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FABRICATION OF MICROINJECTOR SYSTEM (SIM) USING ANODIC ALUMINUM OXIDE

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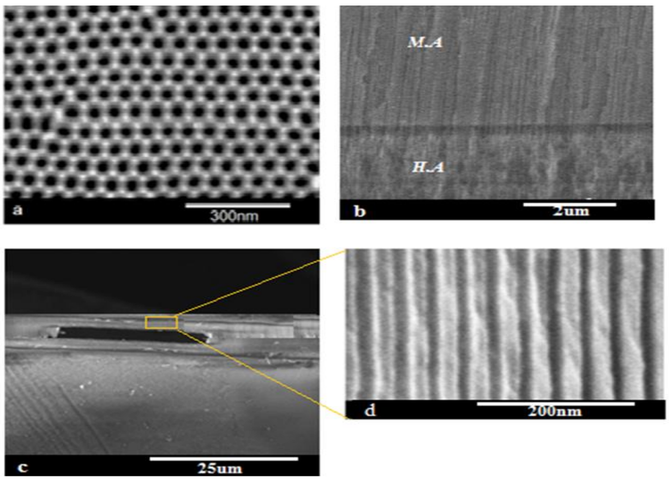
HIGHLIGHTS

- Fabrication of anodic aluminum oxide(AAO) by combing M.A and H.A
- Develope microinjector system (MIS) using anodic aluminum oxide
- First experimental study about embeded micro flow channels with injecting nozzle fabricated inside alumina membrane.

GRAPHICAL ABSTRACT



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ABSTRACT

The present study provide method to construct a novel type of micro-injector system (MIS) for microfliudic delivery inside nanoporous anodic aluminum oxide (AAO) membrane. Now a day's these micro and nano combined structure gain attention in medical and biological applications. This fabricated micro-nano structure consists of a thin film of anodic aluminum oxide having 50-100μm wider and 12μm deeper channel. A thin Anodic Aluminum Oxide (AAO) is fabricated by combing Mild anodization in oxalic and Hard anodization (H.A) in sulphuric acid. The dimension and an anisotropic etching were investigated by scanning electron microscopy (SEM). These channels are used for fluid flow on micro scale. These channels are connected with a nozzle like microchannel which can perform fluid injection function. This microinjector system is simply manufactured by embedded microchannel and there is no moving micro part inside nanoporous membrane. Fabrication process is mainly based on photolithography and wet chemical etching technique. Wet chemical etching takes place in 5 wt% of phosphoric acid solution. Dimensions and the shape of these microchannels depend solely on anodization and etching conditions. The presented system can be used into two way: in first way the fluid can be injected through nozzle like channel inside AAO and in second way the liquid can be purify and filtrate through nanoporous AAO membran. This study provides a low cost fabricated micro-nano combined structure which could be further used in different types of microfluidic devices and enable the device to perform the function of injection, filtration and purification.

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1. INTRODUCTION

Drug delivery is the process of controlling a pharmaceutical compound to acquire tonic effects in human and animals. The importance of drug delivery increases for the treatment of animals and human diseases. For this purpose several drug systems have been constructed like liposomes, microsphere, gel and many others [1]. For in vivo drug delivering applications smart particles have gaining much attention for researchers and these particles are biocapsule [2], microparticles [3] and nanoparticles [4]. Delivery of drugs at controlled rate, targeted delivery is other very significant method and pursued energetically. Microinjection is another well-established cellular technique that enables foreword of exogenous materials into cell in which needles inserted into the cell for injection [5]. Conventional LIGA process was applied to obtain high aspect ratio (HAR) solid microneedles array using biocompatible material such as PMMA. In LIGA process the plannar exposure method and layer by layer technique is used to achieve the 3D microstructure .The sharp tip microneedles was formed with deep x-rays lithography (DXRL) [6]. The device with hollow microneedles for ECG measurement provide comport home health care. The device consist two layer contain silicon die with hollow microneedles second layer is polymer die with pt electrode. The depth and diameter is around 100um which etched by using deep reactive ion etching (RIE) [7]. Current efforts to automate the microinjection technique by replacing operator with robotic but this has come at the expense of instrument and complexity [8]. Complementary efforts have to require using MEMS fabrication process to create devices which improve the reproducibility of injection and integration of numerous functions into a single chip enables simplifications relative to robotic microinjection instrumentation [9]. Since 1990 microfluidic devices has developed into versatile technology and different approaches for integrating membrane functionality in microfluidic chip is getting much attention [10]. Here in this research we purposed micro-nano structure has potential applications in the biology and medical related fields as fluidic delivery. In this paper nanoporous AAO membrane with injecting functionality is fabricated by photolithography and chemical etching technique. This simple and low cost microinjecture system could be integrated on single chip to obtain microfluidic based microinjector system.

2. EXPERIMENTAL AND METHOD

The fabrication of microinjector system was divided into five steps including membrane fabrication, deposition of adhesive layer, photolithography and chemical etching. The fabrication steps of AAO membrane with modulated pore diameter is shown schmetically in Fig 2.

2.1. Fabrication of Anodic Aluminum Oxide (AAO)membrane

Fabrication of micro injector system is based on Anodic Aluminum Oxide (AAO) nanoporous membrane formation on Aluminum (Al). In this work highly pure aluminum sheet (99.999%) of desire shape with thickness of 300um were used and work as working electrodes. Aluminum sheet were cleaned ultrasonically in acetone and DI water for 10 min and then sample were electrochemically polished in a 1:4 (volume ratio) mixture solution of ethanol and perchloric acid under vigorous stirring at 0°C. Electro polishing was done at 20V for 5 min to achieve mirror finished surface. Electro polishing eliminate the influence of oxide film on aluminum surface. After electro polishing the Al sheet were again cleaned ultrasonically in acetone and DI water for 10min.Anodization of surface finished aluminum sheet were performed under potentiostatic mode in 0.3M oxalic acid solution at 32V with vigorous magnetic stirring for 2 h and temperature were kept constant at 1°C. Porous aluminum sheet were immersed into mixture of chromic acid and (1.8 wt% and phosphoric acid 6wt% for 10 min to remove porous oxide layer completely. The resulting textured aluminum sheet was used for second anodization step. The second step of anodization comprises mild anodization (M.A) and hard anodization(H.A).M.A were carried at 32V in oxalic acid solution for 24 h at 0°C. Porous oxide layer were reanodized under potentiostatic condition in electrolyte solution of 0.3M H_2SO_4 at 38V for 15 min.

2.2. Fabrication of microinjector system

Schematically view of microinjector system is shown in Fig 1. This system was formed by selective etching of AAO which was fabricated by combining MA and H.A. A thin adhesive layer of silver was deposit on template by using plasma sputtering. Uniform thin layer of photoresist was obtain by applying positive photoresist on substrate with the help of spin coater at spin speed of 1000 to 3000rmp for10 second. Soft backing was carried at 90°C for 20 min and mask of desire texture was placed in contact with AAO sample. Now the sample was exposed to ultraviolet under 1000w ultraviolet lamp for 1min. After UV exposure hard backing at 135°C for 20 min was done and the exposed substrate were immersed in developer solution. Photoresist and thin layer of silver was removed. For selective etching of H.A layer, the sample was immersed in 5wt% phosphoric acid for 20min at 45°C.

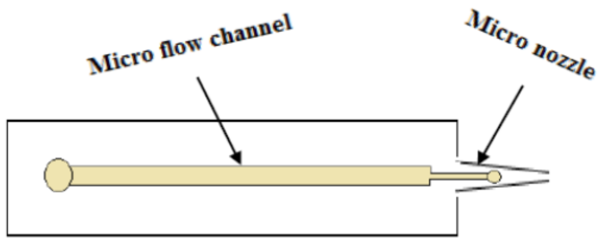

Fig. 1. *Schematic diagram of microinjector system*

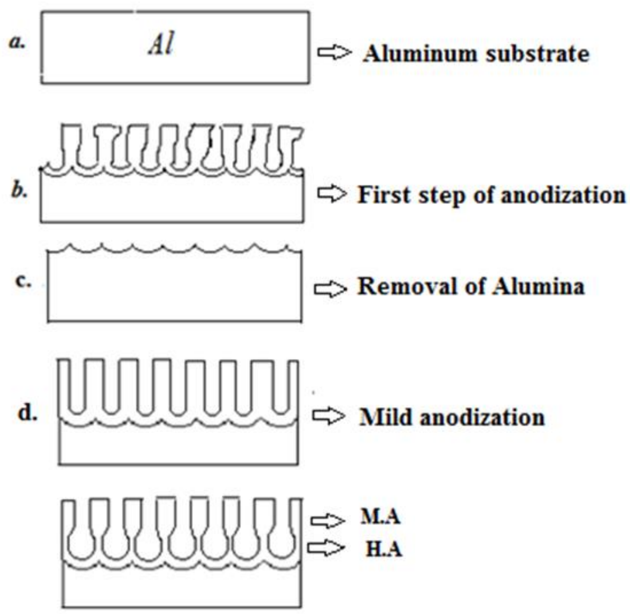

Fig.2. *Schematic representation of fabrication process of AAO membrane by combining M.A and H.A*

3. RESULT AND DISCUSSION

Scanning electron microscope was used for the analysis of AAO membrane and microchannel. Figure 3 shows the SEM analysis of microinjector system prepared in lab. AAO membrane have interpore distance of 80nm for both anodization (M.A and H.A) and thickness of membrane is about 5µm. Figure 3(a) shows upper surface morphology with uniform hexagonal nanoporous structure. The pore size and interpore distance of AAO membrane are highly depends on anodization parameters such as of electrolyte, potential difference and temperature. Fig 3(d) represents the cross sectional image of AAO membrane with well aligned regular and straight nanopores. The cross sectional view in Fig 3(b) shows chemical etching process which carried in 5wt% phosphoric solution for 5 min. The result shows that the etching process is only observed in H.A layer and no any effect of etching is detect in M.A. Microchannel with length of 50-100µm and 12µm in depth is shown in Fig 3(c).

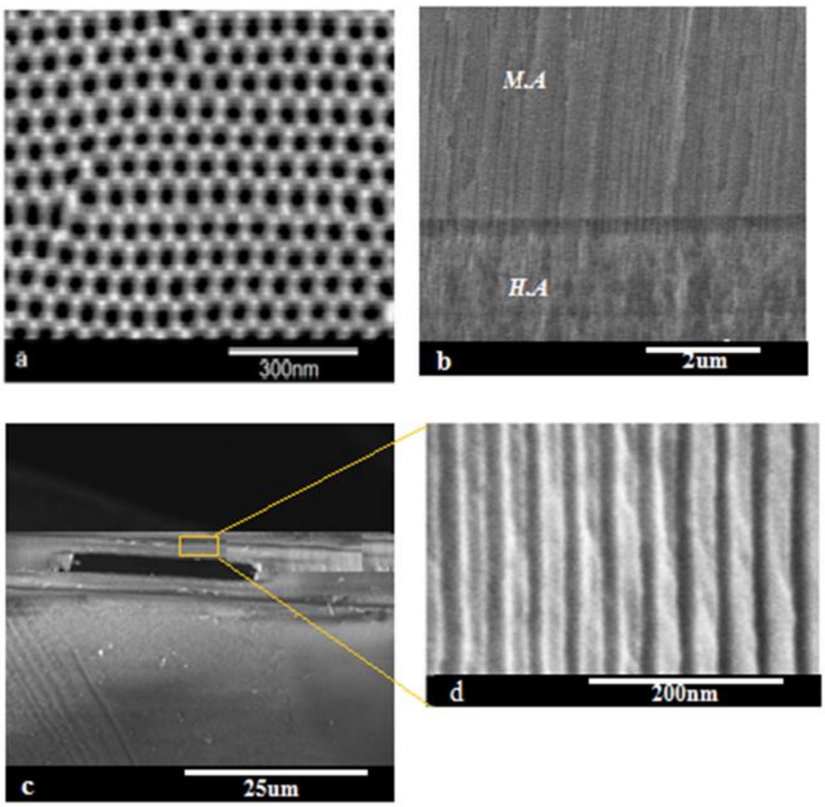

Fig. 3. *SEM images (a) top view of AAO membrane (b)cross-sectional view of Etching process in H.A (c) Cross-sectional view of Microchannel (d) cross-sectional view of upper layer*

Table 1. *The content of anions impurities in some popular electrolytes*

Electrolyte	H_2CrO_4	H_2PO_4	H_2C2O_4	H_2SO_4
Anion content (%)	0.1-0.3	6-8	2-3	10-13

The amount of anion impurities in H.A layer anodized in sulphuric is high so an anisotropic etching process was only observed in H.A layer. In our work a very attractive structure of AAO with combining M.A and H.A was used for the fabrication of microinjector system. Keeping inter pore distance similar it becomes possible to combine M.A and H.A by exchanging the electrolyte solution. The potential of 32V and 37V were applied for mild anodization in oxalic solution and hard anodization in sulphuric respectively. The porosity in oxalic based mild anodization is about 10% and in sulphuric based hard anodization it is about 30%. The interpore distance Din in H_2CO_3 and H_2SO_4 depend upon applied voltage with proportionality constant 2.5nmv^{-1} and 2nmv^{-1} for M.A and H.A. Up to our knowledge these microchannels was prepared first time inside AAO. The mechanical stability of membrane was very high and we can handle it without any damage. The microchannels with the range of 50-100μm in width and 10-15μm in deep was generated in prepared AAO sample by photolithography technique and wet etching was done in 5 wt% H_2PO_4. First the silver layer is coating on AAO membrane by plasma sputtering. The process was simplified by using positive photoresist and mask of desire microstructure. The given pattern on mask was transferred on photosensitive positive resist in UV light and by etching process the micropattern conveys in AAO membrane as show in Fig 3(c). As H.A segment was formed in H_2SO_4 in which the density of pore wall was very less and level of impurities was observed very high. This anion (SO_4^{-2}) impurity in H.A was about 88% higher as compared to the M.A segment [11]. The amount of anion impurities depends on the electrolyte and the condition of anodization. The anionic impurities content (%) for some popular electrolytes is given in table1.The density of pore walls was less and anion impurities were presented in large quantity so the chemical stability of H.A segment of AAO becomes very less in 5 wt% H_3PO_4 etchant. 50-100μm wide and 12μm deep microchannels were fabricated between uniform self-organized nanopores layer of M.A and Al substrate. Al substrate gives mechanical stabilty to microinector system. The depth of microchannel depends on thickness of H.A layer which can vary by changing anodization time. Etching time is another very important parameter so it was maintain up to20 min during etching process in phosphoric. If it increases from limited time the entire AAO membrane was etched.

4. CONCLUSIONS

In this study we reported very simple method for fabrication of micro and nano combined structure. Microchannels were formed inside anodic aluminum oxide by simple photolithography technique and wet etching technique. A thin film of anodic aluminum oxide (AAO) was fabricated by combining both the M.A anodization in oxalic acid and H.A in sulphuric acid. This novel type of microinjector system can be used to construct a microfluidic device by inegrating it on a chip. Our purposed stucture contains microchannels and can enable the devices to perform the function of injecting. This method will make fabrication easier, inexpensive, fast and simple and could be very useful in numerous applications of medical and biology.

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