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Review of Highly Ordered Anodic Porous Alumina Membrane Development

Deepak Mankotia[1], Dr. Y.C. Sharma[2], Dr. S.K.Sharma[3]

[1]Research Scholar, Vivekananda Global University Jaipur
[2]Professor, Dept. of Physics, VIT, Jaipur
[3]Professor, Dept. of Physics, Maharishi Ved Vyas College, Jagadhari

Abstract— **Anodic alumilminium oxide (AAO) formation is an electrochemical process. Using this process hexagonal cylindrical shape nano pore can be produced i.e. nanoporous membrane. Hexagonal nano pore can be obtained in anodic alumina film by repeated anodization. This review paper is an overview of AAO membrane by two step anodization, effect of anodization parameter. A new method for characterization of pore structures of AAO membranes has also been suggested by flow porometry and bubble point method.**

Keywords— **AAO, nanoporous membrane, Two step anodization, flow porometry bubble point.**

I. INTRODUCTION

Anodization is an electrochemical oxidation process. Two type of anodic aluminum oxide (AAO) are formed depending on the electrolyte: one is nonporous barrier type oxide from neutral oxide and other is a porous type oxide from acid electrolyte. The porous type Aluminum anodization received growing attention because AAO widely used for fabrication of various nanostructure materials. A number of studies have been performed in which nanporous structure is used as template in the production of nanowires or nanotubes from various materials, used as a filter for purification of DNA or whole cell from blood. Various structures and devices based on AAO such as carbon nanotubes (CNTs), metal nanowires, nanodotes, nanoholes and other nanodevices and In many applications AAO membranes act as barriers to particles or organisms. (1-5)

The porous anodic alumina may be represented as honeycomb structure of fine channels characterized by a closed packed array of hexagonal cells, each containing cylindrical pore, as shown in fig1.Pore diameter ranging from 30 to 400 nm, oxide thickness from 0.3 to 200µm and pore densities in the range 10^9 to 10^{11} cm^{-2} can be obtained. The size and interval can be easily controlled by changing the processing condition.

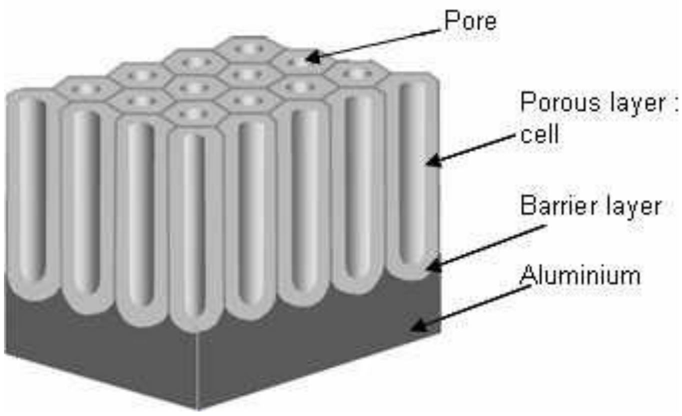


Figure 1: A schematic drawing of porous alumina

In an anodizing process, an aluminum plate is connected to the anode of DC source. A week acid solution used as electrolyte. The cathode can be made from chemically inert conductive material. When apply tension, hydrogen ions are reducing in order to form hydrogen gas at cathode and aluminium is oxidized into Al^{3+} cations. Some of these cations are dissolved in the electrolyte, and some part form oxide layer on the metal surface, For further reaction,oxygen containing anions are supplied by electrolyte (6).

Type of anodic oxide Film:

The anodizing of aluminium can result in two different type of oxide film a barrier type anodic film and porous oxide film. The adherent, non-porous and non conducting barrier type of anodic film can be formed by anodizing aluminium in neutral solution (PH = 5-7) in which anodic oxide layer stays practical in soluble. These film are thin and dielectrically compact .The group of electrolyte used for this film formation include boric acid, ammonium borate, ammonium tartrate and aqueous phosphate solution, as well as tetraborate in ethylene glycol, and some organic electrolyte such as citric, malic, succinic and glycolic acid. Porous oxide film are made using strong electrolyte solution such as sulfuric acid, oxalic, phosphoric acid and chromic acid solution ,where the resulting oxide film can be only sparingly soluble. For anodic porous alumina. The film growth is associated with localized dissolution of the oxide, as a result of which pores are formed in oxide film (6, 7).

Electrochemistry of porous alumina:

Electrolyte	Pore Diameter (nm)	Interpore distance (nm)	Voltage(V)
Sulphuric Acid	30-35	60-70	25-27
Oxalic acid	40-100	80-200	30-80
Phosphoric Acid	130-250	250-500	100-195

Table: 1 Comparison of pore diameter and inter pore distance with different electrolyte.

Porous alumina templates grow gradually in acid solutions that continuously anodize aluminium under the electric field between two electrodes. Different pore diameter and interpore distance can be obtained by using different electrolyte as shown in table1(8).

II. TWO STEP ANODIZATION:

2.1. Experimental procedure:

A pure aluminium sheet (99.99%) was annealed at 500°C for 2hr in an inert atmosphere (N_2) atmosphere and than cooled.
To remove surface oil samples were rinse with acetone and ethanol and dried after twice washing with deionized water.
To remove the natural oxide membrane samples were immersed in 4wt% H_2SO_4 and 7wt%H_2CrO_4 mixed solution by volume 1:1 at 70 ℃ for 2 min.
To neutralize acidic substance samples were immersed into 10wt% NaOH at 20 °C for 15sec.
The samples were dried after twice washing with deionized water, immersed in absolute ethanol at 25 °C for 3 min, and subsequently put into 60 wt% HCLO4 ethanol solution (HClO4: C2H5OH: H2O =4:4:2, v/v/v) at 30 °C to carry out electrochemical polishing at voltage of 2 to 3 V for 2 to 3 min.
The polished samples were immersed in a 0.3 M oxalic acid solution under a current density of 4.0 mA/cm^2 for 2 h (first step of anodic oxidation).
During the first step anodization, hexagonal pore formed on the surface of aluminium foil are nano ordered due to surface roughness. These non ordered pores of AAO should be removed.
To remove the first oxide membrane, the samples were immersed in a mixed solution with a 1:1 volume ratio of 6 wt% H_3PO_4 and 1.8 wt% H_2CrO_4 at 40 °C for 15 h and then dried after washing with deionized water.
The samples were immersed in oxalic acid solutions with various concentrations of oxalic acid at 40 °C under a constant voltage of 40.0 V for 9 h (second step of anodic oxidation).
The unreacted aluminium was removed by dipping the specimen in saturated solution of mercuric chloride ($HgCl_2$) for 10 to 30 min.
The AAO template was then treated with 5wt% phosphoric acid for 30-60 min to remove the bottom barrier oxide layer.
The AAO were again rinse with acetone and ethanol and dried (2, 9).

2.2. The Chemical Reactions Involved In Anodization of Aluminum

During anodization several chemical reactions take place at the interfaces between the metal and the oxide (m/o interface) and the oxide and the electrolyte (o/e interface).
These reactions are important for formation and structuring of the oxide. In the electrolyte there is splitting of water into hydrogen and oxygen ions. The electrolysis of water occurs at the bottom of the pore near the interface of the electrolyte/oxide.

$$H_2O \rightarrow 2H^+ + O^{2-}$$

Due to high electric field produce at the barrier layer, the oxygen ions then migrate through the oxide along their electrical gradient to react with aluminum at the metal/oxide interface and form aluminum oxide

$$2Al + 3O^{2-} \rightarrow Al_2O_3 + 6e^-$$

In the metal there is also formation of aluminum ions at the interface of metal/oxide and its distribution in the oxide layer near the interface.

$$Al \rightarrow Al^{3+} + 3e-$$

These aluminum ions can either react with the oxygen ions at the m/o interface or continue to the o/e interface and form oxide there

$$Al^{3+} + 3O^{2-} \rightarrow Al_2O_3$$

These reactions and movements of ions suggest that oxide forms at both interfaces simultaneously, which has been confirmed by experiments where impurity ions are added to the metal or the electrolyte before anodization. Another important reaction is the dissolution of oxide into the electrolyte at the interface electrolyte/oxide.

$$Al_2O_3 \rightarrow 2Al^{3+} + 3O^{2-}$$

The dissolution of oxide is crucial for the formation of porous alumina. If no dissolution occurs the oxide layer will be of barrier type. (15, 17)
In this process there is equilibrium between the dissolution at the oxide interface electrolyte/oxide and oxide formation at the interface oxide/metal. These balances is critical to the formation of pore alumina structures, therefore maintain the barrier layer thickness constant in the entire process of anodizing and allow the steady state propagation the porous layer. The electric field enhanced dissolution is the driving force of the formation of the porous alumina. This mechanism is not produced when anodize in neutral solution.

The formation of highly ordered hexagonal arrangement of the membrane of pore alumina is a high organization process during anodizing of the Al .It is suggested that the repulsive forces between neighboring pores in the metal/oxide interface promote the formation of hexagonal ordered pore arrangements.

2.3. Kinetics

The current density passing across the oxide film can be written as

$$j = ja + jc + je$$

Where *ja*, *jc* and *je* are the anion-contributing, cation-contributing and electron-contributing current density, respectively. Since the electronic conductivity in the aluminum oxide is very low, the ionic current density ($ji = ja + jc$) is the predominant mode to transport the charges. The relationship between the ionic current, *ji*, and the electric field, *E*, can be expressed in terms of the Guntherschultze-Betz equation

$$ji = j0\exp(\beta E)$$

Where both $j0$ and β are temperature- and metal-dependent parameters. For the aluminum oxide, the electric field E, $j0$ and β are in the range of 10^6 to 10^7 V/cm, 1×10^{-16} to 3×10^{-2} mA/cm2 and 1×10^{-7} to 5.1×10^{-6} cm/V, respectively. Based on the Guntherschultze- Betz equation, the rate-limiting steps of the film formation are determined by the ionic transport either at the metal/oxide interfaces, within the bulk oxide or at the oxide/electrolyte interface. Nowadays, it is generally accepted that the oxides simultaneously grow at both interfaces, e.g., at the metal/oxide interface by Al^{3+} transport and at the oxide/electrolyte interface by oxygen ion transport. (16)

III. ANODIZATION PARAMETERS INFLUENCING SELF – ORDERING:

3.1. Anodic Voltage effect on pore:

Pore diameter and interpore distance can determine by the voltage. The inter pore distance D_{int} and pore diameter (D_{pore}) tend to be proportional to the anodic voltage (U) with Proportionality constants of 2.5 nm/V for the pore spacing and 1.29 nm/V for the pore diameter. (Proportionality constants may slightly vary).

$D_{int} = 2.5U$

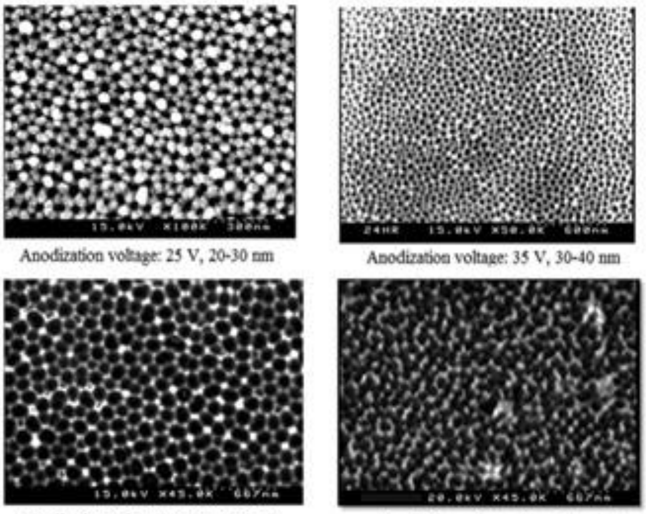
$D_{pore} = 1.29U$

In addition, the thickness of the barrier layer can be approximately estimated as half of the interpore distance.

$D_{int} = 2D_B$

Where D_B is the barrier-layer thickness.

To study the effect of voltage on pore diameter at constant time and temperature 5min &15 °C, a range of voltages (25V, 35V, 40V, and greater than 60V) was selected, and the changes in the pore diameter variation were observed. From the SEM analysis shown in Fig, it is clear that the voltage has predominant effect on uniform pore formation. Pore diameters of ~20- 30 nm, at 40 V, ~30-40 nm at 50 V and ~40-50 nm at 60 were observed. But at 60V or above, the pores are almost collapsed.



Anodization voltage: 25 V, 20-30 nm

Anodization voltage: 35 V, 30-40 nm

Anodization voltage: 40 V, 40-50 nm

Anodization voltage: More than 60 V

Effect of voltage

From the above-mentioned observations, one can conclude that too high voltage will lead to the destruction of the pores, which occurred at or above 60V in this study. (12)

3.2. Pore wall thickness (W):

Pore wall thickness W can be express as

$W = 1/2\,(D_{in} - D_{pore})$

3.3. Barrier layer thickness (B):

During the anodization of aluminum, a very thin, dense and compact dielectric layer is formed at pore bases. The barrier layer has the same nature as an oxide film formed naturally in the atmosphere, and allows the passage of current only due to existing faults in its structure. The existing compact barrier layer at the pore bottoms makes the electrochemical deposition of metals into pores almost impossible. On account of this limit, the thickness of the barrier layer is extremely important and can determine any further applications of nanostructures formed by the anodization of aluminum. The thickness of the barrier layer depends directly on the anodizing potential. The dependence is about 1.3–1.4nmV^{-1} for barrier-type coatings, and 1.15nmV^{-1} for porous structures. (7)

Some variations have been reported in the barrier layer thickness with anodizing potential or the concentration of electrolyte. The values of B_U(known as an anodizing ratio and defined as a ratio between the thickness of the barrier layer and anodizing potential) shows that variation of the barrier layer thickness per volt depends on whether oxide films are formed at a constant potential or at constant current density regimes . The increasing temperature of anodization decreases the thickness of the barrier layer for anodizing at the constant potential. An opposite relationship is observed, however, for the constant current density anodization. The increasing concentration of phosphoric acid at the constant temperature either decreases or increases the barrier layer thickness for the

Potentiostatic or galvanostatic anodizing regime, respectively. This can be attributed to the fact that, at constant current density, a constant electric field is maintained across the given barrier layer. The increasing thickness of the barrier layer with increasing electrolyte concentration suggests that ionic conduction becomes easier under the set current density, and that most of the ionic current passes through micro crystallites in the barrier layer. On the other hand, the observed decrease in barrier layer thickness with increasing electrolyte concentration and anodizing

Temperature is a direct result of an enhanced field-assisted dissolution of oxide at the oxide/electrolyte interface.(7)

3.4. Porosity (α) and pore density (n):

Two other features that characterize the porous oxide layer are its porosity (α) and the pore density (n).

$$\alpha = \pi/3.46(D_P/D_{int})^2$$

$$n = (1.15)10^6/\,(D_c)^2$$

It can be seen that the pore density is inversely proportional to the square of the anodization voltage. (1)

3.5. Effect of electrolyte:

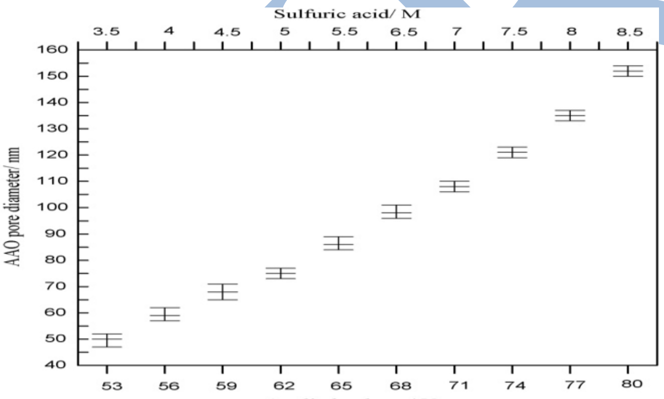
3.5.1. Type and concentration of the electrolyte:

The type and concentration of electrolyte for a given anodic voltage has to be selected carefully to obtain self-ordered pore growth. Most widely used acid are Sulphuric acid (H_2SO_4), Oxalic acid ($H_2C_2O_4$) and Phosphoric acid (H_3PO_4). Anodization of aluminium is usually carried out in H_2SO_4 at low voltage, typically ~25 V.

$H_2C_2O_4$ is suitable for medium voltages of 40 V and H_3PO_4 for high voltages (160-195 V) depending on the concentration of the electrolyte. H_2SO_4 produces the smallest pore and cell sizes, whilst H_3PO_4 results in the largest pore diameters. This restriction is due to the conductivity and PH value of the electrolyte. For Example if aluminum is anodized in sulfuric acid at high potential, break down of the oxide layer take place. In addition, the PH value of the electrolyte determines the size of pore. At lower PH value smaller pore diameter are obtained.Therefor, large pore diameter are obtained by using the phosphoric acid, and smaller pore diameter are obtained by using sulfuric acid.

3.5. 2 Mixture of electrolyte:

The self-ordered AAO films can be effectively formed pure aluminum sheets in an aqueous solution mainly consisting of sulfuric and oxalic acids. The applied voltage and sulfuric acid concentration were found to be the key factors affecting the pore diameter of AAO. The pore diameter of AAO is regularly and monotonously increased from 50 to 150 nm by the simultaneous increase in the applied voltage and the concentration of sulfuric acid from 53 to 80V and from 3.5 to 8M as shown in fig.



The applied voltage, and sulfuric acid concentration were clarified to be the key factors affecting the pore diameter of AAO films while the influences of oxalic acid is very minor. The shape of nanopores is transformed from circle to hexagon when the pore diameter size is above 100 nm (18).

3.6. Effect of temperature:

3.6.1. Effect of temperature on pore diameter:

During anodization, the surface of aluminium heats up (the reaction involved in the anodic oxidation of aluminium is exothermic), leading to a requirement for a cooling system. During the anodization, temperature should be kept lower than room temperature to prevent the formed oxide structure from being dissolved in acidic electrolyte. The Temperature should be set between 5 and 20°C if Oxalic acid is used as the electrolyte at 40 V and between 0 and 5°C for phosphoric acid at 195 V.

The second reason to keep the temperature low as possible is to avoid a local heating at the bottom of pores during anodization. The local heat causes an inhomogeneous electric field distribution at the bottom, leading to electrical breakdown of the oxide. In fact bursts and crack of the oxide film are generated if porous film is produce without temperature controlling. Speed of growth of porous alumina is affected by the temperature. Lower is the temperature lower is the growth rate.

Surface pore density and pore volume increase with temperature, the increase in electrolyte temperature give rise to the increase in no of pore, and their diameters, which result in a porosity increase. The pore size were increased from 10 to 40nm when the temp was increased from 5 to 20 °C.The temperature increase leads to an enhancement of aluminium dissolution rate, which result decrease in ohmic resistance and hence of the potential of the process. As mention previously potential of the process is directly proportional to the pore size and inversely proportional to the pore density .The dissolution of the resulting oxide by the acid electrolyte is endothermic. The excessive heat not only triggers burning or breaks down of an anodic film but also promotes undesired acidic dissolution of the oxide membrane by the electrolyte. (11, 12, 14)

3.6.2 Effect of temperature on barrier layer thickness and inter pore distance:

The barrier layer thickness depends on the anodizing voltage and is independent of the electrolyte temperature <40 °C. At temperature greater than 40 °C, the barrier layer thickness is higher. It is observed that the barrier layer thickness is independent of the electrolyte temperature because, at a given voltage, an increase in electrolyte temperature leads to an increase in aluminum oxide growth which is then balanced by a corresponding increase in its dissolution rate in the electrolyte. The inter-pore distance estimated from SEM monographs. At a given voltage, there was no significant change of the inter-pore distance with temperature. However, the pore size slightly increased with bath temperature (19).

3.7. Effect of anodizing time on pore:

With increase in time pore size increase but pore density decreases. The pore size increase by merging with adjacent pore. The pore density decrease witsh anodizing time. Pore growth may be due to a field assisted hydrogen ion attack on the oxide layer. With increase in anodization time pore depth increases from 5min (Pore depth 130nm)to 15min (pore depth 290nm).with further increase in anodizing time, The pore depth decreased to 139nm.

IV. CHARACTERIZATION OF PORE STRUCTURE:

The novel technique flow porometry has been developed to characterize these relevant pore structures of membranes without distorting the pore structure.

4.1. Principle:

The sample of the membrane is soaked in a wetting liquid that fills the pores spontaneously. The pressure of a non-reacting gas on one side of the sample is gradually increased to empty the pores and allow gas to flow through the sample. The differential pressure needed to empty a pore is determined the pore diameter by following relation.

$D = 4 \gamma \cos \theta / p$

Where,
p= differential pressure on the sample determine by the bubble point.
γ = surface tension of the wetting liquid
θ = contact angle of the wetting liquid on the sample
D= pore diameter. Pore diameter is defined as the diameter of a cylindrical opening

Figure 2 illustrates the characteristics of the membrane obtainable from the measured gas flow rates and differential pressures. (13)

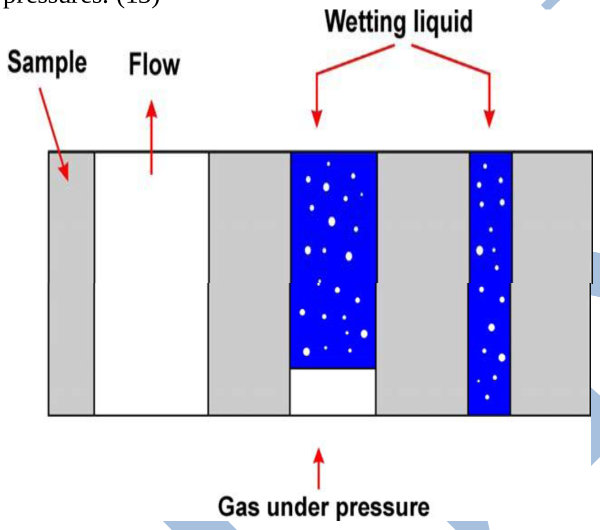


Figure 2 : Principal of capillary flow porometry

4.2. Bubble point:

A bubble point test is used to determine the pressure at which a continuous stream of bubbles is initially seen. To perform a Bubble Point Test, gas is applied to one side of a wetted filter, with the tubing downstream of the filter submerged in a bucket of water. The filter must be wetted uniformly such that water fills all the voids within the filter media. When gas pressure is applied to one side of the membrane, the test gas will dissolve into the water, to an extent determined by the solubility of the gas in water. Downstream of the filter, the pressure is lower. Therefore the gas in the water on the downstream side is driven out of solution. As the applied upstream gas pressure is increased, the diffusive flow downstream increases proportionally. At some point, the pressure becomes great enough to expel the water from one or more passageways establishing a path for the bulk flow of air. As a result, a steady stream of bubbles should be seen exiting the submerged tubing. The pressure at which this steady stream is noticed is referred to as the bubble point.

4.2.1 Test Method

1. Record the filter information. Also include physical observations.
2. Wet the filter to be tested with water or in any compatible chemical.
3. Place the wetted filter in the appropriate housing or holder.
4. Connect the outlet fitting from the compressed air pressure regulator to the upstream side of the test filter. Check that the gauge which is connected to the pressure regulator has subdivisions of at least 0.5 psig, and has the capacity to measure up to 100 psig and error. A digital pressure gauge can also be used.
5. Connect the outlet fitting from the compressed air pressure regulator to the upstream side of the test filter.
6. Connect a piece of flexible tubing from the downstream port of the test filter into a beaker filled with water.
7. Starting from zero pressure, gradually increase the pressure to the test filter using the pressure regulator.
8. Observe the submerged end of the tubing for the production of bubbles as the upstream pressure is slowly increased in 0.5 psig increments. Note the rate that the bubbles appear for the end of the submerged tube.
9. The bubble point of the test filter is reached when bubbles are produced from the tube at a steady rate. Record the pressure to the nearest 0.5 psig as indicated on the pressure gauge.

V. CONCLUSIONS:

Processes for forming self ordered porous alumina have been reviewed. An AAO membrane with well –ordered and uniform pore was obtained by anodizing aluminium in an electrolyte by two step method and technique developed for characterization of membrane by capillary flow porometry.

References:

[1]. Gerrard Eddy jai poinern, Nurshahidah Ali and Derek Fawcett.“Progress in nano-engineered anodic aluminium oxide membrane development ” materials journal,No.4,2011,pp .487-526.
[2]. Yan huang,Hong-yan,Ce Zhao,Ye-qing Qu,and Pin Zhang.“Kinetics Models of Controllaable Pore Growth of anodic aluminum oxide

Membrane"Met.Mater.int.Vol.18,No.3,2012,pp. 433-438.

[3]. Chi Lu and Zhi Chen, "Anodic Aluminum Oxide-Based Nanostructures and Devices",Encyclopedia of Nanoscience and Nanotechnology,Volume 11,2012,pp.235-259.

[4]. HOU PengXiang,LIU Chang,SHI Chao and CHENG HuiMing, "Carbon nano tube prepared by anodic aluminum oxide template method",Chinese Science Bulletin,2012,Vol 57,No. 2-3,pp.187-204.

[5]. Hee Han,Sang-joon park,Jong shik Jang,Hyun Ryu,Kyung Joong Kim,Sunggi Baik,and Woo Lee,"In Situ Determination of the pore opening point during wet-chemical Etching of the Barrier Layer of Porous anodic aluminum oxide:Non uniform impurity Distribution in Anodic Oxide"ACS Appl.No.5,2013,pp.3441-3448.

[6]. D.Nemes,V.Moldovan,E.Bruj,N.Jumate,I.vida-simiti,"Porous Anodic Alumina Films obtained By Two Step Anodization"Engineering Sciences,2011,Vol 4,No 2,pp.75-80.

[7]. Grzegorz D. Sulka,"Highly Ordered Anodic Porous Alumina by Self Organized Anodizing"Nanostructured Materials in Electrochemistry,2008,Weinheim ISBN 978-3-527-31876-6.

[8]. "Report on self-ordered porus Alumina Template Technology"N2T2 Deliverable WPLID3.

[9]. P.Chowdhury,K.Raghuvaran,M.Krishnan,Harish C barshilia and KS Rajam,"Effect of process parameters on growth rate and diameter of nano-porus alumina templates,"Bull,Mater SCL, Vol 34,No. 3,pp. 423-427.

[10]. Gerrard Eddy jain Poinern,Xuan Thi Le,Marilyn Hager,Tom Becker,Derek Fawcett, "Electrohemical Synthesis,Characterisation,and Preliminary Biological evaluation of an Anodic Aluminium Oxide Membrane with a pore size of 100 nanometers for a potential cell culture substrate"American journal of Biomedical Engineering,2013,No. 3(6),pp. 119-131.

[11]. Prof. Dr. Ulrich,Prof.Dr.Helmut,prof.Dr.Patrik Schmuki,"Fabrication Of Monodomain Porous Alumina Using Nanoimprint Lithography And Its Applications"ULB Sachsen-Anhalt,2003,pp.1-103.

[12]. Lan Shen,Mubarak Ali,Zhengbin Gu,bonggi Min,Dongwook Kim,and Chinho park, " Pareparation of anodic aluminum oxide (AAO) nano-template on silicon and its application to one dimensional copper nano-pillar array formation"Korean J.Chem.Eng,2013,30(1),pp. 221-227.

[13]. Akashaya Jena and Krishan Gupta, "Characterization of pore Structure of Filtration Media", Fluid/partical separation journal,Vol.14,No.3,2002,pp.227-241.

[14]. N.V. Myung,J.Lim,P.Fleurial,M.Yun,W.West and D.Choi,Nanotechnology Vol.15,833(2004).

[15]. Ajab Khan, Kasi,Jafer, Khan Kasi, Nitin Afzulpurkar and Erik Bohez "Fabrication of anodic aluminium oxide(AAO) Nano-Porus Membrane on both sideAluminium Sheet" ICMEE,Vol.2,2010,pp.122-126.

[16]. Ian W.M.Brown, Jeremy P.Wu andGeoff Smith, "Fabrication ,Structure and Properties of Nanostructured Ceramic Membranes"The Macdiarmid institute for Advance material and Nanotechnology.pp. 191-228.

[17]. Woo Lee,Ran Ji Ulrich Gosele and Kornelius Nielsch "Fast fabrication of long-range ordered porous alumina membrane by hard anodization"Nature materials,vol 5,2006,pp741-747.

[18]. Allen Bai,Chi –Chang Hu, Yong-Feng Yang, and Chi-Cheng Lin"Pore Diameter Control of anodic aluminum oxide with ordered array of nanopores"Elsevier,2008,pp.2258-2264.

[19]. Alaa M. Abd –Elnaiem, and A.Gaber, "Parametric Study on the Anodization of pure Aluminum thin film Used in fabricating Nano-pores Templates"International journal of electrochemical science,vol 8,2013,pp.9741-9751.