

Research Article

A Simple and Efficient Method for Synthesizing Te Nanowires from CdTe Nanoparticles with EDTA as Shape Controller under Hydrothermal Condition

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We developed a simple and efficient method for synthesizing Te nanowires from CdTe nanoparticles with ethylenediaminetetraacetic acid disodium salt dehydrate (EDTA) as shape controller under hydrothermal condition. The system could both complete the transformation to Te and reduce the interference of CdTeS by adjusting the concentration of EDTA, which was proved by inductively coupled plasma mass spectrometry, X-ray diffraction patterns, and Raman spectra. It was found that the as-prepared Te nanowires display strong fluorescence emission in the blue-violet region. The nanowires exhibit a pretty good morphology with the average diameter of ca. 30 nm and a length up to micrometer scale. Moreover, a possible transformation mechanism of CdTe nanoparticles into Te nanowires is also discussed.

1. Introduction

One-dimensional (1D) semiconductor nanostructures, such as nanowires, rods, tubes, and belts, have been a significant aspect of today's endeavours in nanotechnology, due to their special optical, magnetic, electrical, and chemical properties [1–4]. Up to now, many techniques have been designed to synthesize one-dimensional semiconductor nanostructures, such as electrochemical synthesis [5], microwave-assisted synthesis [6, 7], template method [8], precursor [9], self-seeding solution process [10], polyol process [11], spontaneous route [12, 13], and solution-based chemical process [14, 15].

It is very important to control the formation and morphology of semiconductor nanostructure in the exploitation of novel properties [13, 15–17]. Progress has been made in synthesizing 1D nanostructures using semiconductor nanoparticles [18]. According to a pioneer study conducted in 2002 [3], CdTe nanoparticles could spontaneously reorganize into nanowires after controlled removal of the protective shell of organic stabilizer and storage in darkness at room temperature under ambient conditions for a continuous growth of 7 days. In 2005, to synthesize Se and Te nanowires

and investigate their possible transition mechanism, the researchers used stabilizer-depleted CdTe and CdSe nanoparticles and introduced ethylenediaminetetraacetic acid disodium salt dihydrate (EDTA) to the nanoparticles [12]. In 2006, they continued to synthesize spontaneously angled Te nanocrystals such as checkmarks, X-marks, and other unusual shapes and confirmed the transition mechanism of the system [19, 20]. However, it is necessary to develop a simpler and more efficient method for synthesizing nanowires.

In this study, hydrothermal method was used to synthesize Te nanowire in a hexagonal structure from CdTe nanoparticles with EDTA as shape controller. The effect of EDTA on the chemical transformation and morphology control of the nanowires was explored in details. This technique of nanowire synthesis is safe, and meanwhile the quality and the optical activities of the nanowires are very significant. This method, therefore, will find great potential applications in the processes of chemical transformation and synthesizing 1D nanostructures using semiconductor nanoparticles. In particular, this crystalline Te nanowires display strong fluorescence (FL) emission in the blue-violet region, which is of great importance for optoelectronic applications [21].

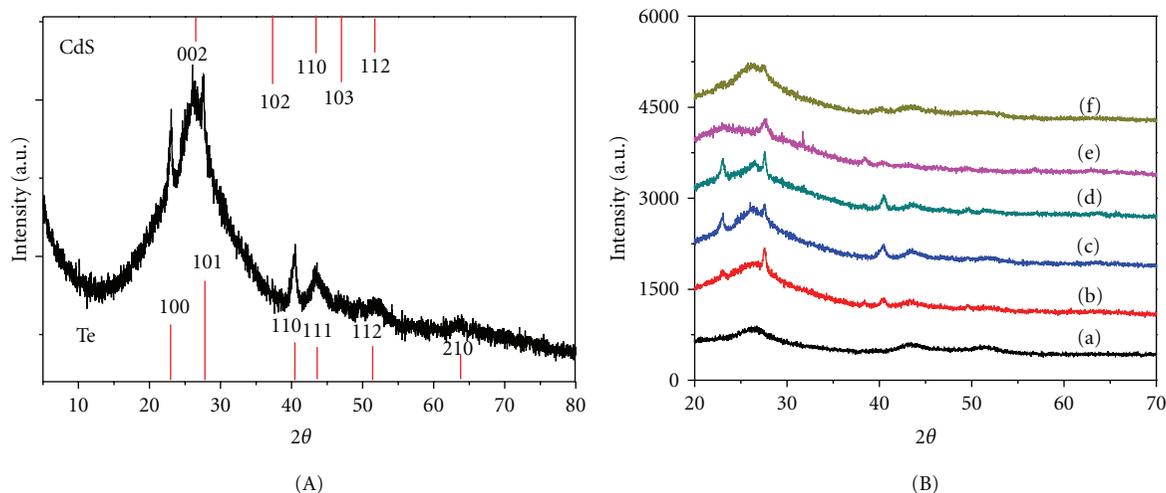


FIGURE 1: (A) XRD patterns of the as-synthesized nanowires with $0.10 \text{ g}\cdot\text{L}^{-1}$ and (B): (a) $0 \text{ g}\cdot\text{L}^{-1}$ EDTA; (b) $0.050 \text{ g}\cdot\text{L}^{-1}$ EDTA; (c) $0.10 \text{ g}\cdot\text{L}^{-1}$ EDTA; (d) $0.50 \text{ g}\cdot\text{L}^{-1}$ EDTA; (e) $1.0 \text{ g}\cdot\text{L}^{-1}$ EDTA; (f) $6.0 \text{ g}\cdot\text{L}^{-1}$ EDTA.

2. Experimental Methods

NaHTe was prepared according to the previously published procedure with several modifications [22, 23]. Briefly, a mixture of 0.0107 g of tellurium (Te) powder and 0.0354 g of sodium borohydride (molar ratio of Te to NaBH_4 is 1 : 3) was first loaded into a 10 mL bottle, then 0.2 mL of N_2 -saturated ultrapure water was added into the bottle to react at 4°C until the black Te powder fully disappeared and white sodium tetraborate precipitation appeared at the bottom of the bottle. The as-prepared NaHTe solution was used as Te precursor in the following reaction.

0.0357 g CdCl_2 and 100 mL ultrapure water were transferred to a three-necked flask with a condenser attached and kept bubbling with highly pure N_2 for 20 mins. This solution was then mixed with $15 \mu\text{L}$ thioglycolic acid (TGA) and 0.0227 g L-cysteine (L-cys). After that, $1.0 \text{ mol}\cdot\text{L}^{-1}$ NaOH was added to adjust its pH to 11 when the mixture became clear. 2.0 mL freshly prepared NaHTe solution was rapidly injected into the solution under vigorous stirring. The obtained solution was heated at 100°C for 3.5 h, and then was transferred to autoclave at 150°C for 1 h with a certain concentration of EDTA solution at pH 9. During the process, the colour turned from orange to deep blue, the typical colour as found previously for Te nanowires synthesized by the hydrothermal method [6]. The as-prepared product was centrifuged with acetone and then stored in a Medical refrigerator at 4°C for future use.

Fluorescence measurements were performed in a 3.0 mL quartz cell with an optical pathlength of 1.0 cm at room temperature using a RF-5301 Luminescence Spectrometer (SHIMADZU, Japan), with the scanning speed set at fast. X-ray diffraction (XRD) patterns were taken on a Rigaku D/MAX-RB diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda = 0.15406 \text{ nm}$). The transmission electron microscopy (TEM) images were performed using H-7650 (Hitachi, Japan) with an accelerating voltage of 200 kV . The scanning electron microscopy (SEM) images of catalysts were obtained by a

Philips XL30 FEG scanning electron microscope. The Raman spectra were acquired with an inVia micro-Raman spectra spectroscopy system (Renishaw, UK) in a spectral range of $100\text{--}2000 \text{ cm}^{-1}$, equipped with a He-Ne laser excitation source emitting wavelength at 633 nm . X-ray photoelectron spectra (XPS) were recorded on a VG multilab 2000 X-ray photoelectron spectrometer using $\text{Al K}\alpha$ radiation ($h\nu = 1486.58 \text{ eV}$) as the exciting source. The inductively coupled plasma mass spectrometry (ICP-MS) instrument used was an Agilent 7500 ce. All pH measurements were tested with a Model MP 120 pH meter (Mettler-Toledo Instruments Ltd., Swiss).

3. Results and Discussion

Panels (A) and (B) of Figure 1 show the XRD patterns of the freshly prepared product with different concentrations of EDTA, which proved that transformation of CdTe-CdS/Te exists and CdTeS could be effectively regulated just by the introduction of EDTA. As shown in Figure 1(A), the spectrum of the sample was in accordance with the values in the standard card of hexagonal Te (JCPDS no. 36-1452) [6], with the major reflections being at 100, 101, 110, 111, 112, and 210, respectively. These peaks can be indexed to perfect hexagonal structure of trigonal Te with the cell constants of $a = 4.4579 \text{ \AA}$ and $c = 5.927 \text{ \AA}$. Strong Te peaks undoubtedly confirmed that hexagonal Te really exists in the product. The four reflections at 002, 110, 103 and 112 are clear and support hexagonal crystal structure of CdS [24]. The reflections at 102 are poor due to the overlapping of peaks. These peaks in this pattern can be indexed to the hexagonal phase of CdS with the cell constants $a = 4.14092 \text{ \AA}$ and $c = 6.7198 \text{ \AA}$, which are in good agreement with the standard literature data (JCPDS no. 77-2306). As previously reported [19], the transformation products were composed of anisotropic Te and CdTeS component after EDTA were added into the crude CdTe nanoparticle (NP) solution without the step of stabilizer depletion. However, because

the ligands bond to the surface atoms were dynamically “on and off” [25], hydrothermal technology was adopted in our study to accelerate the dynamic process. CdTeS without centrifugation in methanol or 2-propanol to deplete the stabilizer shell. The image of Figure 1(b) confirmed the process, which shows that the peaks of CdS gradually disappeared with the increasing EDTA concentration.

The chemical composition of as-prepared 1D nanowires were measured by ICP-MS, which is considered as a powerful technique for trace and ultra-trace element analysis among research fields [26]. Before the ICP-MS measurements, the as-prepared samples were first purified and then dissolved by aqua regia and diluted by ultrapure water. Table 1 displays the relationship between EDTA concentrations and the element content of the nanowires. Along with the increase of the EDTA concentration, the maximum content of Te in nanowires emerged at $0.50 \text{ g}\cdot\text{L}^{-1}$ and then showed a significant decrease, indicating that the components of the nanowires could be effectively accommodated by adjusting the concentration of EDTA.

XPS was also used to characterize the surface chemical composition of the as-prepared nanowires. Shown in Figure 2 are the XPS spectra of the obtained nanowires without EDTA (Figure 2(a)) and with $0.50 \text{ g}\cdot\text{L}^{-1}$ EDTA (Figure 2(b)). The measured Cd/Te/S atomic ratio of 1.00 : 22.9 : 9.53 without EDTA and 1.91 : 56.3 : 1.00 with $0.50 \text{ g}\cdot\text{L}^{-1}$ EDTA indirectly reflect the surface composition of the 1D nanowires. The peak of S could also be detected from the sample with $0.50 \text{ g}\cdot\text{L}^{-1}$ EDTA due to formation of CdTeS. The S atoms can only come from the thiols of stabilizer, and the addition of EDTA was supposed to have partially removed the stabilizers from the nanowires to decrease the thiols in the surface.

The influence of the EDTA concentrations on the shape of the Te nanowires was studied by TEM and SEM. As shown in Figures 3–4, the products are exclusively anisotropic. To observe all the forms of the nanowires present, the nanowires showed noticeable variance while increasing the concentration of EDTA. From the images, it can be seen that, without EDTA, the nanowires are not very uniform and particularly the length and the quantity of the product are very limited. By introducing EDTA, both the morphology and the quantity of the nanowires are improved effectively. When the concentration of EDTA was $0.10 \text{ g}\cdot\text{L}^{-1}$, the uniform and long nanowires were obtained. The images in Figure 3(c) and Figure 4(c) show the long nanowires with high aspect ratios, an average diameter of 30 nm, and lengths up to micrometer scale. When the concentration of EDTA was lower than $0.10 \text{ g}\cdot\text{L}^{-1}$, the morphology tends to become gradually uniform, suggesting that the addition of EDTA improves the transformation of nanoparticles to nanowires, and hence the quantity of nanowires was remarkably enhanced. When the concentration of EDTA was more than $0.10 \text{ g}\cdot\text{L}^{-1}$, an agglomeration occurred and the obtained nanowires became shorter. In addition, the images clearly demonstrate that the resulting nanowires possess good monodispersity in solution. The dispersion of Te nanowires in water was deep blue, which is the typical colour as found previously for Te nanowires [6, 27]. Meanwhile, the flexibility of the material was shown to be pretty good. EDTA,

TABLE 1: The effect of EDTA concentrations on the element content ratio of Te to Cd in nanowires by ICP-MS.

Concentration ($\text{g}\cdot\text{L}^{-1}$) of EDTA	0	0.050	0.10	0.50	1.0	6.0
The content ratio of Te to Cd	0.807	3.321	7.685	88.78	7.752	0.790

therefore, could have played a crucial role in the formation and growth of the Te nanowires.

Figure 5 depicts the Raman scattering spectra of as-prepared nanowires with different concentrations of EDTA. The characteristic vibration peaks at 121.4 , 140.8 , and 260.7 cm^{-1} of Te are close to those reported previously, but have shown an obvious shift to high frequency [28, 29]. The 121.4 cm^{-1} and 140.8 cm^{-1} wavelength bands were assigned to A_1 and E phonon vibration modes of tellurium [30]. As depicted in Figure 5 and the inset figure, in the sample without EDTA, the wavelength number of 139.3 cm^{-1} and 157.7 cm^{-1} was attributed to longitudinal optical (LO) mode and weak transverse optical (TO) mode in CdTe, while the 269.8 cm^{-1} and 291.4 cm^{-1} wavelength bands were related to the CdS phonon vibrational mode. Under hydrothermal conditions, when the thiol compound was hydrolyzed in alkaline solution, CdTeS formed on the surface of original nanowires, which has proved that CdTeS shell actually envelops the nanowires [31]. However, the corresponding peaks in the spectra with EDTA are not so distinct maybe due to the limitation of the content of CdTeS shell. Therefore, all this also has confirmed that only with the help of EDTA can the hydrothermal method regulate the components of CdTeS effectively.

Figure 6 shows the FL emission spectra of nanowires with different concentrations of EDTA. The FL emission spectra excited at 230 nm and measured at room temperature with the nanowires sample dispersed in ultrapure water. The spectra showed two obvious emission peaks: a broad weak peak at around 300 nm due to excitonic emission and a narrow sharp peak at 370 nm due to carrier recombination caused by surface trap states of the nanowires. Different from what was reported previously [27], a narrow sharp peak was detected at 370 nm due to carrier recombination caused by surface trap states of the nanowires. The introduction of the EDTA was assumed to have led to a significant increase in the amount of excitonic emission peak and surface trap state emission. It is well known that the trap state emission relates to the surface chemistry and the structure of the nanowire [32]. Therefore, we infer that the introduction of EDTA results in certain changes in the surface state. The maximum FL intensity of trap state emission was found to emerge at the concentration of $0.50 \text{ g}\cdot\text{L}^{-1}$ (Figure 6(d)), which is consistent with the result of ICP-MS. The strong optical properties in the blue-violet region of the Te nanowires observed suggest that they may have potential applications in optoelectronic scales.

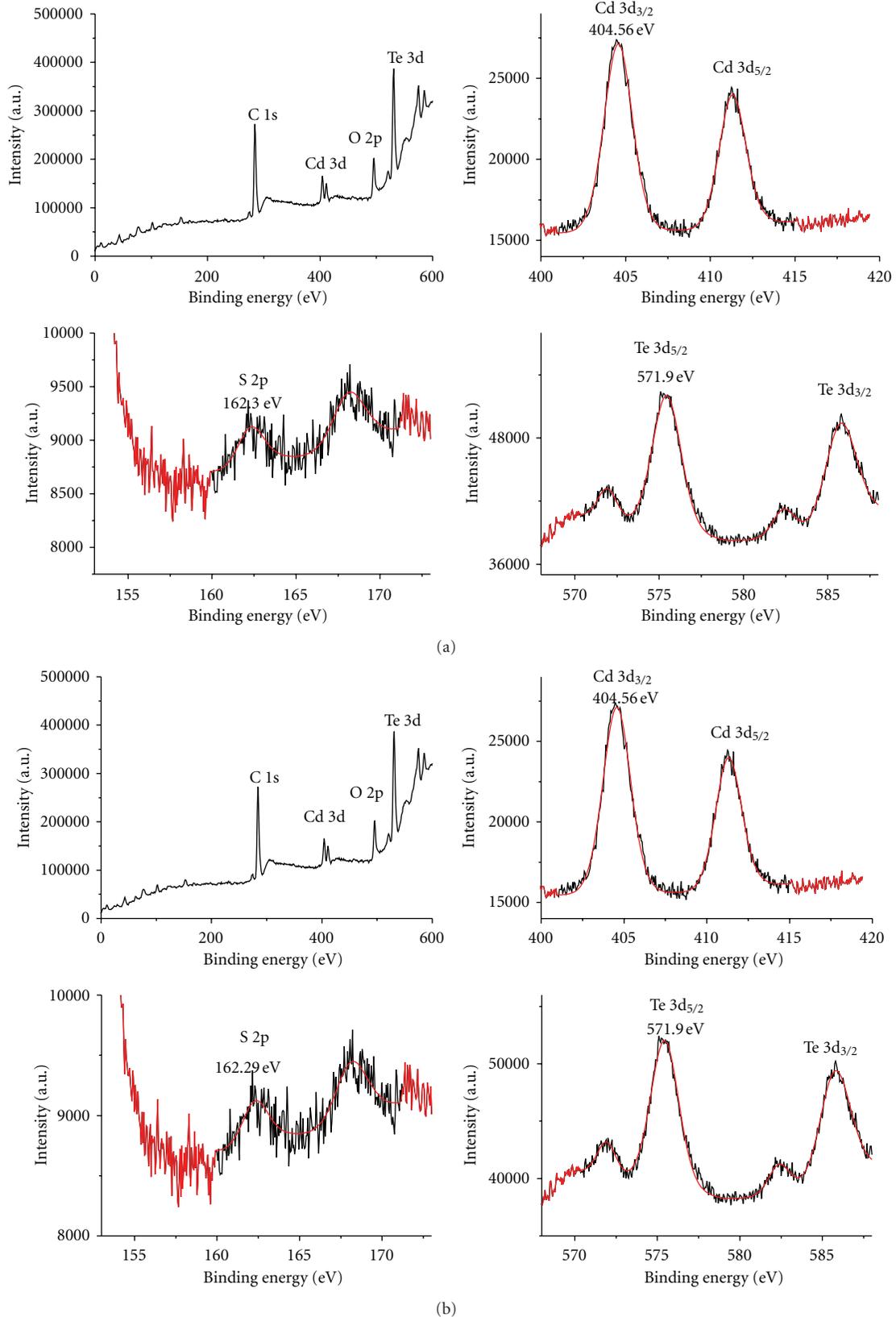


FIGURE 2: XPS spectra of the obtained nanowires (a) without EDTA and (b) with 0.50 g·L⁻¹ EDTA.

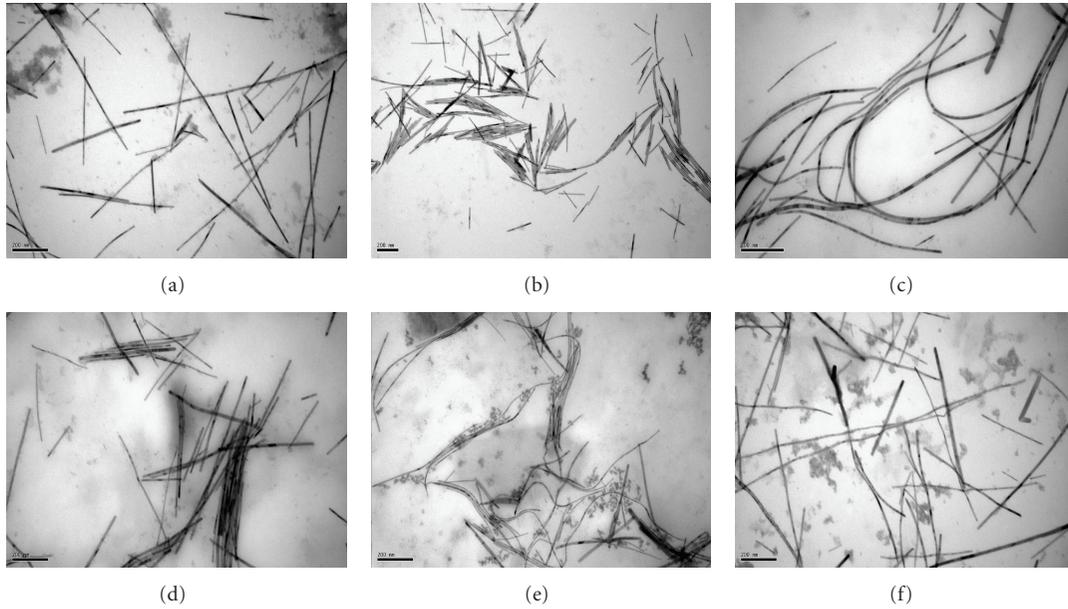


FIGURE 3: TEM images of nanowires with (a) $0 \text{ g}\cdot\text{L}^{-1}$, (b) $0.050 \text{ g}\cdot\text{L}^{-1}$, (c) $0.10 \text{ g}\cdot\text{L}^{-1}$, (d) $0.50 \text{ g}\cdot\text{L}^{-1}$, (e) $1.0 \text{ g}\cdot\text{L}^{-1}$, and (f) $6.0 \text{ g}\cdot\text{L}^{-1}$ EDTA. The scale bar is 200 nm.

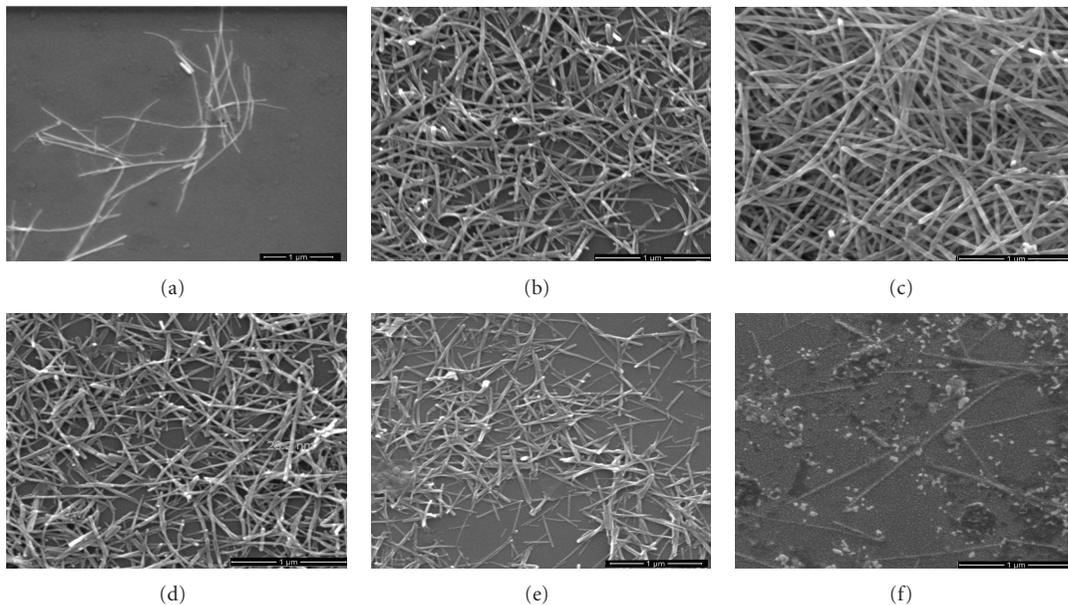
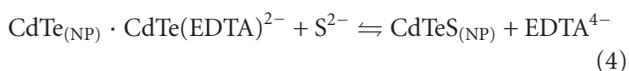
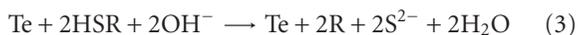
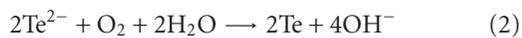
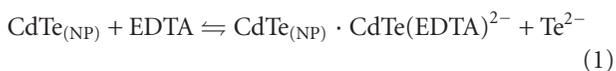


FIGURE 4: SEM images of nanowires with (a) $0 \text{ g}\cdot\text{L}^{-1}$, (b) $0.050 \text{ g}\cdot\text{L}^{-1}$, (c) $0.10 \text{ g}\cdot\text{L}^{-1}$, (d) $0.50 \text{ g}\cdot\text{L}^{-1}$, (e) $1.0 \text{ g}\cdot\text{L}^{-1}$, and (f) $6.0 \text{ g}\cdot\text{L}^{-1}$ EDTA. The scale bar is $1.0 \mu\text{m}$.

A possible mechanism by which CdTe nanoparticles are transformed into Te nanowires is suggested as follows [12, 33–36]:



The as-prepared CdTe nanoparticles stabilized with L-cys and TGA were redispersed into a certain concentration of EDTA solution at pH 9 (1). In the presence of EDTA, inherent instability of Te^{2-} and HTe^- in the solution can be gradually released in water, due to their strong reducing character of being easily oxidized by dissolved oxygen and finally transformed to Te (2) [33]. Due to its relatively low viscosity, water was a good medium for tellurium atoms to diffuse through, and the tellurium atoms were solvated in the solution phase. Meanwhile, because an excessive amount of L-cys and TGA stabilizers were involved in the transformation reaction [19], the modified reagents containing thiol

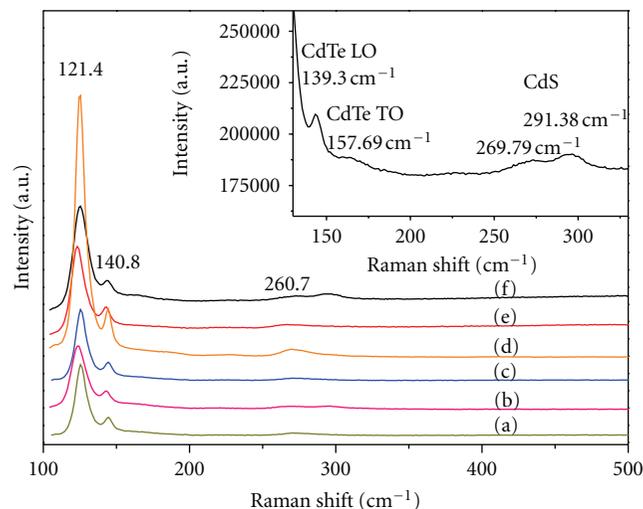


FIGURE 5: Raman scattering spectra of nanowires with (a) $0 \text{ g}\cdot\text{L}^{-1}$, (b) $0.050 \text{ g}\cdot\text{L}^{-1}$, (c) $0.10 \text{ g}\cdot\text{L}^{-1}$, (d) $0.50 \text{ g}\cdot\text{L}^{-1}$, (e) $1.0 \text{ g}\cdot\text{L}^{-1}$ and (f) $6.0 \text{ g}\cdot\text{L}^{-1}$ EDTA. (Inset: the Raman spectrum of as-prepared Te nanowires with the concentration of $0.50 \text{ g}\cdot\text{L}^{-1}$ EDTA.)

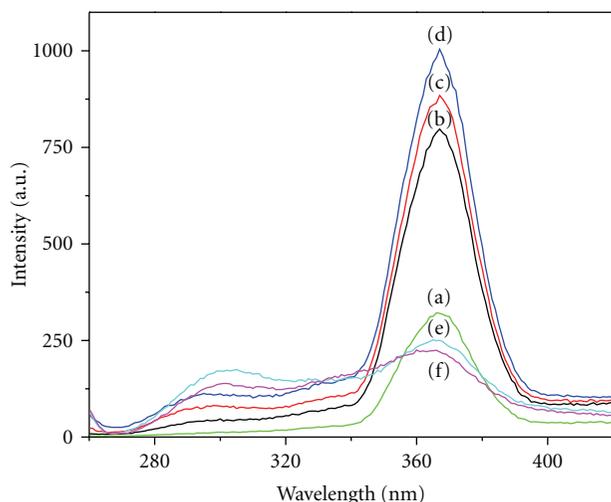


FIGURE 6: FL emission spectra of nanowires with (a) $0 \text{ g}\cdot\text{L}^{-1}$, (b) $0.050 \text{ g}\cdot\text{L}^{-1}$, (c) $0.10 \text{ g}\cdot\text{L}^{-1}$, (d) $0.50 \text{ g}\cdot\text{L}^{-1}$, (e) $1.0 \text{ g}\cdot\text{L}^{-1}$, and (f) $6.0 \text{ g}\cdot\text{L}^{-1}$ EDTA.

reacted with the tellurium atoms, and then released S^{2-} (3). Finally, S^{2-} interacted with the complex to form CdTeS (4), which was proved by XRD results.

What's more, the manipulation of EDTA concentrations can effectively regulate the amount of Te, and the introduction of the hydrothermal route increases the reaction temperature, which in turn results in the acceleration of the dynamic process of the ligands to the surface atoms. Besides, the long- and short-range of interparticle forces lead to the formation, and the mixture of different ligands contributing to the anisotropic growth of nanowires [34–37].

4. Conclusions

In conclusion, we have reported the nonspontaneous synthesis of Te nanowires by using the partially destabilized

hydrothermal method and adjusting the concentration of EDTA. The resulting nanowires exhibit both an excellent uniformity in diameter and a high aspect ratio. The adjustment of the concentration of EDTA offers a convenient means of controlling the morphology of nanowires. The strong optical properties in the blue-violet region may have potential applications in optoelectronic scales labels.

Acknowledgments

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