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Low temperature synthesis of nano alpha-alumina powder by two-step hydrolysis

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ABSTRACT

The ultral fine alpha-alumina powder has been successfully synthesized via two-step hydrolysis of aluminum isopropoxide. Glucose and polyvinyl pyrrolidone were used as surfactants during the appropriate processing step. The alpha-alumina powder was used as seed particles. Several synthesis parameters, such as the amount of seeds, surfactants, and calcination temperature, were studied by X-ray diffraction (XRD), Fourier transform infrared spectra (FTIR), Thermogravimetry-differential scanning calorimetry (TG-DSC), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The experimental results showed that glucose greatly lower the phase transformation temperature of alpha-alumina by impelling the gamma-alumina transformed to alpha-alumina directly, and the seed could improve the phase transformation rate of alpha-alumina, the polyvinylpyrrolidone have an effect on preventing excessive grain growth and agglomeration of alpha-alumina powder. Comparatively well dispersed alpha-alumina powder with particle size less than 50 nm can be synthesized through this method after calcinations at 1000 °C for 2 h.

1. Introduction

Nano α-alumina is one of the most important oxides for a wide range of applications in ceramic, high strength materials, functional materials, adsorbents, catalysts, catalyst support and transparent armours for ballistic performance [1–4], which are attributed to its excellent performance such as high mechanical strength, good wear resistance, low conductivity, high refractoriness, high hardness and high corrosion resistance.

In order to prepare nano α-alumina powder with good performance, a lot of approaches such as mechanical milling [5], vapor phase reaction [6–7], microemulsions [8–9], precipitation [10], sol–gel [11–12], hydrothermal [13–14] and combustion methods [15] have been developed. However, alumina powder exists in a lot of metastable transition phases, the transformation proceeds of α-Al_2O_3 usually through the following sequence: γ-$Al_2O_3 \rightarrow$ s-$Al_2O_3 \rightarrow \theta$-$Al_2O_3 \rightarrow$ bα-Al_2O_3 [16]. While the nanoscale α-alumina powder is difficult to obtain, because of the following reasons: first, the α-Al_2O_3 is the most thermodynamically stable phase after calcination at high temperature, the transformation of α-Al_2O_3 from θ-Al_2O_3 always relates to a significant change in the oxygen sublattice from cubic peaking to hexagonal close packing [17], the reconstructive transformation needs relatively high activation energy compared with γ-$Al_2O_3 \rightarrow \sigma$-$Al_2O_3 \rightarrow \theta$-$Al_2O_3$ transformation. As a result, the phase transformation temperature for almost salt-derived aluminum hydroxides and hydrated alumina form to α-Al_2O_3 is always at 1100–1250 °C. the high temperature easily prompt the grain growth of powder, which makes it difficult to get nanoscale particles; secondly, the phase transformation of α-Al_2O_3 from θ-Al_2O_3 is performed by a nucleation and growth mechanism, the α-Al_2O_3 nuclei form within the ultrafine θ-Al_2O_3 matrix, and then rapidly grow to produce α-Al_2O_3 colonies [18], which inevitably results in certain degree of particle coarsening. For the above reasons, the α-alumina prepared by common methods shows serious agglomeration, so eliminating the agglomeration in the subsequent mechanical treatment is necessary, which inevitably introduced impurities.

Thus, lowering the phase transformation temperatures is the target for preparing the nanoscale α-Al_2O_3. Currently, there are many reports about lowering the phase transformation temperatures of α-Al_2O_3. Li et al. [19] proposed that citrate polymeric precursor derived from aluminum nitrate and citric acid mixed solution promoted the γ-Al_2O_3 transformed to α-Al_2O_3 directly, which greatly reduced the transformation of α-Al_2O_3. Some studies [20–22] indicated that phase transformation temperature of θ-$Al_2O_3 \rightarrow$ can be influenced by some metal cations such as Fe^{2+},

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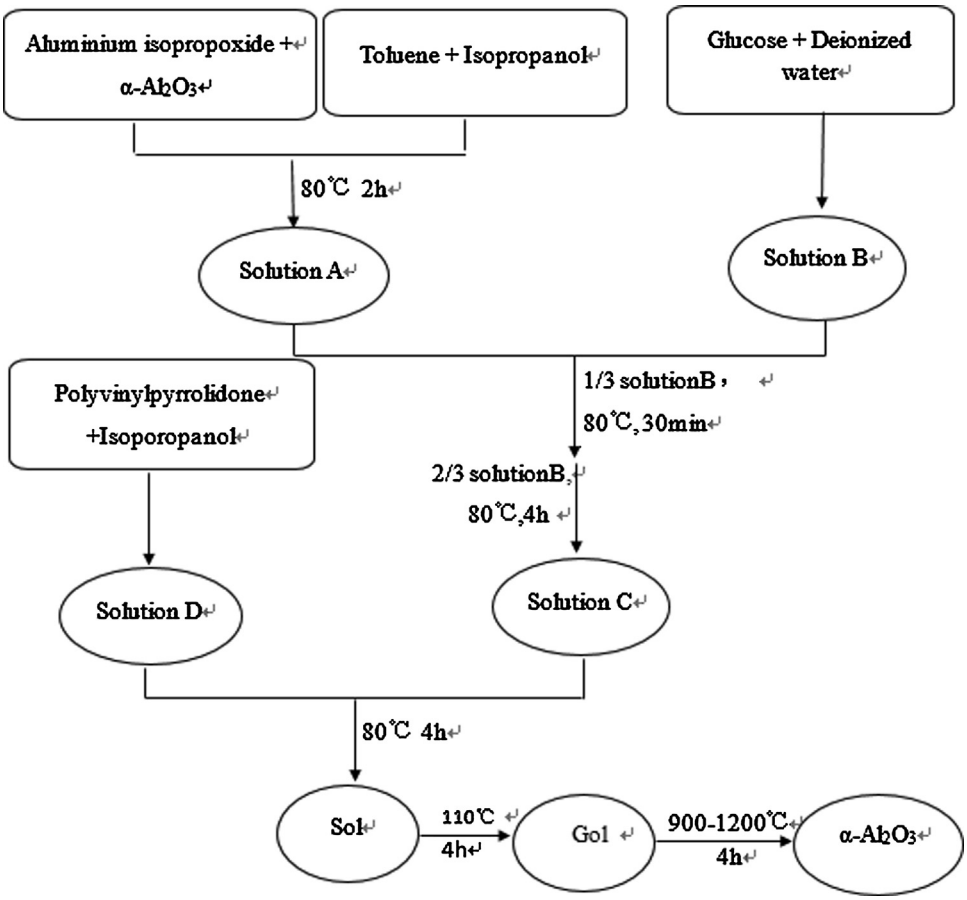

Fig. 1. Flow diagram for preparation of Samples.

$Cr^{3+}$and Ti^{4+}. However, the addition of inorganic ion inevitably introduced impurities, which reduced the purity of α-Al_2O_3, therefor limits its application in some areas. The previous studies [23–25] have indicated that the α-Al_2O_3 seeding can provide the nucleation sites in the process of the phase transition to reduce the activation energy and phase transformation temperature, besides, the α-Al_2O_3 seeding can prevent the formation of vermicular structure during sintering.

Herein, we present an easily accessible, reproducible, and high-throughput method to synthesize high purity and homogenous nano α-Al_2O_3 through two-step hydrolysis of aluminum isopropoxide, the α-Al_2O_3(<100 nm) seeds were introduced to reduce the transformation temperature of α-Al_2O_3. In addition, glucose and polyvinyl pyrrolidone were used as surfactants in the process of hydrolysis. Decomposition of the precursor, phase transformation and morphology of the synthesized alumina powder are investigated.

Table 1
Synthesis conditions of different samples.

Sample	Seed wt%	Glucose wt%	Polyvinlyrrodidone wt%
Al-1	–	30	–
Al-2	–	–	5
Al-3	–	30	5
Al-4	3.5	30	–
Al-5	3.5	–	5
Al-6	2	30	5
Al-7	3.5	30	5
Al-8	5	30	5

2. Experimental

Aluminum isopropoxide $Al(OC_3H_7)_3$ (Xuancheng Jingrui New Material Co., Ltd), glucose($C_6H_{12}O_6$) (A.R, Sinopharm Chemical Reagent Co., Ltd), and polyvinylpyrrolidone (k-30) (A.R., Sinopharm Chemical Reagent Co., Ltd) were used as raw materials to prepare α-Al_2O_3 powder. Solution A was prepared by dissolving the aluminum isopropoxide with or without the seed of α-Al_2O_3

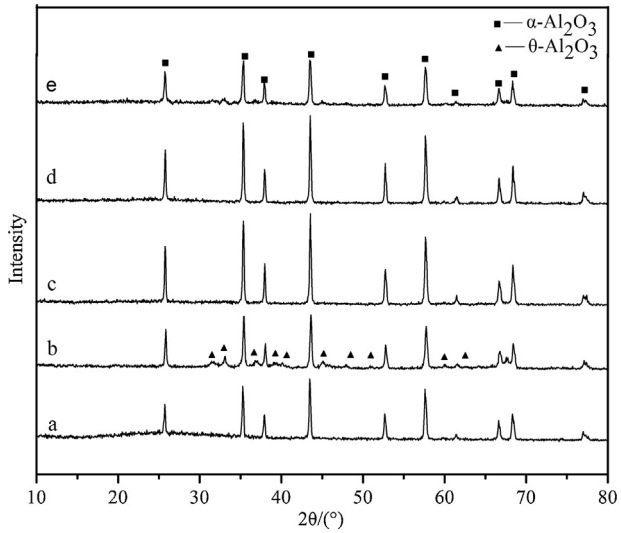

Fig. 2. XRD patterns of samples (a)Al-1, (b)Al-2, (c)Al-3, (d)Al-4, (e) Al-5 after being treated at 1100 °C for 2 h.