



Rapid Communication

Fabrication of well-dispersed Y_2O_3 nano-powders by poly(acrylic acid) low-temperature combustion

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ABSTRACT

A new organic fuel (poly(acrylic acid)) was adopted to fabricate well-dispersed Y_2O_3 nano-powders according to the solution combustion method. The improved combustion method requires no tedious operation or restriction conditions. The effects of organic fuel dosage and calcination temperature on the characteristics of Y_2O_3 powders were investigated. The results indicate that the Y_2O_3 can be formed at 400 °C and well crystallized at 500 °C with a nitrate and organic fuel mass ratio of 7:1. The addition of organic fuel significantly improves the dispersity and reduces the particle size of Y_2O_3 powders, whereas the increase in calcination temperature leads to the slight increase in the particle size of the Y_2O_3 powders. The well-dispersed Y_2O_3 powders with the particle size range of 10–30 nm are obtained at 500 °C when the mass ratio of the organic fuel and nitrate is 7:2. The poly(acrylic acid) is a new and feasible fuel for the solution combustion method to prepare well-dispersed Y_2O_3 nano-powders.

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1. Introduction

Yttrium oxide (Y_2O_3) is an interesting material with excellent properties, such as heat resistance, stability, and optical clarity over a broad spectral region. Therefore, it has been applied in infrared windows, high-power lasers, and radiation detection devices [1–4]. Transparent Y_2O_3 ceramics show potential advantages over single crystals: low cost, simple preparation technology, complex shape fabrication, easy doping process, and enhanced mechanical properties. However, transparent ceramics are required to obtain a final density of nearly 100%. Therefore the sintering characteristics of powders are crucial [5]. Powders are required to possess high purity, superfine grain size, narrow size distribution, uniform microstructure, and good dispersity [6–8]. It is significant to explore a simple and feasible synthesis route to obtain high-quality powders for the preparation of transparent ceramics. Kopylov [9] reported that the most important requirements for the starting nanopowders were grain size (around 100 nm) and grain form (as close to the sphere) in order to enable close grains contact during the sintering process.

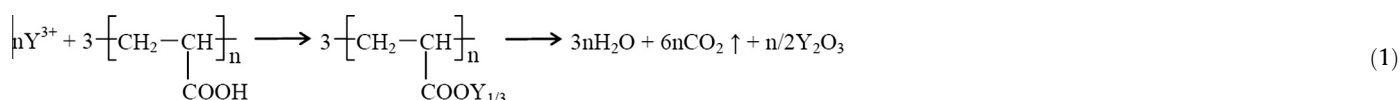
Various methods, such as solid state reactions [10], co-precipitation [11–13], sol–gel techniques [14], and hydrothermal synthesis method [15], have been used to prepare Y_2O_3 powders. Solid-state reactions require a high annealing temperature due to the refractory nature of oxide precursors. In co-precipitation and sol–gel methods, the preparation of precursors requires tedious steps and many restrained conditions. In addition, a high crystallization temperature is required for the resultant pure powders and the particle size is generally larger than 0.1 μm . Compared with the above methods, the combustion method has lots of advantages, such as simple experimental procedure, the relatively low-reaction temperature, and short reaction time. The method can be used to produce complex, fine, chemically homogeneous, and pure nano-powders [16]. Therefore, it is a superior powder processing method to produce Y_2O_3 powders. This process undergoes an exothermic reaction of an oxidizer such as metal nitrates and an organic fuel. The organic fuel has an important effect on the characteristic of final powders. Generally, a qualified organic fuel should produce non-toxic gases in the soft reaction. Typically, urea (CH_4N_2O), carbonylhydrazide (CH_6N_4O), citric acid ($C_6H_8O_7$) [17,18], sucrose ($C_{12}H_{22}O_{11}$) [19], and glycine ($C_2H_5NO_2$) [20–22] were chosen as the organic fuel. However, the above mentioned fuels have some shortcomings respectively. For example, Xu et al. [19] synthesized nano-crystallines using sucrose as a chelating agent, but the process wasn't straightforward which need control the pH of the solution.

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Wang et al. [23] synthesized Eu:Y₂O₃ powders by combustion method with glycine as fuel. However, the results showed that the powders agglomerated seriously. Rekha et al. [18] synthesized Nd:Y₂O₃ nanopowders by combustion with citric acid as the chelating agent under the higher temperature of 1000 °C.

Compared with these fuels, the poly(acrylic acid) possesses higher relative molecular mass than other fuels, resulting in that it can release more gas (with same mole) to make the obtained powders have better dispersibility and smaller particle size than those obtained using other fuel. In addition, the preparation process does not need control the pH if the poly(acrylic acid) was served as the fuels. Therefore, poly(acrylic acid) ([C₃H₄O₂]_n) is a superior organic fuel to prepare Y₂O₃ powders through solution calcination method. The synthesis reaction of Y₂O₃ powders with poly(acrylic acid) and salts containing Y³⁺ can be expressed in Eq. (1). However, so far, there is still no report about polyacrylic acid used as organic fuel to prepare Y₂O₃ powders.



In this paper, a new organic fuel, polyacrylic acid (PAA, MW ~ 3000), was firstly adopted to synthesize nano-sized Y₂O₃ powders according to the solution combustion method. The improved combustion method required no tedious operation or restriction conditions. In addition, the effects of organic fuel dosage and calcination temperature on the characteristics of the powders were studied by XRD, FTIR, DSC-TG, and TEM.

2. Material and methods

Yttrium nitrate hexahydrate (Y(NO₃)₃·6H₂O, 99.9%, Aldrich) and poly(acrylic acid) (PAA, MW ~ 3000, 99.9%, Aldrich) were dissolved in a minimum amount of deionized water (mother solution). The mass ratio of yttrium nitrate hexahydrate (Y) to poly(acrylic acid) (P) was about 7:0 to 7:2 (Y:P = 7:0–7:2). The mother solution containing these two raw materials was stirred and mixed well with a glass rod. The mother solution was transferred into a corundum crucible and then placed in a muffle furnace. The samples were heated to the selected temperature (400, 500, 600, 700 and 800 °C) at the heating rate of 5 °C/min for 60-min dwell. The synthesized powders, without any further process, were investigated by X-ray diffraction (XRD, Rigaku Dmax-rC, Japan), transmission electron micrographs (TEM, JOEL JEM-2100, 200 kV), Fourier Transform Infrared Spectrometer (FTIR, VERTEX 70, Bruker, Germany), and thermogravimetric analysis/differential scanning calorimetry analysis (TG/DSC, Mettler-Toledo 1, Switzerland).

3. Results and discussion

Fig. 1 shows the typical TG/DSC curves for dry gel with Y:P = 7:1. The measurement was performed in air within the temperature range from room temperature to 800 °C under the heating conditions at the heating rate of 5 °C/min. A broad endothermic peak and weight loss can be observed at about 100 °C due to evaporation of water in the precursors. Two obvious exothermic peaks at 299.2 °C and 422.3 °C are observed. These two exothermic processes can be respectively interpreted as the decomposition of PAA and the oxidation reaction of nitrate because two obvious weight loss stages are observed in the corresponding temperature ranges (260–301 °C and 399.2–441.6 °C). The decomposition temperature of PAA is basically same with those reported by other authors [24,25]. Weight loss in the temperature range of

25–550 °C accounts for about 71.6% of total weight. When the temperature is above 550 °C, no weight change or thermal signal is found. Based on the TG/DSC curves analysis, it is reasonably supposed that crystalline Y₂O₃ had been formed before the temperature rose above 550 °C.

According to the TG/DSC curves, Y₂O₃ powders were fabricated under a series of calcination temperatures. The influence of the calcination temperature on the XRD patterns of Y₂O₃ powders obtained with Y:P = 7:1 is shown in Fig. 2. The diffraction peaks of all the samples calcined at 400, 500, 600, 700 and 800 °C are consistent with those in JCPDS card No. 83-0927. As shown in Fig. 2(a), in the sample calcined at 400 °C for 1 h, typical diffraction peaks of the cubic phase Y₂O₃ are observed although these peaks are weak and broad, indicating that the Y₂O₃ has been formed at just 400 °C. The low formation temperature of Y₂O₃ may be interpreted as follows: the exothermic reaction between the metal nitrates and fuel can increase rapidly the surrounding temperature. With the

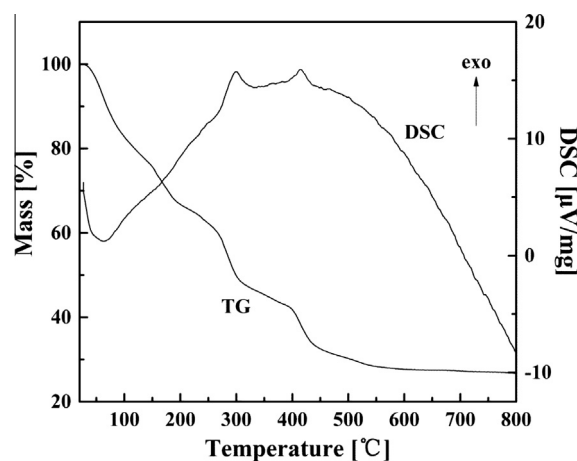


Fig. 1. TG/DSC curves for the precursors of Y₂O₃ (Y:P = 7:1).

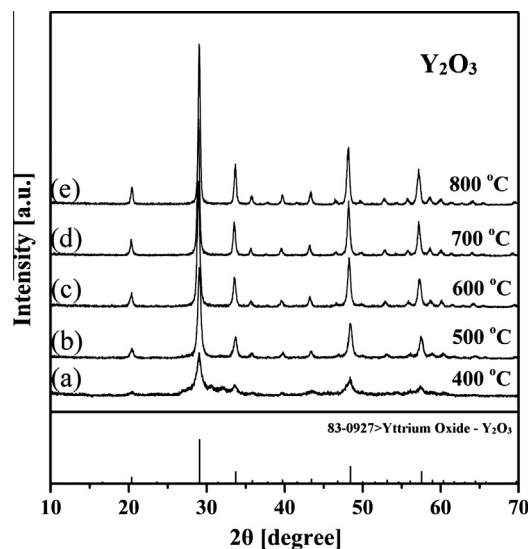


Fig. 2. XRD patterns of Y₂O₃ powders obtained at different calcination temperatures with Y:P = 7:1 (a) 400 °C; (b) 500 °C; (c) 600 °C; (d) 700 °C; (e) 800 °C.

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