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An Efficient Templating Approach for Synthesis of Highly Uniform CdTe and PbTe Nanowires

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Highly uniform CdTe nanowires with a very high aspect ratio of ~1000 and an average diameter of 12 nm can be conveniently synthesized using ultrathin Te nanowires as templates via a low-temperature hydrothermal process. Several other interesting CdTe nanostructures, including tadpolelike, chainlike, and branched nanostructures, can also be fabricated by adjusting precursor concentrations and reaction temperature. The formation mechanism of uniform CdTe nanowires using highly reactive ultrathin Te nanowires as template has been discussed. The synthesized uniform CdTe nanowires can be well-dispersed in water or ethanol, and they may find potential applications in the fields of photovoltaics, biological sensors, and nanoscale electronics. The present templating method can also be extended to synthesize other one-dimensional telluride nanostructures such as PbTe nanowires with uniform diameter and high aspect ratios.

1. Introduction

Over the past two decades, semiconductor nanostructures have drawn a lot of research interests because of their unique electronic and optical properties, which are obviously different from either isolated atoms or bulk materials.[1] It is generally accepted that the precise morphology and dimensionality control of nanomaterials is critical to their chemical and physical properties and device functionality. So far, many methods have been developed for design and growth of semiconductor nanostructures with special shapes,[2] among which semiconductor nanowires are of particular interest because of their importance in both fundamental researches and technological applications.[3,4]

CdTe, as a near-infrared band gap semiconductor (E_g = 1.5 eV, 300 K), is an ideal material for photovoltaics,[5] light emitting diodes,[6] biological sensors,[7] and nanoscale electronics.[8] The common strategy for synthesis of high-quality CdTe nanowires is through the so-called solution−liquid−solid (SLS) route developed by Buhro and coworks, in which solution-phase organometallic precursors decompose to produce the desirable semiconductor in the presence of molten catalyst particles at moderate temperature (240−300 °C).[9] In addition, low-temperature aqueous processes, including spontaneous self-assembly of CdTe nanoparticles[10] and direct synthesis of CdTe nanowires using suitable ligands,[11] have been proven to be effective routes for fabrication of CdTe nanowires or nanorods. However, the above methods normally require air-free manipulations using glove boxes or Schlenk lines because of the use of the air-sensitive tellurium precursors such as tri-n-octylphosphine telluride (TOPTe) and NaHTe. Such air-free manipulations significantly increase the complexity as well as the cost of the synthesis of

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CdTe nanowires. Although several other techniques, including template-directed electrodeposition,[12] solvothermal and hydrothermal process,[13] and chemical vapor deposition,[14] have also been applied to produce CdTe nanowires, these methods failed to yield high-quality CdTe nanowires with uniform diameter and high aspect ratio. Recently, we have used ultrathin Te nanowires as templates to selectively synthesize ultrathin noble metal Pt and Pd nanotubes and nanowires with high aspect ratio.[15]

In this paper, we report a high-yield conversion method for synthesis of highly uniform CdTe and PbTe nanowires using highly reactive ultrathin Te nanowires as templates. The formation mechanism of uniform telluride nanowires using highly reactive ultrathin Te nanowires as template has been discussed.

2. Experimental Section

All chemicals were analytical grade and commercially available from Shanghai Chemical Reagent Co. Ltd. and used as received without further purification.

2.1. Synthesis of Ultrathin Te Nanowires. Highly uniform Te nanowires with only several nanometer diameters and a high aspect ratio were prepared according to a procedure developed by our group.[16] Briefly, 1.000 g of polyvinylpyrrolidone (PVP) and 0.089 g of Na_2TeO_3 (0.4 mmol) were dissolved in 35 mL of double-distilled water to form a homogeneous solution under vigorous magnetic stirring at room temperature. Next, 1.65 mL of hydrazine hydrate (85%, w/w%, 28.9 mmol) and 3.35 mL of aqueous ammonia solution were added into the previous solution. The final solution was transferred into a Teflon-lined stainless steel autoclave (50 mL in total volume), which was closed and maintained at 180 °C for 3 h, and the sample was then allowed to cool to room temperature naturally. The chemical yield of Te nanowire templates was determined by the following method. First, 110 mL of acetone was added into the Te nanowires solution to precipitate the product, which was weighed after being centrifuged and dried. The percentage content of Te in the final product was determined by inductively coupled plasma (ICP) measurements. The yield of Te nanowire can then be calculated by the formula: $Y_{(\mathrm{Te})} = \mathrm{wt}\ \%_{(\mathrm{Te})} \times W_1/W_2$. $Y_{(\mathrm{Te})}$ is the yield of Te nanowire, wt $\%_{(\mathrm{Te})}$ is percentage content of Te in the final product, W_1 is actual weight of final product, and W_2 is theoretical product weight of Te nanowires.

2.2. Synthesis of CdTe Nanowires. In a typical synthesis, a quarter of the above dispersed solution of Te nanowires (10 mL) and 0.016 g of $CdCl_2 \cdot 2.5H_2O$ (0.07 mmol) were mixed with 30 mL of double distilled water. The mixture solution was stirred for 4 h at room temperature, and then transferred into a Teflon-lined stainless steel autoclave (50 mL in total volume), sealed, and maintained at 140 °C for 12 h. After being cooled to room temperature naturally, the product was centrifuged and washed several times with double-distilled water and absolute ethanol.

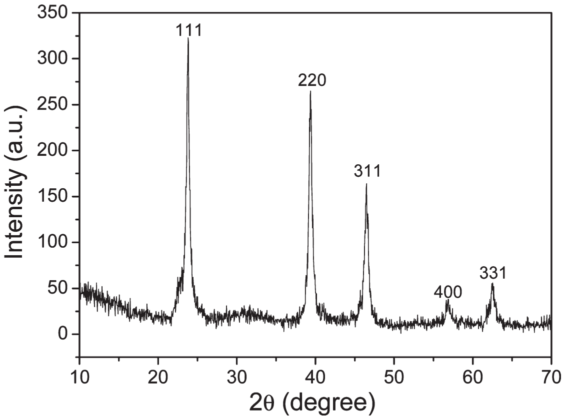

Figure 1. XRD pattern of the product obtained from the reaction of 10 mL of dispersed solution of tellurium nanowires with 0.07 mmol $CdCl_2$ at 140 °C for 12 h.

2.3. Synthesis of PbTe Nanowires. The synthetic route to PbTe nanowires is similar to that of CdTe nanowires. Ten milliliters of the dispersed solution of Te nanowires and 0.07 mmol $Pb(NO_3)_2$ were mixed with 30 mL of double-distilled water. The mixed solution was stirred for 4 h at room temperature, and then transferred into a Teflon-lined stainless steel autoclave (50 mL in total volume), sealed, and maintained at 100 °C for 12 h. After being cooled to room temperature naturally, the product was centrifuged and washed several times with double-distilled water and absolute ethanol.

2.4. Characterization. X-ray power diffraction (XRD) analyses were carried out on a Philips X'Pert PRO SUPER X-ray diffractometer equipped with graphite-monochromatized Cu Kα radiation. Field-emission scanning electron microscopy (FESEM) was carried out with a field-emission scanning electron microanalyzer (JEOL-6700F). Transmission electron microscopy (TEM) and high-resolution TEM (HRTEM) were performed on JEOL-2010 operated at an acceleration voltage of 200 kV. The energy-dispersive X-ray spectroscopy (EDS) analysis was also done with a JEOL-2010 TEM with an Oxford windowless Si (Li) detector equipped with a 4-pulse processor. UV−vis spectra were recorded on a UV-2501PC/2550 at room temperature (Shimadzu Corporation, Japan). X-ray photoelectron spectra (XPS) were recorded on an ESCALab MKII X-ray photoelectron spectrometer, using Mg Kα radiation as the exciting source. Inductively coupled plasma atomic emission spectrometry (ICP-AES) measurements were conducted using an Atomscan Advantage Spectrometer (Thermo Ash Jarrell Corporation, USA).

3. Results and Discussion

Template-directed synthesis has been one of the most popular strategies for fabrication of one-dimensional (1D) nanostructures during the past two decades.[17] Although 1D nanostructures of Se and Te, which can readily form wirelike or tubelike nanostructures because of their anisotropic structure along *c*-axis, have been used as templates to produce various selenide and telluride and noble metal nanostructures with similar morphology,[18] the method described here represents the first attempted synthesis of CdTe nanowires by

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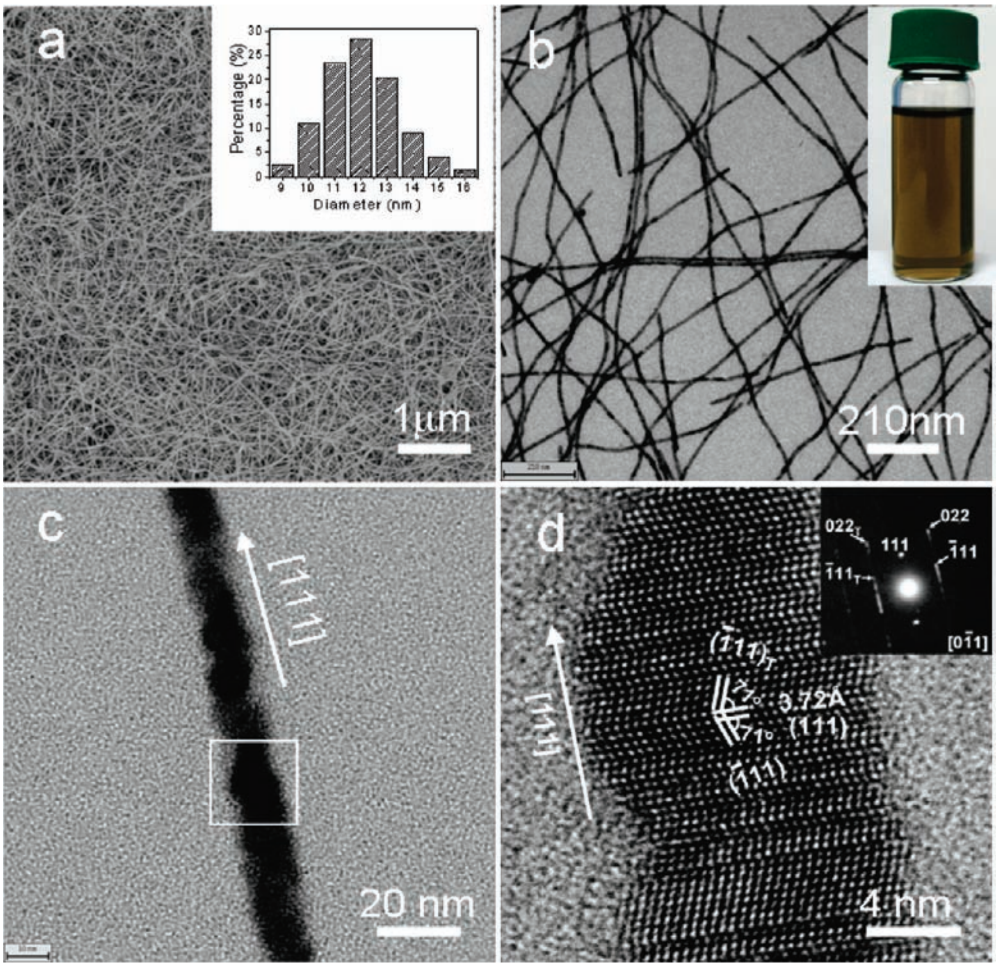

Figure 2. (a) SEM image of the overall morphology of CdTe nanowires. Inset shows the size distribution of CdTe nanowires based on counting 200 nanowires. (b) TEM image of CdTe nanowires. Insert shows the photograph of the dispersed sample. (c) TEM image of an individual nanowire. (d) HRTEM image showing a lattice spacing of 3.72 Å, which was taken from the selected area of the nanowire in (c). Inset in (d) shows the corresponding electron diffraction pattern taken in the individual nanowire.

templating against Te nanowires. High-quality Te nanowires with diameters of only several nanometers and high aspect ratios were first prepared by our group through a polymer-assisted hydrothermal process[16] (see the Supporting Information, Figure S1), which were then used as the templates to synthesize uniform CdTe and PbTe nanowires.

The X-ray diffraction (XRD) pattern of the product in Figure 1 shows that all peaks can be indexed to zinc-blende CdTe with a calculated lattice constant $a = 6.5$ Å, which is consistent with the standard literature values (JCPDS No. 89−3053). The fact that no other diffraction peaks were observed indicates that the obtained product is pure CdTe without impurities, such as Te or $Cd(OH)_2$ crystalline phases. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images a and b in Figure 2 reveal the presence of abundant wirelike nanostructures, which have an average diameter of 12.1 nm with a narrow diameter distribution (inset in Figure 2b). Compared to the template Te nanowires, the resulting CdTe nanowires have a larger diameter as a result of the structural expansion caused by insertion and diffusion of Cd atoms within the structures. These flexible CdTe nanowires can have length up to tens of micrometers, corresponding to an aspect ratio of >1000. These results demonstrate that ultrathin Te nanowires are indeed excellent templates for synthesis of highly uniform CdTe nanowires, which inherit the well-defined 1D morphology and dimensionality of the original templates.

A high-resolution TEM (HRTEM) image in Figure 2d, which was taken from the square part of the single nanowire shown in Figure 2c, shows a lattice spacing of 3.72 Å, corresponding to the lattice spacing of the {111} planes in the zinc-blende CdTe. The result indicates that the nanowires grow along the ⟨111⟩ direction. Also, the angle of planes of (111) and ($\bar{1}$11) is 71°, which is consistent with the calculated value based on its crystal structure. In addition, there are many twins and stacking defects with a high-density perpendicular to the growth direction. The corresponding selected area electron diffraction (SAED) pattern further confirms the above results (inset in Figure 2d). It is noted that planar defects such as twins and staking faults can readily form during crystal growth of metal and semiconductor nanomaterials with a face-centered cubic lattice because of relative low stacking fault or twin boundary energy.[9,19] The energy-dispersive X-ray spectroscopy (EDS) was performed on a random assembly of nanowires, indicating that the product is composed of stoichiometric CdTe (Figure 3).

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The obtained highly uniform CdTe nanowires can be well-dispersed in water and ethanol by ultrasonic treatment for several minutes, which enables subsequent modifications and easy manipulation. The linear absorption spectrum of CdTe nanowires dispersed in ethanol shows an absorption edge at about 800 nm (Figure 4). There is no obvious blue shift of the band edge relative to the bulk band gap of CdTe because the diameter of nanowires described here is close to twice the bulk exciton Bohr radius of CdTe. The fluorescence quantum yield of the obtained CdTe nanowires was so low that the room-temperature photofluorescence was not observed. It has been reported that the photoluminescence of CdTe nanowires with good crystallinity was commonly very weak because of the poor quantum confinement of 1D nanostructures.[9,11]

Generally, the formation of semiconductor nanowires required an anisotropic crystal growth along the wurtzite (001) axis by means of Ostwald ripening or oriented attachment.[10,11] However, in the case of CdTe, the thermodynamically stable phase is zinc-blende. Therefore, the CdTe nanowires or nanorods with wurtzite structure were normally unstable and transferred spontaneously to stable zinc-blende nanoparticles after only several weeks.[11] In contrast, the CdTe nanowires obtained via the template-directed method described here are stable zinc-blende structure and can maintain its 1D structural stability well even after 4 months (see the Supporting Information, Figure S2).

There are three key parameters for successful synthesis of uniform CdTe nanowires with high purity and high aspect ratio, namely, the molar ratio of Na_2TeO_3 to $CdCl_2$, precursor concentration, and reaction temperature. First, the molar ratio of Cd^{2+} ions to Te nanowire precursor had a significant influence on the composition of the products (Figure 5). When the molar ratio of $CdCl_2$ to Na_2TeO_3 was lower than 0.6, Te nanowire templates that were prepared in the first step could not be consumed completely during the next reaction (Figure 5a). Pure CdTe nanowires with uniform diameter can be obtained when the molar ratio of $[CdCl_2]$: $[Na_2TeO_3]$ was increased to 0.7:1, which corresponds to the molar ratio of $[CdCl_2]$: $[Te] = 1:1$, considering that the yield of Te nanowires is about 73% in the synthesis of precursor Te nanowires (Figure 5b and Figure 2). Further increasing the molar ratio of $[CdCl_2]$: $[Na_2TeO_3]$ to 0.8:1 or 1:1 will result in a mixture of CdTe and $Cd(OH)_2$, as shown in patterns c and d in Figure 5. This was because the excessive cadmium ions can combine with hydroxide ions to form $Cd(OH)_2$ precipitate.

Furthermore, the precursor concentration was found to be another important parameter that dramatically influenced the morphologies of the final products (Figure 6). Low precursor concentrations were essential for synthesis of CdTe nanowires with uniform diameters. Upon increasing the concentrations of $[CdCl_2]$ from 1.75 mM to 3.50 or 5.25 mM, a kind of interesting tadpolelike CdTe 1D nanostructures was produced (images b and c in Figure 6). Further increasing the concentration of $[CdCl_2]$ up to 7.00 mM will result in the formation of branched CdTe nanostructures (Figure 6d). It is noteworthy that such CdTe nanostructures, which were produced generally by mixing zinc blende and wurtzite phase in organic media,[20] has potential advantages in hybrid nanocrystal/polymer photovoltaic devices because its 3D structure can provide a direct charge-carrier transportation pathway to improve the device performance.[21]

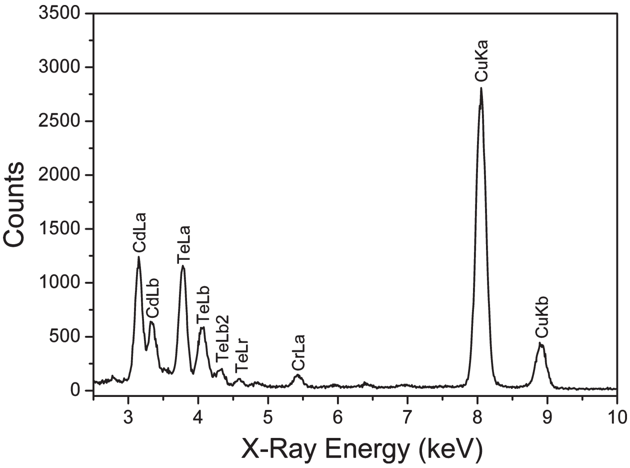

Figure 3. EDS spectrum of the CdTe nanowires. The signals corresponding to Cu and Cr arise from the TEM grid.

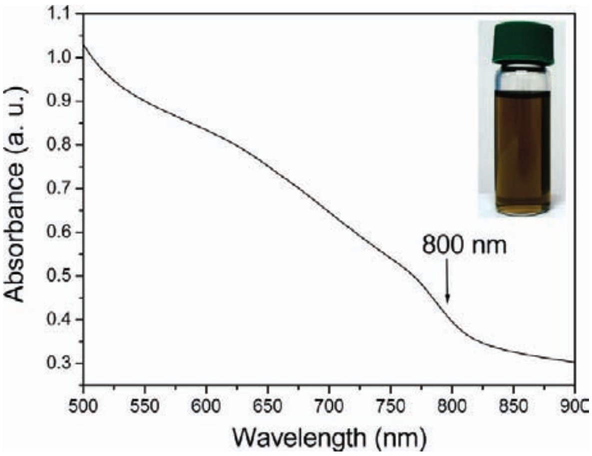

Figure 4. Absorption spectrum of CdTe nanowires dispersed in ethanol. The inset is the optical photograph of the suspension of CdTe nanowires in ethanol.

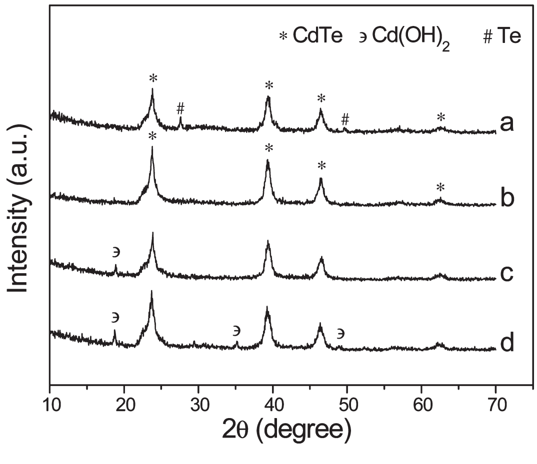

Figure 5. XRD patterns of the products obtained from the reactions of 10 mL of tellurium nanowires solution with various amounts of $CdCl_2$ at 140 °C for 12 h: (a) 0.06, (b) 0.07, (c) 0.08, and (d) 0.10 mmol.

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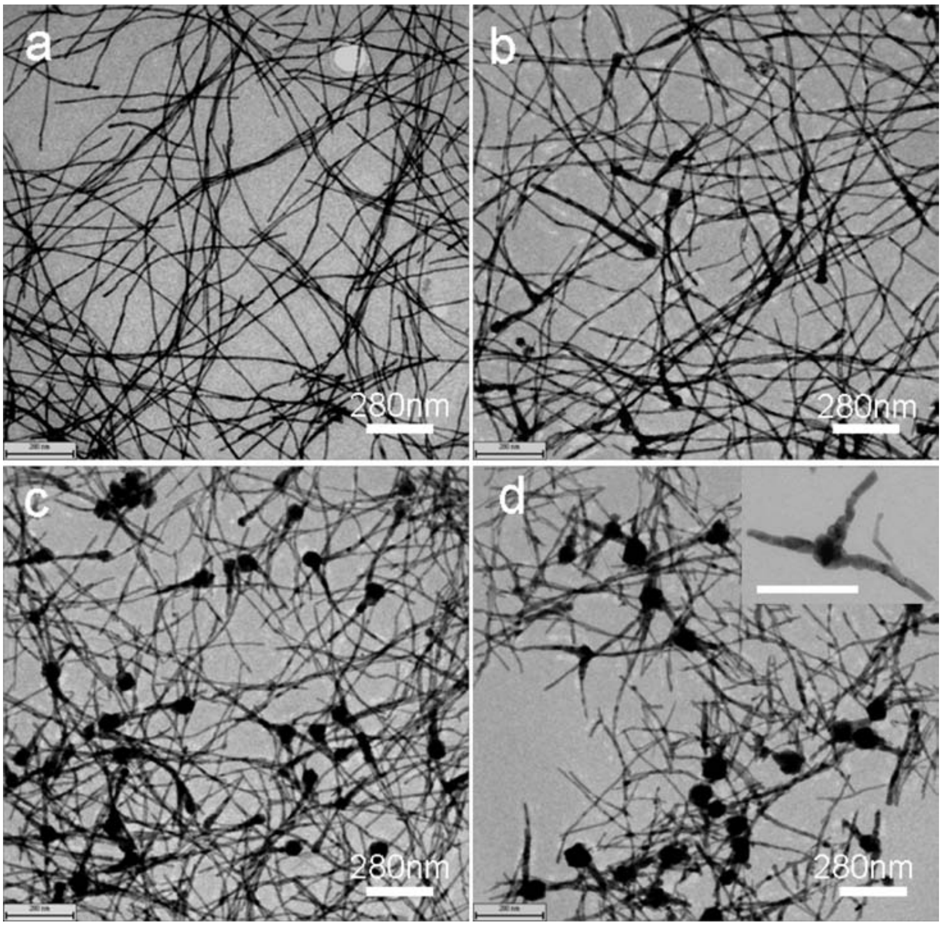

Figure 6. (a) TEM images of four samples obtained by hydrothermal treatment of the precursor solutions with different concentrations at 140 °C for 12 h. The concentration of $CdCl_2$ is (a) 1.75, (b) 3.50, (c) 5.25, and (d) 7.00 mM, respectively. The molar ratio of Na_2TeO_3:$CdCl_2$ was fixed at 1:0.7, which corresponds to the molar ratio of Te:$CdCl_2$ = 1:1 in the conversion reaction. The inset in (d) shows a magnified TEM image of a single branched CdTe nanostructure. The scale bar in (d) is 280 nm.

To obtain CdTe nanowires with high aspect ratio, the reaction should be performed at appropriate temperature, namely, 140 °C (see the Supporting Information, Figure S3a). The reaction cannot occur at the temperatures below 120 °C. When the temperature was increased to 160 °C, some nanowires became rough and broke into chainlike structures (see the Supporting Information, Figure S3b). With further increasing temperatures to 180 or 200 °C, more and more short CdTe nanowires and nanochains were yielded (see the Supporting Information, Figure S3c,d).

In short, different CdTe nanostructures could be selectively synthesized by adjusting the precursor concentration and reaction temperature. Although the exact formation mechanism of these nanostructures is still not completely understood at present, we proposed a plausible explanation described as follows on the basis of the present experimental results. Increasing the precursor concentration or reaction temperature will accelerate the reaction rate, resulting in the fracture of original 1D nanowire templates and further leading to the formation of CdTe nanoparticles or short nanorods. Then those CdTe fragments recombine into nanochains or branched nanostructures via the so-called oriented-attachment mechanism.[22]

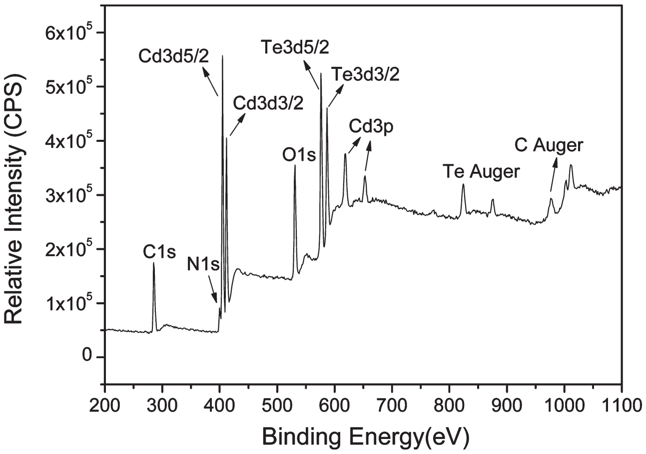

Figure 7. XPS spectrum of the dried sample prepared by separation of Te nanowires from the solution after they adsorbed with Cd^{2+} ions before conversion reaction.

The experiments on examining whether Cd^{2+} ions adsorb on the PVP coated Te nanowires in the reaction or not have been carried out. The $CdCl_2 \cdot 2.5H_2O$ precursor was added into a freshly prepared Te nanowires solution and the mixed solution was stirred for 4 h at room temperature. The product was then dried for the XPS measurement after being centrifuged and washed 3 times with double-distilled water. The XPS spectrum showed that the two strong peaks at 405 and

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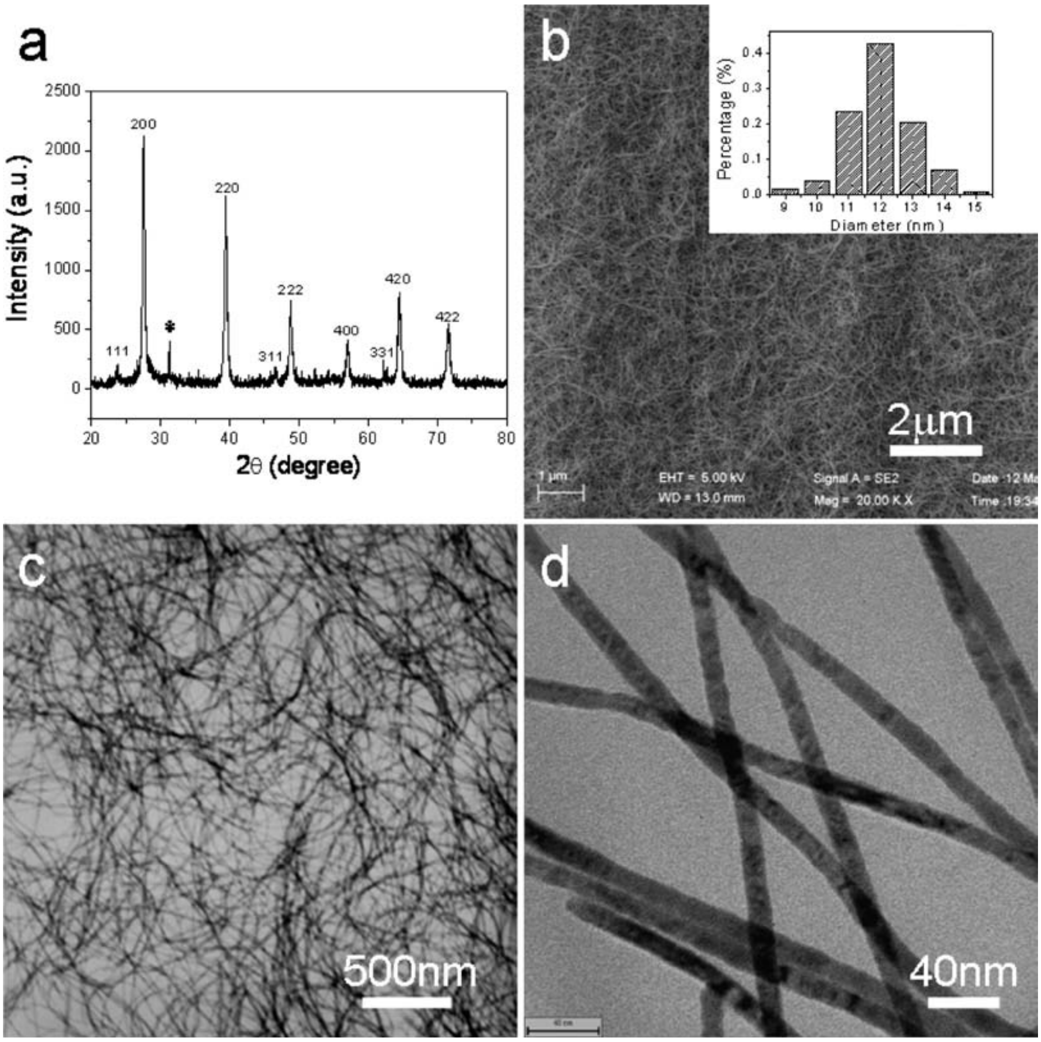
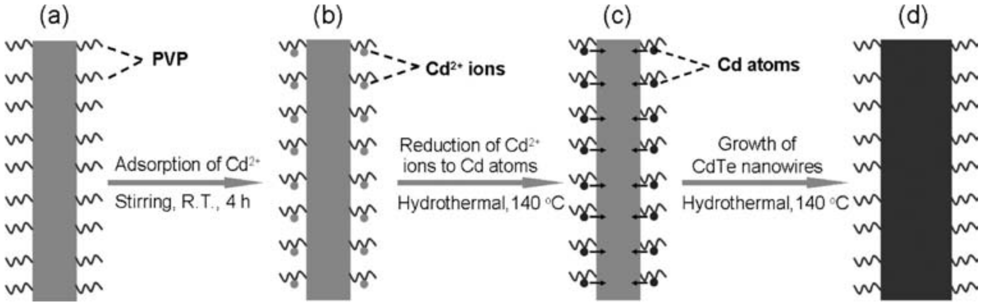


Figure 8. (a) XRD pattern of the product obtained from the reaction of 10 mL of the tellurium nanowire solution with 0.07 mmol $Pb(NO_3)_2$ at 100 °C for 12 h. * indicates a trace amount of Pb_3O_4. (b) SEM image of the overall morphology of PbTe nanowires. Inset shows the size distribution of PbTe nanowires based on counting 200 nanowires. (c, d) TEM images of PbTe nanowires with different magnifications.

Scheme 1. Schematic Illustration of the Fabrication of CdTe Nanowires via a Template-Directed Process



412 eV correspond to Cd(3d) binding energy, indicating that the Cd^{2+} ions can adsorb on the backbone of PVP coated Te nanowires before the conversion reaction occurs (Figure 7). The ICP measurement showed that about 40.6 wt % Cd presents in the dried sample also. These results confirmed that the Cd^{2+} ions can be efficiently loaded on the backbone of Te nanowires coated with PVP.

Up to now, there are two main mechanisms for the transformation from Te (or Se) nanowires to 1D metal telluride (or selenide) nanostructures. One is that Te (or Se) disproportionates into Te^{2-} and TeO_3^{2-} ions (or Se^{2-} and SeO_3^{2-}), and Te^{2-} (or Se^{2-}) reacts with metal ions to form metal telluride or selenide.[18] The other one is that metal ions are reduced to atoms first, and then react with Te or Se nanowires in situ to generate metal telluride or selenide 1D nanostructures.[18,23,24] For understanding the formation process of CdTe nanowires here, the default experiment in the

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absence of $N_2H_4 \cdot H_2O$ and $NH_3 \cdot H_2O$ has been carried out. The fact that no any CdTe crystalline structures were obtained excludes the former mechanism. The latter one is assumed to be reasonable to illustrate the present synthetic process as proposed in Scheme 1. When added $CdCl_2$ into the Te nanowires solution, the cadmium ions were trapped by the PVP near the surface of Te nanowires with vigorous magnetic stirring (illustrations a and b in Scheme 1), which was confirmed by X-ray photoelectron spectrum (XPS) measurement (Figure 7). It is reasonable to believe that $N_2H_4 \cdot H_2O$ is still abundant in the solution compared to Cd or Te precursors after the first step for synthesis of Te nanowires (see the calculation shown in the Supporting Information). In addition, the standard electrode potentials of Cd^{2+}/Cd and N_2/N_2H_4 are −0.403 and −1.15 V, respectively.[24] Thus these trapped cadmium ions could be reduced to cadmium atoms by $N_2H_4 \cdot H_2O$ in the presence $NH_3 \cdot H_2O$, which continuously diffused into the lattice of Te nanowires and react to yield CdTe nanowires (Scheme 1c,d). The reactions occurred under the present conditions can be formulated as follows

$$2Cd^{2+} + N_2H_4 + 4OH^- \rightarrow 2Cd + N_2 + 4H_2O \qquad (1)$$

$$Cd + Te(nanowires) \rightarrow CdTe(nanowires) \qquad (2)$$

Furthermore, the present templating method can also be extended to fabricate other one-dimensional telluride nanostructures such as PbTe nanowires with uniform diameter and high aspect ratio. The reaction of Te nanowires with Pb^{2+} is similar to that of Te with Cd^{2+}

$$2Pb^{2+} + N_2H_4 + 4OH^- \rightarrow 2Pb + N_2 + 4H_2O \qquad (3)$$

$$Pb + Te(nanowires) \rightarrow PbTe(nanowires) \qquad (4)$$

Figure 8a shows the XRD pattern of the product obtained from the reaction tellurium nanowires solution with $Pb(NO_3)_2$. The diffraction peaks can be indexed to face-centered-cubic (fcc) phase of pure PbTe, with a lattice constant of a = 6.5 Å (JCPDS no. 78−1905). The trace amount of tetragonal Pb_3O_4 phase was detected, which is likely resulted from the oxidation of Pb during the hydrothermal process.[18,25] SEM and TEM images reveal the presence of abundant of uniform PbTe nanowires with an average diameter of 12.2 nm and an aspect ratio of several thousand (Figure 7b−d). The EDS spectrum in Figure 9 taken on a random assembly of PbTe nanowires indicates that the molar ratio of Pb to Te is 50.8:49.2, which very closes to 1:1 (Figure 9).

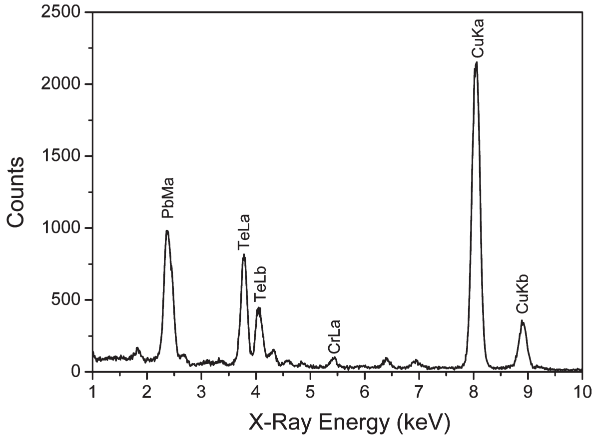

Figure 9. EDS spectrum of the PbTe nanowires. The signals corresponding to Cu and Cr arise from the TEM grid.

4. Conclusions

In conclusion, we have demonstrated a simple templating route to prepare highly uniform CdTe nanowires using ultrathin Te nanowires as the templates via a low-temperature hydrothermal process. The molar ratios between Te and $CdCl_2$, precursor concentrations, and reaction temperatures have crucial influences on the composition and morphology of the products. The resultant zinc-blende CdTe nanowires with a high aspect ratio of ~1000 and an average diameter of 12 nm can maintain their 1D structure well, even after being stored in ethanol for several months. Similarly, this route can be extended to synthesize other one-dimensional telluride nanostructures such as PbTe nanowires with uniform diameters and very high aspect ratios. These one-dimensional telluride nanostructures may find applications in photovoltaics, biological sensors, and nanoscale electronics.

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Supporting Information Available: Size distribution histograms and TEM and SEM images of the samples (PDF). This material is available free of charge via the Internet at http://pubs.acs.org.

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