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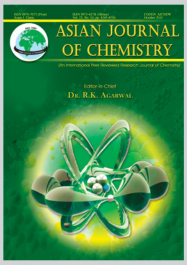

Controlled Synthesis of Different Submicron Crystalline Tellurium Structures by Adjusting Solution pH Value†

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A new approach has been developed for the preparation of tellurium with various morphologies by a simple hydrothermal method using Na_2TeO_3 in ethylene glycol at 180 ºC. In this method, ethylene glycol acts not only as a surfactant but also as a reducing reagent, thus no additional reductants are needed. By control of the reaction conditions, especially, by adjusting of solution pH value, submicro brooms, submicro-channels and micro nanorods have been prepared.

Key Words: Submicro materials, Hydrothermal method, pH value.

INTRODUCTION

There has been a surge of research activity related to the synthesis and characterization of nanorods and nanowires of various inorganic materials such as oxides, sulfides and nitrides as well as elemental materials[1]. Among the elemental materials, different shapes of metals such as Au, Ag and Pd as well as non-metals such as Se have been prepared and characterized[2]. One of the elemental materials of interest is tellurium which has several interesting chemical and physical properties including photoconductivity and catalytic activity as well as piezoelectric, thermoelectric and non-linear optical responses[3]. Recently, some references have reported the preparation of tellurium nanocrystals through different routes[4], tellurium nanotubes and nanorods have been synthesized by a refluxing polyol process, tellurium nanobelts and nanotubes have been prepared *via* a disproportion reaction in ammonia solution[5], tellurium nanocrystals have been prepared by the microwave-assisted ionic liquid and the ultrasonic-assisted method[6].

In this paper,we report the investigation of the reducing ability of ethylene glycol and in the synthesis of tellurium from Na_2TeO_3 by the hydrothermal method using ethylene glycol as both a reducing reagent and a surfactant without any additional reductant. At the same time, through adjusting pH value of the solution, submicro brooms, submicro-channels and micro nanorods have been obtained.

EXPERIMENTAL

All chemicals used in present experiments were purchased and used as received without further purification. In a typical procedure, 0.5 mmol Na_2TeO_3, 20 mL ethylene glycol, 5 mL distilled water and 0.5 mL of 12 M HCl were added into a 30 mL Teflonlined stainless-steel autoclave. The autoclave was sealed and maintained at 180 ºC for 12 h, then allowed to cool to room temperature in air. After the reaction, the solution was filtered, washed by deionized water and absolute ethanol, respectively and dried in a vacuum at 80 ºC for 6 h.

Characterization: X-Ray powder diffraction patterns (XRD) of the products were obtained on a Japan Rigaku D_{max}-γA rotation anode X-ray diffractometer equipped with graphite monochromatized CuK_α radiation ($\lambda = 1.54178$ Å). The field-emission scanning electron microscope (FE-SEM) measurements were carried out with a field-emission microscope (JEOL, 7500B) operated at an acceleration voltage of 10 kV.

RESULTS AND DISCUSSION

Fig. 1 shows the X-ray diffraction pattern of the as-prepared samples. The peak positions are consistent with a hexagonal phase of tellurium (space group: P3121(152)). The lattice constants calculated from the diffraction pattern area = 4.4512, b = 5.9177, compatible with the literature values of a = 0.4458 nm, b = 0.5927 nm (JCPDS 36-1452). The XRD

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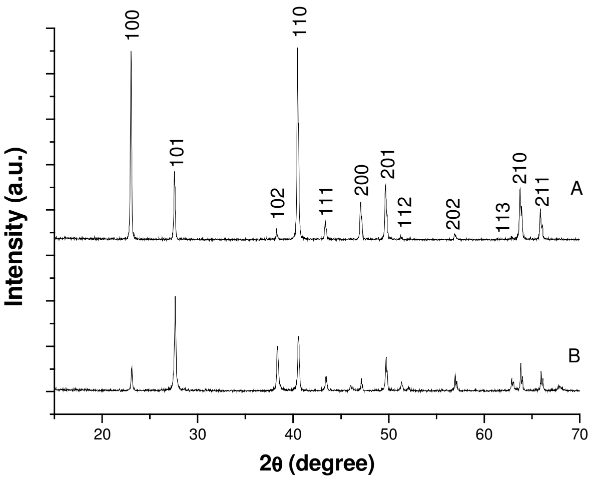
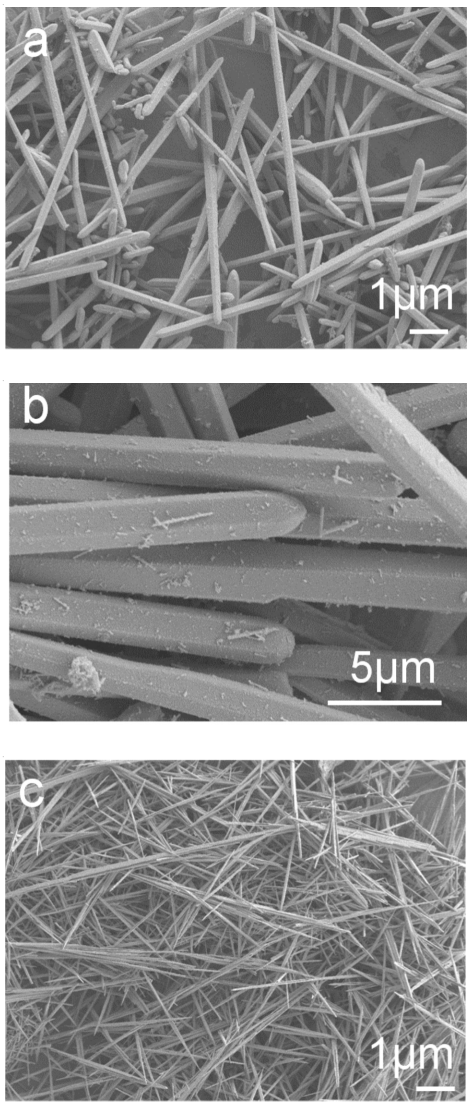


Fig. 1. XRD pattern of the tellurium products, (A) acid treatment, (B) alkali treatment

pattern indicates that pure tellurium products were obtained by acid and alkali treatment.

The morphologies of the prepared samples with acid treatment were investigated by field-emission scanning electron microscopy (SEM) (Fig. 2).



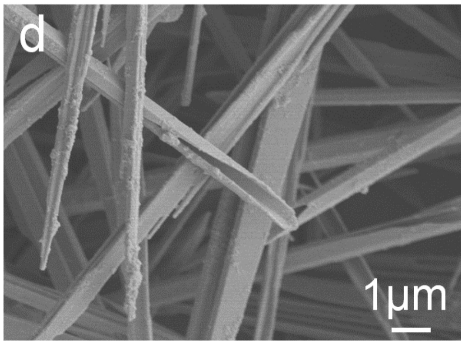
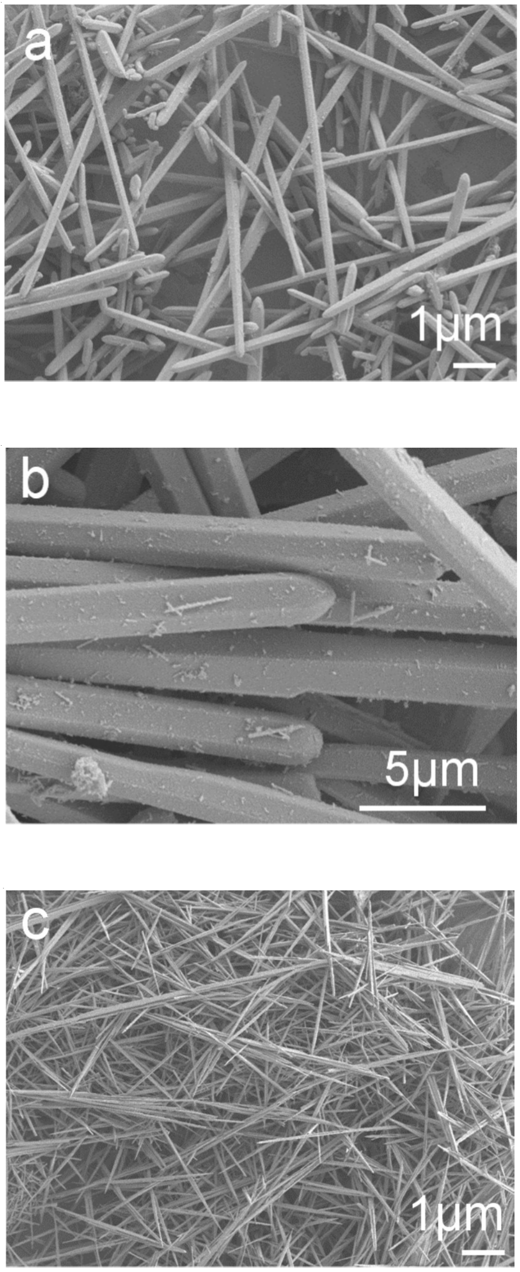


Fig. 2. FE-SEM images of Te synthesized under different conditions by acid treatment. (a,b) Te nano-brooms VEG:Vwater:VHCl = 20:5:0.5, (c,d) Te nanochannals VEG: Vwater:VHCl = 20:5:0.01

The morphologies of the prepared samples with alkali treatment were investigated by field-emission scanning electron microscopy (SEM) (Fig. 3).



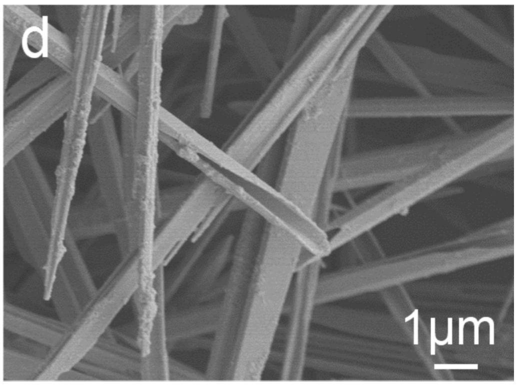


Fig. 3. FE-SEM images of Te synthesized under different conditions by acid treatment. (a, b) Te nano-brooms VEG:Vwater = 20:5, NaOH, 0.5 g (c, d) Te nanochannals VEG: Vwater = 20:5, NaOH, 0.01 g

Conclusion

In summery, we have shown that Te submicro brooms, submicro-channels and micro nanorods can be prepared in liquid phase by a seedless, template-less and hydrothermal method at 180 ºC. The various morphologies of Te can be well controlled by different amouts acid or alkali treatment. The reduction of Na_2TeO_3 by ethylene glycol to form Te was achieved by hydrothermal method at 180 ºC. It may also be extended to synthesize other kinds of nanostructures.

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