Available online at www.sciencedirect.com
ScienceDirect
Materials Research Bulletin 43 (2008) 2736–2741
Materials Research Bulletin
www.elsevier.com/locate/matresbu

Rapid preparation and characterization of $Dy_2Zr_2O_7$ nanocrystals

Yuping Tong, Zongxue Yu, Lude Lu *, Xujie Yang, Xin Wang

Materials Chemistry Laboratory, Nanjing University of Science and Technology, Nanjing 210094, China

Received 27 May 2007; received in revised form 30 September 2007; accepted 19 October 2007
Available online 30 October 2007

Abstract

Ultrafine fluorite type $Dy_2Zr_2O_7$ nanocrystals with cubic structure were fabricated at relatively low temperature by stearic acid method (SAM) using zirconium(IV) butoxide and dysprosium nitrate as raw materials, stearic acid as solvent and dispersant. The fabrication process was monitored by thermogravimetric analysis and differential thermal analysis (TG-DTA) and Fourier transform infrared spectroscopy (FT-IR). The obtained products were characterized by powder X-ray diffraction (XRD), transmission electron microscopy (TEM), high-resolution transmission electron microscopy (HRTEM), energy dispersive X-ray spectrometer (EDS) and UV–vis absorption spectroscopy. A single phase of $Dy_2Zr_2O_7$ with high crystallinity was formed at 800 °C. The interplanar distances measured from the HRTEM image were 0.284 and 0.256 nm, respectively, coinciding with the theoretical values.

Keywords: A. Nanostructures; A. Oxides; B. Sol–gel chemistry; C. X-ray diffraction

1. Introduction

Rare earth zirconium oxides ($Re_2Zr_2O_7$, Re = rare earth) have received considerable attention in recent years [1,2], due to their excellent properties, such as metal-insulator transitions, ferroelectric properties, fluorescent and phosphorescent properties [3]. These compounds contain two structures: the pyrochlore and fluorite structure. The pyrochlore structure belongs to the space group *Fd*3*m* and the space group of the fluorite structure is *Fm*3*m* with O_h symmetry of D_{3d} for both Ln^{3+} and Zr^{4+} ions [4,5]. Traditionally, $Re_2Zr_2O_7$ is prepared by solid-state reaction [6–8], namely the metal oxides are used as precursors and the reaction reagents have to be sintered at very high temperature for long time. Great interests have been focused on the synthesis and investigation of the properties of $Re_2Zr_2O_7$, in recent years, $Re_2Zr_2O_7$ has been synthesized by many methods, including co-precipitation, sol–gel, hydrothermal, hydrazine method and mechanical milling method [9–12].

Precursor method is one of the typical strategies to synthesize nanoscale complex oxide [13]. In our laboratory, the complex oxides nanocrystalline were easily obtained by stearic acid sol–gel method (SAM) using stearic acid as reactant and dispersant, and the reaction temperature was remarkably reduced [14–16]. In this paper, we presented the preparation and characterization of $Dy_2Zr_2O_7$ nanocrystals by SAM.

* Corresponding author. Tel.: +86 25 84315054; fax: +86 25 84315054.
E-mail address: njtong007@yahoo.com.cn (L. Lu).

0025-5408/$ – see front matter © 2007 Elsevier Ltd. All rights reserved.
doi:10.1016/j.materresbull.2007.10.017

2. Experimental

2.1. Preparation of $Dy_2Zr_2O_7$ nanocrystals

All reagents were of analytical grade and used without further purification. The fabrication procedure of $Dy_2Zr_2O_7$ is illustrated in the flowchart in Fig. 1.

$Dy(NO_3)_3{\cdot}nH_2O$ obtained from Dy_2O_3 dissolved in HNO_3 and $Zr(OBu)_4$ were used as the precursors of Dy and Zr, respectively. The molar ratio of K/Dy/Zr is 2/1/1. Stearic acid ($C_{17}H_{35}COOH$) was used as the solvent and dispersant. Firstly, two appropriate portions of stearic acid (20 g) were heated and melted, and then appropriate amounts of $Dy(NO_3)_3{\cdot}nH_2O$ and KOH were added into them, which were named as solution A and B, respectively. These mixtures were thoroughly stirred by the magnetic mixer to eliminate the water. Then solution A was dumped into solution B. After 1 h, $Zr(OBu)_4$ was added into the mixture solution with vigorous stirring. Two hours later, a homogeneous transparent solution was formed. The solution was ignited in air and the obtained powders were calcined at a series of increasing temperatures ranging from 500 to 800 °C for 5 h in air, respectively.

2.2. Instrumentation

The thermal decomposition of the gel was investigated by thermogravimetric and differential thermal analysis (TG-DTA) on a Beijing WCT-2A thermal analyzer. The temperature ranges from 40 to 750 °C, at a heating rate of 20 °C/min under N_2 atmosphere and using Al_2O_3 as the reference. FT-IR spectra of KBr powder-pressed pellets were recorded on a BRUKER VECTOR 22 spectrometer. The crystalline phase structure was determined by Bruker D8 Advance X-ray diffractometer (XRD) using Cu Kα radiation (λ = 0.154184 nm). Transmission electron microscopy (TEM) image and high-resolution transmission electron microscopy (HRTEM) images were recorded on a JEOL JEM-2100 transmission electron microscope. EDS were taken with a JEOL JSM-6380LV electron microscope. The UV–vis absorption spectrum was carried out by Beijing Eraic UV-1201 spectrometer.

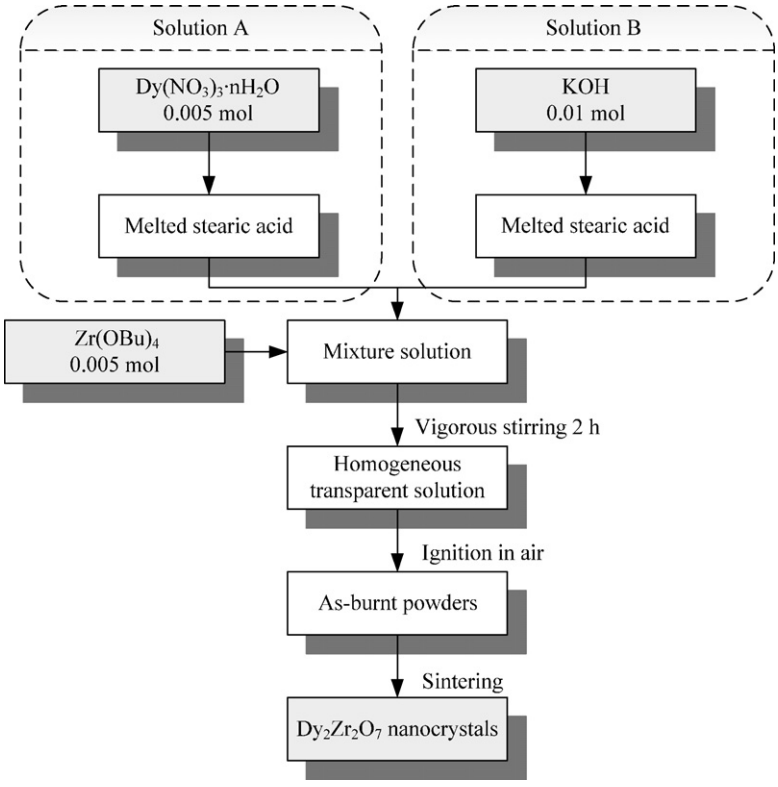

Fig. 1. Preparation procedure of $Dy_2Zr_2O_7$ nanocrystals by stearic acid method.