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Preparation and Characterization of Alumina Nanoparticles by Direct Injection Flame Synthesis

P. Kathirvel ᵃ˙*

ᵃ GRD Centre for Materials Research, Department of Physics, PSG College of Technology, Coimbatore-641004, Tamil Nadu, India.

* Corresponding Author: pkv.phy@psgtech.ac.in

Abstract: The article reports the synthesis of nano alumina particles by instant and continuous one step Direct Injection Flame Synthesis (DIFS) method. The synthesized nanoparticles were highly crystalline with orthorhombic crystal structure belonging to the space group of *P222*. The nano alumina particles have a crystallite size of 36 nm and the particle radius is calculated to be 24 nm. The synthesized nano alumina particle exhibits a spherical morphology with different radius. The particles were subjected to controlled aging process and the formation of encapsulation layer is reported. The thickness of the encapsulation layer is found to be ˜ 4.5 nm.

1. Introduction

Nano alumina (Al_2O_3) has found its application in the transistors, optoelectronic devices, composites, fillers, batteries and other high temperature applications owing to its physical and chemical stability at elevated temperatures, insulating properties and its excellent control over its size and morphology by economical synthesis methods [1-3]. This property has been successfully utilized in ceramic applications such as high temperature crucibles, electrical insulators, temperature resistive coating applications in defense applications [4].

This technologically important material can be synthesized by various routes including Flame synthesis, Hydrothermal, Chemical Vapor Deposition methods [1,4,5]. For this study the flame synthesis method has been chosen due to its ease synthesis process and its control over size and stoichiometry [6]. The demand for nano alumina is increasing over the year continuously and a study on highly valued nano alumina on safe storage and its shelf life period is essential. In this study, the alumina nanoparticles structure and aging effect has been investigated and reported.

2. Experimental Section
2.1 Synthesis of nano alumina

Aluminum (Al) metal powder has been purchased from SRL chem., India with a mesh size of 74 μm is chosen as the reactant material for DIFS. The experimental setup and method are already reported in our previous experiment [4]. The Al metal dust is directly fed into the neutral oxyacetylene flame white cone. This results in the spontaneous combustion and oxidation of Al metal particles into nano alumina which evolves as a white smoke from the neutral oxyacetylene flame. The resulting soot formation is collected and used for further studies. The sample collected is filled up to 20% volume of the air tight container for controlled aging observation.

2.2 Characterization

To identify and understand the crystalline nature of the samples, they were subjected to the powder X-ray Diffraction (PXRD) studies carried out by Empyrean, Malvern Panalytical instrument using the Cu source (λ=0.154 nm). To analyze the composition, structural nature, size and fine morphology of the prepared sample they were dispersed in absolute ethanol and transferred onto the copper grid followed by atmospheric drying for the Energy Dispersive X – Ray Analysis (EDAX) analysis, Selected Area Electron Diffraction (SAED) and High Resolution Transmission Electron Microscope (HRTEM) studies which were carried out using JEOL JEM 2100.
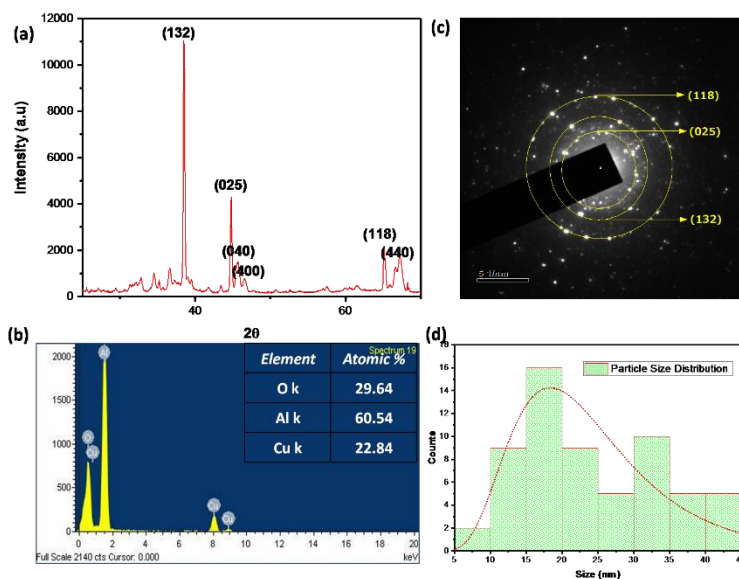
3. Results and Discussion

Figure 1. (a) PXRD pattern of nano alumina. (b) EDAX spectrum of nano alumina particles (insert: elemental composition). (c) SAED pattern of nano alumina particles. (d) Particle size distribution fitted with lognormal distribution.

3.1 Structure and Composition

The powder X-ray diffractogram (Figure 1.a) shows peaks located at 38°49″, 44°74′, 45°42′, 45°69′, 65°10′, 66°53′ and 67°17′ corresponding to the plane (132), (025), (040), (400), (118), (440) and (046) respectively. The peaks were sharp and intense indicating the good crystalline quality of the prepared sample. The peaks align with the standard JCPDS card no: 46-1215 confirming the formation of highly crystalline orthorhombic alumina belonging to the space group *P222*. The relative intensity of the planes does not match with the standard data. The most intense peak is found along (132) contrary to the (046) plane. Other planes relative intensity does not match with the standard data and the predominant growth plane confirmed by HRTEM is along (132) plane. Using the Debye – Scherrer equation (1), the average crystallite size (D) of the particle was found to be 36 nm and the dislocation density (δ) was found to be 7.71×10^{14} lines m^{-2} using equation (2)

$$D = \frac{0.9\lambda}{\beta \cos\theta} \qquad\qquad (1)$$

$$\delta = \frac{1}{D^2} \qquad\qquad (2)$$

where λ is the wavelength of the X-ray source, θ is the peak position and β is the full width half maximum of the peak. No other major peaks were observed in the PXRD indicating the phase purity of the sample. The SAED pattern (Figure 1.c) shows circularly scattered dot pattern unique to powder nanoparticles and shows only the planes of orthorhombic alumina which confirms the formation of nano alumina. The EDAX spectrum (Figure 1.b) shows only the peaks belonging to the aluminum, oxygen and copper. The copper peak arises from the copper grid used for supporting the sample during EDAX study. This proves the prepared nano alumina is free from impurities.
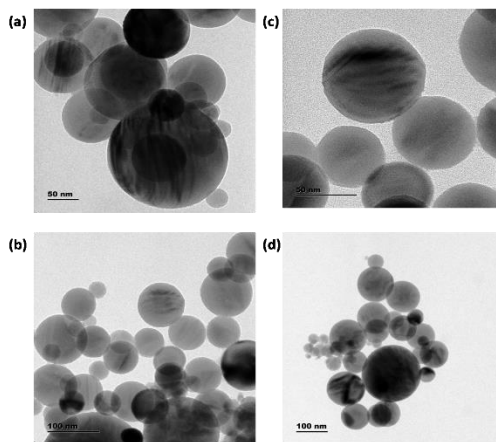
3.2 Morphology

Figure 2. (a-d) HRTEM images of freshly prepared spherical nano alumina particles without any encapsulation layer.

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to be a perfect sphere average surface area and volume of a particle is calculated using the basic

geometry equation and found to be 8.36 μm² and 86.73 μm³ respectively. The surface to volume ratio is found to be 0.9/nm. The nano alumina shows slight agglomeration of adjacent particles which is a signature of flame synthesis but compared to other traditional flame synthesis method our method agglomeration is very low evident from the **HRTEM** image [7,8].
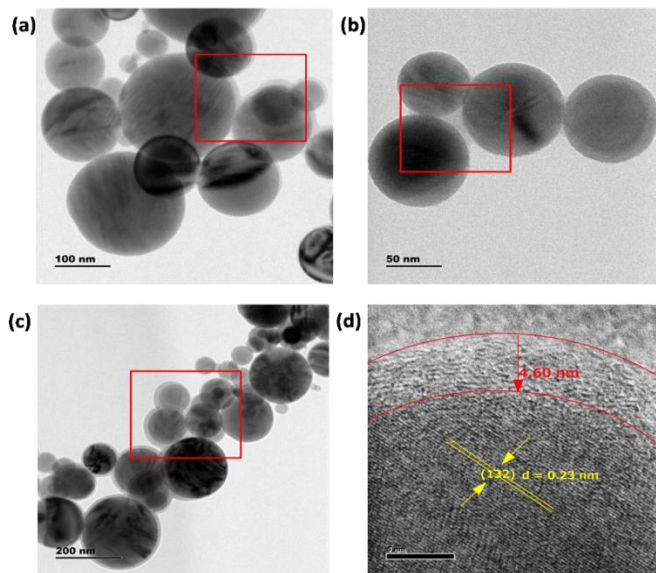
3.3 Aging Layer

Figure 3. (a) Encapsulation layer over nano alumina due to aging effect. (b-c) Growth of encapsulation layer onto adjacent particles. (d) HRTEM image of encapsulation layer.

The freshly sample is devoid of layer formation on the outer surface but after the prolonged storage of the nano alumina there forms a nanolayer (Figure 4.a-c) with the average thickness of 4 to 5 nm around the particle (Figure 4.d). The thickness of the nanolayer is found to be uniform for a single particle but varies for different particles. This shows that the exposure of the nano alumina is different for each particle. This variation may cause due to the packing condition, air pockets between the particles and environmental temperature differences. The exact origin in these differences is unclear but we believe the surface level defects plays a significant role in attracting the atmospheric species to form a layer around it. Other important observation is that after prolonged storage the nanolayer on the alumina surface begins to form contact with other adjacent particles and develops further to encapsulate the total particle system and results in agglomeration.

4. Conclusion

This investigation shows the synthesis and formation of highly crystalline orthorhombic structured nano alumina with a mean radius of 24 nm. The formation of aging effect was confirmed after prolonged storage (6 months) of the synthesized nano alumina by the direct

observation of encapsulated layer around the particle with a average thickness of 4 – 5 nm. After further aging the encapsulated layer forms contact with adjacaent particle to form a agglomerated particle system. Overall, for the prolonged storage of nano alumina particles, inert atmosphere or vacuum environment is favored for pristine condition.

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Conflict of interest: The Author has no conflicts of interest to declare that they are relevant to the content of this article.