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Preparation and characterization of alpha alumina nanoparticles by in-flight oxidation of flame synthesis

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ABSTRACT

Alpha alumina nanoparticles were synthesized from micron-sized commercial aluminium powders by in-flight oxidation of flame synthesis. The synthesized alumina nanoparticles were characterized using X-ray diffraction (XRD) and α phase alumina was confirmed. The average grain sizes were estimated to be 98 nm using Scherer's formula. The formation of alumina nanoparticles was identified by scanning electron microscopy (SEM). Transmission electron microscopy (TEM) study shows the different sized spherical nanoparticles ranging from 70 nm to 150 nm. The energy dispersive X-ray analysis (EDAX) confirms the presence of aluminium and oxygen in the α-Al_2O_3 nanoparticles. The Photoluminescence spectrum of α-Al_2O_3 nanoparticles reveals the presence of a large amount of oxygen vacancies.

1. Introduction

Research on synthesize of nanosized alumina (Al_2O_3) has been extensively investigated due to its excellent properties such as high stability, high hardness, high insulation and transparency [1]. Alumina has several crystalline phases (polymorphs) namely, delta (δ), gamma (γ), theta (θ), and alpha (α). Among them, alpha (α-Al_2O_3, corundum) is one of the most extensively used ceramic materials due to a its significant properties such as; high mechanical strength and hardness, high corrosion, thermal conductivity, and abrasion resistance [2–4]. α-Al_2O_3 with excellent properties possess a promising applications in electronics [5], optoelectronics [6], and reinforcement filler in composites [7,8]. Al_2O_3 nanoparticles can be synthesized by various techniques including ball milling, sol–gel, pyrolysis, sputtering, hydrothermal, laser ablation, and flame synthesis [9–15]. Furthermore, Chandradass and Bae [16] and Chatterjee et al. [17] have synthesized alumina nanoparticles by sol–emulsion–gel method using nonionic surfactants. Mazloumi et al. [18] have synthesized a-alumina nanopowders from synthetic caustic sodium aluminate solution. Synthesis of γ- and α-alumina nanopowders by the combustion method has been reported by Edrissi and Norouzbeigi [19].

So far, only a few works have been devoted to the flame synthesis of Al_2O_3 with limited emphasis on size and morphology control, particularly in the nanoscale range [20]. The principle of flame synthesis is the melting and oxidation of molten metal-particles in a flame, thereby forming stable metal–oxide followed by nucleation, and agglomeration. Among other synthesis methods, flame synthesis method has been initiated to form metal oxide nanoparticles due to their advantages of high yield, better reproducibility, and one step approach with high phase purity [20]. Furthermore, flame synthesis is an easy synthesis method where the size and morphology of the product can be tuned by changing the parameters such as composition of the precursor gas and the flame operating conditions.

Here we report, for the first time, α-Al_2O_3 nanoparticles from commercial aluminium powders using flame synthesis technique. The structure, elemental, surface morphology, particle size of alumina nanoparticles have been analysed and reported.

2. Materials and methods

The schematic diagram of the flame synthesis setup of α-Al_2O_3 nanoparticles is shown in Fig. 1. A horizontal nozzle is fitted so that the commercial aluminium powders from the feeder falls vertically into the flame emanating from the nozzle. A powder collector plate was placed at a distance of 25 cm above the flame. The commercial aluminium powders are fed into the flame which contains oxygen and acetylene gas (O_2:C_2H_2). The commercial aluminium powders powders melt and react with the atmospheric oxygen and the flame oxygen resulting in the formation of alumina nanoparticles on the surface of a powder collector. The process is carried out at three different gas percentage ratios of $25O_2$:$75C_2H_2$, $50O_2$:$50C_2H_2$ and $75O_2$:$25C_2H_2$.

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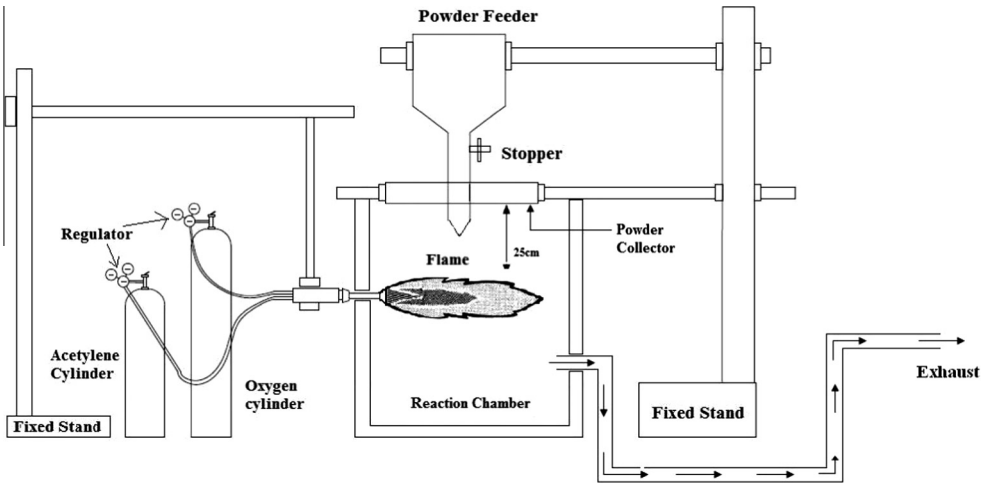

Fig. 1. Schematic diagram of flame synthesis set-up.

The synthesized α-Al_2O_3 nanoparticles from commercial aluminium powders powders are characterized by X-ray diffraction to find out the resultant phases with an X-ray diffraction (XRD) model Siemens D5000 with Cu Kα radiation (λ = 1.5406 Å and θ = 30–80°). The surface morphology of the nanoparticles is examined by scanning electron microscopy (SEM, Hitachi S2300, Tokyo, Japan). The elemental conformation of the synthesized α-Al_2O_3 nanoparticles was confirmed by energy dispersive X-ray fluorescence spectrometers (EDX-720/800HS, North America). In order to analyze the individual nanoparticles, transmission electron microscopy (TEM) is done employing Philips CM200 equipment. The particle size and distribution of the nanoparticles are observed by using Laser particle size analyzer, Fritsch model analysette 22 compact, Idar-Obestein, Germany. The surface defects and overlapping of sub bands are confirmed by photoluminescence (PL) study by using Fluorescence Spectrophotometer AE-F96PRO.

3. Results and discussion

The X-ray diffraction pattern of the commercial aluminium is shown in Fig. 2(a). The strong peaks correspond to the planes (111), (200), (220) and (311) are the characteristic of aluminium metal [21]. Scanning electron micrograph of the commercial aluminium powders is shown in Fig. 2(b). It is seen that the particles are distinguishable, irregular in shape and not agglomerated. The average particle size range estimated from SEM images is 10–20 μm. The energy dispersive X-ray analysis (EDAX) spectrum of the commercial aluminium confirms the presence of aluminium without any impurities, as shown in Fig. 2(c).

The X-ray diffraction pattern of the α-Al_2O_3 nanoparticles synthesized from commercial aluminium powders are shown in Fig. 3. The diffraction peaks of the α-Al_2O_3 nanoparticles with planes (104), (110), (113), (300), (116) and (311) corresponds to α-Al_2O_3 [22].

The grain size is calculated by using Scherer's formula [23].

$$D = 0.9\lambda/\beta \cos\theta$$

where D is the grain size, λ is the wavelength of the X-rays, β is the full-width at half-maximum of the corresponding line and θ is angle of diffraction of the peak.

The dislocation density was calculated by the relation [24].

$$\delta = 1/D^2$$

where D is the grain size.

The micro strain was calculated by the formula [25].

$$\varepsilon = \beta \cos\theta/4$$

The microstructural quantities of α-Al_2O_3 nanoparticles are calculated and reported in Table 1.

Fig. 4 shows the scanning electron microscopy images (SEM) of the Al_2O_3 nanoparticles synthesized from the commercial aluminium powders in three different ratios of oxygen and acetyl (O_2:C_2H_2). The SEM images of the alumina nanoparticles synthesized from the commercial aluminium powders in the ratio of $25O_2$:$75C_2H_2$ shows that alumina nanoparticles are in different size and in the decomposed state (Fig. 4(a)). The SEM images of α-Al_2O_3 nanoparticles with a ratio of $50O_2$:$50C_2H_2$ shows that all the nanoparticles are in almost uniform in size whereas for the SEM images of α-Al_2O_3 nanoparticles synthesized from the commercial aluminium powders in the ratio of $75O_2$:$25C_2H_2$ agglomerates comprising of nanoparticles can be seen, but individual nanoparticles are not distinguishable (Fig. 4(c). The elemental

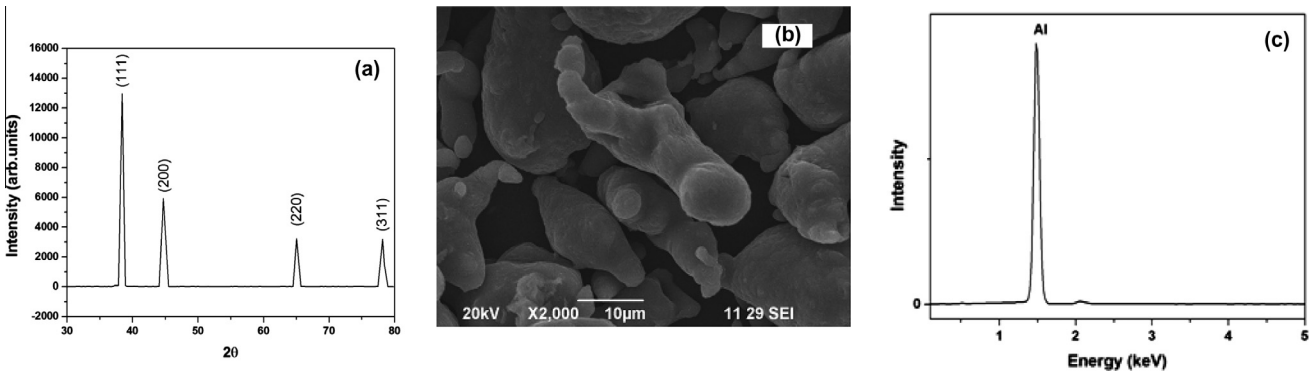

Fig. 2. Commercial aluminium powders (a) X-ray diffraction pattern (b) Scanning electron microscopy image, and (c) EDAX spectrum.