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Fabrication and Characterization of Alumina Thin Film

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2022

Abstract

Aluminum oxide (alumina) have received much attention due to its excellent mechanical, optical, chemical, thermal and electrical properties. Alumina is one of the most important oxide ceramic materials, which finds a wide range of applications in the fields of electronics, catalysis, dielectrics, solar selective coatings, refractory coatings and sensors.

Alumina thin film is prepared by using drop method. The samples prepared with different concentrations (1, 2, 3, 4 gm/L),then it dropped on glass slide. The optical properties (absorbance and transmittance), and microstructure were used to characterize the alumina thin films samples. The experimental results showed the absorbance increases with increasing alumina concentration while the transmittance decreases due to increase the absorption as result the increase of Al_2O_3 particles concentration which absorb the incident light.

(1-1) Introduction

Ceramic materials can be defined as inorganic materials constituted by the combination of metallic and nonmetallic elements whose properties depend on the way in which these elements are linked. Ceramic materials are the most versatile branch of materials. The origin of this versatility lies in the chemical nature of its bonds, since they are mainly constituted by strong ionic and covalent bonds in different proportions. The bonds determine a series of particular properties of ceramic materials among which are relatively high fusion temperatures, high modulus, high wear strength, poor thermal properties, high hardness and fragilities combined with tenacities, and low ductility. In addition to the lack of conduction electrons since they are combined forming chemical bonds, they are good electrical insulators [1].

Ceramic materials can be divided into two large groups: traditional ceramics and technical or advanced ceramics. Traditional ceramics can be defined as those that are based on silicates, among which are cement, clay products, and refractories. Traditional ceramic materials are made with raw materials from natural deposits such as clay materials [2]. The second group, technical or advanced ceramics, is manufactured with artificial raw materials that have undergone an important chemical processing to achieve a high purity and an improvement of their physical characteristics[3]. Among them are carbides, nitrides, borides, pure oxides, and a great variety of ceramics with magnetic, ferroelectric, piezoelectric, and superconducting applications. These ceramics possess excellent mechanical properties under extreme conditions of tension, high wear strength or excellent electrical, magnetic, or optical properties, or exceptional strength to high temperatures and corrosive environments, showing high strength to chemical attack [4].

(1-2) Oxides

Oxide is large and important class of chemical compounds in which oxygen is combined with another element. With the exception of the lighter inert gases (helium [He], neon [Ne], argon [Ar], and krypton [Kr]), oxygen (O) forms at least one binary oxide with each of the elements.

Both metals and nonmetals can attain their highest oxidation states (i.e., donate their maximum number of available valence electrons) in compounds with oxygen. The alkali metals and the alkaline earth metals, as well as the transition metals and the post transition metals (in their lower oxidation states), form ionic oxides—i.e., compounds that contain the O^{2-} anion. Metals with high oxidation states form oxides whose bonds have a more covalent nature. Nonmetals also form covalent oxides. A smooth variation from ionic to covalent in the type of bonding in oxides is observed as the periodic table is traversed from the metals on the left to the nonmetals on the right. This same variation is observed in the reaction of oxides with water and the resulting acid-base character of the products. Ionic metal oxides react with water to give hydroxides (compounds containing the ^{-}OH ion) and resultant basic solutions, whereas most nonmetal oxides react with water to form acids and resultant acidic solutions [5].

(1-3) Aims of the work

The present work aims to:

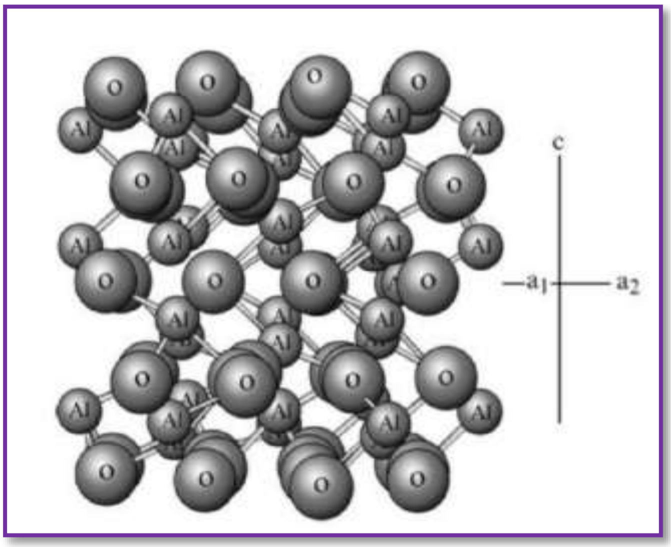
1) Preparation alumina thin film by drop method which used as gas sensors.
2) Studying the microstructure and optical properties of Al_2O_3thin films.

(2-1)Introduction

Alumina (Al_2O_3) or Aluminum Oxide is the only oxide formed by the metal aluminum and occurs in nature as the minerals corundum (Al_2O_3); diaspore ($Al2O_3$•H_2O); gibbsite (Al_2O_3•$3H_2O$); and most commonly as bauxite, which is an impure form of gibbsite [6].

(2-2) Alumina (Al_2O_3)

The alumina exists with different phases depending on the alumina purity and its mechanical and physical properties. The most important, and common, alumina polymorphs (crystalline phase) are denoted as (α, γ, θ, and κ) [3]. The α phase is thermodynamically stable polymorph (as shown in figure (2-1))and occurs naturally as corundum or sapphire, while the other phases are metastable in bulk form (but can still be produced in certain processes where thermal equilibrium is not reached, e.g., thin film growth). The differences in properties between the phases make them important in different applications. For example, the α and κ phases are widely used as wear resistant coatings due to their high hardness and thermal stability, while γ and θ-alumina are more suited for catalytic applications due to their lower surface energies, leading to larger active surface areas available for catalytic reactions. Crystalline phase are responsible for different properties of alumina yet dopants also effects on alumina phase stability. As an atom of the alumina lattice is substituted by another element, the energy of the lattice would change due to differences in chemical bonding and/or size of the substituting atom compared to the original. It has been shown that it is possible to increase or decrease the relative stability of the α-alumina and even make it less stable than the θ alumina by dopant atoms [6]. Consequently, the results predicted that it should be possible to control the thermal stability of doped alumina thin films in order to increase the thermal stability of metastable alumina phases. However, it is difficult, to predict the exact effect of doping in an experimental situation, since it was also shown that phase separations of the doped alumina are energetically favored, and will most likely occur at elevated temperatures. The common alumina polymorphs formed within typical synthesis temperatures range from room temperature up to about 1000°C. This complicates the study and growth of alumina, since it becomes difficult to control the process so that the desired phase is achieved. All alumina phases are involved in transformation sequences, which all have in common that they end in α phase alumina at high temperature. Transformations into α phase are irreversible and typically take place at above 1000°C [6].



Figure(2-1). The aluminum atoms occupy two-thirds of the octahedral interstices in a hexagonal close packed array of oxygen atoms, which is distorted because the octahedral share faces in pairs [6].

(2-3)Properties of Alumina

Alumina shares several characteristics with other polycrystalline ceramic materials, such as moderate tensile and bending resistance and the brittle fracture behavior, which is the main disadvantage of the mechanical properties of alumina[7]. The strong chemical bonds in alumina are the roots of several of its characteristics such as the low electric and thermal conductivity[8], the high meltingpoint that makes it practically impossible to shape alumina by casting, and the high hardness that characterizes this material and makes its machining complex and costly [9].

(2-4)Applications of Alumina

More than 90% of alumina produced worldwide is utilized in the production of Aluminum. This is because converting the naturally occurring bauxite into alumina is the necessary first step before it can be converted into Aluminum[10]. It is estimated that over 4 million tons of alumina is used in material applications worldwide (excluding the alumina used for aluminum production). The varied applications of alumina are due to its abundance and its multiple forms as well as its properties of stability, purity, refractoriness, and chemical inertia[11]. The applications of alumina are:

1. Display devices.
2. Separation Membrane Applications
3. Data Storage.
4. Electronics and Insulation.
5. Mechanical and chemical Ceramics.
6. Bio Medical
7. Refractory and Enameling [12].

8. It is utilized as fiber composite to improve properties, catalyst in industries and to separate or removal of water from gas steam.

9. It is used in the abrasive industry and in the manufacturing ofsandpaper owing to its significant properties such as wear and abrasion resistance[7&13].

Alumina used for wide range of applications in the fields of opto-electronics, catalysis, dielectrics, optics, sensors and tribology. It also finds applications in optically transparent ceramic coatings, organic light emitting devices (OLED), solar selective coatings, optical lenses and windows, refractory coatings, antireflection coatings and optical wave guides[13]. All these applications require films with good homogeneity, low surface roughness and good control of thickness. It is also pointed out that most of the properties depend on the microstructure and morphology of the films, process parameters and the properties could lead to the development of thin films of advanced properties [8].

3-1 Introduction

This chapter focuses on the preparation of alumina thin film by using drop method. As well as the optical, microstructure and atomic force microscope tests have been done to evaluate properties of the all samples.

3-2 Equipments

Devices and tools which are used in this study are as follows:

1. Sensitive four digits balance with accuracy (± 0.001), (Germany).
2. Magnetic stirring.
3. Optical microscope (China).
4. UV- spectrophotometer (shimadzu 1800, Japan).
5. Particle size analyzer, type (Bettersize2000 laser particle size analyzer) measured particle size in the range (0.02-2000µm), (China).
6. Atomic Force Microscope(AFM).

3-3 Preparation of alumina (Al_2O_3) thin film

Alumina thin film is prepared by using drop method. The samples prepared with different concentrations (1, 2, 3 ,4 gm/L),then it dropped on glass slide after clean it by ethanol and distilled water.

3-4 Tests

3-4-1 Particle Size Analyzer

Laser particle size analyzer has been used to determine the particle size of alumina by using the following steps :-

1) The dispersion cell is filled with clean, deionised water.

2) A few amount of powders are dispersed into deionised water that circulated across a quartz measurement cell illuminated by a laser beam; figure (3-1) shows laser particle size analyzer.

Figure (3-1).Laser particle size analyzer**.**

3-4-2 UV- spectrophotometer

UV spectroscopy involves the transitions of electrons within molecule or ion from a lower to a higher electronic energy level or vice-versa by the absorption or emission of radiation's falling in the UV-visible range of electromagnetic spectrum.

ultraviolet–visible spectrophotometer refers to absorption spectroscopy or reflectance spectroscopy in part of the ultraviolet and the full, adjacent visible spectral regions. This means it uses light in the visible and adjacent ranges. The absorption or reflectance in the visible range directly affects the perceived color of the chemicals involved. In this region of the electromagnetic spectrum, atoms and molecules undergo electronic transitions. Absorption spectroscopy is complementary to fluorescence spectroscopy, in that fluorescence deals with transitions from the excited state to the ground state, while absorption measures transitions from the ground state to the excited state.

Figure (3-2) shows UV- spectrophotometer.

3-4-3 Optical microscope

The optical microscope, also referred to as a light microscope, is a type of microscope that commonly uses visible light and system of lenses to generate magnified images of small objects. the microstructure of Al_2O_3 thin film obtained at magnification (100X).

Figure(3-3) shows optical microscope.

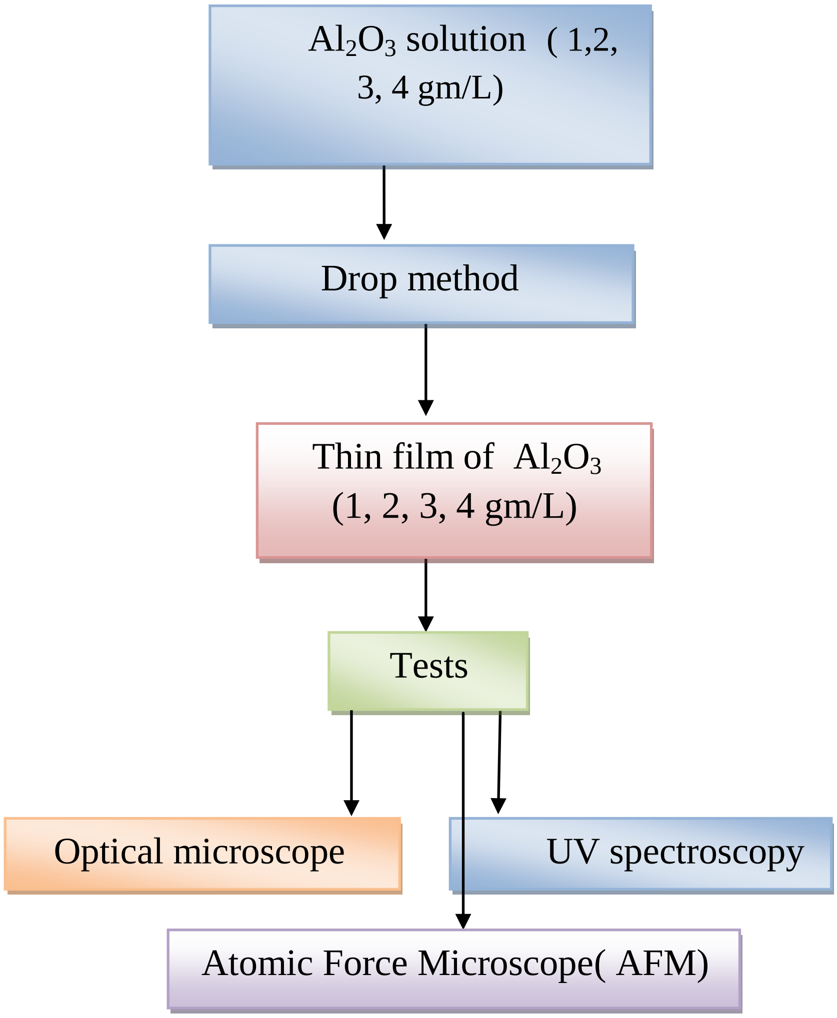


Figure (3-4) Flow diagram of Al_2O_3 thin film preparation.

4-1 Introduction

The results consist of the optical properties and microstructure of prepared Al_2O_3 thin film.

4-2 Particle Size Analyzer

Particle size of Al_2O_3 has been measured by using particle size analyzer as shown in figure (4-1) . The average particle size of alumina is (3.603µm) and particle size range is (0.212-34.56µm).

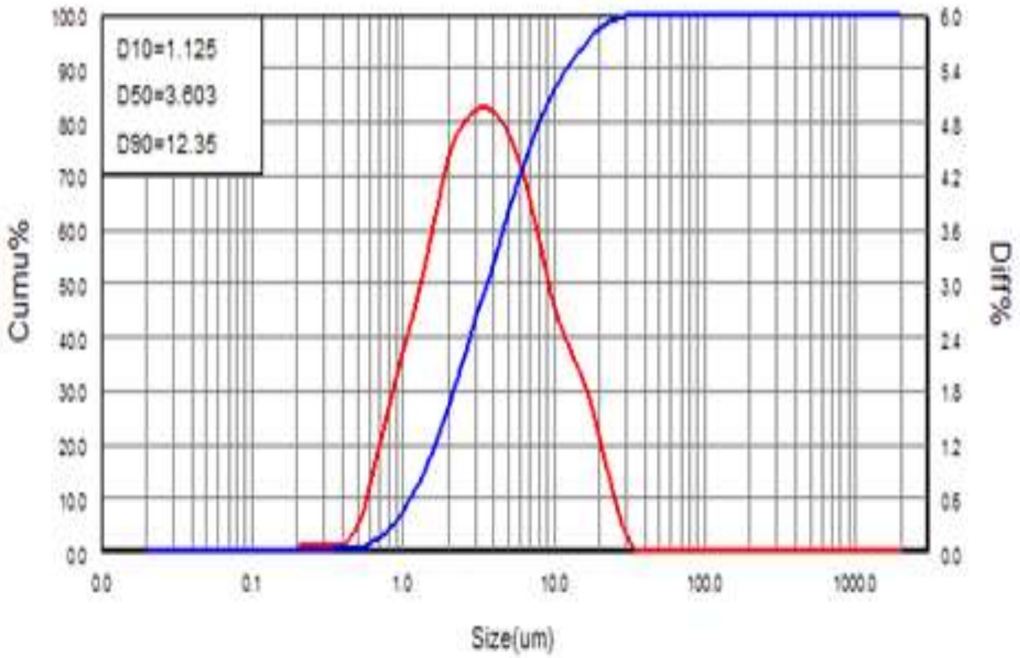


Figure (4-1)shows particle size analysis of Al_2O_3.

4-3 Optical properties and microstructure

The relationship between absorbance and wavelength has been shown in figure (4-2). The absorbance increases with increasing alumina concentration from (1 g/L to 4 g/ L). The raise of absorption connected to increase the charges carries number in Al_2O_3 which absorb the photons.

The huge absorbance power in UV region; it explains the value for band gap attributed to interaction of charge transfer. The absorption edge of a deteriorate semiconductor is shifted to shorter wavelengths with rising concentration of carrier. The primary absorption indicates to transition of electron from the valance to the conduction band which be used to determine the value and nature of the band gap[14&15]. The relationship between transmittance and wavelength has been shown in figure (4-3). The transmittance decreases with increasing alumina concentration from 1 g/L to 4 g/ L. the decreasing of transmittance due to increase the absorption as result the increase of Al_2O_3 particles concentration which absorb the incident light as shown in figure (4-4)which shows distribution of Al_2O_3 particles for different concentrations(1, 2, 3 and 4 g/ L). It can be noted the light absorption with the increase in Al_2O_3 particles concentrations.

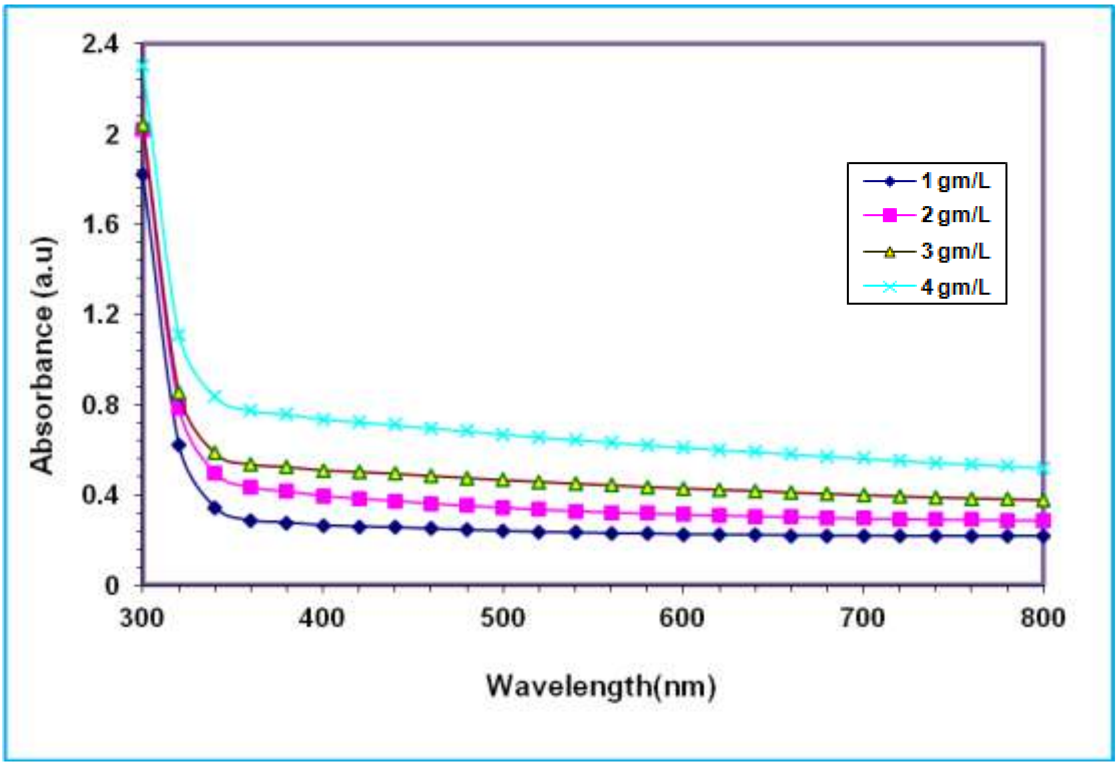
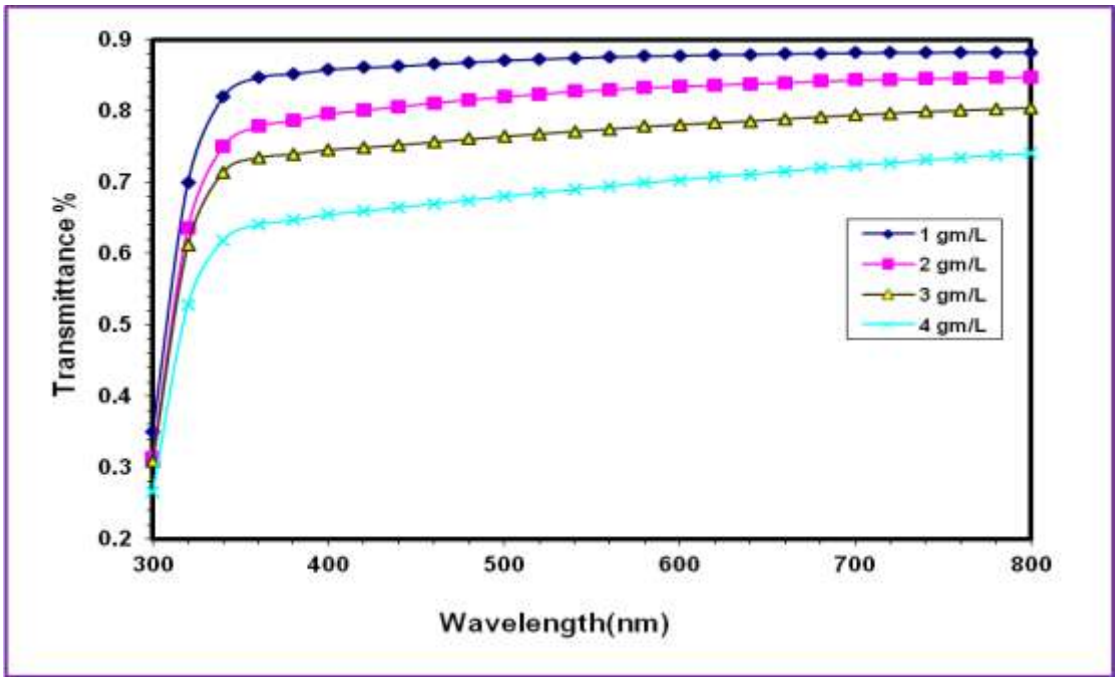


Figure (4-2) shows the relation between absorbance and wavelength.



Figure(4-3) shows the relation between transmittance and wavelength.

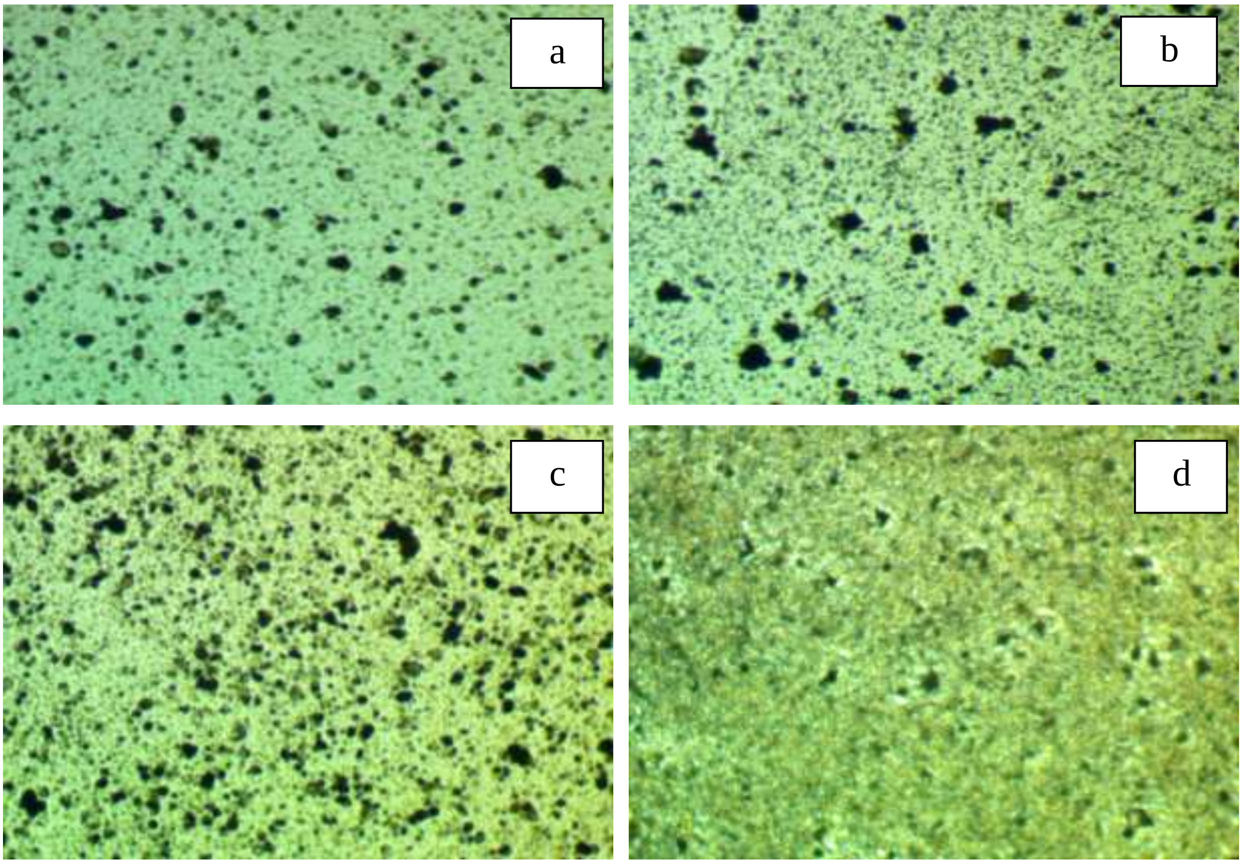


Figure (4-4) shows the microstructure of alumina thin film at different concentrations.
(a) 1 gm/L. (b) 2 gm /L. (c) 3 gm/L. (d) 4 gm/L. at magnification (100X)

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