

# Effect of Laser fluence on Structure Properties of α-Al<sub>2</sub>O<sub>3</sub> Prepared by Sol Gel

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Received: 5 March 2024 Accepted: 1 May 2024

Published: July 2025

**DOI:** https://dx.doi.org/10.24237/ASJ.03.03.867C

### **Abstract**

The aim of this study to show the effect of laser fluence on Structure Properties Of  $\alpha$ -Alumina nanoparticle ( $\alpha$ -Al2O3) which had been synthesized by sol gel method. A laser Nd :YAG (1064 nm,1500 mJ) was used for irradiation the samples at (1 minute for 100 pulses) and (3 minutes for 300 pulses). The characteristic features of the samples had been improved directly with the increasing the pulse and irradiation time of the laser as shown in these tests (XRD, FESEM, FT-IR and Hardness). The XRD analysis show diffraction peaks remained unchanged when the laser irradiation was increased but the clearest effect of the laser on the samples was a decrease in the size of the crystals. FESEM of the irradiated samples decrease in particle size when increase laser irradiation .FTIR showed no changes for all sample after laser irradiation. Also, the hardness of the irradiated samples is harder than the original sample, and the hardness increases with increasing laser pulses and irradiation time.

**Keywords:** Nd: YAG laser, irradiation, Al2O3, sol gel, structure properties.

#### Introduction

Laser is one of the most significant inventions of the 20th century. The history of science, engineering, and technology has seen an exciting chapter in its continued development. The laser is a highly concentrated source of pure energy that has become a popular research tool and tool with applications in an amazing range of fields. Einstein created the first groundwork



for laser theory [1]. Einstein's prediction was then confirmed experimentally for the first time by Kopfer-mann & Ladenburg [2]. In 1960, Maiman, created the first-ever ruby laser. Much more fundamental laser development took place between 1962 and 1968. This period saw the invention of nearly all significant types of lasers, such as semiconductor, Nd:YAG, CO2 gas, dye, and other gas lasers. The current lasers were created and manufactured with increased dependability and durability after 1968. By the middle of the 1970s, more dependable lasers were becoming available for industrial uses including drilling, cutting, welding ,and marking .Lasers were investigated for surface-related applications including heat treatment, cladding, alloying, glazing, and thin layer deposition in the 1980s and early 1990s [3]. The acronym "laser" stands for the term "light amplification by stimulated emission of radiation" [4]. Laser-matter interaction has been applied to both metallic and non-metallic materials in surface research for many years [5]. It is known that the particular way that laser light interacts with a substance can cause long-lasting modifications to that substance's characteristics that are difficult to achieve in other ways[6]. Laser radiation can be distinguished from the light from conventional sources on the basis of its special characteristics and the effects it is able to produce because of these characteristics. It is these characteristics that have led to explosive growth in the usage of laser devices since the invention of this magic source of light in 1960. These include: (monochromaticity, coherence, temporal and spatial, Directionality) [7]. Numerous applications, such as the creation and control of high brightness, ultrafast electromagnetic and particle sources, laboratory simulation of astrophysical scenarios, application of extreme shocks to materials, etc., have resulted from the rapid advancements in high-power laser and high energy density science [8].

#### **Materials**

Nonahydrate aluminum nitrate (Al(NO<sub>3</sub>)3.9.H<sub>2</sub>O): It is in the form of grains of a white color and is quick to dissolve in deionized water, It is a product of the Indian company THOMAS BAKER.

Urea (NH<sub>2</sub>.CO.NH<sub>2</sub>): It is in the form of a white powder that dissolves in deionized water.

It is a product of the Indian company THOMAS BAKER.

P-ISSN: 2958-4612 Volume: 3, Issue: 3, July 2025 E-ISSN: 2959-5568 83



#### **Experimental Procedure**

#### Synthesis of $(\alpha - Al_2O_3)$ by (sol-gel method)

After dissolving 35 grams of  $Al(NO_3)_3.9H_2O$  in 35 milliliters of room temperature deionized water, the beaker is set on a magnetic stirrer, and 72 grams of urea are added. The mixture is then agitated for 20 minutes. The mixture was heated to  $100^{\circ}C$  for twelve hours, producing a transparent gel[9]. The gel was then dried for five hours at  $250^{\circ}C$ , producing a white powder, and it was then calcined for three hours at  $1000^{\circ}C$  to generate ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>). A hydraulic press device was used to compress the powder.

### Irradiation (α -Al<sub>2</sub>O<sub>3</sub>) with Nd:YAG laser

Nd:YAG laser used to irradiation ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>) at 1064 nm with 6 Hz Repetition frequency, 1500 mJ pulse energy and the distance between the laser and ( $\alpha$ -Al<sub>2</sub>O<sub>3</sub>) was 10 cm. For the laser irradiation, we used different pulses 100,300 pulse, as shown in the figure (1).



**Figure 1:** Laser irradiation of  $(\alpha-Al_2O_3)$  a)100 pulses b) 300 pulses

### **Results and Discussion**

#### X-ray diffraction analysis

For both irradiated and non-irradiated lasers, the XRD patterns were recorded with 100 and 300 laser pulses of Al2O3- $\alpha$  are shown in Figure (2). The reflections from the (012), (104), (110), (113), (024), (116), (122), (214), (300), (1010), (220) and (312) planes correspond to the diffraction peaks at (25.4678), (35.0431), (37.6701), (43.2457), (52.441), (57.3871), (61.1301), (66.4082), (68.1068), (77.1544), (80.5804), and (86.2972), respectively. The results is agreement with the card (JCPDS) files No.(46. 1212) [10]. According to this outcome, the structure of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> has hexagonal [11].



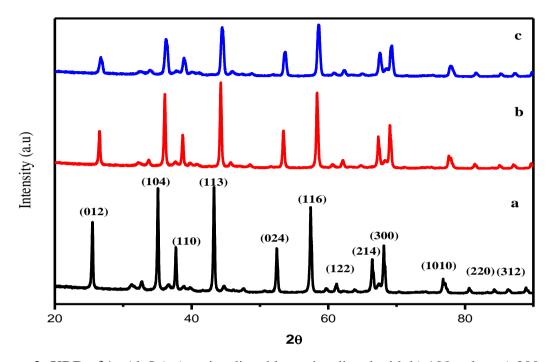


Figure 2: XRD of (α-Al<sub>2</sub>O<sub>3</sub>) a) un irradiated laser, irradiated with b) 100 pulses c) 300 pulses

Crystallite size was determined by utilizing Scherer's formula, which is provided in equation bellow:

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

#### Where:

D is the average size of the crystallite,  $\lambda$  is the X-ray wavelength falling on the target (1.5406 A),  $\theta$ : is X-ray angle of incidence, K is the factor of the body or formation and its value (0.94-0.9),  $\beta$ : Is the peak broadening(FWHM) Full Width at Half Maximum Measured in units of radius (Rad) [12]. The average crystallite size From this calculation of samples as shown the table (1):

**Table 1:** crystallite size of samples

Samples	20	(hkl)	Interplanar	crystallite size
			Spacing 'd' (Å)	(D)
$\alpha$ -Al <sub>2</sub> O <sub>3</sub>	35.8278	(104)	2.5044	66
α-Al <sub>2</sub> O <sub>3</sub> irradiated with 100 pulses	35.0431	(104)	2.5585	45
α-Al <sub>2</sub> O <sub>3</sub> irradiated with 300 pulses	35.6724	(104)	2.5149	35

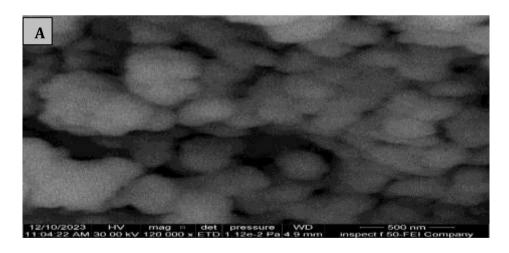


The location and number of diffraction peaks on the samples remained unchanged when the laser irradiation was increased, as shown by the X-ray measurement. However, there was a shift in the FWHM of the peaks, which affected the crystallite size. According to this calculation, there is a positive effect of decreasing crystallite size when there is more laser irradiation [13], and the peaks' broadening as laser irradiation increases, which causes the grains' size to decrease [14]. The intensity variation is caused by the diffraction and scattering of the incident radiation. [15].

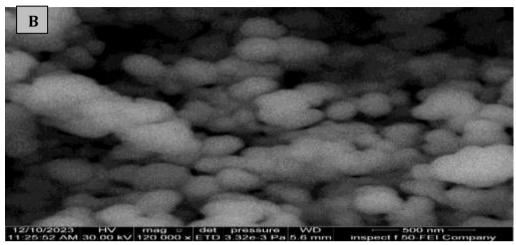
#### **FE-SEM Measurements**

The Field Emission Scanning Electron Microscopy (FE-SEM) used to investigate the surface morphology of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> before and after exposure to laser irradiation with 120,000 x magnifications. FE-SEM images of the samples were analyzed using Image J software.

In Figures (3-a) explain the morphology of the surface of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> before laser irradiation its appears to be similar to a cauliflower, spherical and the particle size is (89 nm), and following exposure to laser radiation for (1 minute (100 pulses)) the particle size decrease to (80 nm) as Figures (3-b), and when increase the time irradiation to (3 minutes (300 pulses)) the particle size decrease more to (71 nm) as the Figures (3-c).







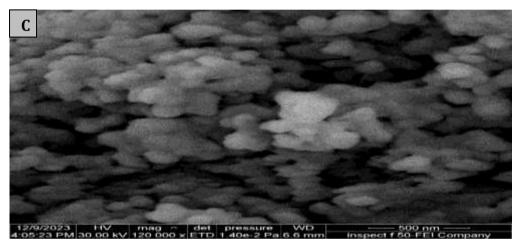


Figure 3: FESEM of  $\alpha$ - Al<sub>2</sub>O<sub>3</sub> at **a**) un irradiated laser, b) irradiated laser with (1 minute 100 pulse), c) (3 minutes 300 pulse)

### The Infrared Fourier Transform Spectroscopy (FTIR)

The compounds and functional groups present in the metallic nanoparticles that were produced are identified using FTIR. The chemical relationships between  $\alpha$ - Al<sub>2</sub>O<sub>3</sub> in the range of 4000 to 400 cm-1 were shown in Figure (4). The O-H groups produce the band at 3436 cm-1, the C-H groups produce the absorption picks at 2924 and 2860 cm-1, the C-N group produces the picks at 1383 cm-1, and the bending vibration of C = O produces the picks at 1623 cm-1 for absorption [16,17]. The Al-O stretching associated with the stable and transitional phases of Al2O3 is responsible for the bands (593, 490, 638 cm-1) [18].



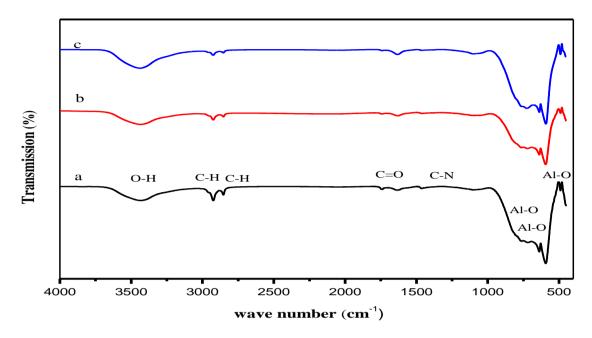


Figure 4: FTIR of α-Al<sub>2</sub>O<sub>3</sub> at a) un irradiated laser, irradiated laser with b)100 pulses c) 300 pulses

#### **Hardness Test**

The hardness of  $\alpha$ - Al<sub>2</sub>O<sub>3</sub> was measured by using (Shore D) device before and after laser irradiation. Before laser irradiation the hardness of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> was (10.5), when exposed to laser fluence for (1 minute 100 pulses) the hardness rise to (12.1) and when increase time irradiation to (3 minutes 300 pulses) the hardness rose even further to (12.9) as displayed in the table (2) and this is consistent with the findings of other researchers [19,20]. The Figure (5) shows the hardness increase when increase pulses laser.

**Table 2:** hardness for  $\alpha$ - Al<sub>2</sub>O<sub>3</sub> before and after laser

Sample of α-Al <sub>2</sub> O <sub>3</sub>	un irradiated	Irradiated with 100 pulses	irradiated with 300 pulses
Hardness	10.5	12.1	12.9



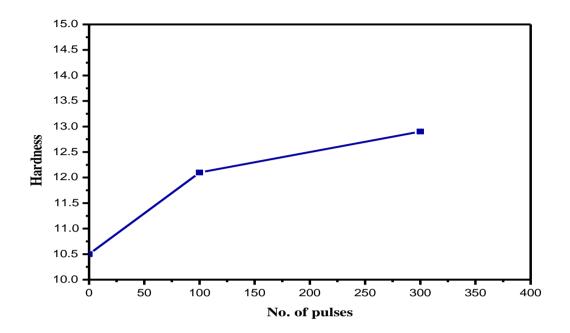


Figure 5: Hardness as a function of laser pulses number for α-Al<sub>2</sub>O<sub>3</sub>

## **Conclusions**

In summary,  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> nanoparticles were exposed to high power Nd:YAG laser fluence in this experiment, and the results demonstrate that , the XRD analysis confirmed that there was no phase shift after laser fluence and that the crystallite size decreased with increased laser irradiation .FESEM show that when increasing laser fluence the particle size decreased. Moreover, FTIR measurements following laser fluence showed no changes in any of the samples and the hardness examination showed that the alumina before laser fluence was low hardness value (10.5) , and after laser irradiation the hardness increased to (12.9) . This study showed that laser fluence led to a decrease in crystallite size and particle size according to analysis (XRD, FESEM). It was also shown that the hardness of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> increased after laser irradiation, which enhances its mechanical and chemical properties, making it a "good" choice for use in a wide range of industrial applications.

**Source of funding: none** 

Conflict of interest: none

**Ethical clearance: none** 



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