



Flash synthesis of Li_2TiO_3 powder by microwave-induced solution combustion



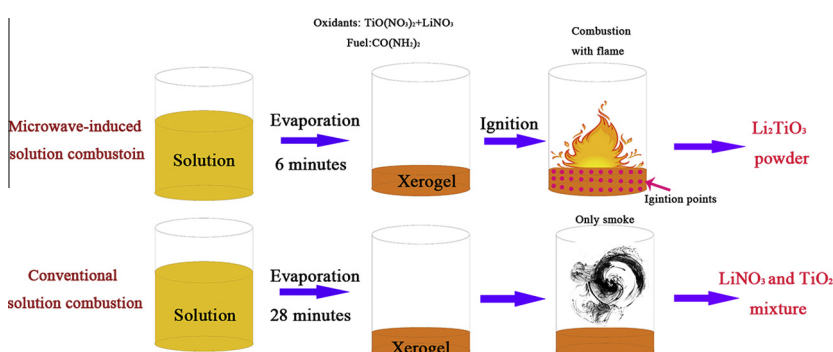
Qilai Zhou, Liyao Tao, Yue Gao, Lihong Xue*, Youwei Yan

State Key Laboratory of Materials Processing and Die and Mould Technology, Huazhong University of Science and Technology, Wuhan 430074, PR China

HIGHLIGHTS

- We synthesized Li_2TiO_3 powder by microwave-induced solution combustion in one step.
- The combustion reaction only happened by microwave heating.
- Changing total metal ion concentration will influence combustion process.
- The as-synthesized powder shows good sinterability.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Available online 17 May 2014

ABSTRACT

Nano-crystalline Li_2TiO_3 powder was prepared by a microwave-induced solution combustion synthesis (MSCS) route using urea as fuel. It is observed that combustion reaction, which did not occur by conventional heating, happened when microwave heating was induced. The as-synthesized Li_2TiO_3 powder exhibits a narrow size distribution. In MSCS, the total metal ion concentration (C_m) in the starting solution plays an important role. By changing C_m values in starting solution, SCS process including ignition time, combustion period and reaction rate can be controlled. The as-prepared powder could be sintered up to 92.6% of the theoretical density at 1223 K.

© 2014 Elsevier B.V. All rights reserved.

1. Introduction

Simple and efficient synthesis of tritium breeding materials is an important experimental task. Among the lithium based oxide ceramics, such as Li_2O , Li_4SiO_4 , LiAlO_2 , Li_2ZrO_3 and Li_2TiO_3 , it is found that Li_2TiO_3 is a preferable tritium breeding material candidate because of its reasonable lithium atom density, low activation, excellent tritium release performance, and chemical stability [1,2]. However, the densification of Li_2TiO_3 powder is not easy due to the fast grain growth during sintering which results in large pore traps in over-sized grains or on grain boundaries. Therefore,

densification should take place at a relatively low temperature before rapid grain growth occurs [3].

Powders obtained by wet chemical methods usually possess high surface activity and small crystallite sizes, which can reduce sintering temperatures, avoiding excessive grain growth. Wu et al. [4] synthesized nano-sized Li_2TiO_3 powder with a crystallite size of 40 nm using sol-gel method. Sinha [5] reported a solid-liquid combustion route to produce high purity Li_2TiO_3 powder. Li et al. [6] devised an in situ hydrolysis method using TiCl_4 and $\text{LiOH}\cdot\text{H}_2\text{O}$ as the raw materials. The precursor was subsequently converted to Li_2TiO_3 at 1073 K for 6 h. In general, nearly all Li_2TiO_3 powders fabricated by the as-mentioned wet chemical techniques can be densely sintered at temperature ranging from 1273 K to 1373 K.

* Corresponding author. Tel.: +86 27 87543876; fax: +86 27 87541922.

E-mail address: xuelh@mail.hust.edu.cn (L. Xue).

However, in comparison with common high-temperature solid state reaction, there are some shortcomings in these wet chemical methods. It is not an easy task to produce Li_2TiO_3 powders in large scales via hydrolysis. The sol-gel method usually needs multi-steps to obtain the final product. Thus, research on a simple and economic way to prepare Li_2TiO_3 powder has become an urgent concern. It is widely known that solution combustion synthesis (denoted as SCS) is an effective, rapid and energy-efficient method for mass production of various nanomaterials [7–9]. In SCS, the combustion reaction is self-sustained by its own exothermic reaction and the whole synthesis procedure lasts just a few minutes [10].

Recently, microwaves have been used for processing advanced materials [11–15]. Microwave-processing of materials is fundamentally different from conventional processing in its heating mechanism. In conventional heating, heat is generated by heating elements and then transferred to the sample surface through thermal conduction [16]. But in a microwave reactor, the heat is generated within the sample itself due to the interaction of microwaves with the material. So, microwave heating has the following advantages: (i) homogeneous heating; (ii) higher reaction rate; (iii) no contact between the heating source and reactants; (iv) selective heating if the solution contains ions with different microwave absorbing properties; (v) higher phase purity with better yields, Bilecka and Niederberger [17]. In addition, microwave heating and electromagnetic effects sometimes induce complicated chemical reactions to occur, which accelerate the reaction rate. Thus, target materials are obtained [18–20].

This paper presents the synthesis of Li_2TiO_3 nanoparticles via a microwave-induced solution combustion method using urea as

fuel. It is interesting to mention that this redox reaction did not happen under conventional heating condition, which was also revealed by Jung [21].

2. Experimental

2.1. Fabrication

In a typical process, titanyl nitrate ($\text{TiO}(\text{NO}_3)_2$) and lithium nitrate (LiNO_3) were used as oxidizers, urea ($\text{CO}(\text{NH}_2)_2$) as a reductant and also a complexing agent. Typically, 0.005 mol tetrabutyl titanate ($\text{Ti}(\text{OC}_4\text{H}_9)_4$) was added into deionized water to obtain a white precipitation, which was washed with deionized water and dissolved in concentrated nitric acid to get a clear $\text{TiO}(\text{NO}_3)_2$ solution. Then 0.01 mol LiNO_3 together with 0.025 mol $\text{CO}(\text{NH}_2)_2$ were added to the solution to form the starting precursor (100 mL, the total metal ion concentration (denoted as C_m) is 0.15 mol/L).

The transparent solution was transferred into a microwave-induced solution combustion device (Type: MZY3000S, designed by our lab, maximum power 3000 W, microwave frequency 2.45 GHz) and a muffle furnace preheated to 873 K (for comparison), respectively. The power of microwave was set at 1300 W. At the early stage in MSCS, the solution was heated to boiling. Within a few minutes, the solution ignited to produce self-propagating combustion, which only lasted for several seconds. The combusted powder was ground in a mortar and washed with deionized water.

In order to study the sinterability of the as-synthesized Li_2TiO_3 powder, pellets with 14 mm in diameter and 2 mm in thickness

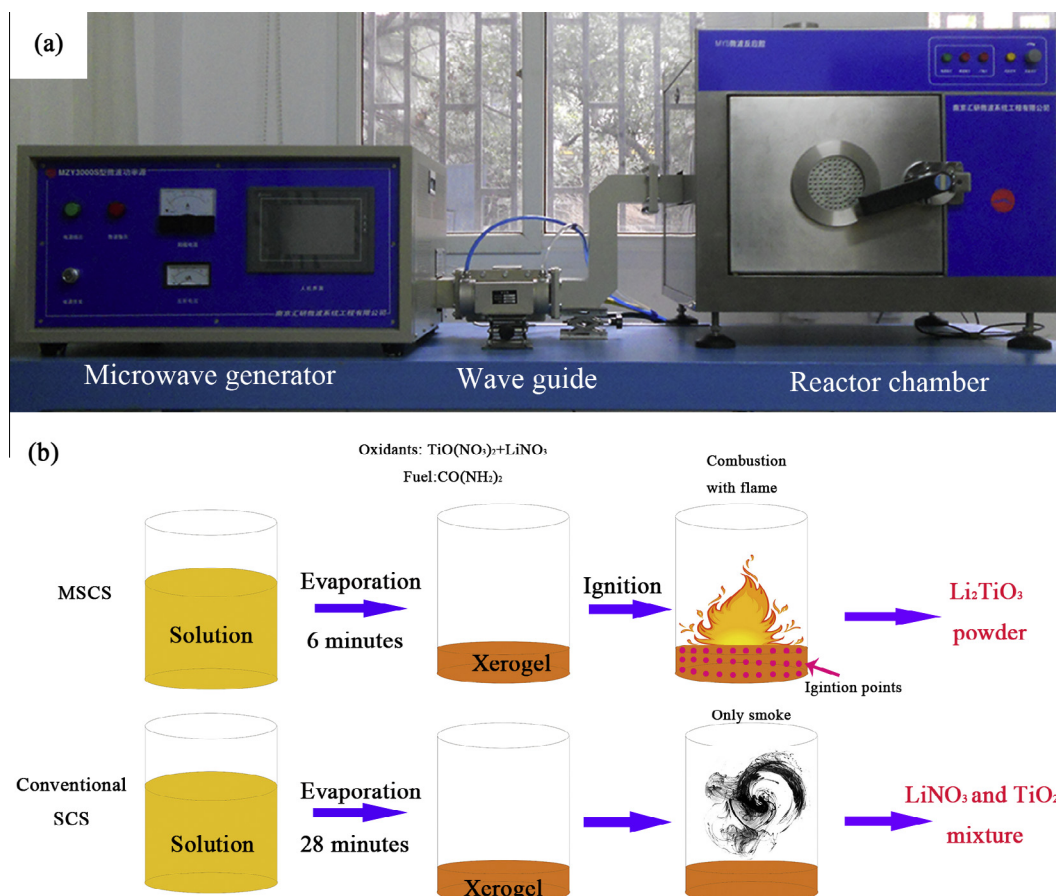


Fig. 1. (a) MZY3000S device custom designed for MSCS and (b) a schematic illustration of the different experimental phenomenon by microwave-induced solution combustion synthesis (MSCS) and conventional solution combustion synthesis (SCS).

Download English Version:

<https://daneshyari.com/en/article/1564948>

Download Persian Version:

<https://daneshyari.com/article/1564948>

[Daneshyari.com](https://daneshyari.com)