulk quantities. We adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.25 In this work, such strategy was adopted a simple modified VD approach by the evaporation of ZnSe powders onto Si substrates seeded with CdSe NCs. This seeded growth approach has been used to prepare zeolite membranes23,24 and thin films.

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degree of vapor supersaturation. Since fine control is possible, this novel one-step synthetic route is expected to be developed in order to synthesize other semiconductor nanostructures.

Experimental Section

Materials. Analytical-grade reagents including Cadmium acetate, methanol, chloroform, toluene, and high-purity Selenium powder (99.9%) were purchased from Beijing Chemical Reagent Ltd. Co. of China. Trioctylphosphine (TOP, 90%, Alfa), Stearic acid (SA, 96%, Aldrich), and Zinc Selenium powder (99.99%, Alfa) were used as received without further purification.

Synthesis of CdSe Nanocrystals (NCs). The CdSe nanocrystals were synthesized according to the method of literature. Typically, the synthetic processes were conducted in an inert atmosphere and were performed using standard air-free techniques. A mixture of a specified amount of Cd (Ac) 2 and 5.5 g of ligand SA in a 25 mL three-neck flask was heated to about 150 °C to obtain a colorless clear solution of Cd-precursor; then the solution was cooled to room temperature and aged for 24 h, and this system was heated to 290 °C. At this temperature, 0.12 g of selenium in 2.0 mL of TOP solution was quickly injected, and then the system was reacted for 5 min at 270 °C. After reaction, the obtained colloidal solutions were cooled, and precipitated by methanol. The formed floculent precipitate was centrifuged and the upper layer liquid was decanted, then the isolated solid was dispersed in toluene. The purified nanocrystals were re-dispersed in toluene, methanol, chloroform, toluene, and high-purity Selenium to about 150 °C. Since fine control is possible, this novel one-step synthetic route is expected to be developed in order to synthesize other semiconductor nanostructures.

Synthesis of ZnSe Sub-Microstructures. The ZnSe sub-microstructures were synthesized in a conventional tube furnace with a 50 mm inner-diameter quartz tube mounted inside, as described in detail elsewhere. In short, high-purity ZnSe powders were put in a quartz boat located at the center of the tube. Single-crystal p-type Si wafers covered with a thin film of CdSe nanocrystals were placed downstream to collect the products (Figure 1). After evacuation of the tube to $2 \times 10^{-2}$ Torr, a carrier gas of high-purity N$_2$ was kept flowing through the tube. The flow rate and pressure inside of the tube were kept, respectively, at 50 sccm and 1 Torr throughout the experiment. The furnace was first heated to 600 °C at a rate of 15 °C/min and held at that temperature for 30 min, and then further raised to 850 °C and kept for 60 min. After that, the furnace was cooled to room-temperature slowly. The sub-microstructures were collected on the Si wafers at different positions in the furnace.

Measurement. The synthesized products were characterized by scanning electron microscopy (SEM, Hitachi F-4300) with an X-ray energy dispensed spectrometer (EDS), X-ray diffraction (XRD, Rigaku D/max-2400PC) with Cu K$_\alpha$ radiation, transmission electron microscope (TEM, JEOL JEM-2010), and high-resolution TEM (HRTEM, Philips Tecnai F30). The Raman spectra were recorded in the backscattering geometry by using a Renishaw-2000 Raman spectrometer with the 514.5 nm line of an Ar ion laser as the excitation source. X-ray photoelectron spectrum was measured using an ESCALab220i-XL electron spectrometer from VG Scientific using 300 W Al Ka radiations. The binding energies were referenced to the C 1s line at 284.6 eV from adventitious carbon. Luminescent spectra of individual structures were measured at room temperature by a cathodoluminescence (CL) system installed on the SEM (SEM, Quanta 200F).

Results and Discussion

Morphological Evolution of ZnSe. On the basis of SEM images and the plot of temperature at different deposition distance, three distinctive temperatures zones were identified. From 760 to 350 °C, the morphologies of ZnSe structures change from nanobelts (zone I, 760–450 °C) to meso-scale rods (zone II, 450–400 °C), and finally sub-microwires (zone III, 400–350 °C). Figure 1a schematically depicts the position of three zones inside of the tube. The corresponding plot of temperature and distance is shown in Figure 1b.

Figure 2a,b shows typical SEM images of the as-prepared ZnSe nanobelts deposited on a p-type Si substrate covered with a thin film of CdSe NCs, placed in zone I. The ZnSe nanobelts have thickness of about 20–50 nm, width of 0.5–6.0 um (width-distribution of the belts is shown in Figure 2f), and lengths up to several hundreds of micrometers. The lower-right inset of Figure 2b shows a high-magnification SEM image of typical nanobelts with a thickness of ~30 nm and a width of ~800 nm. The nanobelts have smooth surfaces, and their morphologies show little variation with the substrate temperature ranging from 450 to 760 °C. The structure and morphologies of the nanobelts were further characterized by transmission electron microscopy (TEM). Figure 2c shows a TEM image of an individual ZnSe nanobelt, which is almost transparent to the electron beam suggesting that the thickness of the nanobelt is small. Dark lines on the belt are due to bending contours, which are common as observed by TEM in slightly bent crystals. Figure 2d is a HRTEM image of the nanobelt, which shows perfect wurtzite structure with a [001] zone axis, with the corresponding selected-area electron diffraction (SAED) displayed in the inset. The fringe spacings shown in the image are about 3.45 Å and 6.53 Å, which agree with the (010) and (001) lattice planes of the hexagonal ZnSe, respectively. The [01−10] direction is parallel to the length of the nanobelts, e.g., the growth direction, which is perpendicular to the [001] direction. The perfect diffraction pattern and lattice fringes indicate that the nanobelt is well-crystallized. The energy dispersive X-ray spectroscopy (EDS) in the Figure 2e confirm that the nanobelt compositions are
ZnSe with no detectable traces of other elements, except for the weak Pt and O signals originate from the substrate. With the decrease of the substrate temperature, the shape of the products changed significantly. The SEM images of the products formed in zone II show that they are meso-scale rods (Figure 3a), in contrast to the nanobelts formed in zone I. Figure 3b shows that these rods have an average diameter of 620 nm and lengths up to several micrometers, exhibiting a cross-section with the hexagonal prismatic morphology. Figure 3c shows a TEM image of a single ZnSe rod with the diameter of about 600 nm, which is consistent with the SEM observations. The SAED pattern (inset of Figure 3d) and HRTEM image reveal that the ZnSe meso-scale rods have a single crystalline structure growing along [0001] direction which is different from the nano-belts. EDS spectrum taken in the SEM shows that the rods are ZnSe (Figure 3e) with negligible signals from CdSe. A statistical analysis of the diameter distribution of ZnSe rods is conducted by measuring over 200 rods in SEM images and the results are shown in Figure 3f. It can be seen that the sub-microrods are uniform, giving an average diameter of 622 ± 10 nm.

With further decrease of the substrate temperature, the morphologies of the products changes from meso-scale rods to wires. The general morphologies of the ZnSe sub-microwires formed in zone III are shown in Figure 4a. A dense carpet of meso-scale wires, having a typical length of several micrometers, covers the substrate. High-magnification SEM images (Figure 4b,c) show that the sub-microwires appear to have a relative uniform diameter of 325 nm and circular cross-section. In Figure 4f, histograms representing the diameter of these rods are shown. Furthermore, we found interesting structure morphology of ZnSe crystallites as shown in Figure 4d,e, which can be called branched wires. The branched wires also appear to be uniform over the whole length. Detailed structure analysis of the as-synthesized ZnSe wires was performed with HRTEM. Figure 5a shows a TEM image of a single meso-scale wire with the diameter of 325 nm. It is obvious that the ZnSe sub-microwire exhibits a uniform diameter throughout the length. The corresponding HRTEM and SAED shown in Figure 5b demonstrate the single crystalline nature of the wire and show that it grows along the [0001] direction similar to the meso-scale rods. Figure 5c,d shows the TEM images of ZnSe branched sub-microwires. Extensive HRTEM and ED examinations verify that all arms of each wire are structurally uniform single crystals. Figure 5e1 is an image of a single branch within the architecture. The lattice fringes of the [010] and [001] planes are clearly seen with d spacing of ~0.346 and ~0.654 nm, respectively, which are characteristic of the wurtzite ZnSe crystal structure. The growth direction of the branch is parallel to the [001] crystallographic axis; in other words, each branch of the complex anisotropic nanocrystal grows parallel to its own unique c-axis but not along other directions. The ED pattern (Figure 5e2) taken from the branch can be indexed as the [100] zone axis of a wurtzite ZnSe crystal; the out-of-focus diffraction pattern also suggests that the branch grows along the [0001] direction. Panels f1 and g of Figure 5 are HRTEM images of the interface between two arms of a ZnSe branched sub-microwire, and Figure 5f2 is the corresponding ED pattern. Both HRTEM and ED examination confirm that the arms of a branched-wire ZnSe crystal are single-crystalline.

The X-ray diffraction (XRD) patterns of the samples obtained at different positions are shown in Figure 6a. All the diffraction peaks can be readily indexed as hexagonal wurtzite-structured ZnSe with lattice parameters of a = 3.97 Å, c = 6.506 Å, in
good agreement with the literature value (JCPDS Card, No. 80-0008). No characteristic peaks of other impurities were detected in these patterns. The sharp diffraction peaks indicate the good crystalline of the prepared crystals. It is noted that the relative intensities of the peaks differ from the standard pattern of the bulk material, which should be caused by preferred orientation and distribution of ZnSe nanoparticles. Patterns of sub-micron rods and wires all show a strong \{002\} diffraction peak, which might be due to the growth of wurtzite-structured ZnSe along the [0001] direction. Therefore, the XRD pattern may be indicative of the anisotropic shapes of the three ZnSe samples.

Figure 6b shows the Raman spectra of these structures measured at room temperature. Apart from the Raman peak of CdSe at 200 cm\(^{-1}\), the peaks observed at 246 cm\(^{-1}\) can be assigned to the longitudinal optical (LO) phonon modes of ZnSe crystal. \(^{11,15,35–37}\) In comparison with the values of bulk hexagonal ZnSe powders, it is worth noting that the LO phonon peaks of the ZnSe sub-microproducts show a shift toward the higher energy. The quality and composition of as-prepared samples were also studied by X-ray photoelectron spectroscopy (XPS) analysis. Figure S2 shows the survey spectrum of the nanobelts formed at position I. No peaks of other elements except Zn, Se, C, and
the furnace, respectively. The vertical lines at the bottom in Figure 6a corresponding to the standard XRD pattern of wurtzite ZnSe (JSPDS No. 80-0008)

Figure 6. (a) XRD patterns and (b) Raman spectra of ZnSe nanobelts, sub-micron rods and wires, which were obtained at different positions in the furnace, respectively. The vertical lines at the bottom in Figure 6a corresponding to the standard XRD pattern of wurtzite ZnSe (JSPDS No. 80-0008)

Figure 7. XPS analysis of the ZnSe nanobelts formed at position I: (a) zinc region; (b) selenium region.

O were observed. The presence of C and O comes mainly from atmospheric contamination due to the exposure of the sample to air. The binding energies in all the XPS spectra were corrected for specimen charging by reference to C 1s at 284.60 eV. The Zn 2p3 and Se 3d5 features shown in panels a and b of Figure 7 have a binding energy of EB = 1022.0 and 53.9 eV, respectively. These results are in agreement with those reported previously. Those data of the sub-micron rods and wires are similar to that of the nanobelts. All XRD, Raman and XPS analysis indicate that the as-prepared products are ZnSe.

Control Mechanism. One unique aspect of the current study is using CdSe NCs as the seeds. Without the seeds, only some faceted ZnSe grains were observed and no belt, rod or wire-shaped ZnSe crystals were prepared (Figure 8). It is obvious that CdSe film acts as a seed layer for secondary crystal growth of ZnSe structures. In general, surface nucleation is favored when the crystal/surface interfacial free energy is low. As we know, deposition of a seed layer helps to promote surface nucleation. Furthermore, the lattice parameter mismatch between ZnSe and CdSe is relatively small. In accordance with the lattice-matching theory, effective epitaxial growth of ZnSe on the CdSe seeded film should be possible, since CdSe always used to be the core of ZnSe shell in the core−shell nanocrystals. Thus, the CdSe NCs play a critical role in the formation of ZnSe sub-microstructures.

For the growth of nanostructures in various material systems, two types of growth mechanisms have been proposed, namely, the vapor−solid (VS) and vapor−liquid−solid (VLS) mechanisms. The VLS mechanism can be ruled out because neither metal is used nor alloy particles are found on the tips of the nanobelts, sub-micro rods and wires, which is the symbol of the VLS model. So we suggest that the VS mechanism may be dominant in the growth process. In the VS method, the source materials vaporize to the molecular level with stoichiometric cation−anion molecules, and the molecules will be arranged in such a way that the proper local charge balance and the structure symmetry are maintained, resulting in a nucleation center. At position I, the temperature is relatively high and concentration of ZnSe is also high because of diffusion. Nanobelts are then nucleated and elongated along the [01−10] direction. The growth mechanism of ZnSe nanobelts is similar to that of other nanobelts. Wang and co-workers demonstrated that the end surfaces of the nanobelts may have a relatively higher surface energy than the sides, which may lead to a faster growth rate normal to the end surfaces of the nanobelts, forming a belt structure. With further intake of the molecules, the surfaces that have lower energy, e.g., side surfaces, start to form and tend to be flat. As the molecules have higher mobility at the high growth temperature, the newly arrived molecules tend to diffuse to the rough growth front, resulting in the increase in the surface area, which leads to the formation of the nanobelts. On decreasing the temperature, both the degree of supersaturation and diffusion rate of ZnSe molecules descend, which results in the formation of few ZnSe nuclei. At the same time, the ZnSe molecules have a higher chance of finding preferential positions with high energy on the ZnSe nuclei, causing the ZnSe solid-state nuclei to grow along preferential directions to form sub-micron rods/ wires. Since few ZnSe molecules are not enough to provide another growth direction for the ZnSe nuclei. The morphologies of the structures may be determined by a combination of growth kinetics and thermodynamics. The low-energy surface defines the planes that can be present in the final product from the thermodynamic point of view, while the growth kinetics determines the growth directions and the morphology of the product.

Differences in growth direction can be explained as follows. Generally, the zinc blende (ZB) phase of ZnSe is stable at low temperature, while the wurtzite (W) phase is a high temperature form. Previous studies show that W-type nanowires grow along two distinct (010) and (002) directions (Figure 9). It is possible that the (002) direction assigned for a minority of ZnSe structures could correspond to the (111) direction of a ZB structure. However, XRD measurements made on bulk microwire samples are consistent with the W assignment. Considering all these factors above, we conclude that ZnSe structures may prefer to grow along (010) direction at higher temperatures and along the (001) direction at lower temperatures. At position II and III, these temperatures are relatively lower, thereby favoring the growth along (001) direction.

It should be noted here that the dimensions of the ZnSe wires are much smaller than those of the rods and belts. According
to basic thermodynamics, eq 1 gives the driving force of growth ($f$) for the crystal prepared via the vapor phase:22,47

$$f = \frac{kT \ln \frac{p}{p_s}}{\Omega_s}$$

where $k$ is the Boltzmann constant, $T$ is the temperature, $p$ is the vapor pressure of the reaction site (a high degree of supersaturation means large $p$), $p_s$ is the saturation pressure of the vapor, and $\Omega_s$ is the molecular volume. It is evident that the crystal growth will be accelerated as the ZnSe vapor pressure and temperature increases. Obviously, from position I to III, both the temperature and vapor pressure descent. According to eq 1, $f$ for ZnSe wires is smaller than that for rods and belts. This makes the ZnSe sub-micron structures formed at position I larger than those formed at position II and III.

**Cathodoluminescence.** To investigate the optical properties of the ZnSe meso-scale structures, the nanoscale cathodoluminescence (CL) spectroscopy and CL imaging were conducted at an acceleration voltage of 15 KV. To visualize the spatial distribution of luminescence from single ZnSe products, CL images were recorded at room temperature, as shown in Figure S3. In the CL images, all the meso-scale structures appear bright. Figure 10 shows CL spectra of individual ZnSe wire, rod and belt. As can be seen from the dashed-dotted line curve, the as-grown ZnSe meso-scale wire displays a sharp and strong blue emission at 2.68 eV, which is usually attributed to the band edge emission of ZnSe.48 The measured 71 meV width of the peak is a result of temperature broadening convoluted with an instrument resolution, which was narrower than that of reported.16 The strong band edge luminescence shows that the ZnSe sub-microwires contain few, if any, dislocations or stacking faults, as these defects tend to quench band edge radiative recombination. The stable orange emission band at 1.93 eV was also observed, which is assigned to so-called “self-activated emission.”13,15,49probably as a result of some donor—acceptor pairs that are related to zinc vacancy50–51 and interstitial

states,52–54 associated with dislocations, stacking faults, and nonstoichiometric defects,21,55 the nature of which is not clear or worthy of further study.

The different optical properties for different shapes and phase structures of the semiconductor crystals has attracted significant attention recently,56–58 so comparing the optical properties of the ZnSe nanobelt, meso-scale rod and wire may help us better understand the intrinsic CL properties of our deposited ZnSe structures. In case of ZnSe sub-microrod, a new band at 2.17 eV was obtained, which is most probably attributed to neutral associative pairs ($V_{Zn} - D_{Zn}$).48 A ZnSe nanobelt with a thickness of $\sim$30 nm and a width of about 4.2 um was detected as shown in Figure S3e. It is obvious that the emission band of the ZnSe nanobelt is blue-shifted to 2.74 eV with respect to bulk ZnSe, which may be related to quantum-confinement effects. From these spectra, we found that the differences in the intensity distribution of the red, green, and blue CL bands may be important in practice, since color temperature of the white emission can be varied based on this fact. One may obtain “warmer” or “colder” white light by regulating the morphology of the ZnSe meso-scale structures.

**Conclusion**

In summary, various morphologies of ZnSe, including nanobelts, sub-micron rods, and wires/branched wires, have been synthesized by using CdSe nanocrystals as the seeds. CdSe nanocrystals play a critical role in the formation of ZnSe meso-scale structures. By manipulating the growth driving force (i.e., the degree of supersaturation), the morphology of the ZnSe crystals can be varied sequentially from belts to rods to wires/branched wires. Although the detailed growth mechanism
requires more systematic investigations, the present results suggest that this simple method might be useful for the synthesis of other semiconductor nanostructures to meet the growing demands of nanoscale science and technology.

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Supporting Information Available: XRD pattern and TEM images of the cubic CdSe NCs, XPS analysis of the ZnSe nanobelts and CL images of ZnSe products. This material is available free of charge via the Internet at http://pubs.acs.org.

References and Notes