Fusion Engineering and Design 113 (2016) 146–150

Contents lists available at ScienceDirect

Fusion Engineering and Design

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

Sol–gel followed by urea–acetone spherodization for preparation of lithium titanate ceramics pebbles and preliminary characterization

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ARTICLE INFO

Article history:
Received 25 September 2015
Received in revised form
13 September 2016
Accepted 28 October 2016
Available online 9 November 2016

Keywords:
Lithium titanate
Sol–gel followed by urea-acetone technique
Thermal conductivity
Crush strength

ABSTRACT

Lithium titanate ceramics pebbles were prepared by chemical method of sol. gel followed by urea-aceton spherodization technique. Lithium titanate having monoclinic phase was obtained. Laser flash method was used to study thermal conductivity and diffusivity of the ceramics. At near 472 K the average value of diffusivity was around 0.0052 $cm^2\ s^{-1}$ while the specific heat capacity was 3101.3 $J\ kg^{-1}\ K^{-1}$ while the same two parameters had values of 0.0041 $cm^2\ s^{-1}$ and 3894.4 $J\ kg^{-1}\ K^{-1}$ at a temperature 1050 K. The crush strength of pebble was measured on Universal Testing Machine (UTM) and was found as 37.08 N. The density was measured and found to be 2.8 g/cc (81.63% of TD of value 3.43 g/cc).

1. Introduction

Lithium-based ceramics have been recognized as promising tritium breeder material for fusion research reactor blankets [1–8]. These ceramics have excellent potential as tritium breeder because of the ease of tritium recovery, excellent thermo-mechanical, thermo physical properties and good irradiation behavior under long-term irradiation at high temperature and large temperature gradients. They are Li_2O, Li_2ZrO_3, $LiAlO_2$, Li_4SiO_4 and Li_2TiO_3 and others. These materials are potentially regarded as the breeder material for breeder blanket application in fusion reactor. Indeed their inherent thermal stability and chemical inertness are significant safety advantages. The breeder blanket is a key component of the fusion reactor because it directly involves tritium breeder and energy extraction, both of which are critical to development of fusion power.

Recently, attention shifted to lithium titanate (Li_2TiO_3), when it was proposed as the potential candidate material in a breeder blanket because of several advantages over other similar materials [4,5]. One of them is the ease with which tritium is recovered at low temperature. Li_2TiO_3 is especially a good material from the viewpoint of reprocessing because lithium titanate Li_2TiO_3 can be selectively dissolved in low HNO_3 concentration [6]. Apart from that lithium density also play a significant role in tritium breeding and its recovery, and also Li_2TiO_3 pebbles have been supposed to use in the blanket so it is required to produce/fabricate desirable dense pebbles with desired thermal and mechanical properties. It has been further recognized that lithium recovery is proportional to some extent to its electrical conductivity so attention is also shifted to fabricate desired geometry ceramics with required electrical conductivity [7], microstructure and activation energy for better production and recovery of tritium.

Fabrication of lithium titanate ceramics in pebble form is reported to be much more suitable for breeder blanket application as it produces optimum tritium in comparison of other shape or state. K.Tsuchiya et al. were reported the fabrication of Li_2TiO_3 pebbles by wet processes and achieved 80–85% TD, further they reported successful control over particle size and density of the pebbles by adopting dehydration and substitution reactions [1,2]. T. Hoshino et al. had projected a sol. gel. method by help of which mass decrease in Li_2TiO_3 pebbles at elevated temperature can be prevented by adding $Li(Li_{2+x}TiO_{3+y})$, they further reported a method of emulsion for fabrication and achieved pebble density in the rage of 85% TD [3,4]. Different way of fabrication for Li_2TiO_3 pebbles are reported [1–10]. For breeder blanket application, one of the foremost requirements of pebble is related to proper mechanical crush strength such that it can sustain in harsh environmental condition of fusion reactor. Mandal et al. [11] had reported the fabrication of lithium titanate pebbles by solid state method having dia 2 mm and

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http://dx.doi.org/10.1016/j.fusengdes.2016.10.018
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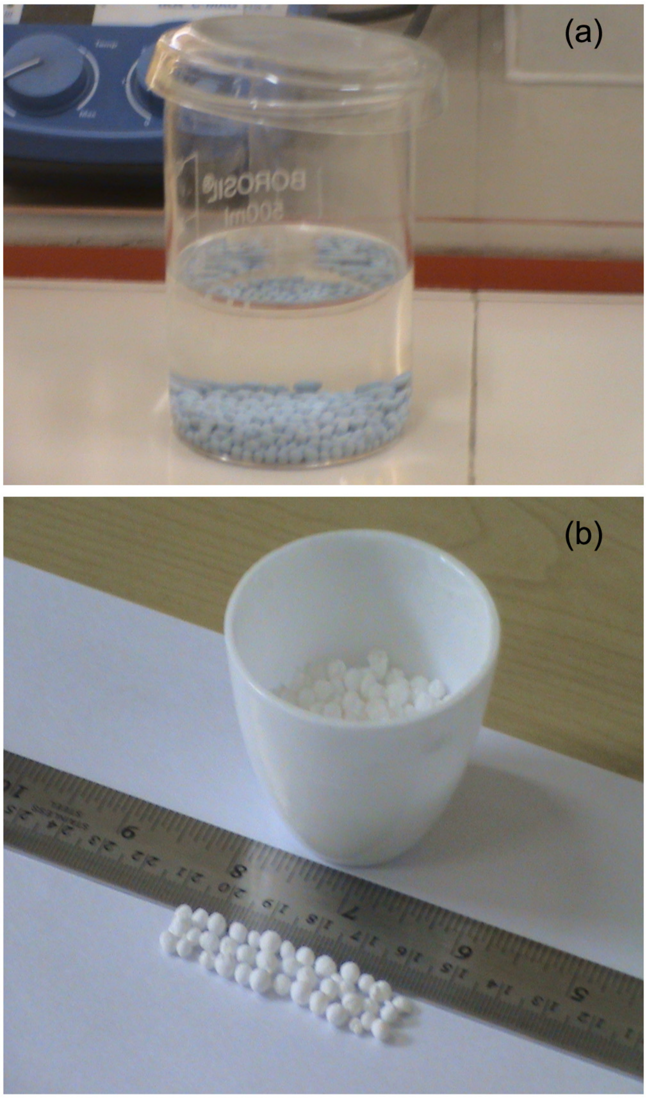

Fig. 1. (a): Photograph of spheridization of lithium titanate pebbles. (b): Lithium titanate pebbles after sintering at 1100 °C.

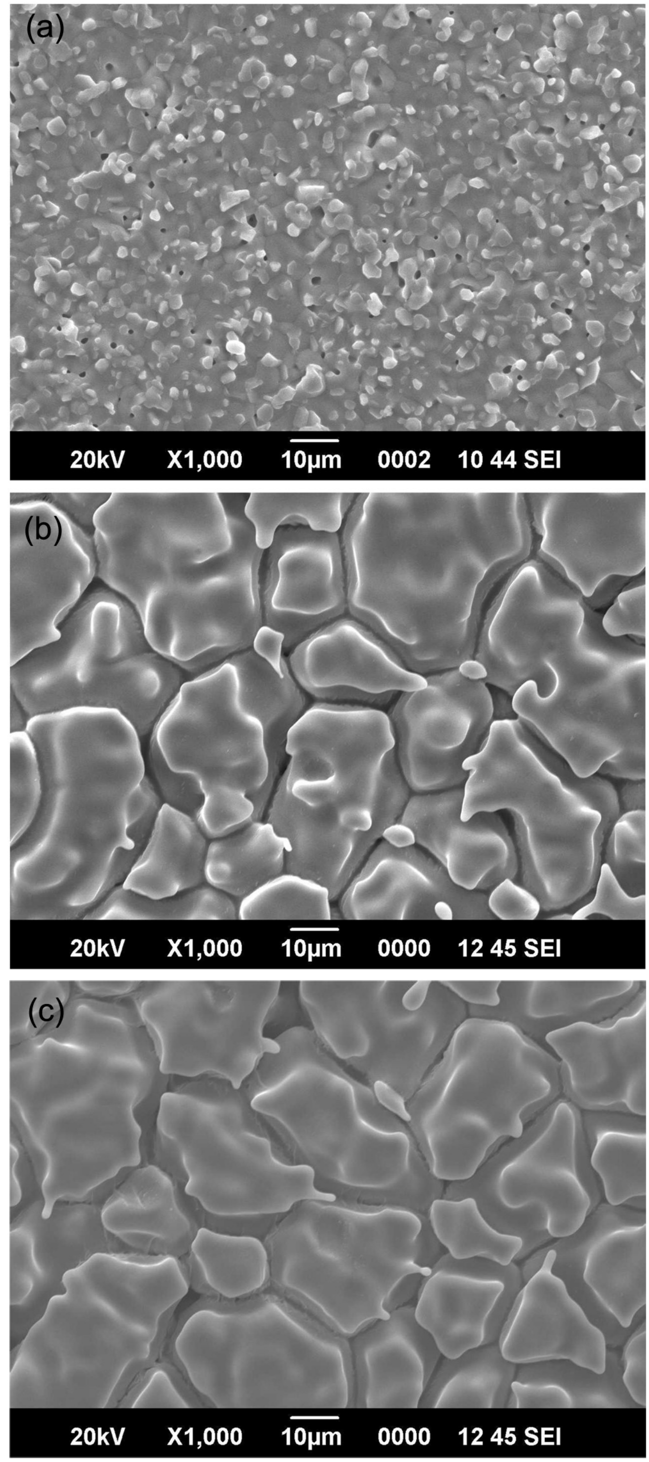

Fig. 2. (a) SEM micrograph of pebble sintered at 1100 °C for three hours. (b) SEM micrograph of pebble sintered at 1150 °C for 3 h. (c) SEM micrograph of pebble sintered at 1200 °C for 3 h.

crush strength 25 N. In present work, chemical method of sol. gel. technique followed by spherodization of lithium titanate with the help of urea-acetone solution was adopted. Lithium titanate monoclinic phase was obtained. No high temperature calcination was needed in obtaining required phase. Laser flash method was used to study thermal conductivity and diffusivity of the sample. At 472 K the average value of diffusivity was observed as 0.0052 $cm^2\ s^{-1}$ while the specific heat capacity was found as 3101.3 $J\ kg^{-1}\ K^{-1}$. The crush strength of pebble was measured on UTM Machine and was found as 37.08 N. The density was measured and found to be 2.8 g/cc (81.63% of TD of value 3.43 g/cc) [12–14].

2. Material and methods

2.1. Fabrication of lithium titanate

Required weight of Titanium (IV) butoxide was taken and dissolved in a small amount of ethanol to make the solution concentrated. Limited amount of citric acid was weighed and diluted with minimum amount of distilled water to prepare the citrate solution. Ammonia solution was added to the citrate solution to adjust the pH of the solution around 9.0. Solutions as obtained were mixed under vigorous and continuous stirring for more than half an hour while keeping the temperature of the hot plate around 30 °C. pH of the titanium sol so obtained was monitored and kept at 9.0 throughout the process. pH value was monitored by taking out sample from hot plate and checking the pH value manually. Ammonia solution was added to maintain the pH value. Lithium nitrate solution was obtained by dissolving the required amount of lithium nitrate powder to the distilled water. The lithium nitrate solution was added drop wise to the titanium sol by simultaneously monitoring the pH