Note

Negative Feedback Effect of Microwave Irradiation in the Microwave-assisted Hydrothermal Synthesis of Bi_2S_3 Nanorods

TAO, Xiu-Cheng(陶秀成) **SHAO, Ming-Wang***(邵名望)

College of Chemistry and Material Science, Anhui Normal University, Wuhu, Anhui 241000, China

The microwave-assisted hydrothermal synthesis of Bi_2S_3 nanorods was reported. The result showed that microwave irradiation can help to produce Bi_2S_3 nanorods in very short time. There is a negative feedback effect which increases the degree of crystallinity in the reaction.

Keywords Bi_2S_3 nanorods, hydrothermal synthesis, microwave irradiation

Introduction

Nanocrystalline materials display the unique properties as compared with bulk phase. Group V—VI compounds, especially Bi_2S_3 with energy gap (E_g) of 1.3 eV, have widely been studied because of their excellent properties like photoconductivity, photosensitivity, infrared (IR) spectroscopy and thermoelectric power.[1-4]

There are many ways to prepare Bi_2S_3: direct method of bismuth and sulfur vapor in a quartz vessel at high temperature;[5] thermal degradation of metal complexes needing to be processed in a stream of H_2S or H_2/H_2S at high temperature;[6] single-source method using expansive precursor,[7] solvothermal method with organic solvent,[8] and high energy methods such as electrochemical fabrication[9] and asynchronous-pulseultrasonic spray pyrolysis.[10]

Hydrothermal method is in a distinctive position to prepare nanocrystalline materials. It is one of the most promising solution chemical methods, by which organometallic or toxic precursors are not required and reactions may be carried out at comparatively low temperature.

However, to our knowledge, many reactors in hydrothermal method are static. It may bring large colloid size distribution, low crystallization rate and worse crystallization condition.[11] So the liquid solution in the syntheses of Bi_2S_3 often yield products that are mostly amorphous, poorly crystallized, or colloidal particles.[12,13]

In this study, we employ microwave-assisted hydrothermal method to produce Bi_2S_3 nanorods in short time.

Experimental

Experiment details were as follows: 0.01 mol of analytical pure $BiCl_3$ (0.005 mol of EDTA was added to prevent $BiCl_3$ from hydrolysis) and 0.015 mol of Na_2S were dissolved in distilled water respectively. Then solutions were mixed while stirring. The mixture was filled in a Teflon-lined autoclave of 50 mL capacity to 90% of the total volume.

The reactor was under 500 W microwave (2450 MHz) irradiation for 8 min and then cooled to room temperature naturally. The precipitate was washed with distilled water. The product was dried *in vacuo* at 60 ℃ for 1 h.

Results and discussion

The product was characterized by powder X-ray diffraction (XRD) patterns at a scanning rate of $0.02° \cdot s^{-1}$ in the 2θ range from 10° to 70°, using a Rigaku

* E-mail: smwsq@mail.ahwhptt.net.cn

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(Japan) D/Max-rA X-ray diffractometer with Cu Kα radiation ($\lambda = 0.154178$ nm).

The XRD pattern is shown in Fig. 1. All the intense peaks can be indexed with orthorhombic lattice with cell constants $a = 1.115$ nm, $b = 1.132$ nm and $c = 0.399$ nm, which are in agreement with the reported values $a = 1.1149$ nm, $b = 1.1304$ nm and $c = 0.3981$ nm (JCPDS 17-320).

The transmission electron microscope (TEM) image and selected area electron diffraction (SAED) pattern were taken with a Hitachi Model H-800 transmission electron microscope.

TEM image, shown in Fig. 2a, is the Bi_2S_3 nanorods. They were uniformed particles with average diameter of 30 nm and length of 100 nm. The SAED pattern, shown in Fig. 2b, reveals that the nanorods have satisfied crystallinity with only 8 min of microwave irradiation.

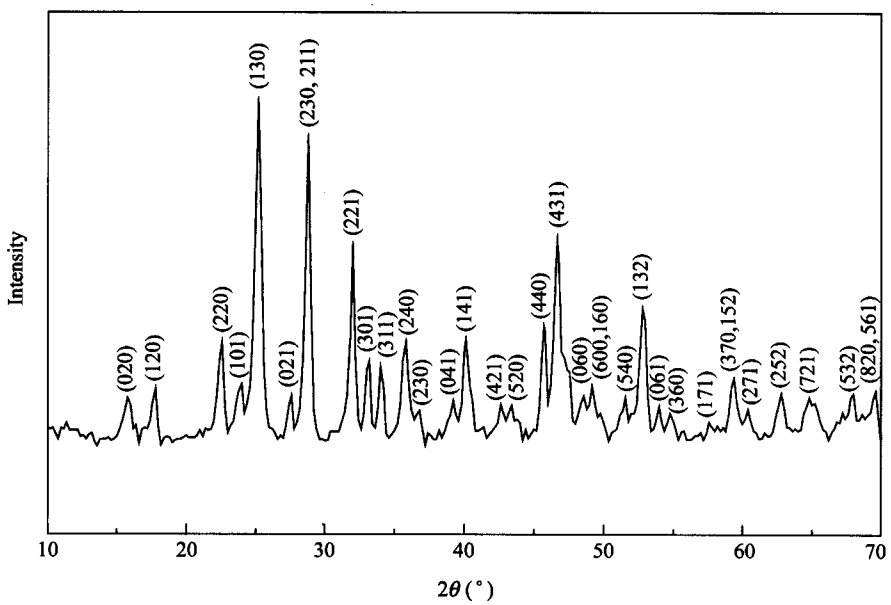

Fig. 1 XRD pattern of the product in very short time.

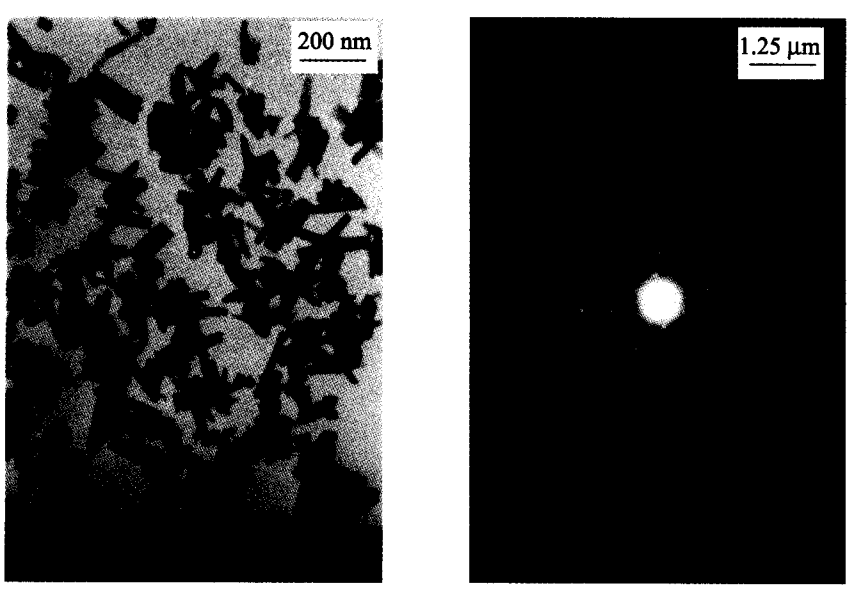

Fig. 2 TEM image and SAED pattern of the product. (a) Transmission electron microscope image; (b) selected area electron diffraction pattern.

The microwave of 2450 MHz has a particular property. The higher the temperature is, the less the microwave is absorbed by water. This negative feedback supplies a uniform temperature distribution in reactor and has a similar function of agitation, which provides better conditions for reaction and crystallization and produce Bi_2S_3 nanorods.

Conclusion

In summary, we discussed the microwave irradiation mediated hydrothermal synthesis of Bi_2S_3 nanorods. Microwave irradiation, which not only accelerates the reaction rate but also provides better condition for crystallization, helps to produce uniform nanorods. And this may be useful in commercial application.

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