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Morphology investigation of alumina nano-powders prepared by a sol-gel combustion method

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ABSTRACT

Highly sinterable alumina nano-powders have been synthesized by a sol-gel combustion method with glycine as fuel. The preparation involved the thermal decomposition of a chelating agent (fuel)-nitrate gel and the formation of amorphous precursors. The nanopowders calcined at 1100°C were characterized by X-ray powder diffraction (XRD) and Scanning Electron Microscopy (SEM). It was found that the chelating agent and fuel used had a significant influence on the average grain size of α-Al_2O_3 nanopowder. The rate of combustion reaction between chelating agent (fuel) and nitrate was responsible for the growth of the grain.

Keywords: Alumina Nanopowder; Sol-gel Combustion Method; Glycine; Fuel

INTRODUCTION

Ceramics have many application in high technology from structural to electrical and electronic because of their excellent properties, but sometimes their low mechanical properties limit their wide applications. However, nanocrystalline ceramics can enhance their mechanical properties. So nanometer size powder processing is of great importance in the range of nanotechnology since it affords to fabricate various kinds of nanocrystalline materials and the nanocomposite ceramics have such advantages over monolithic ceramics as high strength and high toughness [1, 2].

Surface activity of ultrafine powders is one of the major driving forces in the densification process during solid state sintering. Improved sinterability is attained by using powder with controlled morphology, size and size distribution. These requirements are achieved by using synthesized powders. The common precursors include either alkoxides or inorganic salt.

Yoldas [3-7] dealt with the preparation of alumina sols from alkoxides and adjusted the conditions of preparation. Al-isopropoxide and Al-sec- butoxide were used as precursors. Hydrolysis and peptization reactions of the alkoxide governed the particle size achieved. In a review, Blendell et al. [8], they demonstrated how sub-micron, monosized particles could be formed by heating Al-chloride and perchloride solutions. Boehmite is the alumina phase obtained. Johnson [9] recorded that alumina powder can be made by spray drying with an average particle size 10μm. However, smaller sizes around 1μm could be obtained by aerosol technique. Cornilsen and Reed [10] used urea as an agent to produce aluminium hydroxide precipitate that is easier to filter than that obtained by rapid addition of a base. Precipitation depends on the hydrolysis of urea and the gradual rise in pH which can precipitate a basic Al salt on heating at 80-90°C. In comparison with urea, formamide is a better precipitating agent. It does not yield a gas allowing better control of contamination. It may be better than urea for producing monosized particles because of the high initial rate of pH

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change as reported by Blendell et al. [8]. Djurcis [11] used glucose and urea 1:2 in molar ratio to produce fine alumina powder. Thus the organic complex was made by melting at 150°C.

To obtain nanostructure ceramics, nanoscale powders with high performance are necessary. However, nanoscale α-alumina (α-Al_2O_3) powder is difficult to obtain, because of two reasons: First, α-Al_2O_3 is in a stable phase after calcining at high temperature, which easily prompt the grain growth of powder, and make it difficult to get nanoscale particles; secondly, α-Al_2O_3 particles tend to aggregate during dehydration process in wet chemistry method. Therefore, it is necessary to develop new methods to overcome this problem [12, 13].

Conventional synthesise process of α-Al_2O_3 involve mechanical milling, vapor phase reaction, precipitation, sol-gel, hydrothermal and combustion methods. Mechanical synthesise of α-Al_2O_3 requires extensive mechanical ball milling which easily introduces impurities. Vapor phase reaction for preparation fine α-Al_2O_3 powder from a gas phase precursor demands high temperature above 1200°C. The precipitation method suffers from its complexity and time consuming (long washing times and aging time) [14, 15]. The direct formation of α-Al_2O_3 via the hydrothermal method needs high temperature and pressure. The combustion method has been used to yield α-Al_2O_3 powders, whereas the powder obtained from the process is usually hard aggregated but contained nano-sized primary particles [16]. Sol-gel, a commonly used technique, involves the formation of an amorphous gel from a precursor solution. This method based on molecular precursors usually makes use of metal alkoxides as raw material. Some advantages of the sol-gel method are better homogeneity and purity from raw material, lower preparation temperature which save energy cost and the ability to form unique composition [17]. The purpose of this work is to senthesize nano-size α-Al_2O_3 by controlling the particle size distribution and shape.

EXPERIMENTAL

Nanoparticle of α-Al_2O_3 are prepared by sol-gel auto combustion method [18-24]. Appropriate amounts of analytical grade $Al(NO_3)_3.9H_2O$ is taken and mixed with glycine that were collected from merck. Shematic flow chart of sol-gel processing that was applied in this study is shown in Fig. 1. The molar ratio of aluminium nitrate to glycine was calculated as 1:3. The mixture is then dissolved in deionized water. The addition of glycine helps the homogenous distribution and segregation of the metal ions. A small amount of ammonium hydroxide is added carefully to the solution to change the pH value to 7. The solution was refluxed under magnetic stirring at 80°C for 6 h. Condensation reaction occurs between the adjacent metal nitrate and the molecules of glycine yielding a polymer network in colloidal dimenstion known as sol. The resultant sol is poured in a silica crucible and heated at 135°C under constant stirring to transform into a xerogel. Continuous heating of xerogel leads to the formation of nanopowders of α-Al_2O_3through a self-propagating combution process. The powder obtained after auto-ignition were calcined at 1100°C for 1h to obtain pure and well crystalline α-Al_2O_3 powders.

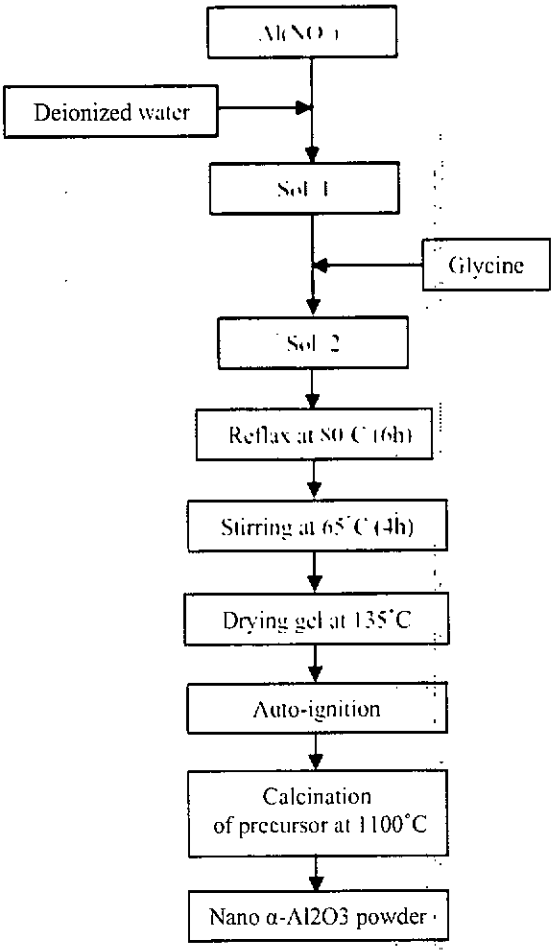

Fig. 1. Flow chart of the α-alumina sol-gel processing

Phase identifications were performed by X-ray diffraction XRD pattems of nanopowders were obtained with an X-ray diffractometer (Model: XPERT-MPD, philips) using Cu Kα radiation (λ= 10540 Å) with operated at 40 KV and current of 40 mA. The shape and morphology of powder were analyzed by Scanning Electron Microscopy (SEM-philips XL 30).

RESULTS AND DISCUSSION

The XRD pattems of the α-Al_2O_3 nano crystalline powders is shown in Fig. 2. The particle size of the samples has been determined employing the Scherrer equation:

$$D= K\lambda/\beta\cos\theta \qquad (1)$$

Where β is the full width half maximum (rad.), λ the wavelenght of the X-ray, θ the angle between the incident and diffracted beams (degree) and D the particle size of the sample (nm). The result of XRD shows at lower temperature the diffraction lines have confirmed the formation of single phase of α-Al_2O_3 nanoparticles.

The XRD analysis in Fig. 2 showed the most stable phase, α-Al_2O_3 occurred dominantly at 1100°C for 1h. Although the thermodynamically stable α-Al_2O_3 phase can be obtained through a sequence of topotactic and reconstructive transformations, the morphology remains unchanged and the final products have the same shape as the initial phases. The grain sizes of the prepared Alumina are found to be in the range 22-27nm.

CONCLUSIONS

The sol-gel method was used for the synthesis of nano α-Al_2O_3 from aluminum nitrate. Selection and control of the precise reflax and stirring time together proved to be important in controlling the particle size, degree of aggregation and the particle shape. Nano α-Al_2O_3 was checked at 1100°C for 1h, when the transformation was just completed. The shape of the α-Al_2O_3 nanoparticles was basically spherical. The particle size of α-Al_2O_3 nanoparticle was between 22-27nm.

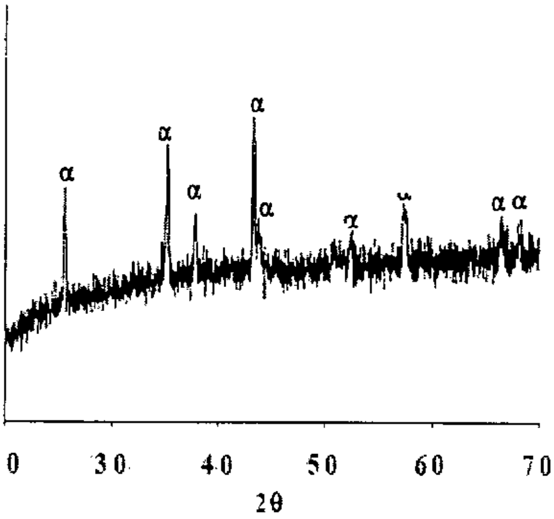

Fig. 2. The XRD pathern for α–alumina nanoparticles.

The structural morphology of the nanoparticles was investigated using Scaning Electron Microscopy (SEM). Fig. 3 shows SEM micrograph. Besides that, the particle size and shape of the α-Al_2O_3 is determined by the crystal structure of the series of phase transformations which occur during calcinations.

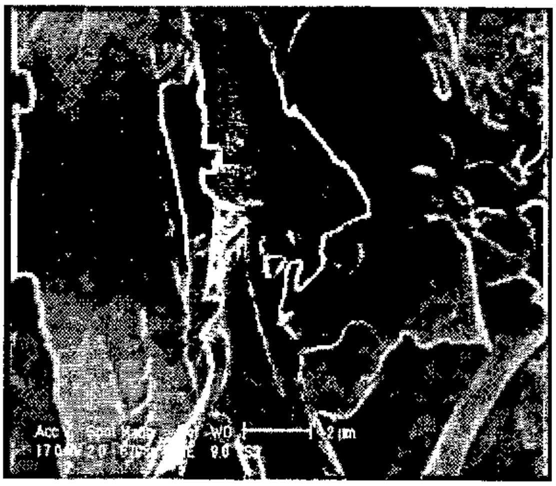

Fig. 3. SEM image of α-alumina nanoparticles.

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