



Effect of acidity on microstructure and spectroscopic properties of $Y_2O_3:Eu^{3+}$ powders and ceramics

Chao-Nan Wang^{a,b}, Yong Li^{a,b}, Wei-Ping Zhang^{a,c}, Min Yin^{a,b,*}

^a Hefei National Laboratory for Physical Sciences at the Microscale, Hefei 230026, PR China

^b Department of Physics, University of Science and Technology of China, Hefei 230026, PR China

^c National Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei 230026, PR China

ARTICLE INFO

Article history:

Received 30 October 2008

Received in revised form 22 April 2009

Accepted 13 May 2009

Keywords:

Homogeneous precipitation

Y_2O_3

Ceramic

VUV spectra

ABSTRACT

$Y_2O_3:Eu^{3+}$ nano-powders were synthesized by homogeneous precipitation and the influence of solution acidity was investigated. IR and TG–DTA analysis showed that in different acidic solutions different kinds of sediments were generated. TEM images revealed distinct morphologies of obtained powders. The resultant powders were sintered into ceramics in vacuum and then in N_2 atmosphere without any additives. SEM images indicated that the morphologies of powders have significant impact on the microstructures of sintering-derived ceramics. Low-agglomerated and uniform powders were in favor of production of ceramics with homogeneous microstructures. Excitation and emission spectra of both powders and ceramics were measured and some changes were observed.

© 2009 Elsevier B.V. All rights reserved.

1. Introduction

Transparent ceramics have been attracting ever-growing attention in the fields of scintillators and solid-state lasers for the past few years. Compared with single crystals, transparent ceramic materials have many advantages, such as easy fabrication, inexpensiveness, and mass producibility. Moreover, ceramic materials have possibility of fabricating large size and high concentration samples with multi-layer and multi-functional ceramic structure [1].

Yttria finds potential applications because of its high corrosion resistivity, thermal stability and transparency over a wide wavelength region, i.e. from violet to infrared light. In order to fabricate transparent ceramics it is essential to synthesize ultrafine, mono-sized, low-agglomerated and spherical powders. In recent years, many methods have been developed to prepare yttria powders, such as combustion [2], precipitation [3], hydrothermal synthesis [4], spray pyrolysis [5], and sol–gel technique [6]. Among various synthesis routes, homogeneous precipitation is one of the most effective methods. Because nano-powders prepared by homogeneous precipitation usually have high purity, good dispersivity and homogeneity in atomic size level. In the homogeneous precipitation process, precipitation is brought about uniformly throughout the solution at the molecular level by thermal decomposition of

urea. The gradual and uniform release of ammonia and carbonate ions results in the nucleation and growth of uniformly sized and shaped particles. In this paper, $Y_2O_3:Eu^{3+}$ powders were synthesized by means of homogeneous precipitation. The influence of solution acidity on morphologies of powders and microstructures of ceramics, as well as the spectroscopic properties of both powders and ceramics were reported.

2. Experimental

2.1. Preparation of samples

Y_2O_3 (99.99%), Eu_2O_3 (99.99%) and urea of analytical grade were used as starting materials. Y_2O_3 and Eu_2O_3 were dissolved in dilute nitric acid under stirring and heating, while urea was dissolved in distilled water to obtain corresponding aqueous solutions. Then they were blended in required molar ratio and uniformly stirred to get transparent precursor solutions. Here, the concentration of urea was kept as 1 M/L, and that of rare earth ions was 0.02 M/L. The molar concentration of Eu^{3+} ion for all prepared samples was 5%. The pH value of the precursor solution was varied. For one precursor solution, the pH value was measured as 4 and it was directly heated on a water bath at 85 °C under stirring. For the other one, appropriate amount of NH_4OH was added in advance to adjust the pH value to 6.5. After a complete reaction, the precipitates were separated, washed with distilled water to remove the soluble ions and ethanol to reduce agglomeration, and then dried in an oven at 120 °C for 4 h. The dried precursor powders were crushed and calcined at 800 °C for 2 h in air. Here, we named the two resultant

* Corresponding author at: Department of Physics, University of Science and Technology of China, Hefei 230026, PR China. Tel.: +86 551 3607412; fax: +86 551 3607417.

E-mail address: yinmin@ustc.edu.cn (M. Yin).

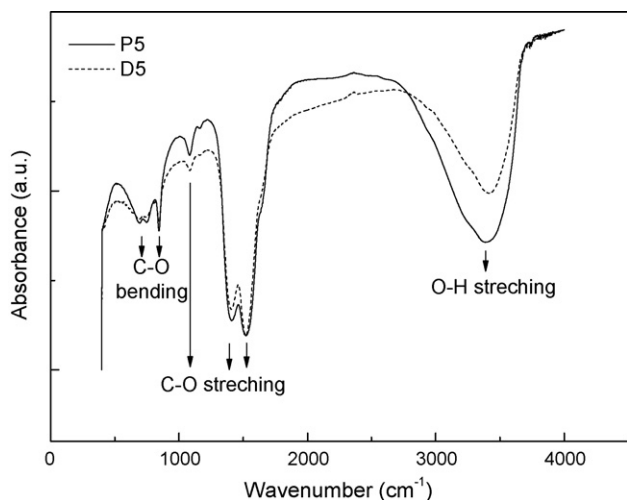


Fig. 1. FT-IR spectra of the dried precipitates from solutions of different pH values.

samples as D5 and P5, respectively, in which 5 means the doping concentration of Eu^{3+} is 5%. D indicates that the powder was prepared by direct homogeneous precipitation without adjusting the pH value of the solution and P denotes that the pH value of the precursor solution was adjusted before the homogeneous precipitation process.

The powders D5 and P5 served as starting materials for fabrication of ceramics. Firstly, the powders were pressed into 8-mm diameter disks by uniaxial cold pressing under a load of 2.4 tons without any additives. Then such pellets were sintered in two separate heating processes: in vacuum at 1150°C for 5 h and then in N_2 atmosphere at 1700°C for 2 h and 1600°C for 10 h.

2.2. Characterization

Infrared (IR) spectra of the precursors were obtained with a Fourier transform infrared (FT-IR) spectrometer (MAGNA-IR 750, USA). X-ray diffraction studies ($10\text{--}70^\circ$) were carried out on the precursors and powders using $\text{Cu K}\alpha$ radiation on a Philips X'PERT PRO X-ray diffractometer. The morphological features of the powders and ceramics were examined by a JSM-6700F Field Emission Scan Electron Microanalyzer (FESEM) and a JEOL-2010 High-Resolution Transmission Electron Microscope (HRTEM). Thermal gravimetric and differential thermal analysis (TG-DTA) of the precursors was conducted from room temperature to 1000°C on a TG-DTA thermal analyzer (TA-50, SHIMADZU) with a heating rate of $10^\circ\text{C}/\text{min}$. The emission and excitation spectra of both powders and ceramics were measured at VUV spectroscopy station of National Synchrotron Radiation Laboratory (Hefei). The excitation spectra were corrected for the incident light intensity by using sodium salicylate standard sample. All the measurements were conducted at room temperature.

3. Results and discussion

3.1. Thermal analysis and morphologies of $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ powders

The FT-IR spectra of precursors of D5 and P5 were presented in Fig. 1. The peaks at 750 cm^{-1} , 844 cm^{-1} and the ones at 1084 cm^{-1} , 1410 cm^{-1} , 1520 cm^{-1} are due to C-O bond bending and stretching vibration, respectively. The broad band at 3400 cm^{-1} is assigned to O-H stretching vibration. It is indicated that there is no considerable difference in the chemical composition between the precipitates derived from solutions of different acidities. However the absorbance of O-H bond in precursor of P5 is relatively stronger

than that in precursor of D5.

The TG-DTA analysis was conducted for precursors of D5 and P5 to examine the conversion process and elucidate the transformation to crystal yttria during calcination, as shown in Fig. 2. For both precursors, the weight loss (10% for D5 and 11% for P5) before 240°C is mainly attributed to the elimination of physically absorbed water and chemically bonded water, corresponding to the endothermic peaks around 165°C in the DTA curves. The weight loss between 270°C and 800°C is ascribed to the decomposition of precipitates. Obviously, the precursors of D5 and P5 behave differently during this process.

For precursor of D5, weight loss during this temperature range is 28.55%, which is very close to the theoretical weight loss of $\text{Y}_2\text{O}(\text{CO}_3)_2:\text{Eu}^{3+}$ (27.5%). The slight disparity is caused by the residual crystal water in the compound. The endothermic peak at 424°C in the DTA curve is associated with the decomposition of carbonate and the exothermic peak at 630°C is resulted by the crystallization of $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$.

For precursor of P5, the weight loss between 270°C and 800°C is 24.99%. Taking into account all the possible compounds that may be produced during the precipitation process, the precursor of sample P5 is a mixture of $\text{Y}_2\text{O}(\text{CO}_3)_2:\text{Eu}^{3+}$ and $\text{Y}(\text{OH})_3:\text{Eu}^{3+}$ (theoretical weight loss: 18.89%). This can also be validated by the DTA curve. The endothermic peak at 429°C and exothermic peak at 672°C are assigned to the decomposition of carbonate and crystallization of $\text{Y}_2\text{O}_3:\text{Eu}^{3+}$ in $\text{Y}_2\text{O}(\text{CO}_3)_2:\text{Eu}^{3+}$, respectively, just as demonstrated in sample D5. The endothermic peak at 345°C is corresponded to the decomposition of hydroxide and the exothermic peak at 560°C is

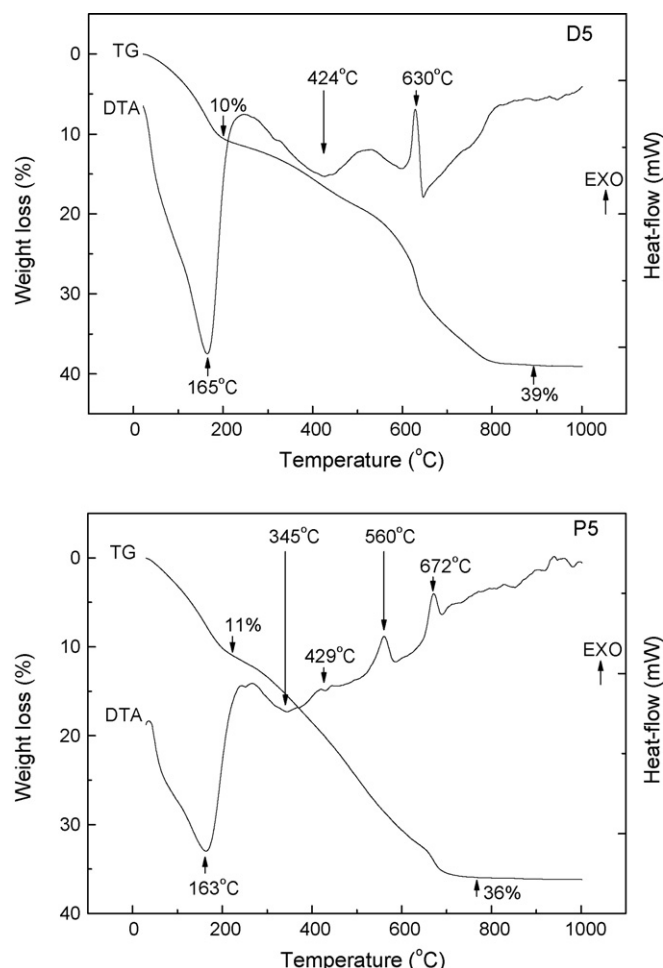


Fig. 2. Thermal behaviors of precursors of D5 and P5.

Download English Version:

<https://daneshyari.com/en/article/1234176>

Download Persian Version:

<https://daneshyari.com/article/1234176>

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