Chapter 2
Synthesis of One-Dimensional Te Nanostructures

2.1 Introduction

In the recent years, one-dimensional semiconductors, such as nanowires, nanorods, and nanotubes, have received wide attention because of their promising potential applications in basic scientific research. The nanowires with diameter of 1–100 nm, a large surface area, and one-dimensional conduction have great prospects in the preparation of numerous devices. Moreover, nanowires exhibit high reactivity and can act as ideal platform to transmit microscopic particles due to their unique structural characteristics.

In general, tellurium is a spiral p-type semiconductor with a configuration of $4d^{10}\ 5s^2\ 5p^4$ and band gap energy of 0.35 eV at room temperature [1]. The crystal structure of tellurium shows highly anisotropic nature, comprising helical chains of covalently bound atoms resulting in t-Te inherent chirality, as well as a strong tendency toward 1D growth. Moreover, the peculiar properties of tellurium such as unidirectional image conductivity, nonlinear optical response, and high thermal conditions to produce piezoelectric response make it promising material for use in optoelectronic devices, gas sensors, field-effect devices, photonic crystals, radiation-cooled instruments, self-developing holographic instruments, and infrared acoustic-optic deflectors [2, 3].

Over the last several years, the several methods have been developed for the successful synthesis of Te nanocrystals in a rich variety of shapes such as zero-dimensional nanoparticles, one-dimensional nanostructures, two-dimensional nanostructures, and even more complex hierarchical structures. By using a variety of synthesis methods such as solution-phase approach, refluxing process [4], microwave-assisted synthesis in ionic liquids [5], biomolecule-assisted routes [6], visible-light-assisted technique, and vapor-phase growth [7], many Te nanostructures have been synthesized very efficiently.

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2.2 Microwave-Assisted Synthesis of Te Nanowires

The microwave chemistry has been accepted as a promising method for the liquid-phase preparation of in organic materials due to its definite advantages such as high reaction rate, low processing cost, and high yield. This rapid, efficient, and nonpolluting strategy quickly provides the homogenous solvent, uniform nucleation, and growth by the microwave irradiation of medium. It is widely used for the synthesis of a large number of nanostructures. Microwave synthesis of nanostructures has been studied by many scientists, for example, Hu and coworkers have developed the microwave-assisted ionic liquid (MAIL) method for the production of Te nanorods and nanowires [5]. Up to now, it has not been possible to synthesize high-yield, high-quality tellurium nanowires in the aqueous phase by microwave method.

With the utilization of microwave approach, Yu and coworkers have synthesized high-quality Te nanowires in water phase in high yield without using ionic liquids (Fig. 2.1a, b) [8]. The monocrystalline tellurium nanowires with a diameter of 20 nm and a length of several tens of micrometers can be synthesized by the microwave-assisted reaction by using Na_2TeO_3 as tellurium source, polyvinylpyrrolidone (PVP) as a surfactant and hydrazine hydrate as reducing agent in a microwave-sealed vessel made of quartz at 150 °C by microwave. The construction of these high-quality tellurium nanowires is extremely dependent on the reaction conditions such as the amount of PVP, the reaction time, the pH of the initial solution, and the choice of surfactants.

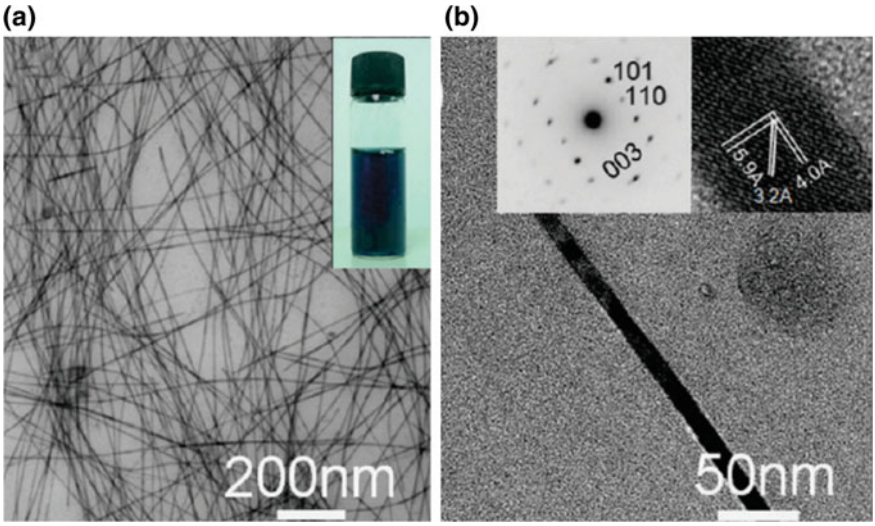


Fig. 2.1 **a** TEM image of the Te nanowires; **b** a single Te nanowire. Reprinted with permission from Ref. [8] Copyright 2010 American Chemical Society

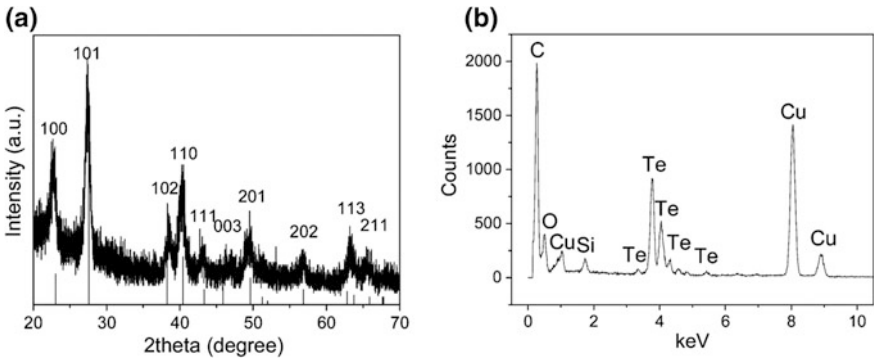


Fig. 2.2 XRD and EDS spectra of synthesized nanowires. Reprinted with permission from Ref. [8] Copyright 2010 American Chemical Society

The reaction proceeds with the reduction of Na_2TeO_3 by hydrazine. The XRD spectra presented in Fig. 2.2 shows the formation of Te nanowires with a constant hexagonal tellurium phase by $a = 4.0$ Å and $b = 5.9$ Å. Similarly, an energy-dispersive X-ray spectroscopy (EDS) spectrum shows the strong tellurium peaks, exhibiting the product as pure Tellurium.

Tellurium nanowires synthesized by microwave methods form a deep blue color solution on dispersion in water that is a characteristic color as found previously for ultrathin Te nanowires synthesized by the hydrothermal method, indicating the successful synthesis of Te nanowires by microwave method [9]. Moreover, Te nanowires show high aspect ratio with the diameter of 20 nm, and lengths up to tens of micrometers (Fig. 2.1a) with the lattice spacing of ca. 5.9, 4.0, and 3.2 Å, analogous to the lattice spacings of the (001), (100), and (101) planes for hexagonal tellurium, respectively. The plane perpendicular to the axis of the nanowire has a spacing of 0.59 nm, which is in agreement with the (001) crystal plane, showing the monocrystalline nature of tellurium nanowire (Fig. 2.1b).

2.3 Solution-Based Synthesis of Te Nanostructure

Te nanostructure with different morphologies such as nanoparticles, nanorods, and nanowires can be synthesized by using a simple solution-based approach that involves the hydrothermal treatment of a mixture containing PVP, Na_2TeO_3, NaOH, hydrazine hydrate, and ethylene glycol (EG) at 70 °C for 15 s to 3 h [10]. Moreover, the diameter of nanowires changes drastically by the addition of acetone to the reaction mixture. Figure 2.3a–e shows the SEM and TEM images of Te nanostructure indicating the change in morphology with an increase in time. Initially, at the time of 15 s, Te nanoparticles with the diameter of 7 ± 3 nm are obtained, which change to nanorods as the reaction time increases gradually. The

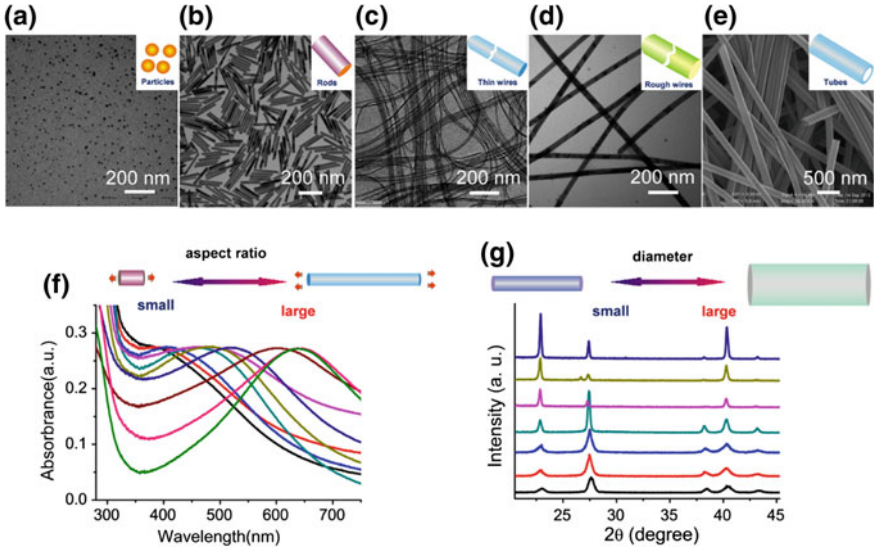


Fig. 2.3 **a**–**d** TEM images of Te nanostructures with different morphologies. **e** SEM image of Te nanotubes. **f** UV–Vis spectrum of Te nanostructures, from nanoparticles to nanorods with different aspect ratio. **g** XRD patterns of the as-synthesized Te nanowires with different diameters. Reprinted with the permission from Ref. [10]. Copyright 2016 WILEY-VCH

formation of Te nanorods is due to the anisotropic crystal structure of tellurium, which contains helical chains of covalently bound tellurium atoms. The chains have a strong tendency to grow along the *c*-axis direction into a one-dimensional structure [11]. Furthermore, with the increase in aspect ratio of nanorods, the absorption peak of *UV–Vis* spectra shifts from 388 to 639 nm, indicating that the UV–Vis spectra track the increase in reaction time (Fig. 2.3f). Also the diameter of nanowire changes from 7 to 150 nm with the addition of acetone and the nanotubes will be formed as the amount of acetone continues to increase (more than 50% of the total system) due to the increase in (100) proportion compared to (001) (Fig. 2.3g).

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