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## **Historical Analysis and Applications of Nanoporous Anodized Aluminium Oxide**

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### **Abstract**

*Anodic oxidation is one of the advantageous techniques used to form a thicker oxide on the surface of aluminium. The history of anodization of aluminum dates back to the beginning of the last century. Several patents had been published before the 1950's, relating to the anodization of aluminum for decorative and coloring purposes. Anodized aluminium has pores and it is probable to generate self organized nanopores in this oxide layer by controlled anodization process. The remarkable characteristics of nanoporous anodic alumina such as highly orderings, large and small pore diameter, high pore density and longer pore channel length or thickness give marvellous results in engineering applications. For instance highly ordered and having large diameter nanopores of anodic aluminium oxide (AAO) enhances electrical, optical, and photoelectric properties. A leading example is the fabrication of ordered metallic nanowires by using AAO template, those can be applied to lithium ion batteries, magnetic devices, energy storage devices, solar cells and nano-reactors. However, high density followed by smaller-sized nanopores of AAO is crucial to enhance infiltration, ion transportation, molecular separation and catalysis. Furthermore, longer pore channel length or thickness enhances chemical resistance and adsorption properties. This property makes the surface wear resistant, corrosion resistant and suitable for load bearing applications which proves as a boon for tribology. The aim of present paper is to review on versatile applications of nanoporous anodized aluminium oxide template (NPAAO).*

**Keywords:** *Anodization, Aluminium, Aluminium Oxide, Nanoporous*

### **Introduction**

When a fresh aluminium (oxide free) surface is exposed to air at room temperature, an oxide layer (native oxide film) forms immediately because of the high affinity of the aluminium surfaces to oxygen. This oxide film protects the surface from further oxidation and therefore is called self-protecting native film. The thickness of such native film is only about 15 to 18 nm, which is susceptible to breakage. Thus, the thickness of this oxide film could be artificially increased by anodization 'Keller et al. (1953)'.

Anodization is defined as a process to generate an oxide film on metals by electrolysis. During anodization process the metal to be oxidized is made of anode in an electrolytic cell and its surface is oxidized electrochemically. Anodization of metals, especially aluminium has received a lot of attention since the beginning of the last century, due to its diverse applications, such as protective, decorative coatings and dielectrics 'Diggle et al. (1969)'. It has also found applications also in micro filters and catalytic membrane reactors 'Bocchetta

et al. (2003), Broughton and Davies (1995)'. Thin film deposition and anodization are used for manufacturing embedded capacitors 'Samudrala and Bandyopadhyay (2009)'. Nowadays, PAAO film is used as important templates for the synthesis of nanowires and nanotubes with monodisperse controllable diameter and high aspect ratio 'Montero et al. (2007), Sarkar et al. (2007), Piao et al. (2005)'. Moreover, PAAO can also be used as two dimensional photonic crystals 'Choi et al. (2003(a)), Brown et al. (2006)' and humidity sensors. Porous anodic alumina template has the necessary large number and diversity of attributes to play a vital role in the outlook of nanotechnology. PAAO is using as a lubricating reservoir to improve tribology properties 'Wang et al. (2015), Zhao et al. (2003)'.

### **Anodic Oxidation Of Aluminium: Previous Work**

The anodized film is formed by the passage of direct current in appropriate electrolytes. During anodization, the aluminium serves as an anode and a chemically stable metal such as platinum, carbon etc serves as a cathode. Various electrolytes such as phosphoric acid Wu et al. (2000)', chromic acid, oxalic acid 'Sui et al. (2002), Douglas et al. (2005), Chu et al. (2005), Keshavarz et al. (2013)', sulphuric acid can be used for anodic oxidation 'Diggle (1969), Yi et al. (2011) and (2012), Sanz et al. (2011), Sulka et al. (2006), Zhu et al. (2013), Daryakenari et al. (2016)'.

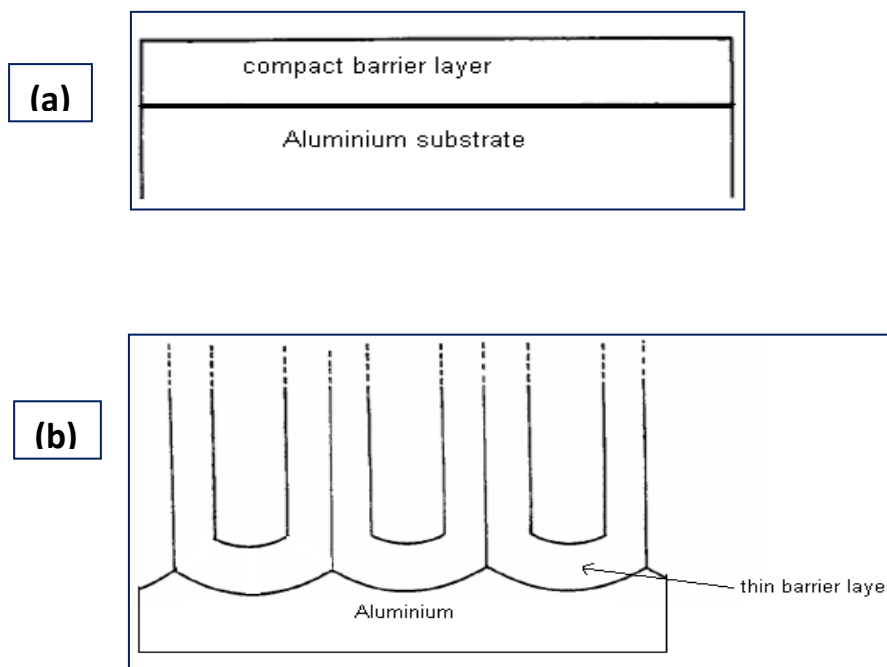
A brief introduction and a schematic diagram of the anodization cell is shown below in Fig. 2.3. The aluminium sample (shown in dark blue colour, Fig. 2.3) to be anodized is connected to the positive terminal A (anode) of the electrolytic cell. Cathode 'C' is made of any electrically conducting material. Direct current (d.c.) is passed through the cell under 'constant cell potential' or 'constant current density' mode. The current density / potential ranges, specific to the type of acid solution (electrolyte), is applied for a fixed time period at a fixed temperature of bath which kept constant by using thermostatic controlled arrangement.

### **Types of Anodic Oxide Films, Their Structure and Properties**

Two types of anodic oxide films viz. (1) barrier or non-porous oxide film and (2) porous or duplex film are formed depending on the type of the electrolyte and its pH value. Barrier films are used as dielectric material in capacitors while porous films have numerous applications. Formation and morphology of porous films are extensively researched these days, due to its extra-ordinary ability to attain well-ordered, hexagonal (also as triangular lattice) porous morphology under some recipes of anodization parameters, termed as 'self-ordering regime'. This film has got very high potential for many industrial applications and its use as a template in fabrication of many nano-devices e.g. synthesis of magnetic, superconducting and semiconducting nano-structures 'Martin et al. (1994), Saito et al. (1989)'.

Anodization of aluminium in a neutral or weak acidic solution, such as neutral boric acid, ammonium-borate and ammonium tetraborate in ethylene glycol (pH 5-7) leads to formation of a flat nonporous aluminium oxide film; commonly called a barrier-type film whose thickness depends on voltage (~1.4 nm/V) and maximum thickness is limited to few microns only at a voltage of about 500-700 V 'Keller et al. (1953)'.

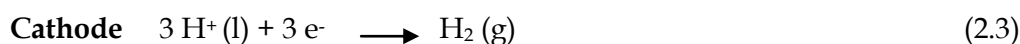
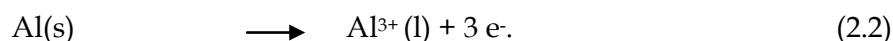
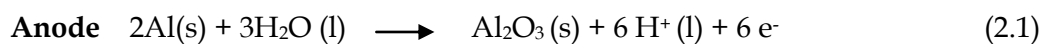
A thicker but porous layer of aluminium oxide (up to 200 microns) can be formed by anodization of aluminium in a dilute acidic electrolyte (such as sulphuric, oxalic, chromic or phosphoric acids) in which alumina is partially soluble, due to a competing mechanism of oxide growth and partial dissolution of the aluminium oxide by the hydrogen ions 'Thompson (1997)'. Fig 3.1 shows a schematic representation of the two types of anodic layers discussed.



**Fig. 3.1: A schematic of the (a) Barrier and (b) Porous types of films (Thompson et al, 1997)**

The formation of self-organized porous structure using neutral electrolytes containing fluoride has been reported only recently. The thickness of the porous layer depends on the charge density

The reactions involved during anodization are as follows:



In case of barrier type oxide film formation, electrochemical oxidization of Al takes place by reaction (2.1) and negligible amount of aluminium dissolution into the solution is given by the reaction (2.2). However, for porous oxide films, both of the reactions, viz., (2.1) and (2.2) take place simultaneously. The hydrogen evolution reaction at the cathode (2.3) is common to both types of films formation 'Runge and pomis (2000), (2002), (2006), Daryakenari et al. (2016), Keshavarz et al. (2013)'.

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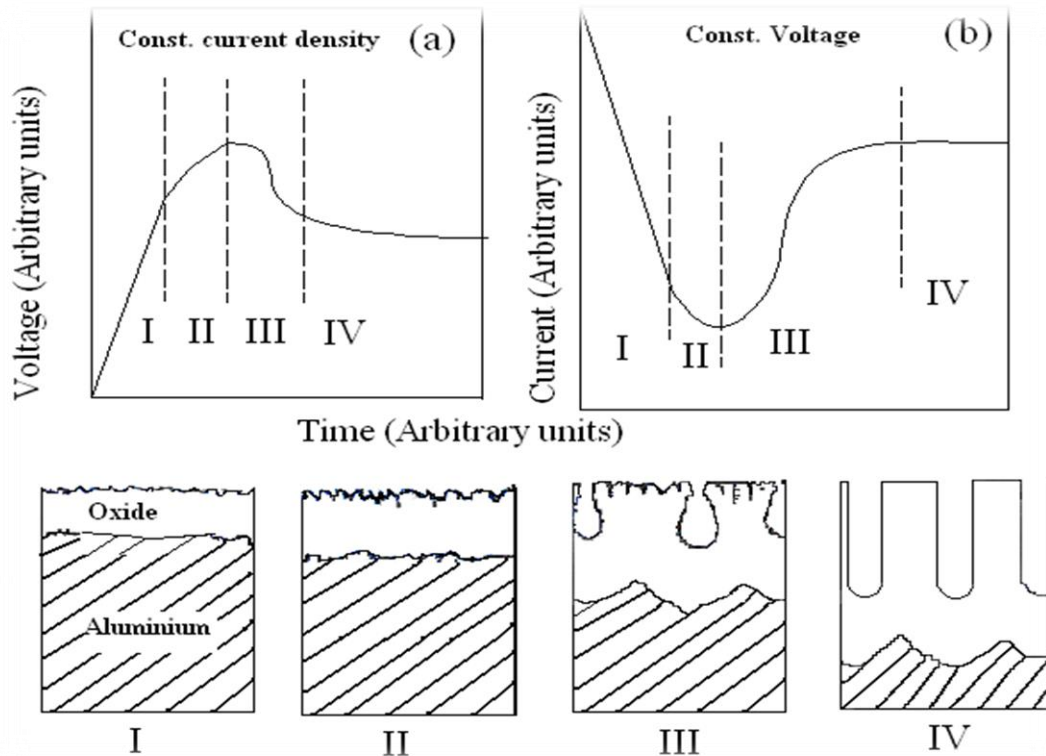
## Mechanisms Of Synthesis Of Pao Films

The synthesis of porous alumina with self-organized pore size, spacing (tailor-made) and ordering by various techniques became possible recently due to advent of scanning probe lithography 'Shingubara et al. (2003)', focussed-ion beam 'Luo et al. (2003)', electron beam lithography and direct writing laser lithography 'Stasi et al. (2007)'.

The formation of porous alumina has been studied in an extensive manner by many researchers in last five decades, but till date, there is no consensus opinion about the mechanism, which can conclusively explain pore formation. The shapes of the current-time and voltage-time transient curves are well established by 'Diggle et al. (1969), Patermarakis (2006)' and can provide insight into the growth mechanisms and kinetics 'Thamida et al. (2002), Patermarakis (1996)'. PAAO films can be formed at constant voltage or current density mode. At constant current, the voltage starts increases linearly with time until a critical value is reached when transition from barrier to porous type films occurs. The voltage decreases slightly, due to pore formation, and then reaches a steady state whose characteristics depend on the pH and the applied current density. At constant voltage, the current decreases rapidly over a short period of time due to a sharp increase in barrier layer thickness and just after the initiation of pore formation, the current increases and reaches steady values after a long time. However, it is frequently observed that during steady state growth, the current density reduces a little in a consistent manner.

The reason behind this is diffusion limits in the long pore channels. The decrease in voltage and the increase in current after pore formation are related to the increase in the active surface area due to the pores. 'Parkhutik et al. (1992)' have explained these processes in four stages (I to IV) by the following sketch (Fig. 2.7), in which, the status of electrical transients as well as physical changes that occurs in the PAAO during anodization are depicted. The four stages of porous structure growth are shown in the lower diagram.

1. Barrier layer formation
2. Pore-initiation
3. Pore growth and
4. Steady state pore growth.



**Fig. 5.1: Schematic diagram showing the mechanism and kinetics of porous oxide growth on aluminium in Galvanostatic (Constant current) and (b) potentiostatic (Constant voltage) regimes (Parkhucic et al, 1992)**

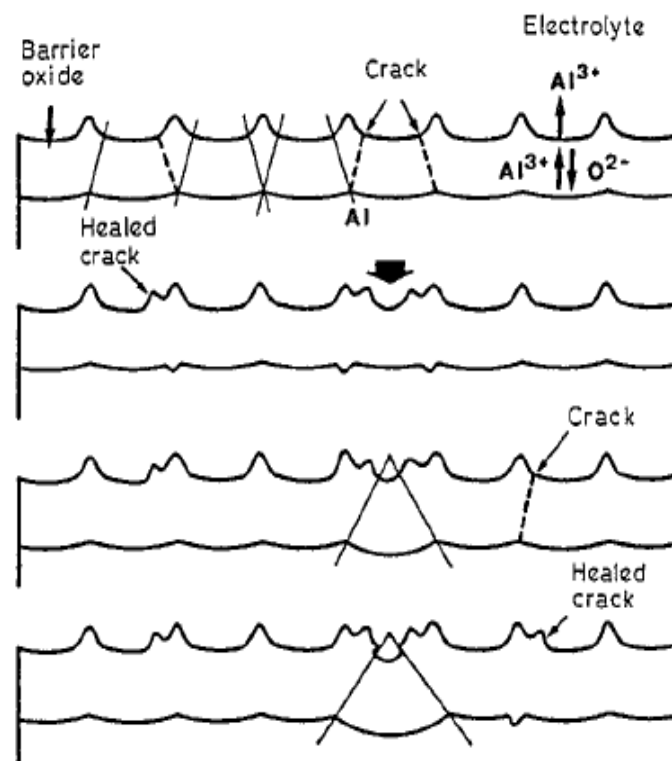
The electric field applied during the anodization causes the thickening of the native oxide layer (barrier layer) in the beginning followed by on dissolution of this pre-formed barrier oxide layer at some weak points such as at grain boundaries 'Bandyopadhyay et al. (1996)' at the site of accumulated impurities, at the site where oxide thickness is minimum, or the site at which the surface is concave shaped. Electric field gets concentrated at these sites 'Sullivan et al. (1970)' resulting in initiation and growth of the pores due to self-perpetuating mechanism or due to positive feedback mechanism 'Bandyopadhyay et al. (1996)'. In the start, dissolution takes place at a particular point. At this particular point, the thickness of oxide gets reduced and current flows to repair the damage to the oxide layer or oxide formation. This increases the electrolyte temperature at this point, and more rapid dissolution of oxide occurs at the same point. Therefore, both the oxide formation and dissolution are assisted by electric field. This mechanism would perpetuate a pore once it is formed. As the current continues to flow through the single pore, additional oxide forms such that an oxide cell is created 'Keller et al. (1953)'.

In the stage/process of PAAO formation, due to the competition that prevails among the growing pores, all pores cannot grow to the same extent due to different degrees of heterogeneity in the metal just below the growing pores. A few of the pores stop growing



while others will continue to grow. A dynamic equilibrium is established between oxide formation at the metal /oxide interface and field-enhanced dissolution of the oxide at the electrolyte/oxide interface. Oxidation occurs at the metal/oxide interface by the migration of  $O^{2-}$  or  $OH^-$  from the electrolyte 'Uchi et al. 2001'. Dissolution of the oxide layer is caused mainly by the hydration reaction of the formed oxide layer as shown below 'Shimizu et al. (1992)'.

The field assisted dissolution rate, first proposed by 'Hoar et al. (1959)' depends on the pore geometry and therefore, it is not possible to quantify the dissolution through a simple model. 'Shimizu et al. (1992)' concluded that the buildup of tensile stress in the oxide contributed to local cracking of the film above pre-existing metal ridges (from electropolishing) on the metal surface. They also suggested that cracked regions are repaired by oxidation processes; however this leads to non-uniform film growth. The locally thinner regions then become preferred regions for pore development (Fig 2.10).

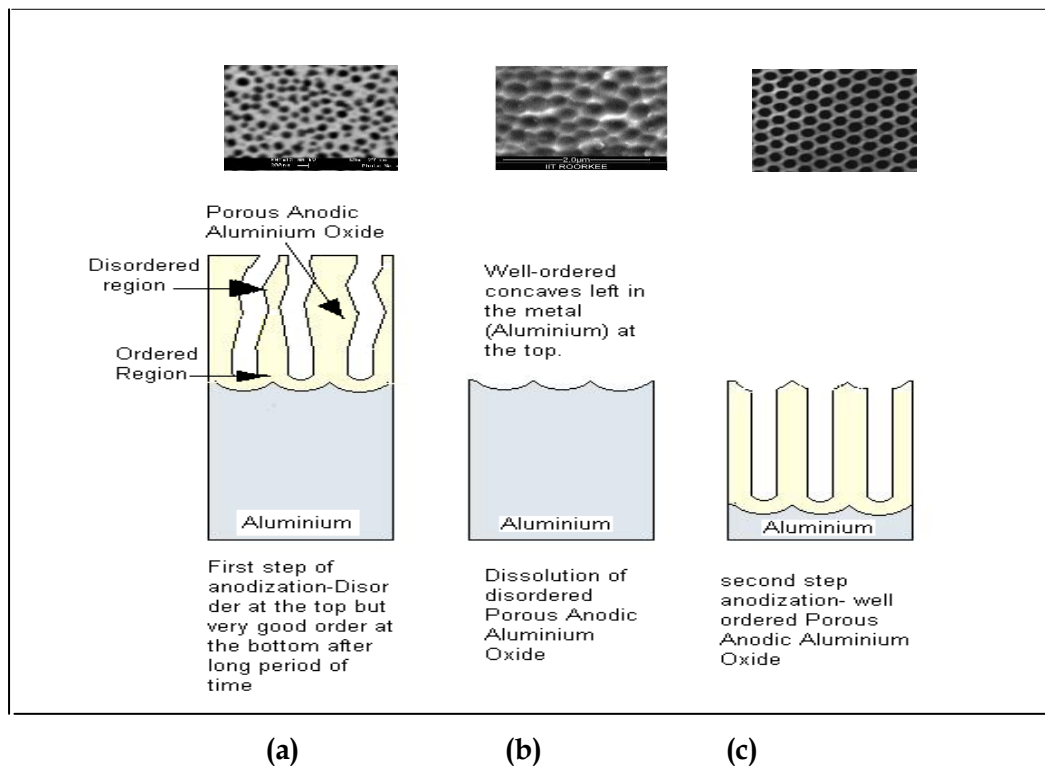


**Fig. 5.2: Schematic Diagram Showing Pore Development during Anodizing of Aluminium, Oxide Cracking and Healing Process that Occur due to Tensile Stress in Oxide during Porous Alumina Formation as Explained by Shimizu et al. (1992)**

However, they neither confirmed the presence of stress by experimental measurements nor explained the mechanism of formation of pores with a characteristic spacing dependent on the applied voltage / current, pH and temperature. 'Belwalker et al. (2008), Ilango et al. (2016).

## 6. TWO-STEP ANODIZATION

'Masuda et al. (1996)' stated that a highly ordered nanoholes array could be attained by two-step anodization of high purity aluminium using a 0.3 M oxalic acid solution using constant voltage of 40V at 0°C. The first step anodization is taken out for 76 hours. Although cell arrangement at the surface is not so regular, nanoholes consistency increases as film thickness increases. Admirable regularity can be realised at the hole bottom afterwards long anodization period as shown in Fig. 2.11(a). Then the first anodic alumina film is selectively etched away by a mixture of 35 ml/l 85% H<sub>3</sub>PO<sub>4</sub> and 20 g/l CrO<sub>3</sub> at 80° C as established by 'Schwartz and Platter (1975)'. The etched surface has an irregular surface roughness, with highly regular array of nanohole bottoms (Fig 2.11 (b)). Subsequently, the nanohole array made by the second anodic oxidation reveals outstanding regularity as a result of initial surface Fig. 2.11(c) 'Say and Chen (2007)'.



**Fig. 6.1: Mechanism of two step anodization; (a) First step of anodization forms disordered region of PAAO at the top surface and ordered region at the bottom, (b) The oxide part (yellow coloured) is etched away and metal part (light grey) is left having concave shaped impressions (well-ordered)**

### Conclusion

By anodization technique, one can obtain a highly ordered PAAO, which is characterized by a long anodizing period. There are some reports of multi-step anodization, where three or more steps of anodization have been conducted 'Sulka et al. (2002), Zhou et al.

(2006), Zhang et al. (1998), but the improvement in ordering as well as size of well ordered domains is not significant after the second step.

However recently some researcher have successfully fabricated ideally ordered nanoporous alumina films by high-field anodization realizing the need to produce highly ordered porous anodic alumina film with low cost and by a simple process on a large scale 'Zaraska et al. (2009), (2010), (2013), (2014), Nazemi et al. (2014), Ilango et al. (2015)'.

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