

Study on the formation process of Al_2O_3 – TiO_2 composite powders

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Abstract

Fine particles of anatase were suspended in solutions of ammonium alum with $\text{Al}_2\text{O}_3/\text{TiO}_2$ molar ratios from 0.1:1 to 7:1. By spray drying the suspensions and calcining the spray-dried powders, Al_2O_3 – TiO_2 composite particles were obtained. The results show that after the spray drying, coatings of ammonium alum are formed on the surface of the anatase particles, leading to composite precursor powders (CCPs) with larger particle sizes. Upon calcining the CCPs, ammonium alum pyrolyzes to amorphous Al_2O_3 and anatase transforms into rutile. Both are mainly responsible for the observed particle size reductions as well as the densification of each composite particle. The in-situ formed α - Al_2O_3 and rutile may have higher reactivities, forming aluminum titanate at 1150 °C, about 130 °C lower than the theoretical temperature for the formation of Al_2TiO_5 by solid reaction. The reaction between α - Al_2O_3 and rutile starts from the interface between the anatase and the alum coating and mainly takes place in the single particles formed by spray drying. The molar ratio of Al_2O_3 to TiO_2 influences the final crystalline phases in the composite powders, but not stoichiometrically.

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1. Introduction

Al_2O_3 ceramics have been widely used in many fields because of their good mechanical properties, such as high hardness and super abrasion resistance [1,2]. Recently, researchers have tried to improve their performance through additions of other oxides [3–6]. Xiao and his coworkers found that protective coatings on metals, which were prepared from Al_2O_3 – TiO_2 composite powders by flame-spraying, have much better abrasion resistance than those consisting of pure Al_2O_3 powders [6]. Wunderlich et al. pointed out that nano-hybrid Al_2O_3 – TiO_2 might be applied as catalysts [7]. In addition, ceramics made from Al_2O_3 – TiO_2 composite powders show attractive perspectives. Aluminum titanate ceramic is a good example. Owing to its excellent thermal expansion behavior [8–10], it can be used as thermal resistant materials, such as catalyst carriers for purification of fume produced by cars, as containers and tubes for storing or conveying high temperature steel liquid and as protective tube for thermal couples, etc.

Traditionally, Al_2O_3 and TiO_2 are ball-milled to form composite powder mixtures for the subsequent shaping and sintering process [10]. Other authors have tried new methods such as wet chemical synthesis and high temperature oxidation [7,11,12]. We use spray drying, an industrial technology, to form composite precursor powders (CPPs) of ammonium alum and titania. After calcining the precursor powders, Al_2O_3 – TiO_2 composite particles with different crystalline phases are obtained. The most obvious character of the spray drying is that the as-prepared powders are uniformly spherical, which is beneficial to the following conveying and shaping procedure in the manufacturing of ceramic products. In addition, the spray drying introduces less impurity and saves time and energy compared to the traditional ball-milling. We also find that upon calcination the in-situ formed components inside the CPPs show higher chemical reactivities.

2. Experimental

Fine anatase powder produced by the sulfate process (Yuxing Chemical Industrial Factory, Jinan) was suspended

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Table 1

Designed compositions of Al_2O_3 – TiO_2 composite powders

Sample no.	AT1	AT2	AT3	AT4	AT5
$\text{Al}_2\text{O}_3/\text{TiO}_2$ molar ratio	0.1:1	0.6:1	1:1	1.4:1	7:1

in the solutions of ammonium alum (Dagang Yizhong Chemical Industrial Factory, Tianjing). The molar ratios of Al_2O_3 to TiO_2 (A/T ratio) were changed from 0.1:1 to 7:1 (see Table 1).

Spray drying was performed on a QP-3 spray-drier (Research Institute of Chemical Industry, Beijing). The conditions for the spray drying were as follows: pressure of the atomizing air, 0.1–0.2 MPa; flow of feeding, 30 ml/min; temperatures at the inlet and outlet of the spray-drier, 200 ± 10 °C and 100 ± 10 °C, respectively.

Powders collected from the spray-drier were calcined in an electrical furnace with a temperature precision of ± 5 °C. The temperatures were set at 700, 864, 1150 and 1368 °C, respectively, with a heating rate of 10 °C/min. The calcined samples were cooled in air directly from high temperatures.

X-ray diffractograms (XRD) were recorded on a D/max-rA diffractometer (Rigaku, Japan) to identify the crystalline phase in the calcined samples. A S-2500 Scanning Electron Microscope (SEM) (Hitachi, Japan) equipped with an Energy Dispersive Spectrum (EDS) analyzer (Oxford Co., Britain) was used to analyze the morphology and composition of the powders. The measurements of the powder particle size and its distribution were performed on a FAM Laser Particle Diameter Analyzer (Pike Instrument Co., Shanghai). Histograms of the particle size distribution (PSD) based on the mass frequency were drawn according to the original data. Modes, representing the values that occur most frequently in the distributions, were labeled on the histograms.

3. Results and discussion

3.1. Formation of the composite precursor powders (CPPs) upon spray drying

Most of the particles prepared by the spray drying are dispersed and spherical (Fig. 1a).

A comparison among the mean particle sizes (d_{50}) of the original anatase and the spray-dried powder shows an increase of d_{50} from 4.48 to 17.24 μm , indicating a significant increment of the particle size. Taking AT3 as an example, Figs. 2a and b depict the histograms of particle size distribution of the original anatase powder and the spray-dried powder. As compared with the columns in Fig. 2a, the heights of the first eight columns in Fig. 2b decrease in contrast with increases of the height of the subsequent columns, suggesting that after the spray drying, larger particles are formed. It can also be seen that after the spray drying, the mode shifts to a larger particle size value (Fig. 2b). Meanwhile, the particle size distribution becomes broader, indicated by new columns in the size fractions above 53.5 μm .

According to the mechanism of spray drying [6], ammonium alum coating is supposed to be formed on the surface of TiO_2 particles. Moreover, EDS analysis (Fig. 1b) reveals that these particles, bigger or smaller in size, all contain elements of O, Al, S and Ti, indicating that spray drying the suspension with anatase powder in the alum solution successfully leads to the formation of composite powder consisting of TiO_2 and ammonium alum.

TGA analyses reveal that upon heating, the weight losses of the CPPs depend on the A/T ratios (Fig. 3). The larger the A/T ratio is, the more the weight loss is observed. Since the weight loss is mainly due to the thermal decomposition of alum in the CCP, this correlation between the weight losses and the A/T ratios indicates that thicker alum coatings are formed on the surface of anatase in the case of higher A/T

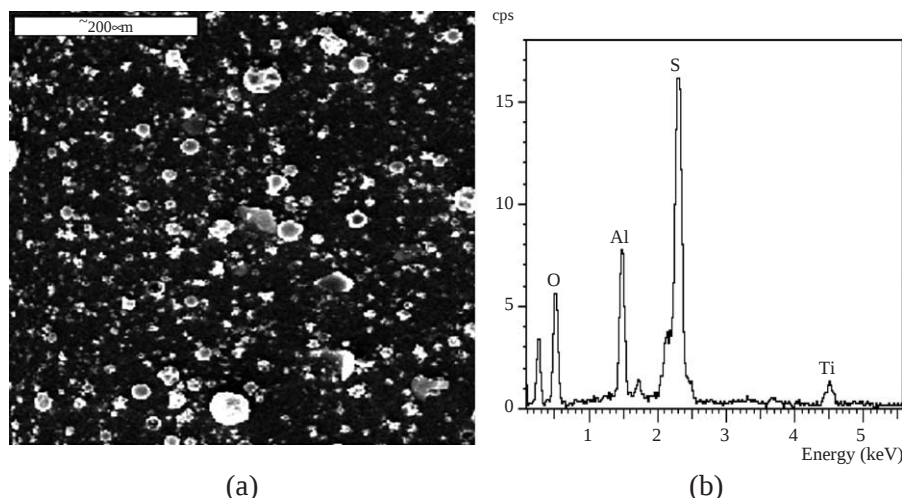


Fig. 1. SEM graph (a) and EDS (b) of the composite precursor powder.

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