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Synthesis of yttria nano-powders by the precipitation method: The influence of ammonium hydrogen carbonate to metal ions molar ratio and ammonium sulfate addition

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A B S T R A C T

Yttria (Y_2O_3) nano-powders were synthesized by the normal-strike precipitation method. The influences of R (NH_4HCO_3/M^{3+}, molar ratio) and ammonium sulfate addition were studied throughout the preparation process. Several analytic techniques such as XRD, TG/DSC, SEM and FTIR were used to determine the characteristics of the precursors and Y_2O_3 powders. It was found that morphologies and phases of the precursors were evidently influenced by the R value. The phases of the precursors were $Y_2(CO_3)_3\cdot 2.79H_2O$ when R was 3–4, while the composition of the precursors with R = 4–5 was $(NH_4)_aY(OH)_b(CO_3)_c\cdot xH_2O$. Homogeneous needle-flake shaped particles composed of coadjacent grains with diameters of about 70 nm were obtained as the R = 3–4. The rhombohedral flake-shaped particles were the products with R = 5–6. The mechanism of the influence of R value on the precipitation of the precursor was also investigated in this work. Pure Y_2O_3 nano-powders were obtained by calcining the precursors at 1000 °C for 4 h regardless of what the R value was. Sulfate ions affect not only dispersion of nanopowders, but also their grain size. Results obtained in this work are contributed to the controllable synthesis of yttria powders.

1. Introduction

Yttria finds potential applications in high-temperature-resistance windows [1], lighting and display applications [2,3], host materials for solid state laser or scintillator [4–9] and so on because of its excellent physical and chemical properties, such as high temperature stability, high corrosion resistance, and nice transparency over a wide wavelength region (from violet to infrared light) [10,11]. Conventionally, Y_2O_3 transparent ceramics are prepared by hot pressing or by normal sintering in a reducing atmosphere with ThO_2, La_2O_3, HfO_2, and LiF as sintering aids [12–16]. The temperature needs to be 2000 °C under normal pressure with long periods, or at about 1500 °C under high pressure. Generally speaking, the fabrication of Y_2O_3 transparent ceramics is considered to be very difficult. A well sinterable powder with a small particle size and low-agglomeration is crucial for the fabrication of Y_2O_3 transparent ceramics. Nanoscale powders as starting materials are found to have tremendous potential to fabricate transparent ceramics at lower sintering temperature. Well-sinterable Y_2O_3 powders have been successfully prepared by some methods, such as sol–gel [17,18], combustion [19,20], precipitation [21,22], homogeneous precipitation [23,24], laser synthesis [25,26] and so on. Among them, precipitation is one of the most promising techniques for the preparation of Y_2O_3 nano-powders due to its feasibility, low-cost and environmental protecting features. Ammonia water and ammonium hydrogen carbonate are usually selected as precipitant in the precipitation [21,22]. A gelatinous hydroxide precursor with severe agglomeration which causes poor sinterability of the resultant powders is the main product by using ammonia water as precipitant. Comparing with ammonia water, the precursor obtained by ammonium

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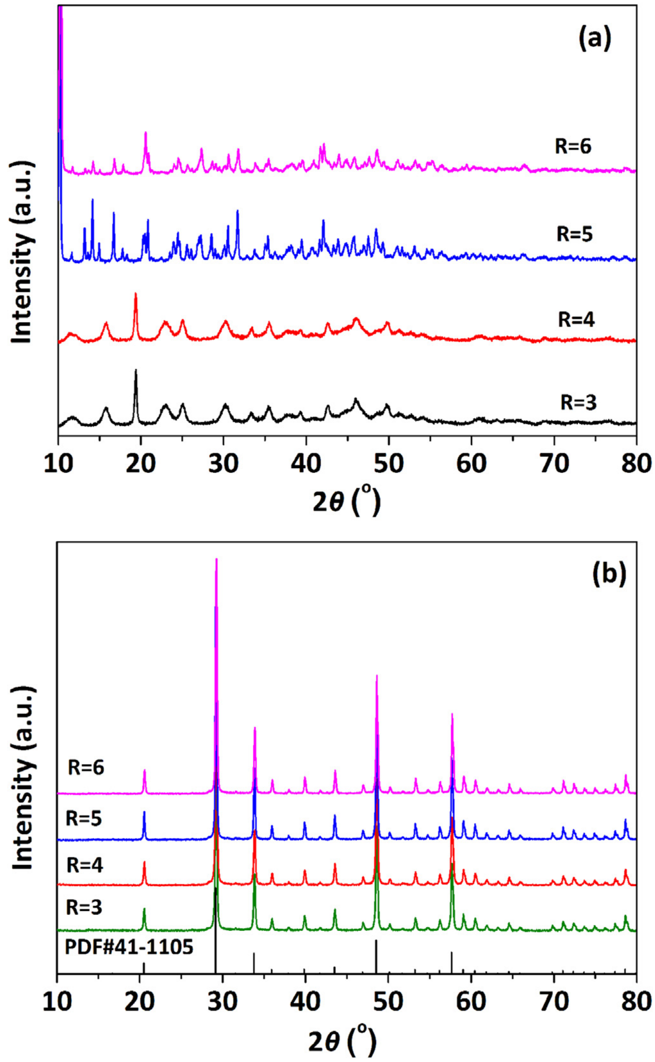

Fig. 1. XRD patterns of (a) precursors synthesized with different R value and (b) yttria powders calcined at 1000 °C for 4 h.

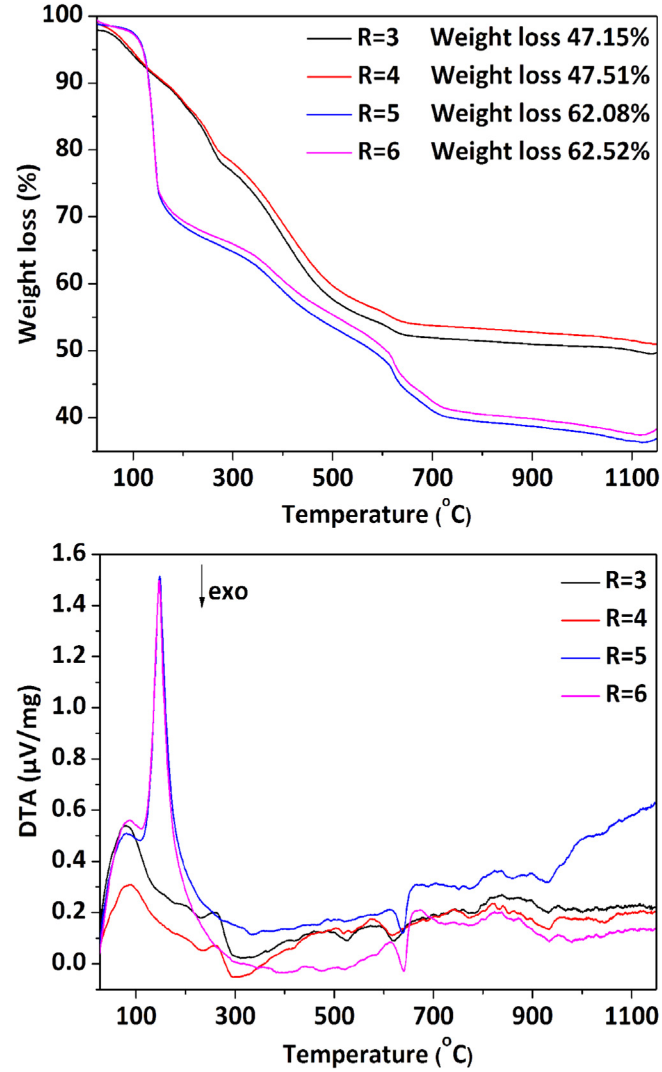

Fig. 3. TG-DTA curves of the synthesized precursors with different R values.

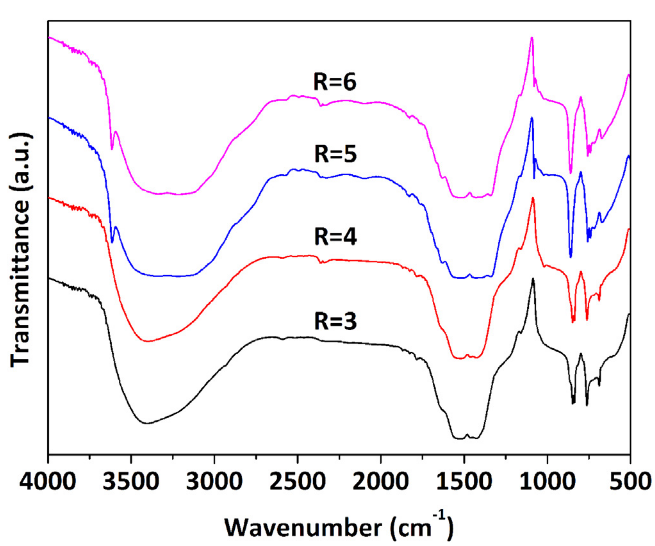

Fig. 2. FTIR spectra of the synthesized precursors with different R values.

hydrogen carbonate is loosely agglomerated after drying [27,28], and the resultant powders showed good dispersion and sinterability. The addition of ammonium sulfate to the powder (measured by weight) plays an important role in the morphology of Y_2O_3 powders [29]. It is well known that precipitant has great influence on morphology, phase and microstructure of the precursor and the resultant Y_2O_3 powder and ammonium hydrogen carbonate is favorable because of its advantages. According to Dulina [30] and Li [31], ammonium hydrogen carbonate to metal ions ratio can influence chemical and phase composition of precursor. However, there is less report on the influence of ammonium hydrogen carbonate to metal ions ratio on morphology and phase of Y_2O_3 precursor and Y_2O_3 powder. In this research, ammonium hydrogen carbonate has been employed as precipitant and $(NH_4)_2SO_4$ as dispersing agent. Normal-strike precipitation method was used to prepare Y_2O_3 precursor. The influence of R (NH_4HCO_3)/(M^{3+}, molar ratio) was studied via the precipitation process. The mechanism of the R value influence on the morphologies of the precursors was also investigated. To obtain favorable dispersibility Y_2O_3 nanopowders, the optimal dosage of ammonium sulfate was studied in this work.