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SYNTHESIS OF Al₂O₃ NANOPARTICLES BY LASER ABLATION OF Al IN DEIONIZED WATER

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Abstract: Alumina nanoparticles were synthesized by laser ablation of aluminium target using Nd:YAG laser with a wavelength of 1064nm. The laser beam was irradiated to the aluminium immersed in deionized water. Physical and chemical properties of the nanoparticles were investigated by field emission scanning electron microscope (FE-SEM), energy-dispersive X-ray spectroscopy (EDX), UV-visible spectroscopy and X-ray diffraction (XRD). FE-SEM images showed that most of spherical nanoparticles are less than 70 nm in diameter. EDX indicated that a ratio of atomic percentage of aluminium to oxygen is approximately 2:3. UV-Visible spectroscopy was used to evaluate the optical property of the particle suspension, representing a strong peak at 213 nm. XRD analysis showed that the nanoparticles are β-Al₂O₃, which would be very useful for many applications such as capacitor and battery.

1. Introduction

Metal oxide nanoparticles have been developed broadly in the past decades. Many applications of metal oxide nanoparticles have been investigated such as catalyst, sensor, semi-conductor, medical science, capacitors and batteries [1-6]. Among them, aluminum and its compounds have been long been known for more than century, e.g. aluminum oxide hydrate (AlOOH), aluminum trihydroxide (Al(OH)₃), aluminum oxide (Al₂O₃) [7].

Aluminium oxide or alumina normally refers to corundum. It is a white and odorless oxide. Alumina has several phases such as alpha, gamma, theta, kappa and beta. Alpha alumina phase is the most thermodynamically stable phase. In general, alumina has many interesting properties; for example high hardness, high stability, high insulation and transparency [8]. Alumina is also widely used in fire retard, catalyst, insulator, surface protective coating and composite materials [9-13].

Alumina nanoparticles can be synthesized by many techniques including ball milling, sol-gel, pyrolysis, sputtering, hydrothermal and laser ablation [14-19]. Among them, laser ablation is a widespread method for synthesis of nanoparticles that can be synthesized in gas, vacuum or liquid. This technique offers several unique advantages comparable to other chemical methods such as rapid and high purity process [20], especially, laser ablation in liquid that has more efficiency than ablation under gas atmosphere [21]. However, recently, most of researchers synthesized alumina nanoparticles using short pulse laser ablation which the pulse length of microsecond to nanosecond [22-23].

In this paper, β-Al₂O₃ nanoparticles were synthesized from bulk aluminum in deionized water using long pulsed laser ablation which barely reports the literatures. Size and morphology of alumina nanoparticles were investigated by field emission scanning electron microscopy (FE-SEM). The chemical composition of the nanoparticles was analyzed using energy-dispersive X-ray spectroscopy (EDX). Their optical properties were carried out using UV-Visible spectroscopy. The phase of the alumina nanoparticles was investigated using X-ray diffraction analysis (XRD).

2. Experimental Setup

2.1 Material preparation

Aluminum powder of 99.7% purity with an average particle size of 35μm was pressed into a tablet by a hydraulic pressed with a constant pressure of 100 bar as shown in Figure 1.

Figure 1. Aluminum powder (left) with an average diameter size of 35μm and aluminum tablet (right).
2.2 Laser ablation

The experimental setup on laser ablation is shown in Figure 2. The Nd:YAG laser used is the Miyachi ML-2331B model. The first harmonic Nd:YAG has a wavelength of 1064 nm with a pulse duration of 5 ms and a repetition rate of 2 Hz. The beam was focused using a 50mm focal-length lens onto the aluminium tablet, which is placed at the bottom of a glass vessel. Deionized water was poured into the vessel until its level is approximately 5 mm above the target. The laser energy used was 3 J and the target was ablated for 5,000 pulses.

2.3 Characterization

After laser ablation, alumina nanoparticles in the suspension were centrifuged at 10,000 rpm for 20 minutes to remove large particles. Approximately 3 ml of the supernatant was used for optical analysis using UV-Vis spectrophotometer (JASCO V570). The supernatant was then collected and dropped on silicon substrate and dried at 40°C for 1 h for the SEM investigation. The size, shape and chemical composition of the nanoparticles were investigated using FE-SEM (Hitachi model S4700 and EDS2006 model 550i analyzer) and EDX. Phase of synthesized alumina nanoparticles was characterized by XRD (PANalytical’s X’Pert Pro).

3. Results and discussion

Figure 3 shows the morphology of aluminum powders before becoming a tablet. After laser ablation, color of the suspension changed from colorless to opaque (inset Figure 4), indicating alumina nanoparticles suspended in deionized water. The particle size can be confirmed by FE-SEM as shown in Figure 4. The particles with spherical shape have diameters sizes ranging from 15-70 nm. The particles size distribution obtained by measuring 500 particle (100%) is shown in Figure 5. The dominant size of the nanoparticles is 37 nm.

Figure 2. Experimental setup for synthesizing alumina nanoparticles by laser ablation in deionized water.

The laser ablation in liquid has three main steps [24] to form nanostructures. First, the target surface was heated up by laser beam to high temperature in which the plasma of aluminium is formed. Plasma then expands adiabatically and finally alumina nanoparticles were generated aftercondensation. During the above phenomena, nucleation of the nanoparticles takes place and then fine nuclei stick together to grow up.

Figure 3. FE-SEM image of raw aluminum powder.

Figure 4. FE-SEM image of alumina nanoparticles. The inset is the suspension after ablation.
Figure 5. Size distributions of alumina nanoparticles.

Chemical compositions were analysed using EDX in FE-SEM by area scanning mode. The results are shown in Table 1. Two elements of aluminum and oxygen were detected, exclusive of silicon substrate. The average ratio of aluminum and oxygen was nearly 2:3 that confirmed the stoichiometric was $\text{Al}_2\text{O}_3$.

Table 1. EDX results of chemical compositions.

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<th>Element</th>
<th>Atomic percentage %</th>
<th>Mean value</th>
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<td>Al</td>
<td>40.1 39.4 41.5</td>
<td>40.33</td>
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<tr>
<td>O</td>
<td>59.9 60.6 58.5</td>
<td>59.67</td>
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Figure 6 shows the absorption spectrum of alumina nanoparticles in the suspension. The absorption spectrum of the particles shows an absorption peak in the UV range with the absorbance at around 213 nm.

Figure 6. Absorption spectrum of aluminanano-particles in the suspension.

Figure 7 shows the XRD pattern of alumina nanoparticles deposited on silicon wafer substrate. The pattern was compared with that of the aluminium target and JCPDS data of three different reference numbers: 85-1327 (Aluminum), and 51-0769 ($\beta$-$\text{Al}_2\text{O}_3$). The result indicates that the synthesized nanoparticles consist of $\beta$-$\text{Al}_2\text{O}_3$.

Figure 7. XRD pattern of (a) silicon substrate, (b) alumina nanoparticles, (c) aluminium target, (d) JCPDS number 85-1327.

4. Conclusions

In summary, we have successfully demonstrated that $\beta$-$\text{Al}_2\text{O}_3$-nanoparticles with a size of less than 70 nm can be easily synthesized via long pulsed laser ablation in deionized water, which allows us to precisely control over size of the nanoparticles. The dominant size is 37 nm.XRD analysis shows that the nanoparticles are $\beta$-$\text{Al}_2\text{O}_3$, which would be very useful for various applications, such as capacitor and battery.

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References