Tellurium Nanorods and Nanowires Prepared by the Microwave-Polyol Method

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Tellurium nanorods and nanowires have been successfully prepared in liquid phase by the microwave-polyol method without using any seed, or template or surfactant. The reduction of tellurium dioxide by ethylene glycol and formation of Te nanorods and nanowires were achieved by microwave heating at 185 °C for 30 min. The as-prepared samples were characterized by X-ray powder diffraction and transmission electron microscope.

The properties of nanostructured materials show a strong dependence not only on size but also on shape of building blocks.[1–3] Since the discovery of carbon nanotubes in 1991,[4] one-dimensional (1-D) nanostructures, such as nanorods, nanowires, and nanotubes, have been intensively studied because of their potential applications as important components and interconnects in nanodevices.[5–8]

Tellurium is a p-type semiconductor with many useful and interesting properties, for example, photoconductivity, catalytic activity toward some reactions, high piezoelectricity, thermoelectricity, and nonlinear optical responses. In addition, Te reacts readily with other elements to generate many functional materials. Recent progress has been made with the preparation of 1-D nanostructures of Te.[9–13]

Microwave heating is promising because of its unique effects compared with the conventional heating, such as rapid volumetric heating, higher reaction rates, shortened reaction time, and energy saving. The microwave-polyol method has recently been used to prepare nanoparticles of Pt, Ag,[14] CdSe,[15] $Cd_{1-x}Zn_xSe$,[16] TiO_2,[17] $CuInSe_2$, $CuInTe_2$,[18] HgS, and PbS.[19] However, these nanoparticles prepared were spherical in shape instead of nanorods or nanowires. In this paper, we demonstrate that the microwave-polyol method can be used to prepare Te nanorods and nanowires without using any seed or template or surfactant. This makes it possible to avoid subsequent complicated workup procedures for the removal of templates, seeds or surfactants.

Analytical grade of tellurium dioxide and ethylene glycol (EG) were purchased and used as received without further purification. The mixture of the reagents was placed in a 100-mL round-bottomed flask and heated to 185 °C and held at this temperature for 30 min by microwave heating. The microwave oven used was a focused single-mode microwave synthesis system (Discover, CEM, USA). The unique, circular single-mode cavity focuses the microwave on the reactants, ensuring the sample is in a homogenous highly dense microwave field. Temperature was accurately controlled by automatic adjusting of microwave power. The microwave synthesis system was equipped with a water-cooled condenser outside the microwave cavity and a magnetic stirring system. White reactant TeO_2 was transformed to black suspension after microwave heating. The products were separated by centrifugation, washed with absolute ethanol several times, and dried at 80 °C in vacuum.

X-ray powder diffraction (XRD) patterns were recorded in 2θ range from 20 to 70°, using a D/max 2550V X-ray diffractometer with high-intensity Cu Kα radiation ($\lambda = 1.54178$ Å) and a graphite monochromator. Transmission electron microscope (TEM) images were taken with JEOL JEM-2010 and JEM-200CX instruments, using an accelerating voltage of 200 kV.

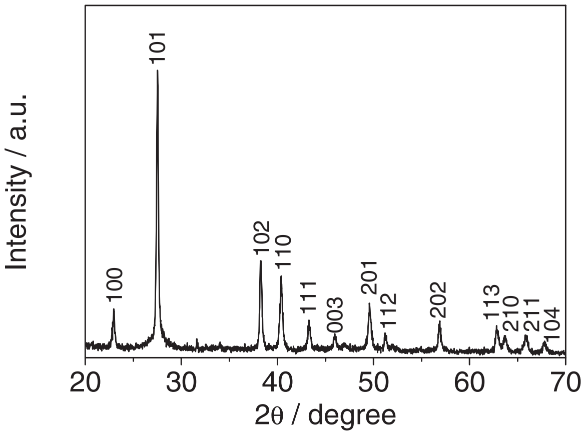

Figure 1. XRD pattern of a typical sample (sample 1) prepared by microwave-heating a mixture of 0.638 g TeO_2 and 40 mL EG at 185 °C for 30 min.

Figure 1 shows XRD pattern of sample 1 prepared by microwave-polyol method. It indicates a single phase of well-crystallized elemental Te with the hexagonal structure. No TeO_2 was detected by XRD. Other samples prepared by changing the amount of TeO_2 have XRD patterns similar to Figure 1. The yield of Te powder collected after washing and drying was usually higher than 90%.

The morphologies of samples were investigated with TEM. Figure 2 shows TEM micrographs and electron diffraction patterns of as-prepared Te samples. Figures 2a and 2b are for samples 2 and 1, respectively. From Figure 2a, one can see Te nanorods with diameters of 30 to 80 nm and with lengths ranging from several hundreds nanometers to ≈1 µm. The aspect ratios of these nanorods are in the range of ≈3 to ≈20. The aspect ratio is defined as the length of the major axis divided by the width of the minor axis. Nanorods are defined as structures that have widths of 1–100 nm and aspect ratios greater than 1 but less than 20; and nanowires are analogous structures that have aspect ratios greater than 20.[20] The aspect ratio of Te nanostrucrures increased by increasing the amount of TeO_2 (see Figures 2a and 2b). Te nanowires (sample 1) with diameters of ≈40 to ≈110 nm and lengths up to ≈10 µm formed (Figure 2b). Figure

2c shows TEM image of an individual Te nanorod (sample 2) with a diameter of ≈70 nm and a length of ≈0.75 μm, its corresponding electron-diffraction pattern is shown in Figure 2d, which was obtained by orienting the crystal along the [210] direction. It confirms the hexagonal structure of Te nanorods, consistent with the result obtained from XRD. The electron-diffraction patterns of different individual Te nanorods and nanowires were essentially the same, indicating that Te nanorods and nanowires were single-crystalline in structure and that Te nanorods and nanowires had preferential growth direction along the [001] (*c* axis of the crystal lattice).

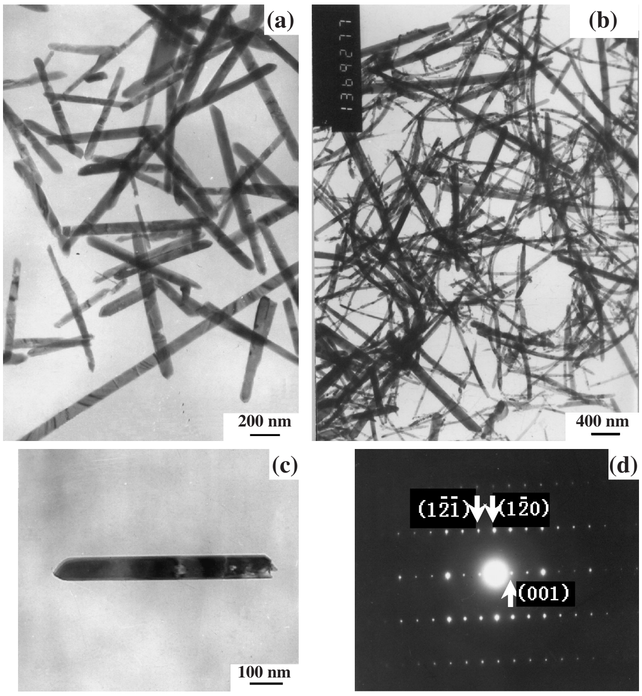

Figure 2. TEM micrographs of Te prepared by the microwave-polyol method. (a) is for sample 2 prepared by microwave-heating of a mixture of 0.128 g TeO_2 and 40 mL EG at 185 °C for 30 min. (b) is for the same sample as in Figure 1 (sample 1). (c) TEM micrograph of an individual Te nanorod from sample 2. (d) Electron-diffraction pattern of the same individual Te nanorod as shown in Figure 2c.

In summary, we have shown that Te nanorods and nanowires can be prepared in liquid phase by a seedless, template-less and surfactant-free microwave-polyol method at 185 °C in a relatively short processing time (30 min). The aspect ratio of Te nanostructures can be well controlled by adjusting experimental parameters. The reduction of TeO_2 by EG to form Te was achieved by microwave-heating at 185 °C. The yield of Te powder collected after washing and drying was usually higher than 90%. This simple, fast and high-yield method is suitable for large-scale production of Te nanorods and nanowires. It may also be extended to synthesize other kinds of 1-D nanostructures.

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