Low cost Nano-membrane Fabrication and Electro-polishing System

Ajab Khan Kasi, Muhammad Waseem Ashraf, Jafar Khan Kasi, Shahzadi Tayyaba, and Nitin Afzulpurkar

***Abstract*—**This paper presents the development of low cost Nano membrane fabrication system. The system is specially designed for anodic aluminum oxide membrane. This system is capable to perform the processes such as anodization and electro-polishing. The designed machine was successfully tested for 'mild anodization' (MA) for 48 hours and 'hard anodization' (HA) for 3 hours at constant 0^{o}C. The system is digitally controlled and guided for temperature maintenance during anodization and electro-polishing. The total cost of the developed machine is 20 times less than the multi-cooling systems available in the market which are generally used for this purpose.

***Keywords*—**Anodic aluminum oxide, Nano-membrane, hard anodization, mild anodization, electro-polishing.

I. Introduction

NANO-POROUS membranes are growing interest in the field of science and technology. Various nano-porous membranes has been introduced, which can be classified as inorganic, organic, and composite. The nano-membranes can further classified according to its material such as polycarbonate (PC), polyethylene terephthalate (PET), polysulfone(PS), polyether sulfone (PES), nafion, alumina, titania, zirconia, and silica [1]. Synthetic nano-mambranes are fabricated using different fabrication techniques including ion track etching, lithography, phase separation, micromachining, powder sintering, sol-gel, and anodization [1], [2], [3]. Anodic aluminum oxide (AAO) membrane is one of the popular membrane which is fabricated using anodization process [2], [3], [4], [5]. The anodic aluminum oxide (AAO) membrane is popular for its regular and controllable pores for many applications including sensors, hemodialysis, protein separation, photovoltics, high density memories, and synthesis of functional nanostructures [2], [3], [4], [5], [6], [7]. Since the discovery of AAO membrane [8], various researchers have been focusing its fabrication under different constraints to characterize main parameter including pore size, pore depth, inter-pore distance, and thickness of membrane [9], [10], [11], [12], [13], [14], [15], [16]. Mostly, the fabrication of AAO is done with constant temperature at 0

A. K. Kasi (Phone: +66869043089; Fax: +66 02 524 5697; e.mail: Ajab.Khan.Kasi@ait.ac.th), M. W. Ashraf (Muhammad.Waseem.Ashraf @ait.ac.th), J. K. Kasi (Jafar.Khan.Kasi@ait.ac.th), S. Tayyaba (shahzadi_tayyaba @ait.ac.th), N. Afzupurkar (nitin@ait.ac.th), are with School of Engineering and Technology, Asian Institute of Technology (AIT), P.O. Box 4, Klong Luang, Pathumthani, 12120, Thailand.
.

to $1^{o}C$ because AAO gives better results at this low temperature [8], [9], [12], [15], [16], [17]. The major requirement for fabrication of AAO membrane is to maintain the low and constant temperature because the mild anodization (MA) is for long time process and large amount of heat is generated in hard anodization (HA) process [16].

In this paper the authors present the new design and development of low cost machine for nano-membrane fabrication. The developed machine has an automatic temperature control system. This machine is tested successfully for MA and HA processes and the temperature remained constant during anodization process. This machine can also be used to perform electro-polishing.

The magnetic stirrer used in the developed machine maintains the chemical concentration and temperature variation in the chemical bath. The chemical bath is made of glass. This bath is suitable for large variety of chemicals and can be easily removed or changed with another bath. The gasses formed in the cooling bath can be removed by the fume extracting facility. Due to this reason all the processes can be observed from the top with out any toxic effect.

Development of this machine is part of the project in which AAO membrane will be used for wearable hemodialysis device.

II. Design and Development

The block diagram of developed machine is given in Fig. 1. It consist of six main components such as cooling chamber, magnetic stirrer, digital temperature controller, electrode system, gas remover, and variable DC power supply.

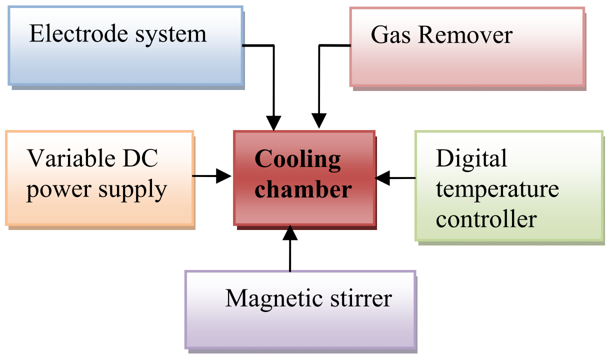

Fig. 1 Block diagram of low cost nano-membrane fabrication and electro-polishing machine.

A. Cooling Chamber

The cooling chamber consists of a cylindrical shape copper coil. A copper pipe with external diameter of 6.5 *mm* and length of 5 *m* is used to make the cylindrical coil. The coil is wounded on a stainless steel jar having 15.3 *cm* diameter and 18 *cm* height. The copper coil is insulated from outside using insulating jacket. The insulating jacket is finally covered by an aluminum sheet. The aluminum sheet is used as heat reflector. The copper coil acts as evaporator and is connected with compressor and condensing coil. The refrigerant used in developed system is R-134a. The cooling chamber was installed in a small size Hitachi refrigerator by replacing its compressor, condenser and evaporator because that was not producing the desired cooling. The condenser was mounted outside the refrigerator body. The new compressor used in system is 200*W*. A glass jar with external diameter of 15 *cm* and height of 26 *cm* is fitted in the stainless steel jar. The glass jar is easily removable from the top of refrigerator as shown in Fig. 2.

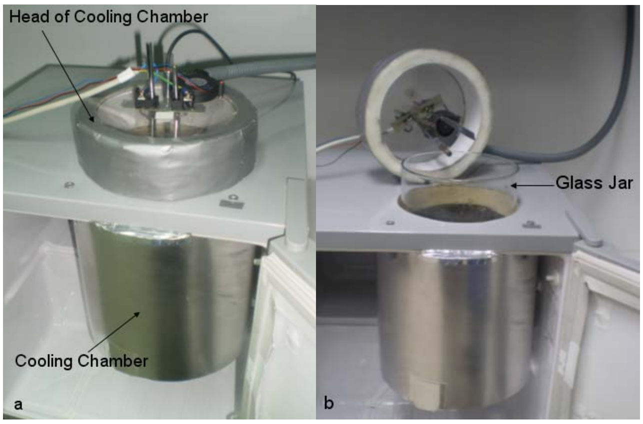

Fig. 2 (a) Cooling chamber with closed head (b) Cooling chamber with opened head for removing glass jar

B. Magnetic Stirrer

A magnetic stirrer was fitted at the bottom of stainless steel jar. The magnet used in magnetic stirrer is of an old CD room and the gear system used to move the magnet. The gear system is attached with 6*V* DC analog motor which is operated with variable DC power supply for different speeds.

C. Electrode System and Gas Remover

The head of cooling bath consists of transparent glass and insulating walls. A small size air blower was installed in the head to remove the gas formed during anodization or electro polishing process. Two copper electrodes which can be adjusted in horizontal and vertical direction are used as anode and cathode. A led sheet was attached at the top of cathode. The top of anode was made as a clip, that is used to hold aluminum sheet for membrane fabrication as shown in Fig. 3.

D. Digital Temperature Controller

The compressor is controlled by a digital circuit which monitors the temperature and switch on or off compressor. The digital system was taken from Sacco, microcontroller Model: S-616D, which is normally used in deep freezers. The digital circuit also controls the blower and a bulb on the top of cooling chamber. The digital system is used to control the operating temperature and change in temperature.

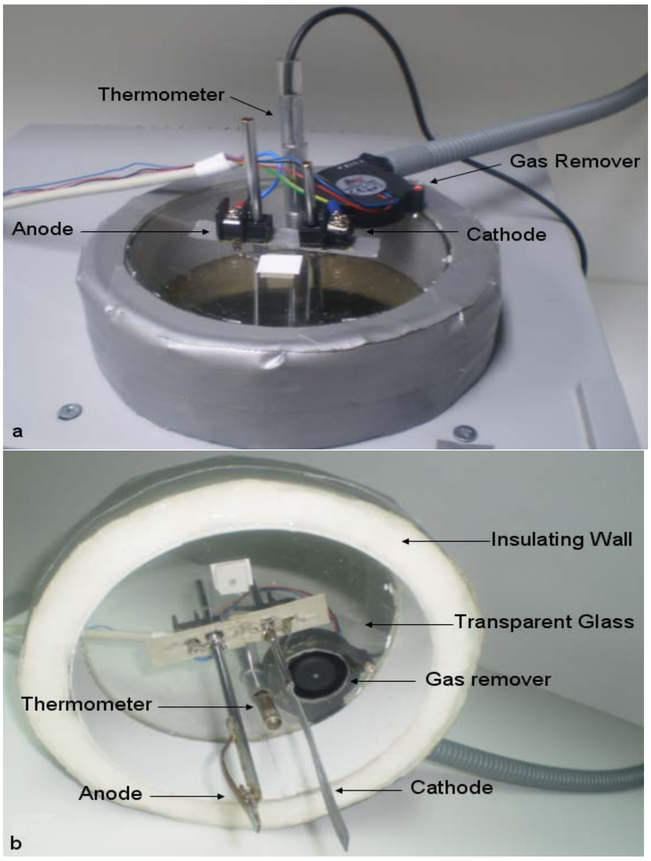

Fig. 3 Head of cooling chamber (a) External side (b) Internal side

E. Variable DC Power Supply

The developed system is attached with a variable power supply which was purchased from SwInstek. The power supply has two 60*V* variable voltage sources which can be operated in series to get 120*V* maximum or in parallel to get 60*V* maximum or independently variable voltages. The voltage sources are operated in series for HA, independently for MA and in parallel for electro-polishing. It also provides 5*V* for gas remover. The magnetic stirrer was attached with one of the variable voltage sources for MA and with constant 5*V* voltage source for HA. The power supply can support maximum of 3A current. An internal voltmeter and ammeter are available in power supply to monitor voltage and current. The complete setup of developed system is shown in Fig. 4.

Fig. 4 Complete setup of low cost nano-membrane and electro-polishing machine

III. Results and Discussion

The developed system has been tested for the MA, HA, and electro polishing. For the MA, system was operated continuously for 48 hours. It was operated 3 hours continuously for HA. The sample size was 2 cm^2 and maximum current density recorded was 250 mA/cm^2. In both anodization processes temperature was maintained at 0^oC with the variation of $\pm 1^oC$. The chemical bath was filled with 3000 ml of 0.3 M oxalic acid. The anodization potential was applied after getting 0^oC. During MA the magnetic stirrer's motor was burnt after 46 hours. Then the gear system was replaced by servomotor and operated for 48 hours successfully. For electro-polishing the current density was maintained at 300 mA/cm^2 for 3 minutes. The sample size was 2 cm^2. The temperature during electro-polishing was maintained at 0^oC. During each process the air blower at the head was continuously removing the gasses formed inside the cooling bath.

IV. Conclusion

In this paper a new design of nano-membrane fabrication and electro-polishing machine is presented. This system can be made in any small lab with very low cost. The machine utilizes common and low cost components. Most of these components are available at any refrigerator repairing shop. The developed machine is user friendly even that it can be used at school and college level to fabricate AAO nano-membrane. The chemical bath (glass jar) used in the machine is easily removable, so the used chemical can be restored. The electrode system is easily adjustable in X and Y direction. The iodization or polishing process can be done even with less amount of chemical. The system is automatic and does not need user to take care for the anodization process for long time. The present work provides the useful information to develop the low cost biomedical system.

References

[1] [1] S. P. Adiga, C. Jin, L. A. Curtiss, N.A. Monteire-Riviere, R. J. Narayan, " Nanoporous membranes for medical and biological applications," *WIREs Nanomedicine and Nanobiotechnology*, vol. 1, pp568-581, 2009.

[2] [2] G. Gorokh, A. Mozalev, D. Solovei, V. Khatko, E. Llobet, X. correig, " Anodic formation of low aspect ratio porous alumina film for metal oxide sensor application," *ELSEVIER, Electrochimica Acta*, pp1771-1780, 2006.

[3] A. C. Attaluri, Z. Huang, A. Belwalkar, W. V. Geertruyden, D. Gao, W. Misiolek, " Evaluation of Nano-Porous Alumina Membrane for hemodialysis application," *ASAIO J. KidneySupport*, , vol 55, pp217-223, 2009.

[4] W. Shi, Y. Shen, D. Ge, M. Xue, H. Cao, S. Huang, J. Wang, G. Zhang, F. Zhang, " Functionalized anodic aluminum oxide (AAO) membrane for affinity protein separation," *ELSEVIER. Junrnal of Membrane Science*, 325, pp801-808, 2008.

[5] Z. Fan, H. Razavi, J. Do, A. Moriwaki, O. Ergen, Y. Chueh, P. L. Leu, J. C. Ho, T. Takashi, L. A. Reichertz, S. Neale, K. Yu, M. Wu, J. W. Ager, A. Javey, " Three dinmensional nanopillar-array photovoltics on low-cost and flexible substrates," *Nature, Materials*, pp648-653, July 2009.

[6] S. Z. Chu, S. Inoue, K. Kurashima, "Fabrication of integrated arrays of ultrahigh density magnetic nanowires on glass by anodization and electrodeposition," *ELSEVIER,* Electrochimica Acta, 51, pp820-826, 2005.

[7] W. Lee, R. Scholz, K. Nielsch, U. Gosele, "A template-based electrochemical method for the synthesis of multisegmented metallic nanotubes," *Angew. Chem. Int*, , 44, pp 6050–6054, 2005

[8] H. Masuda, K. Fukuda, "Ordered metal nanohole arrays made by a two-step replication of honeycomb structures of anodic alumina," *Science* 268, pp1466–1468,1995.

[9] O. Jessenky, F. Muller, U. Gosele, " Self-organized formation of hexagonal pore arrays in anodic alumina," *Applied Physics Letters*, vol. 10, pp1173-1175, number 1998.

[10] H. Masuda, F. Hasegwa, " self ordering of cell arrangement of anodic porous alumina formed in sulfuric acid solution," *J. Electrochem.Soc*, vol.144, may 1997.

[11] Y. Zhao, M Chen, Y. Zhang, T. Xu, W. Liu, " a facile approach to formation of through-hole porous anodic aluminum oxide film," *ELSEVIER, material letters*, vol. 59, pp40-43, 2005.

[12] M. Ghorbani, F. Nasipouri, A. I. Zad, A. Saedi, " on the grouth sequence of highly ordered nanoporous anodic aluminum oxide," *ELSEVIER, Materials and Design*, vol.27, pp983-988, 2006.

[13] H. Asoh, S. Ono, T. Hirose, M. Nakao, H. Masuda, "growth of anodic porous alumina with square cells," *PERGAMON, Electrochimica Acta*, vol.48, pp3171-3174, 2003.

[14] C.U. Yu, C. C. Hu, A. Bai, Y.F. Yang, "pore size dependence of AAO films on surface roughness of Al-1050 sheets controlled by electropolishing coupled with fractional factorial design," *ELSEVIER, Surface and Coatings technology*, vol.201, pp7259-7265, 2007.

[15] W. Lee, R. Ji, U. Gosele, K. Nielsch, "fast fabrication of long-range ordered porous alumina membranes by hard anodization," *Nature Materials*, vol.5, pp741-747, September 2006.

[16] A. Belwalkar, E. Grasing, W.V. Geertruyden, Z. Huang, W. Z. Misiolek, "effect of processing parameters on pore structure and thickness of anodic aluminum oxide(AAO) tabular membranes," *ELSEVIER, Journa of membrane Science*, vol.319, pp192-198, 2008

[17] N. Tasaltin, S.Ozturk, H. Yuzer, Z. Z. Ozturk, " simple fabrication of highly ordered AAO nanotubes," *Juournal of Optoelectronics and Biomedical Materials,* vol.1, pp79-84, March 2009.