

Case Report



Aluminum oxide nano porous: Synthesis, properties, and applications

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ABSTRACT

In recent years, most researchers are trying to seek out and develop effective and comparatively simple methods for producing nanostructures. In this regard, the methods of manufacturing self-regulated nanostructures with alternating arrays are given special attention. The anodization, which ends up within the formation of a porous metal oxide layer consisting of an everyday array of nanoporous, is one such method. The numerous applications of aluminum, research on the anodization has focused more on this metal. Anodic corundum may be a key sample material for creating nanostructures like nanowires, nanotubes. The anodic aluminum, which ends within the formation of porous alumina samples, is comparatively easy and ultimately leads to an array of high-density parallel nanoporous. During this paper, the final structure of porous anoxic alumina further because the growth mechanism of this layer within the presence of electrical field and therefore Penetration of Electrolyte species into the oxide layer, furthermore as explaining the expansion mechanism of porous oxide layer in steady state and hexagonal plasma membrane structure. Affecting the geometric parameters of the porous alumina structure like the pore diameter, the interpore distance, the wall thickness, and also Thickness of the Barrier Layer are discussed.

1. Introduction

Anodization is an anodic electrochemical process accustomed increase the thickness of the oxide layer that forms naturally surface metals. This process was first used on an industrial scale in 1923; it then

expanded rapidly and was first performed in 1927 by Grower and O'Brien on vitriol electrolytes [1–7]. The anodization has many applications within the production of dielectric layers for the manufacture of electrolytic capacitors, sensors, and optical instruments. The resistance of anodic layers against corrosion and abrasion, likewise providing an

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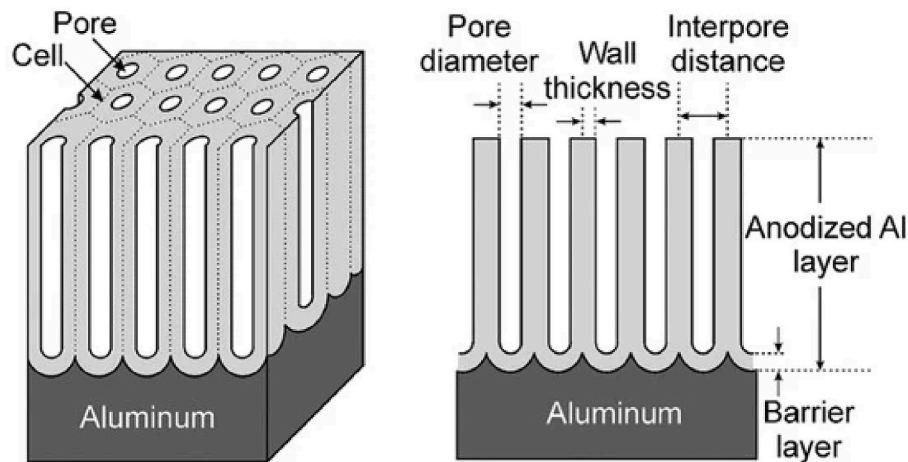


Fig. 1. (a) Structure Porous Anodic Alumina, (b) Cross Section anodization Layer [1].

appropriate substrate for colored and adhesive primers on metal surfaces, has made the anodization additionally to industrial applications, in fields like architecture [8–12]. Metal surface anodization changes surface texture changes the crystal structure of the metal near the surface [13–16]. The formation of a porous oxide layer in electrolytes with very strong acidic properties like acid, Ethane dioic acid has been reported; This layer is partially soluble [17–22]. In porous anoxic alumina, the expansion of the layer is the local Dissolution of Oxide, which eventually Results from the formation of holes within the oxide layer [23–28]. The expansion of the anoxic alumina layer at steady-state results from the interaction of two competing processes, which occur

simultaneously [29–35]. One of these processes is the dissolution oxide layer at the top of the cavities at the interface between the oxide layer and therefore the electrolyte, and therefore the other is the formation of the oxide at the interface between the oxide and metal layers [36–41]. The Aim study analyze the link between anodization conditions, like applied potential, temperature, electrolyte concentration, and therefore the geometric properties of porous alumina structures. In other words, by changing the anodization conditions, the geometric parameters of porous alumina will be controlled and thus the required structure is often achieved [42–47].

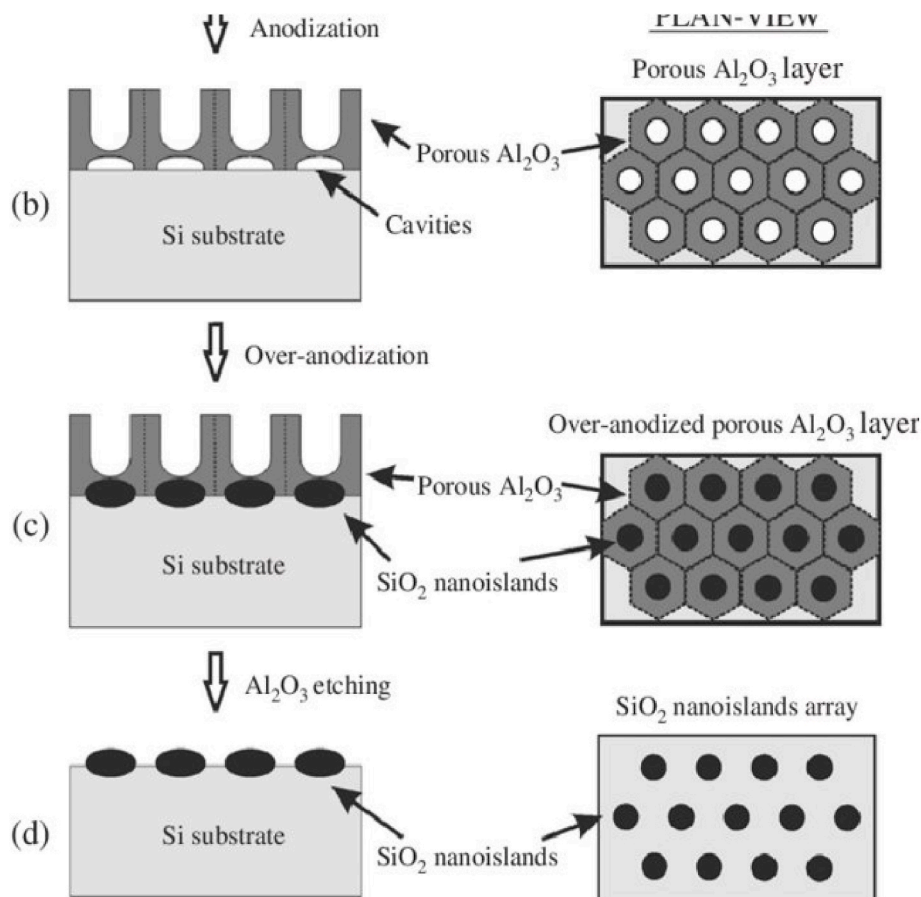


Fig. 2. The Increase of SiO_2 Nano islands on silicon using a porous anodic alumina template is depicted schematically [2].

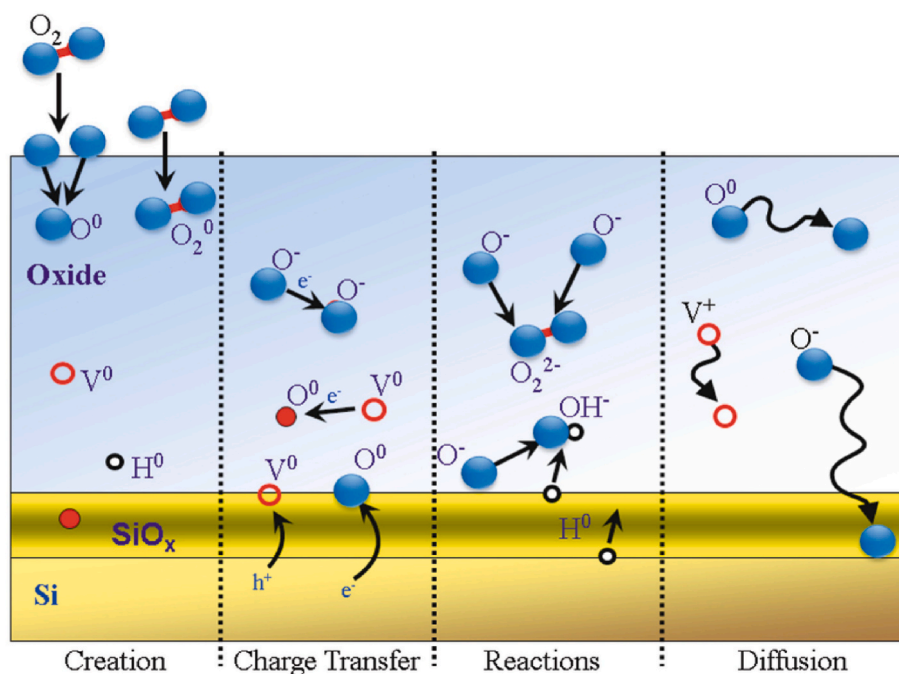


Fig. 3. The formation of O^{2-} and OH^- ions at the oxide and electrolyte border, as a result of the reaction of water with absorbed O^{2-} ions from the source [4].

2. Structure of porous anodic alumina

The formation of alumina thin film isn't only associated with the anodization and is even achieved in an exceedingly simple oxidation process. But what has attracted plenty of attention is the special nature of the porous layer of aluminum oxide, which is created under certain conditions and has many applications in nanotechnology [48–53]. The structure of the self-assembled porous anodic alumina layer, formed under anodic aluminum, is described as an array of tightly packed hexagonal cells, each cavity centered within the center of a cell [54–60]. The porous alumina coating is illustrated schematically in Fig. 1. Various theories are suggested for the formation of nano porous within the alumina crust; nevertheless, what's clear is that the oxide coating grows at the tip of the cavities, on the metal-oxides bound substrates. In this way, the present substance on the surface is converted into a shield layer, which is followed by a porous oxide layer [61–65]. A skinny and compact barrier layer dissolve on the electrolyte's joint surface and also the cavity's rim during the formation of porous oxide, and a brand-new barrier layer forms on the oxides and metals joint s surfaces. Within the steady-state of layer growth, a dynamic equilibrium is made between the speed of layer growth and therefore the rate of dissolution by the ground [66–72]. Because the oxide layer grows steady state, the anode current density remains almost unchanged at constant potential (or anode potential under constant current density). The cylindrical part that appears at the cross-section of the cavities is the result of growth within the steady-state [73–80]. The standardized pore diameter may be easily managed by changing the anodization conditions from some nanometers to some 100 nm [81–85].

3. Mechanism of growth of porous oxide layer in the presence field

The structure of porous alumina begins and extends from the physical phenomenon that emerges at the beginning of anodization on the aluminum surface. Solid ionic conduction induces physical phenomenon formation. This happens within the presence of a constant-power field; the magnitude of the electrical field is defined because of the ratio potential along with the barrier layer thickness [86–90]. As seen in Fig. 2, the barrier oxide layer extends evenly over the surface with a

standardized field distribution controlled by a continuing field [91–96]. The rough surface of the aluminum becomes smoother as a result test of uniform growth. Local changes in field pressure, on the opposite hand, may manifest themselves on the surface as defects, impurities, or surface features like subgrain borders, ridges, and troughs [97–105]. Increased field dissolution to the oxide layer and native thickening porous layer result from this non-uniform flow distribution (Fig. 2-b). A Joule local heating is followed by more current above the metal ridges, leading to a thicker barrier oxide layer [106–114]. The bottom breakdown of the oxide layer, on the opposite hand, helps to flatten the oxide-metal interface. This local heat increases the sphere breakdown of the oxide at the cavities' ends, increasing the current's local density. This mechanism continues because the local current density rises, causing the oxide within the lower part of the cavities to dissolve. The oxide layer that grows on the ridges (on the surface's deficient points) is vulnerable to high local pressure [115–123]. This causes consecutive cracks within the oxide layer, but these cracks suddenly strengthen at high local current densities (Fig. 2-c, 2-d). Cracking-healing events are typical where aluminum is employed as a base and oxide layer walls are built above the defective areas. This enables a part of the wall to crumble, increasing the spherical curvature of the cavities on the metal and oxide joint surface (Fig. 2-e). The flow is localized within the thinner section of the film at the top of the following cavity, leading to preferential development of the oxide over the deficient areas and thickening of the barrier layer. Consequence, event cavities begin the initial cavil ties to preserve a uniform area. The steady-state of cavity growth happens because the curvature of the oxide layer at the metal-oxide joint surface increases enough and therefore the intersection of the hemispherical regions occurs [124–128].

4. Permeability anions

Anions' permeability to the structure of the anodization oxide layer is extremely influenced by the kind of oxide layer produced [129–135]. The penetrating anions within the porous oxide layer are much greater than the boundary oxide layer's penetrating anions. The expansion of the oxide happens concurrently at the oxide-metal boundary and at the electrolyte oxide boundary during the formation of a barrier oxide layer within the steady-state, which is the migration of Al^{3+} and HO^-/O^{2-} ions

in opposite directions. Al^{3+} ions, on the opposite hand, are directly introduced into the electrolyte solution and play no part within the forming of the oxide layer [136–143]. The amorphous oxide film is built he aluminum. The penetration of anions into the oxide layer at the underside of the cavities, which may be a direct result of electrolyte species migration, is required to develop a layer of porous alumina. Since electrolyte kinds may be positively, negatively, or neutrally charged, they will either because immobile on the oxide surface or migrate at or specific rate, which varies counting on electrolyte [144–150]. PO_4^{3-} , SO_4^{2-} , oxalate anions, for instance, migrate into the oxide layer, while chromate anions migrate outward [151–156]. The oxide layer grows only at the interface between the oxide and therefore the metal during the formation of porous alumina, and also the anions move through the barrier layer because the presence of an electrical field shanks the circular structure of the underside of the openings, the electrical field within the barrier layer isn't uniform [100]. The electrical field at the underside of the cavities, where the oxide meets the electrolyte, is far stronger than the electrical field at the underside of the cell, where the oxide meets the metal [157–163]. As a result, the electrolyte anions have a straightforward time managing to infiltrate. The longer the oxide walls are subjected to the acid's aggressive penetration; the more anions penetrate into the porous alumina base [164–170].

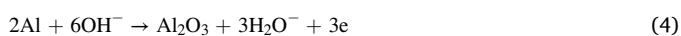
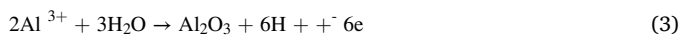
5. Anodization, steady growth of the porous oxide layer

The following reactions release OH^- ions in the electrolyte solution: quick decomposition of water or cathode reduction of dissolved water and oxygen:



O^{2-} ions, on the other hand, are formed at the oxide-electrolyte interface due to the loss of oxygen vacancy, which contributes to the adsorption of OH^- ions [171–176], either in the simple decomposition of water or in the reaction of water with the adsorbed electrolyte ions. Fig. 3 depicts the mechanism of producing O^{2-} and OH^- ions at the interface between the oxide and the electrolyte.

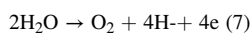
As aluminum is anodization in an acid electrolyte, the following reactions occur, resulting in the formation of an amorphous oxide layer:



In anodization flow, the following reaction is important:



For the anodization of aluminum in different electrolytes, the movement of oxygen near the oxide-metal boundary has been recorded [177–184]. The following are some of the reactions:



Due to the presence of impurities, the production of oxygen bubbles within the anodization alumina, according to reaction (9) near the boundary of the oxide and metal substrate, will begin [185–192]. It's also been proposed that the development of the porous alumina layer is linked to oxygen transport. The presence of electrolyte anions, which are vulnerable to oxidation, complicates the anodic process:

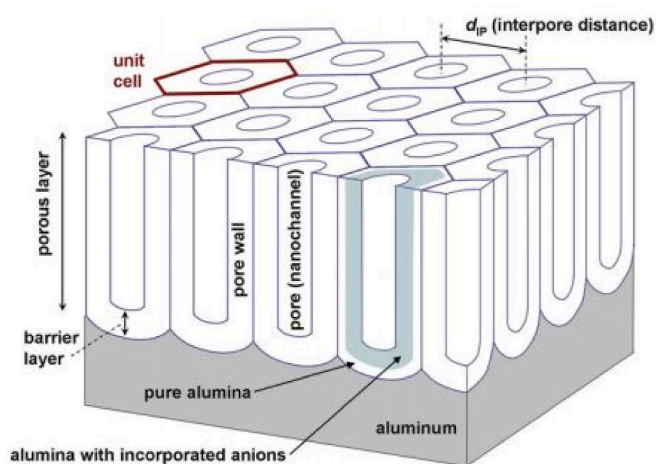


Fig. 4. The ideal structure of the porous alumina layer on the aluminum substrate [7].

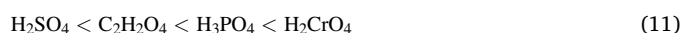
Cavities are formed as a result of thermal dissolution caused by Presence of an oxide layer field at the cavity's end. The expansion of the oxide layer in typical acid electrolytes, like vitriol, oxygen acid, and acid, occurs primarily at the Interface between the oxide and therefore the metal. The movement of oxygen-carrying ions (O^{2-} and OH^-) from electrolyte solution into the oxide layer induces expansion of porous alumina at the underside of the cavities. At Interface between the oxide layer and also the electrolyte, Al^{3+} ions that are progressing through the oxide layer are forced into the electrolyte solution. In fact, metal ion migration into electrolyte solution could be a required condition for the forming of a porous oxide layer, since when these ions meet the common surface of the oxide layer and also an electrolyte, they permit the barrier layer to expand and thus contribute to the formation of the oxide layer [193–200].

Just a little portion of the Al^{3+} ion flux at the oxide/metal and oxide/electrolyte interfaces, however, contributes to the forming of the oxide layer. Electrolyte ions can also move through barrier layer and interact within the formation of metal oxide compounds. Dissolution of the oxide layer at the underside of the cavities is caused by the local increase of the sphere at the favored boundary of the oxide and therefore the metal for the event of the solid-state of porous oxide [201–205]. The complex equilibrium between the oxide field dissolution rate and also the oxide-forming rate increases the expansion rate of the oxide at the interface between oxide and therefore metal because the field dissolution rate of the oxide layer is increased. The polarization of Al–O bonds initiates field breakdown of the oxide, by separation of Al^{3+} ions from oxide structure [206–214]. It's also simpler to tell apart Al^{3+} ions within the presence of the bottom [215–221].

6. Cell wall structure

Structure of the cell membrane the penetration of electrolyte Kinds into oxide walls determines properties of porous alumina layer formed during the anodization. Furthermore, anions' penetration affects the mechanical properties of the anodization alumina layer, like stability, stiffness, and abrasion resistance [222–230]. Anodization factors like anodization potential and temperature influence the sum of infiltrating organisms and their distribution. Fig. 4 illustrates the twin arrangement of the cell membrane.

Thomson et al. proposed that this structure is created from two layers, one among almost pure alumina and therefore the other with electrolyte penetration anions [231–236]. Reckoning on the shape of acid used, the thickness of the inner layer increases as follows:



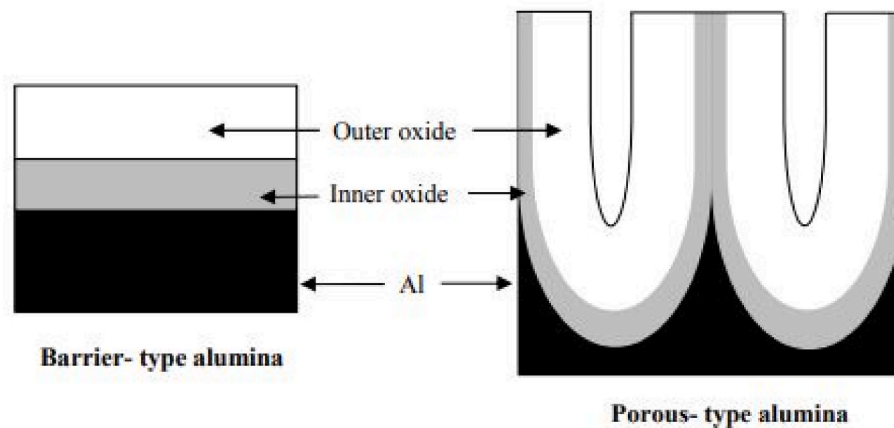


Fig. 5. Vacancies in the structure of porous Anodic alumina [8].

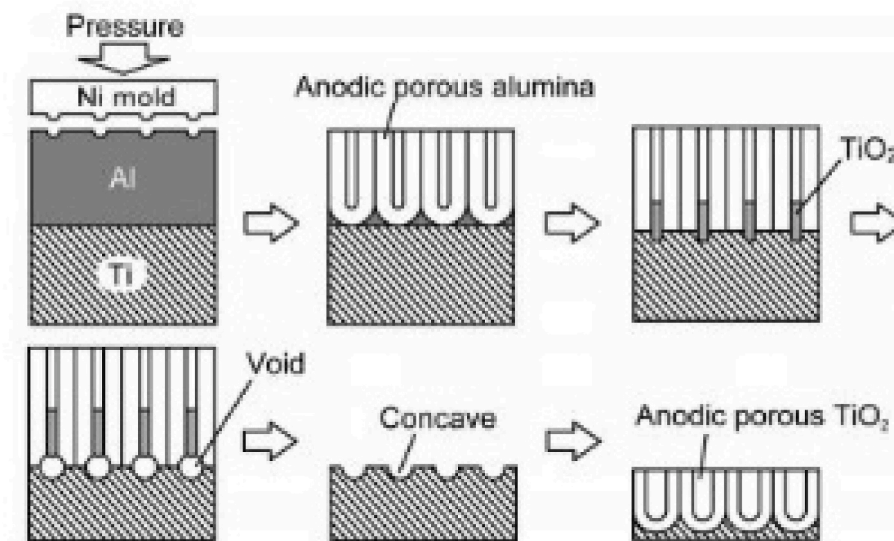


Fig. 6. Steps of formation of voids in the structure of porous anodic [9].

Table 1

Set of nanowires made of metals with their deposition techniques and investigated properties [286–290].

Metal or alloy	Deposition technique	Investigated properties/applications/remarks
Al	Pulse electrodeposition	–
Al	Surface redox reaction (cementation)	–
Ag	DC electrodeposition	–
Ag	AC electrodeposition	Antibacterial properties
Ag	AC electrodeposition	–
Ag	AC electrodeposition	Plasmonic properties were strongly related to the NW diameter
Ag	DC electrodeposition	Diameter-modulated NWs
Ag	Solid state reduction	–
Ag–Au	AC electrodeposition	UV–vis absorption spectra
Au	DC electrodeposition	Optical properties
Au	AC electrodeposition	DNA arranging on the Au NWs surface
Au	DC electrodeposition	–
Au	AC and subsequent DC electrodeposition	–
Au	DC electrodeposition	Diameter-modulated porous NWs
Au	DC electrodeposition	Y-branched NWs

Moving over the membrane and into the cavity, there's a transition from a solid to a jelly-like state. For oil of vitriol, ethane dioic acid, and oxygen acid electrolytes, the thickness ratio of the inner layer to the outer layer is 0.05, 0.1, and 0.5, respectively [11]. The cell wall's triangular shape, as seen in Figs. 5 and 6, was recently stated [12]. Electrolyte species, mostly protons, and anions are impurities within the outer and middle layers. Anions and protons are abundant within the outer layer, while anions dominate the center layer. The cell wall's triangular shape, as seen in Figs. 5 and 6, was recently stated [12]. Electrolyte species, mostly protons, and anions, are impurities within the outer and middle layers. Anions and protons are abundant within the outer layer, while anions dominate the center layer. Oil of vitriol electrolytes produces porous oxide layers that are practically dry [14]. Within the porous alumina structure, voids are found at the information of the aluminum protrusions, at the interface between the metal and also the oxide, within the inner layer of the cell walls, these voids, which are formed by the passage of oxygen, are thought to be created to survive the strain within the layer [237–240]. With increasing anodization potential, the quantity of voids created within the porous oxide layer increases [241–246].

To form nanowires (high-aspect ratio 1D nanostructures), AAO template has to be thick enough and experimental procedure has to provide relatively large charge during the electrodeposition (Table 1).

DC electrodeposition, AC electrodeposition, and pulse electrodeposition are used to obtain highly ordered arrays of nanowires made of Al, Ag, Ag–Au, Au, Bi, Co, and its alloys, Cu, Fe, and its alloys, Ni, and its alloys, Sn, Pd, and Pt (Table 1).

7. Geometric parameters characteristic of nano porous anodic alumina

Regular nanostructures, often with parameters such as pore diameter, interpose distance, wall thickness, barrier layer thickness, porosity, pore density, and thickness of the oxide layer is described [247–252].

7.1. Pore diameter

In general, the pore diameter of porous anodic alumina structures anodization linearly with probability, and the constant of proportionality in this respect, p is equal to 9.0 nm volts for soft anodized and 0.4 nm V for hard anodized [253–257]:

$$D_p = \lambda_p \cdot U \quad (12)$$

The hole diameter (nm) is represented by p , and also the anode potential is represented by U (V). Electrolyte shift has no effect on the complete diameter's dependency on the anodization potential. Researchers often use the terms outer layer for areas near the oxide layer surface and inner layer for areas near the top of cavities when defining the composition of porous anodic alumina [148–151]. The pore diameter of the outer oxide layer doesn't change substantially with time at steady state; the greater diameter of the pores at the surface of the oxide layer is that the product of early development and irregularity of the porous alumina layer within the early stages of growth [8,10,15]. The diameter of the holes is clearly sick with the electrolyte temperature and hydrodynamic conditions within the electrochemical cell. A rise within the speed of chemical dissolution of the outer oxide layer is predicted when anodization at higher temperatures (near room temperature), particularly in electrolytes with high acidity [258–262]. Reducing the stirring speed of the electrolyte during anodization under constant potential, on the choice hand, increases the local temperature of the inner oxide layer and hence this density [18,263–268]. As a result, chemical dissolution of the inner oxide layer and electrochemical forming of the anodic oxide layer happens more quickly, and also the pore diameter increases.

7.2. Distance between holes

It is generally accepted that the distance between holes in a porous anodic alumina structure is linearly related to the growth potential of the oxide layer, and the proportionality constant, λ_c , is approximately 2.5 nm for soft anodization and 1.5–2 nm for hard anodization. Is in nanometers per volt [19]:

$$D_c = \lambda_c \cdot U \quad (13)$$

The distance between the cavities can be partially, at constant potential, dependent on the anode temperature or independent of it. If anodization is performed in the presence of oxalic acid as the electrolyte, the distance between the cavities will not be temperature dependent [20]. Conversely, if the anodized electrolyte is sulfuric acid, the distance between the cavities will be temperature dependent [21]. Research by O'Sullivan and Wood has shown that increasing the electrolyte concentration reduces the distance between cavities [2]. They performed anodization on sulfuric acid electrolyte at constant current density and observed that increasing the anodization temperature also increased the distance between the cavities by increasing the electrolyte concentration.

7.3. Wall thickness

According to the research of O'Sullivan and Wood, the hole diameter for anodization under constant potential is calculated from the following equation [2]:

$$D_p = D_c - 2W \quad (14)$$

Where D_p is the pore diameter, D_c is the distance between the cavities and W is the wall thickness. In this way, the wall thickness can be calculated from the above equation, as follows:

$$W = (D_c - D_p) / 2 \quad (15)$$

They also showed that for anodization in phosphoric acid, wall thickness was linearly related to the thickness of the barrier layer:

$$W = 0.71B \quad (16)$$

$$B \approx D_c / 2 \quad (17)$$

They reported that the ratio between the wall thickness and the thickness of the barrier layer at the anode in the oxalic acid electrolyte varies slightly in the voltage range of 5–40 V. For voltages between 5 and 20 V, this proportionality constant is 0.66, which as the voltage rises to 40 V, this value also increases slowly and eventually reaches 0.89 [2, 106,107,152–156].

7.4. Thickness of the barrier layer

The barrier layer forms at the top of the cavities during the anodization of aluminum. It's a really thin, thick, flexible dielectric layer. This coating is analogous to the oxide layer that normally occurs within the atmosphere therein it only allows current to circulate if its composition includes defects. The electrochemical accumulation of metals into the cavities is nearly unlikely because of the inclusion of this lightweight membrane sheet at the top of the cavities. Given this restriction, the thickness of the barrier layer is very important, and it'll be a deciding think about the next applications of anodized Nano pores [157–161, 269–272]. The anodization ability defines how dense the barrier layer. Changes within the thickness of the barrier layer are identified in some studies as a result of changes in electrolyte concentration [32–34, 108–110,273–280]. The researchers inferred from their experimental findings that the thickness of the barrier layer varies with anode voltage counting on whether the anode was performed within the constant potential or constant current density regime [35,36,281–283]. Within the constant potential regime, increasing anode temperature ends up in a decrease within the thickness of the boundary layer; within the constant current regime, the alternative relationship is found. In anodization at constant potential, decreasing the thickness of the barrier sheet, and anodization at constant current density, increasing the concentration of oxyacid at constant temperature increases this amount [37–40, 285–285]. The anodization rate, BU (ratio between barrier layer thickness and anodization potential), up to 1 nm/volt, is shown by the diagonal line of the line during this diagram. In general, the anodization rate is incredibly the same as 1 nm for various electrolytes and over the complete anodization potential spectrum. This result suggests a general relationship between anodization rate and anodization potential. For the most effective self-regulated anodization conditions, Nielsch et al. suggested the subsequent relationship for the barrier layer thickness and therefore the distance between the cavities [3,117–120], which ends in 10% nanostructure porosity and therefore the arrangement of six full Nano porous cells: The thickness of the physical phenomenon is denoted by the letter B.

7.5. Porosity

Porosity is defined as the ratio of the surface occupied by cavities to

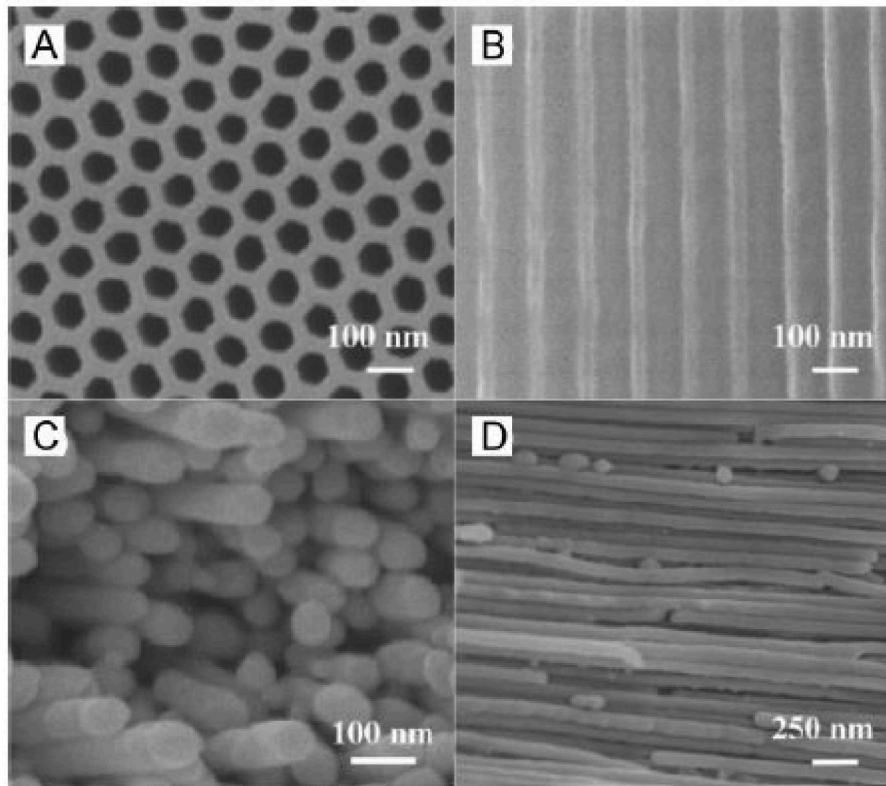


Fig. 7. SEM images of the surface (A), the cross-sectional views (B) of the ordered porous anodic alumina template, the top image of as-prepared 70 nm AgI nanowires after partial removal of AAO (C), and the cross-sectional view of AgI nanowires after complete removal of AAO (D) [1].

the total surface area of the porous oxide layer. For a regular hexagon with a hole in the center, the porosity formula is written as follows:

$$\alpha = S_{\text{pores}} / S = S_p / S_h \quad (18)$$

Assume that each single hole is a complete circle. So for S_p and S_h we have:

$$S_p = \pi (D_p / 2)^2 \quad (19)$$

$$S_h = (\sqrt{3} / 2) D_c^2 \quad (20)$$

By placing equations (19) and (20) in equation (18), the following expression is obtained to calculate the nanostructured porosity consisting of an array of six cell phones:

$$\alpha = (\pi / 2\sqrt{3}) (D_p / D_c) \quad (21)$$

The growth rate of the oxide layer and its chemical dissolution rate within the acid electrolyte, furthermore as anodization conditions like electrolyte form, electrolyte concentration, potential, and anodization temperature, all influence the porosity of nanostructures created with anodization aluminum. The anodization potential and pH of the electrolyte used are the foremost significant variables in assessing the porosity of anodic alumina nanostructure. With increasing anodization potential in acid and Ethanedioic acid, an exponential decline in porosity has been observed [41,121–123]. Porosity nanostructures will influence with anodization time, as predicted. In tetra borate and oxyacid, prolonged anodization time often leads to increased nanostructured porosity [42,43,124–126].

7.6. Cavity density

High-order nanomaterials with a structure composed of compact clusters of Nano pores or nanotubes are of significant importance in the microelectronics industry. Porous anodic alumina was chosen because

of the hexagonal symmetry of the cells. The number of cavities that develop during anodization is one of the most significant properties of porous alumina since nanopores have the highest density and compaction. The density of cavities is defined as the total number of cavities occupying a 1cm^2 region for the distribution of six cell phones in nanostructures [44,45,127–130]. The following is the suggested relationship for measuring the density of cavities:

$$n = 10^{14} / P_h = (2 \times 10^{14} / \sqrt{3}) D_c^2 \quad (22)$$

The P_h is the surface area of a single six-cell phone (nm^2) and the D_c is nanometers. By inserting D_c from the relation $D_c = \lambda c \cdot U$, we will have:

$$n = (2 \times 10^{14} / \sqrt{3} \lambda_c^2 \times U^2) \approx 18.475 \times 10^{14} / U^2 \quad (23)$$

Increasing the anodization potential or the distance between the cavities reduces the amount of cavities formed in the porous alumina structure [7], as shown by Equations (22) and (23).

7.7. Volume expansion

The Pilling-Bosworth Ratio is the volume expansion of porous anodic alumina (R) (PBR). The amount of aluminum oxide formed in the anodization process divided by the volume of aluminum consumed is known as volume expansion:

$$RV_{\text{Alumina}} / V_{\text{Al}} = M_{\text{Alumina}} / M_{\text{Al}} \cdot d_{\text{Al}} \quad (24)$$

(M)Alumina represents the molecular weight of aluminum oxide, (M) Al represents the atomic weight of aluminum, (d) Alumina represents the density of porous alumina ($3.2\text{gr}/\text{cm}^3$), and (d) Al represents the density of aluminum ($2.7\text{gr}/\text{cm}^3$). PBR has a potential value of 1.6 for the formation of porous anodic alumina with a flow efficiency of 100%. Due to the utility below 100% of the flow, the experimental value

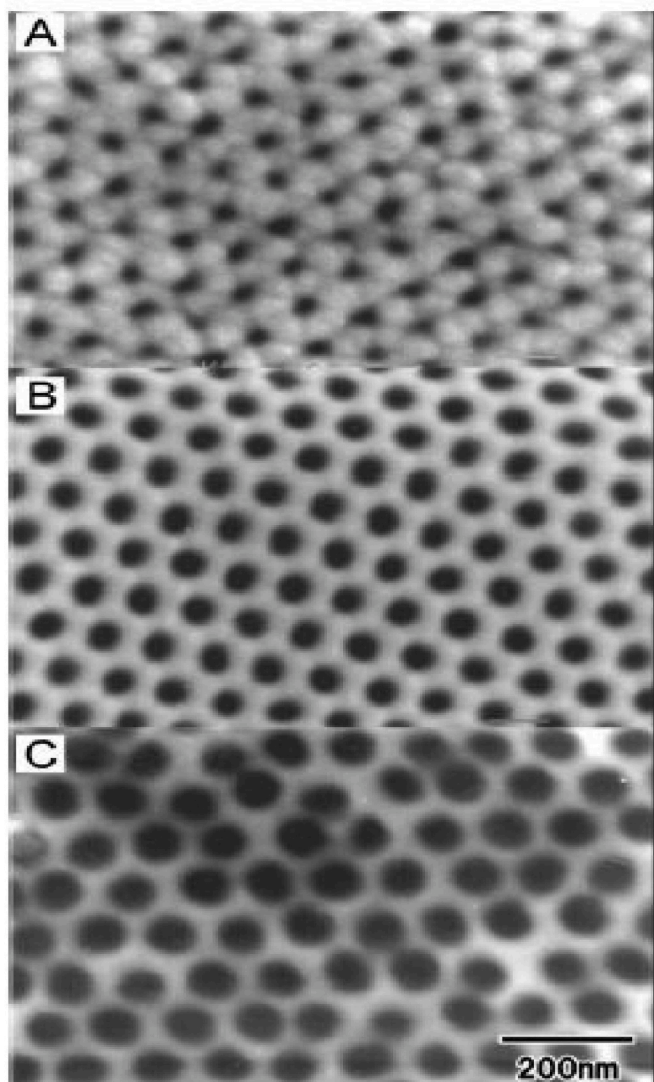


Fig. 8. SEM images of WO₃ antidot arrays with different mean hole sizes: (A) 38 nm, (B) 53 nm, and (C) 65 nm fabricated by a two-step replication process employing negative-type PMMA templates. The PMMA templates have been prepared from highly ordered AAO membranes with different pore diameters [1].

of volumetric expansion differs significantly from the theoretical value and normally ranges from 0.9 to 1.6 [14].

8. Factors affecting the geometric parameters of porous alumina structure

Anodization conditions, which include electrolyte type, electrolyte density, anodization potential, temperature, electrolyte stirring, and anodization time, have a big effect on a range of porous anodic alumina structural parameters like pore diameter, pore spacing, pore density, and pore density [131–135,169–172]. The arrangement of cavities produced under different conditions with the identical magnification (Fig. 7).

8.1. Potential effect

The anodization potential is proportional to the gap between the cavities, because the potential rises, the interpore distance rises likewise. Figs. 10 and 11 precisely demonstrate this. It's worth noting that anodization capacity features a smaller impact on cavity diameter than

it does on cavity spacing [46–48].

The Nano-cavity arrangement principle and structural properties of porous anodize alumina were investigated for anodized samples at different cell potentials from 100 to 200 V [291–295]. Two-stage anodization of aluminum was performed during a solution of 0.3 M H₃PO₄ or a methanol-water mixture. (1: 4 vol) at 0 a d –5 °C. Emission Scanning Electron Microscopes views of porous structures with images Fast Fourier Transform intensity profiles for anodized samples at 0.3 M H₃PO₄ at the bottom (100V) and highest (200V) anodization potentials are shown in Fig. 8, respectively [296–300]. Once again, an exquisite agreement is observed between the experimental fit constant and also the theoretical value for anodized samples in methanol-water mixture (1: 4 vol) [301–304]. These results indicate that the anodization of aluminum within the methanol-water system can cause porous structures with better pore arrangement. The particular proven fact that the porous anodize alumina films obtained with anodization aluminum in antacid show a fancy structure with sub-pores formed below the surface (clearly visible in Fig. 8), the common pore diameter isn't estimated. And potential impact analysis on pore diameter wasn't per.

8.2. Effect of electrolyte type

Each electrolyte has a specific anodization potential spectrum. Anodization with sulfuric acid electrolyte produces the greatest density of cavities, according to the evidence [49,136,137]. Anodization is an electrochemical process that can be applied to some metals. This process, depending on the nature of the electrolyte used, leads to the formation of two types of oxide and porous layers on the surface of metals [138–143,173–176]. When the anodization is performed in the presence of strong acids, the resulting oxide layer will be porous. In general, anodization is done in two ways: anodization with a guide template and self-organizing anodization. In anodization the metal, with a very high percentage of purity as anode and other metals such as aluminum, titanium, platinum, nickel and, sit in the cathode position and the nature of the electrolyte depends on the type of oxide layer and its properties such as hole diameter, distance It varies between cavities and cavity size. Cavity size (either average pore size or pore size distribution) is one of the properties of the material that is not well defined but is often the most useful and fundamental property of Nano process materials. The shape and size of the cavities are controlled by several independent variables, which leads to the distribution of the size and shape of the cavities, but in practice, these contents are never taken into account and the cavities are usually assumed to be narrow holes or cylinders [177–180]. This assumption leads to the definition of a specific parameter for size, which is the distance between the walls or the radius of the cylinder. The pore size distribution is often interpreted qualitatively to determine how regular material is, or in other words, how narrow the pore size distribution is.

8.3. Effect of electrolyte stirring

The anodization is critical and unquestionable. In fact, without stirring the electrolyte, the temperature at the top of the cavities rises dramatically [50]. Poor chilling can easily cause the breakdown of the oxide layer or anodic solution, especially at high currents (hard regime). Additionally, the electrolyte compositions at the underside of the cavities differ from the electrolyte bulk [50,51]. In general, by increasing the stirring speed of the electrolyte and decreasing its acid concentration, self-regulating growth of cavities occurs and better amounts of potential are often applied [181–183]. Electrolyte agitation is one of the important parameters within the geometry of the structure of porous alumina the diameter of the holes.

8.4. Effect of anodization temperature

As the anode temperature increases, the anode potential range

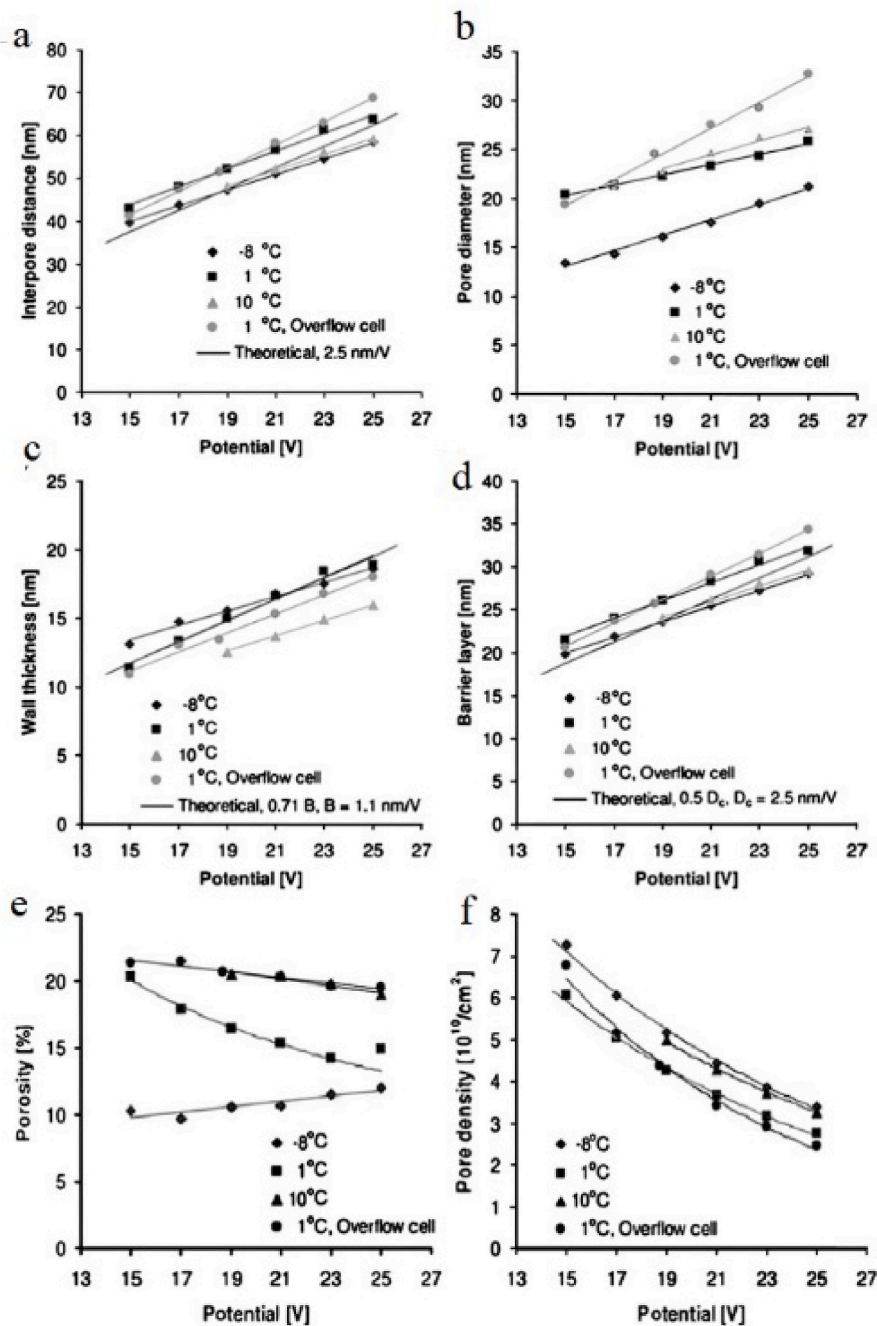


Fig. 9. The effect of anodization temperature, in terms of potential changes, on the parameters (a) the distance between cavities, (b) the pore diameter, (c) the wall thickness, (d) the thickness of the dam layer, (e) the porosity and (f) the density of the cavities; For example, anodized in 2.4 M sulfuric acid electrolyte [11].

decreases. At 10 °C, porous alumina only forms within the range of 17–25 V. Examination of experimental results shows that anodization temperature has little effect on changing the interpore distance and therefore the thickness of the barrier layer [2]; however, changes in cavity diameter and wall thickness are significant with changes in anodization temperature. In general, because the anode temperature increases, the pore diameter increases, and also the wall thickness decreases. The very cheap dissolution rate of the oxide layer occurs at -8 °C and therefore the smallest cavity produced is reported at the identical temperature. Similarly, the thinnest cell walls are observed at the best temperature studied. Anodization temperature also affects the porosity of the anodic alumina layer and also the cavity density [184–186]. The porosity of the corundum layer is between 10 and 20% within the whole

potential range and for all studied temperatures. Fig. 9 shows the effect of anodization temperature on the gap between the cavities, the pore diameter, the wall thickness, and also the thickness of the barrier layer, the porosity, and also the density of the anodic alumina cavities. However, the analysis of the pore arrangement performed on the premise of FFT images doesn't provide a transparent answer about the effect. Fig. 9 shows that increasing the duration of both anodization steps improves the regularity of pore arrangement and uniformity of pore shapes. This can be attributed to the actual fact that within the two-step self-organized anodization of aluminum a giant extension of the first anodization step improves considerably the regularity of the array of Nano-periodic concave, consisting of a triangular lattice structure, on the aluminum substrate after the oxide layer removal [187–190]. The

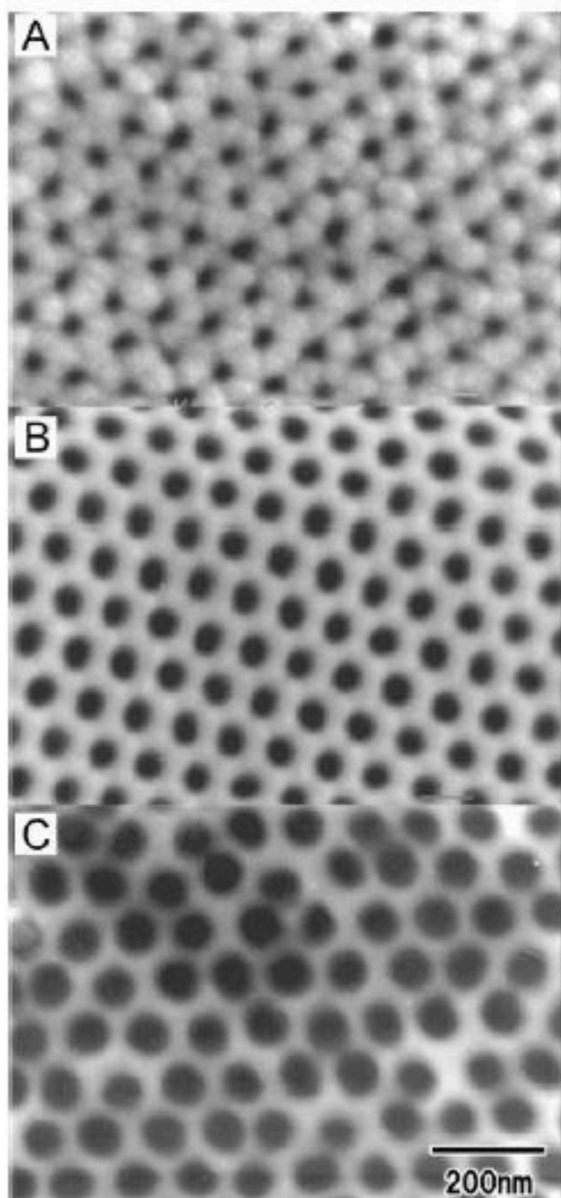


Fig. 10. SEM images of WO₃ antidot arrays with different mean hole sizes: (A) 38 nm, (B) 53 nm, and (C) 65 nm fabricated by a two-step replication process employing negative-type PMMA templates. The PMMA templates have been prepared from highly ordered AAO membranes with different pore diameters [1].

periodic concave triangular features formed on the aluminum surface function nucleation sites for the formation of pores during the second anodization step. In consequence, the regularity of pore arrangement observed after the second anodization is extremely improved.

8.5. Effect of anodization time

While anodization time influences the pore diameter, it's widely agreed that it's little effect on cell size [21,138]. The pore diameter increases because the anodization time increases; this process is caused not only by the chemical breakdown of the oxide layer, but also by the coalescence and forming of a solid wall [21]. The order of the cavities is additionally suffering from the anodization time; because the anode time in various electrolytes increases, the array of Nano pores becomes more uniform [17]. Furthermore, the number of cracks and dislocations

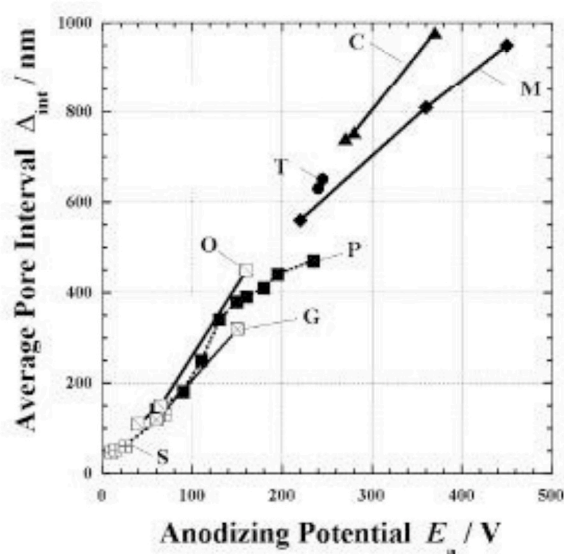


Fig. 11. Effect of anodization potential on the cavity distance for porous anodic alumina formed in different electrolytes. Dotted arrows are related to hard anodization in sulfuric acid, oxalic acid and phosphoric acid electrolytes [10].

within the porous alumina system reduces over time.

9. Luminescent properties of anodic aluminum oxide

As it was mentioned above, during anodic oxide growth, positively polarized anode attracts negatively charged anions [284]. Next, the anions are being adsorbed on the oxide's surface and then incorporated into the AAO walls. Thus, anodic alumina poses anionic impurities in its walls. According to Rauf et al. [305], intensity of PL originating from F-centers can be also controlled. The worse the nanopores' arrangement, the more the defects, like F-centers present in the AAO walls. Therefore, the worse the nanopores' arrangement, the more intensive the PL from the F-centers. Both pore diameter and nanoporous array arrangement influence the PL of the AAO. Simultaneously, morphology and arrangement of the nanopores and chemical composition of the AAO walls are controlled by the operating conditions during the anodization process. Hence, luminescent properties of AAO can be steered by the operating conditions.

10. Anodization of aluminum alloys

Anodic alumina is also being formed on technical purity aluminum, containing various alloying elements and on the aluminum alloys. Anodization of technical purity aluminum provides poor arrangement of the nanopores due to the substrate chemical inhomogeneities [306]. Nevertheless, various approaches are being undertaken to conduct successful anodization of aluminum-containing alloys.

Zhou et al. [307] reported anodization of zinc-containing aluminum alloy. Three main various anodic layers have been distinguished. The upper layer was made of pure alumina, middle one was made of alumina with incorporated zinc ions, and the bottom layer was made of unoxidized zinc nanopillars.

Additionally, Kondo et al. [308] reported anodization of purple gold, AuAl₂ intermetallic alloy. After anodic alumina removal, there were hexagonally arranged hemispherical gold nanodots with diameter of about 30 nm.

11. Macroscopic effects

AAO pores on convex surfaces attempt to achieve minimal energy by

two types of processes. Pore merging occurs, reducing the pore densities and hence volume expansion associated with the AAO growth. Alternately, pore branching occurs, reducing radially inward growth rates since the pores now grow in an angled direction. When adjacent growth fronts meet, they create weakened interfaces which ultimately are regions from which cracks nucleate and propagate. Cracks release built-up stresses. In our experiments, we find that the AAO films are crack free for up to 10 mm thickness for all anodization voltages. This thickness therefore, provides an upper limit for creating viable AAO structures on curved surfaces of 100 mm diameter Al wires. Once the cracks originate, independent 'sectors' of growth are formed. A gradual planarization of the curved AAO fronts is observed and for long anodization, fully planar growth fronts of ordered nanopores are observed. Planarization of the growth front is also an effective way to reduce volume expansion as the number of pores per unit area is reduced and this reduces volume expansion and associated stresses [309].

12. Conclusion and future challenges and perspectives

1. The formation of a porous alumina layer starts and expands from the state of the barrier layer formed on the Al surface at the start of anodization. The barrier oxide layer spreads uniformly throughout the surface, with a consistent distribution of current controlled by a continuing field [190–193]. However, the non-uniform distribution of current within the protruding areas of the aluminum substrate ends up in a rise within the field dissolution of the oxide layer and therefore the local thickening of the porous layer, thus beginning the method of cavity formation. During the formation of the oxide layer within the steady state, therefore migration of Al^{3+} and OH^- and O^{2-} ions in opposite directions, and a part of the Al^{3+} ions, which are driven directly into the electrolyte solution, haven't any role within the formation of the oxide layer.

2. OH^- and O^{2-} ions are the foremost important factors within the formation of the porous oxide layer, each of which enters the anodization through its own reactions. These ions that penetrate the plasma membrane define the properties of the porous alumina layer. Binary and tertiary structures are proposed for the membrane; in both cases the inner layer of the wall consists of pure alumina. Within the structure of porous alumina, empty spaces are observed on the ideas of the protrusions of the aluminum substrate, on the joint surface of the metal and therefore the oxide, within the inner layer of the cell walls, which are created to face up to the pressure within the layer. Given the above, geometric parameters like the pore diameter, the gap between the cavities and therefore the wall thickness of the cells may be controlled by factors like anodization voltage, electrolyte temperature, electrolyte concentration and other factors.

Authors' contributions

Ahmed Mahdi Rheima, Zainab sabri Abbas, Mustafa M. Kadhim, Srwa Hashim Mohammed: reviewing and editing.

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