Materials Chemistry and Physics 175 (2016) 1–5

Contents lists available at ScienceDirect

Materials Chemistry and Physics

journal homepage: www.elsevier.com/locate/matchemphys

Materials science communication

Rapid synthesis of single-phase bismuth ferrite by microwave-assisted hydrothermal method

Wenqian Cao [a], Zhi Chen [a, *], Tong Gao [a], Dantong Zhou [a], Xiaonan Leng [a], Feng Niu [a], Yuxiang Zhu [a, b], Laishun Qin [a, **], Jiangying Wang [a], Yuexiang Huang [a]

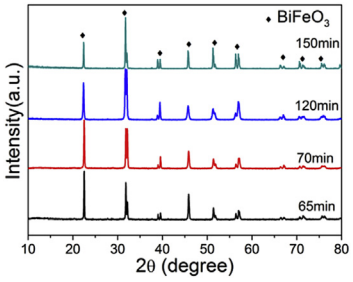
[a] College of Materials Science and Engineering, China Jiliang University, 258 Xueyuan Street, Xiasha Higher Education District, Hangzhou 310018, Zhejiang Province, PR China

[b] Tianjin Key Laboratory of Marine Resources and Chemistry, Tianjin University of Science and Technology, Tianjin, PR China

HIGHLIGHTS

- Rapid synthesis (65 min) of $BiFeO_3$ by microwave-assisted hydrothermal method.
- Reaction time has influence on the purity and preferred growth facets.
- FTIR and magnetic measurement further confirm the pure phase.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 12 June 2015
Received in revised form
23 January 2016
Accepted 25 February 2016
Available online 12 March 2016

Keywords:
Semiconductors
Chemical synthesis
Phase transitions
Magnetic properties
X-ray diffraction
Infrared spectroscopy

ABSTRACT

This paper describes on the fast synthesis of bismuth ferrite by the simple microwave-assisted hydrothermal method. The phase transformation and the preferred growth facets during the synthetic process have been investigated by X-ray diffraction. Bismuth ferrite can be quickly prepared by microwave hydrothermal method by simply controlling the reaction time, which is further confirmed by Fourier Transform infrared spectroscopy and magnetic measurement.

1. Introduction

Perovskite bismuth ferrite is widely investigated for its multiferroic, magnetic and photocatalytic properties with broad applications [1–4]. Recently, various procedures have been developed to synthesize $BiFeO_3$ with destined size and morphology. $BiFeO_3$ is commonly prepared by solid–sate reactions at temperature about 1000 °C using bismuth and iron oxides as the starting material. Chen et al. synthesized the single-phase $BiFeO_3$ nanoparticles by Sol–gel method at 500 °C [5]. Peng et al. reported

* Corresponding author.
** Corresponding author.
E-mail addresses: zchen0@gmail.com (Z. Chen), qinlaishun@yeah.net (L. Qin).

http://dx.doi.org/10.1016/j.matchemphys.2016.02.067
0254-0584/

hydrothermal synthesis of pure-phase $BiFeO_3$ by selecting appropriate parameters [6]. Rapid liquid-phase sintering, pulsed laser deposition, electro-spinning, magnetron sputtering methods have also been reported to synthesize single-phase $BiFeO_3$ powders [7–10]. Microwave synthesis method is an efficient way to ensure phase selectivity as well as fast crystallization [11]. Komarneni et al. reported the microwave hydrothermal synthesis of highly crystalline $BiFeO_3$ at 194 °C for 2 h [12]. However, the fast synthesis has not been reported and the phase transition of $BiFeO_3$ during the synthetic process is not clarified.

In this paper, we report the synthesis of bismuth ferrite by microwave hydrothermal method and the single phase could be rapidly achieved at 65 min. The phase transitions and preferred growth facets in the synthetic process have also been investigated and the pure phase is further identified by FTIR analysis and magnetic measurement.

2. Materials and methods

All the reagents were purchased from Sinopharm Chemical Reagent Co., Ltd in A.R grade. $Bi(NO_3)_3 \cdot 5H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ in 1:1 M ratio were dissolved in 10 mL distilled water. KOH was dropped into the above solution to coprecipitate Bi^{3+} and Fe^{3+} ions with a mechanical stirring for 2 h. Then, the mixture was transferred into a 100 mL Teflon reactor and placed in a microwave oven (Sineo MDS-6G). The reaction was carried out at 200 °C for different reaction time. After naturally cooling down to room temperature, the products were washed several times with distilled water and alcohol.

XRD and SEM were operated on Bruker Axs D2 PHASER diffractometer using Cu–Ka radiation and Hitachi SU8010 field-emission scanning electron microscope, respectively. FTIR spectra were recorded on a Bruker Tensor 27 spectrometer. Magnetic property was measured on VSM, Xpert Philips PW1830 magnetometer. Particle size was measured in water system on Beckman coulter Delsa Nano instrument. To reduce the sedimentation during the measurement, the sample is dispersed in the water by ultrasonic, and DLS measurement is operated immediately after the stop of ultrasonic.

3. Results and discussion

As shown in Fig. 1, all the XRD patterns are dominated by the diffraction peaks from bismuth ferrite. When the reaction time is less than 65 min, the observed impurity phases are assigned to Fe_2O_3 (JPCDS No. 52-1449) and $Bi_{25}FeO_{40}$ (JPCDS No. 46-0416). All diffraction peaks of $BiFeO_3$ can be perfectly indexed to a rhombohedral phase of the R3c space group (JCPDS No. 86-1518), as shown in Fig. 1(a). By prolonging the reaction time, the peaks from $BiFeO_3$ phase increase, while the peaks of $Bi_{25}FeO_{40}$ reduce simultaneously. This indicates that $Bi_{25}FeO_{40}$ impurity can transfer into $BiFeO_3$ phase during the extended heating process. However, $BiFeO_3$ phase almost disappears when the reaction time is up to 50 min. This means that $BiFeO_3$ phase can also convert into $Bi_{25}FeO_{40}$ phase under proper thermodynamic conditions. By further extending the reaction time, $Bi_{25}FeO_{40}$ impurity reduces again until it disappears completely at 65 min. It is evident that the products are single-phase and highly crystalline $BiFeO_3$ after 65 min reaction, shown in Fig. 1(b). After that, only single-phase $BiFeO_3$ powders appear at 65 min, 70 min, 120 min and 150 min. It is interesting to find that the relative facet intensities changes along with the reaction time. The ratio of integrated diffraction intensity at different reaction time (65 min, 70 min, 120 min and 150 min) is listed as below: I (104)/I (110) = 1.8, I (104)/I (110) = 1.2, I (104)/I (110) = 1.1, I (104)/I (110) = 2.2. It indicates that all the

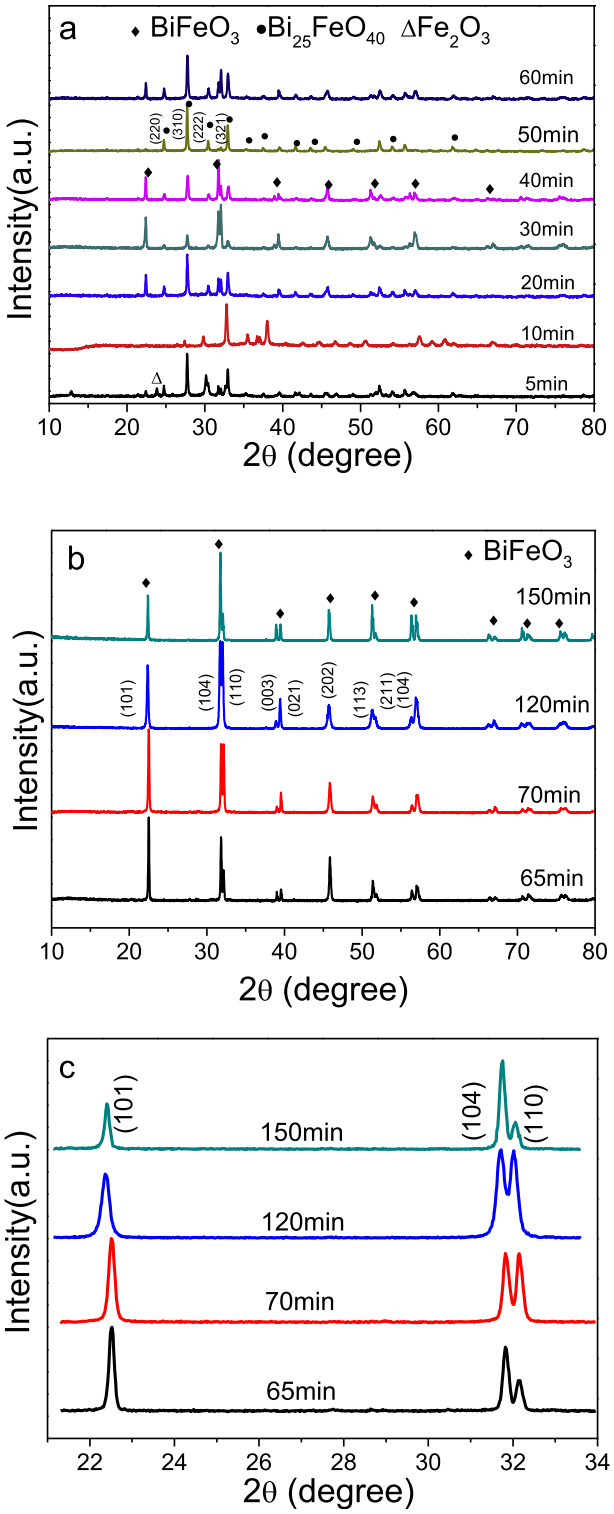


Fig. 1. XRD patterns of microwave-synthesized bismuth ferrite at different time: (a) before 65 min (b) above 65 min (c) the magnification of 2θ between 21 and 33.

single-phase $BiFeO_3$ powders prepared at different reaction time selectively grow along (110) and (104) directions and (104) facet becomes the main crystal growth direction along with reaction continuing, as the Fig. 1c shown. It is interesting to note that the FWHM also changes along with the reaction time in which the FWHM decreases with the increase of reaction time except the