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Design and Fabrication of Uniform Aluminium Pores for Planar Microfiltration System

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ABSTRACT

This paper presents a design and fabrication of a fluid filtration system for separating biological molecules through uniform pores in silicon-based planar microfiltration system. The study is aimed to find a simple process for the fabrication of a microfiltration system. The uniform pores are made of aluminium (Al) material deposited on a silicon substrate, whereas the fluid chamber is fabricated using deep reactive ion etching (DRIE) process of the substrate by utilising SF_6 *as the reaction gas. Afterwards, the prepared filtration membrane is directly bonded with PDMS based microfluidic part using a corona discharge. Tygon S3™ tubings are used as the fluid interconnection between the PDMS sealing layer and the silicon-based Al membrane in order to make up complete silicon-based planar microfiltration system with uniform pores for separating molecules in fluid based on molecule size. The silicon-based planar microfiltration system consists of assembled silicon-based Al membrane, PDMS sealing layer and Tygon tubes. From the initial test conducted, no leaking is detected from the PDMS sealing, and the initial test using DI water shows that the DI water can flow through the uniform filtration pores. The silicon-based planar microfiltration system with uniform pores has the potential to be used in the filtration of biological fluid in separating molecules based on size.*

Keywords: Etching, MEMS, Planar Microfiltration System, Silicon, S1813 Photoresist, Uniform Pore.

1. INTRODUCTION

Currently, the silicon substrate is amongst all materials widely considered in research for the fabrication of filtration membrane, as reported in [1]–[4]. One of the applications possible for these new filtration membranes is in microelectromechanical systems (MEMS) research [5] for the replacement or alternative material to the conventional blood filtration membrane in a dialysis machine [4], [6]. The filtration membrane in dialysis machine works in size-based selective removal of fluid that passes through the openings or pores in the filtration membrane [7]–[10]. The filtration membrane in dialysis machine works to filter out waste molecules in the blood where molecules that retained in the blood will be pumped back into blood capillary of people with kidney failure, and separate them from molecules that are smaller in size compared to the size of the filtration membrane pores [7], [11]–[13]. Therefore, the filtration membrane is one of the vital elements in a dialysis machine in providing improved health care to people with failure kidney.

To obtain a filtration membrane that has better molecules separation capability, currently, several characteristics of the silicon substrate have been studied. One of them is the mechanical strength characteristic where its capability of withstanding applied pressure caused by fluid entering and flowing out through its filtration pores is simulated using COMSOL Multiphysics

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software [2], [7], [14]. The simulation work is conducted at 10 until 55 mmHg applied pressure to mimic the pressure faced by the filtration membrane in native kidney caused by blood flow [2][7]. The effect of the fabricated pores' shapes on the silicon substrate is also explored, as explained in the previous report [2].

Silicon-based filtration membrane structures have also been studied such as The Kidney Project that is led by Roy and Fissel team from the University of California, San Francisco (UCSF), and Vanderbilt University in Nashville, Tennessee, for possible application for wearable or implantable artificial kidney [10], [15]–[19]. In the year 2006, Humes and team highlighted the importance of the silicon membrane as the alternative to the conventional dialysis membranes [20]. Following continuous researches, vertical structures of silicon-based microfiltration system have also been reported previously [12], [21]. In vertical structures, the fluidic flow is perpendicular to the filtration membrane structures so that only molecules with a size smaller than the membrane's pore size pass through the filtration pores, while larger ones are blocked and retained in the fluid chamber. These systems with the vertical structures consist of an inlet port on the top side of the membrane, the filtration membrane itself and the fluid exit at the bottom. These two different discussed fabrication methods; electrochemical etching and deep reactive ion etching (DRIE) produce discrete filtration pore structures.

Creation of filtration pores in the silicon membrane using electrochemical etching method produced ununiform pore size on the silicon substrate [21], [22]. Thus, this may reduce the capability of molecules separation when the filtration mechanism used is mainly based on molecule size. On the other hand, the fabrication method using DRIE produces a uniform structure [12], which is better for molecule separation capability. Nevertheless, the fabrication method outlined in [12] requires several lithography processes and etching processes at both sides of the substrate's surface, making it complicated and thus induced higher fabrication [23] of a microfiltration system that has uniform filtration pores. Using a planar system could simplify the integration processes of membranes into fluidic devices using only one surface of the silicon substrate. Hence, no need to conduct a bulk micromachining process to fabricate a fluid microfiltration system [24].

Thus, to tackle this problem, a silicon-based planar microfiltration system with uniform pore arrays is aimed for better molecule separation capability and reduced fabrication processes. By using a planar microfiltration structure, the requirement to undergo a tedious bulk micromachining process to obtain a filtration membrane could be omitted [23], [25]. In this paper, the idea of design and fabrication of uniform pores for the planar microfiltration system on a silicon substrate is further elaborated in the following sections.

2. DESIGN OF SILICON-BASED PLANAR MICROFILTRATION SYSTEM

The complete schematic for the design of uniform pores for silicon-based planar microfiltration system is shown in Figure 1. The system comprises of the Al pores on a silicon substrate, silicon-based trapping chamber, a fluid inlet, a retained fluid exit, a filtered fluid exit, and a PDMS sealing layer. The filtration membrane is made of Al and silicon dioxide layers having a thickness of 120 nm and 6 μm, respectively. The filtration membrane layers also have the function as the mask for the etching process. The silicon-based trapping chamber is produced from the silicon etching process.

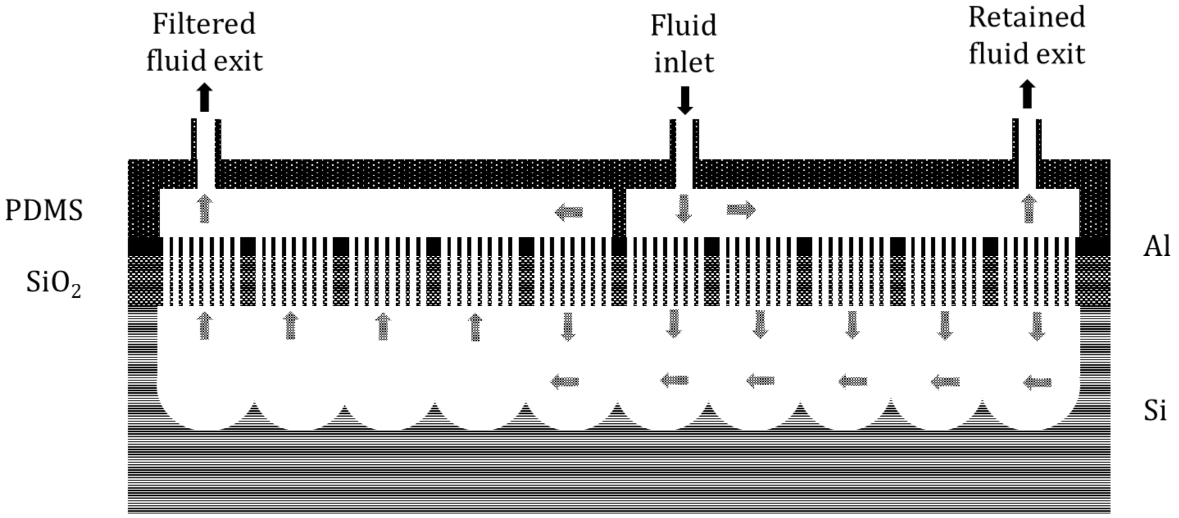


Figure 1. Schematic of Al uniform micropores for silicon-based planar microfiltration system (drawing is not to scale).

The fabrication processes to obtain a uniform pore array on the silicon substrate is further elaborated in section: Fabrication of Uniform Micropores on Silicon Substrate. Once fabrication of Al uniform pores on the silicon substrate and the removal of oxide sacrificial layer underneath the Al layer are completed, the silicon substrate is subjected to ion etching for formation of a fluid trapping chamber. Then, it is cleaned from contaminants before assembled with the PDMS sealing layer and Tygon tubings in order to make up complete silicon-based planar microfiltration system with uniform Al filtration pores.

The Al etched pore structure on the silicon substrate acts as the primary fluid filtration part where the openings will selectively block molecules larger than its opening size from entering the gap. Oxide sacrificial layer underneath the etched Al micropore is patterned using the Al micropore as the mask using BOE solution which is injected through the Al uniform micropore openings. This oxide layer works as the holder to the Al micropore thin layer and at the same time as the supporting mask for subsequent ion etching process of the silicon substrate beneath the oxide layer that will then act as the trapping chamber for molecules smaller than the uniform pore size to be collected later through Tygon tube exit.

The PDMS sealing layer is made up of a mixture of PDMS oligomer and the crosslinking agent. The PDMS oligomer and crosslinking agent is mixed in a ratio of 10:1 in weight ratio. The mixture is thoroughly mixed using a disposable plastic pipette tip for two minutes until the mixture condition is full of air bubbles. A degassing process is then performed to remove those air bubbles under vacuum condition completely. After the degassing process is completed, the PDMS mixture is poured into a Perspex mould to obtain the desired sealing shape with fluid inlet, retained fluid exit and filtered fluid exit connected to Tygon tubes [12], [26]. Then, the Perspex mould containing the PDMS mixture is put into the oven and baked at 65°C for one hour to crosslink the polymer. The fully cured PDMS sealing layer is then peeled from the Perspex mould before it is bonded to the silicon substrate.

As shown in Figure 1, a PDMS sealing layer is bonded on top of the etched Al uniform micropore surface to form a complete silicon-based planar microfiltration system. Another PDMS sealing layer may also be bonded to the bottom of the silicon-based planar microfiltration system to protect the silicon substrate from outside pressure that may cause harm to the microfiltration system. The PDMS sealing layer is beforehand treated with a corona discharge to improve its adhesion to the silicon substrate [26].

Test fluid will be injected into the planar microfiltration system through fluid inlet made up from Tygon tube and PDMS sealing's opening. Molecules in fluid with a size that is smaller than the silicon-based Al uniform micropore will flow through the openings and travels through the trapping chamber in the silicon substrate etched by the ion etching process, and then exits the opening in another side of the etched silicon substrate area through connected Tygon tube acts as the filtered fluid exit. Fluid collected from the filtered fluid exit has a molecule size smaller than the uniform Al micropore openings. The fluid of molecule size larger than the silicon-based Al uniform micropores are accumulated in the retained fluid chamber and is collected through the provided retained fluid exit, as shown in **Figure 1**.

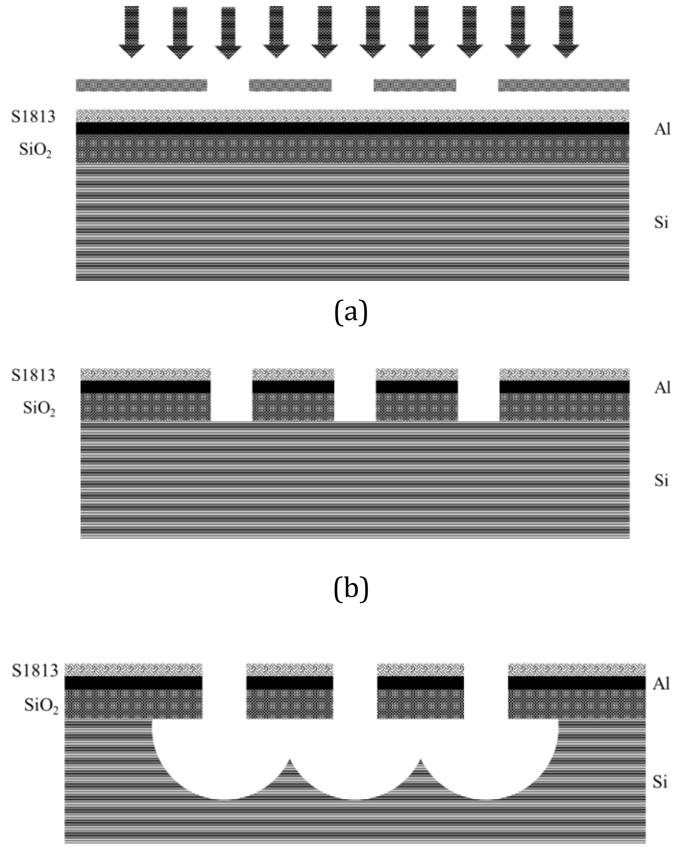
3. FABRICATION OF UNIFORM MICROPORES ON SILICON SUBSTRATE

Prior to fabrication of the uniform pores, its shape is first designed in L-Edit software before the designed pattern is transferred into a Chrome mask. The diameter of the designed uniform micropore is set to 1.8 µm, while the pore-to-pore gap is set to 5 µm. The Chrome mask is then used to transfer the designed uniform pore pattern onto the photoresist layer on the spin-coated photoresist layer during the photolithography process.

Figure 2 shows the schematic of the fabrication process of the microfiltration system. Uniform pores are fabricated on a silicon substrate using several fabrication steps, including photolithography process by spin-coating of photoresist layer for pattern transfer from chrome mask onto the photoresist layer and followed by pattern development process and ion etching process.

For the fabrication of Al uniform pores on the silicon substrate, a <100>-oriented silicon wafer with a silicon oxide layer may be used as the starting substrate. The silicon wafer is then diced into a size of 2 cm x 2 cm before subjected to a standard cleaning procedure using acetone and IPA ultrasonic bath for five minutes each and later blow-dried with nitrogen gas. Al layer with a predetermined thickness is then deposited using either thermal evaporator or a sputtering method. Then, the substrate is sent to the photolithography process to transfer the designed uniform pore pattern on Chrome mask onto the spin-coated photoresist layer. After the uniform micropore pattern is fully developed through the photoresist layer, the uniform micropore on Al deposited layer is then patterned using Al etchant. The oxide sacrificial layer underneath the etched Al uniform micropore is later removed using BOE solution. Figure 2 demonstrates a schematic of the main fabrication process flow to produce micromachined uniform Al micropore arrays on the prepared silicon substrate. Parameters for photoresist spin coating and optimum UV exposure processes are discussed in [27].

S1813 positive photoresist is first spin-coated at the decided 3000 rpm to form a photolithography mask layer to create a uniform micropore pattern on the Al layer. From our previous study [28], this 3000 rpm spin-coat speed is the optimal speed to obtain a constant photoresist mask layer with a thickness of 1.35 µm, which is suitable to act as a mask layer for 1.8 µm sized micropore arrays patterning. Following the photoresist spin-coating process, photoresist soft bake process is conducted at 115°C for 90 s on a hot plate. Later, the prepared substrate with spin-coated S1813 photoresist layer is ready for uniform micropore pattern transfer by photolithography using Midas mask aligner available in IMEN Cleanroom to transfer pattern in prepared Chrome mask. The substrate is exposed to UV light for 15 s, and then the uniform micropore pattern is developed in undiluted AZ726MIF developer.



(a)

(b)

(c)

Figure 2. Cross-section view of the fabrication process of uniform micropores on silicon substrate: (a) Al deposition and pattern transfer onto S1813 photoresist layer by photolithography; (b) Al etching and oxide layer removal; (c) fluid trapping chamber etching on silicon substrate by DRIE.

Once the S1813 photoresist layer is fully developed with uniform micropore pattern, the substrate is rinsed with DI water and blow-dried with nitrogen gas before it is subjected to post bake process at 120 °C for 60 s to harden the photoresist before ready for subsequent Al etching process. From observation using an optical microscope as shown in Figure 3, it is observed that the fully developed uniform micropore pattern has a size of 1.8 µm and pore-to-pore gap of 5.0 µm which is the same size as of initial design size. BOE solution is then injected through the openings in the Al uniform micropore layer to remove the oxide sacrificial layer beneath the etched Al uniform micropore structure. Obtained Al uniform micropore and oxide pattern is then used as the mask for DRIE process in order to etch silicon substrate underneath.

The fluid trapping chamber on the silicon substrate could be etched using Oxford Instrument Plasmalab 100 System, utilising SF$_6$ as the etchant. RF and ICP forward power are set at 80 W and 600 W, respectively. The process is set up at a cryogenic temperature of -115±5 °C, an SF$_6$ flow rate of 60 sccm and O$_2$ flow rate of 13.5 sccm. Large lateral etching in the DRIE process will cause the size of silicon substrate etching is larger than the pore size in Al and oxide layers and eventually those etched pores are connected to each adjacent pore, producing a trapping chamber for fluid transport. The large lateral etch will happen due to incomplete etching and oxidation cycles in the DRIE process. The duration of DRIE process is controlled so that desired etch depth which will be the fluid trapping chamber height is obtained [12] to form a chamber for filtered fluid molecules which is smaller than the uniform size micropore, which will be collected using a Tygon tube and PDMS sealing layer assembly in the system.

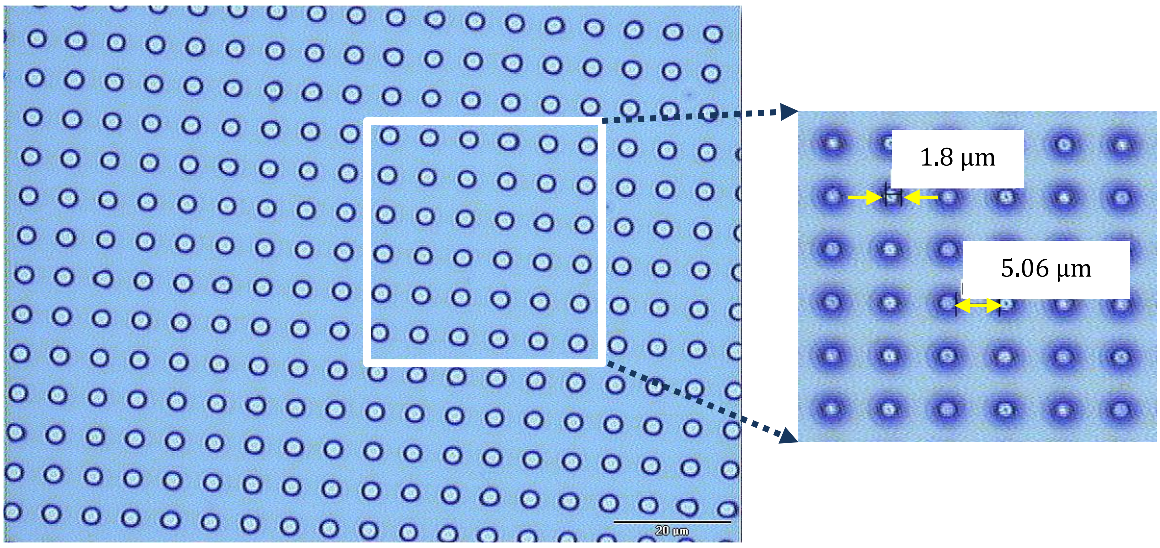


Figure 3. Fully developed uniform micropore pattern.

As shown in Figure 4 from FESEM observation after DRIE process, the size of the DRIE etched micropore is the same as the designed size of 1.8 µm, and also is the same size as of the fully developed uniform micropore pattern in the pattern development process. Meanwhile, **Figure 5** shows the SEM cross-section of the fabricated Al uniform pore filtration membrane with a silicon-based fluid passageway chamber is formed underneath [12]. This show that by using the optimised parameters from our previous study, the final DRIE etched micropore with a size of 1.8 µm could be realised, which is the same size as of initial design uniform micropore size.

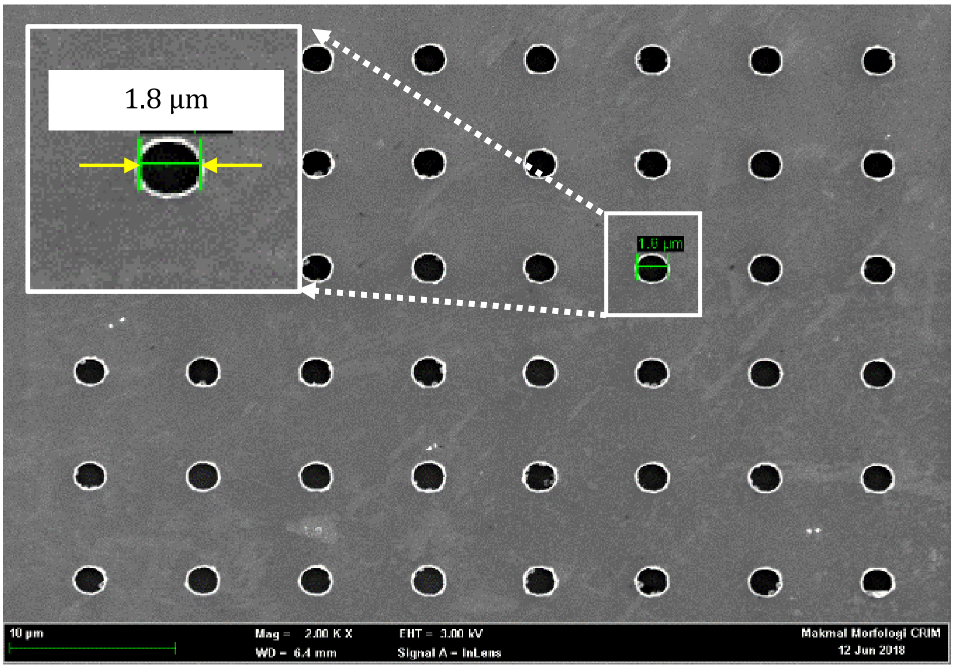


Figure 4. DRIE etched micropore.

PDMS sealing layer which is prepared in advance and Tygon tube is then assembled with the fabricated Al uniform pore filtration membrane using corona discharge to form complete silicon-based planar microfiltration system. In a condition where the uniform micropores are made from silicon instead of Al uniform pore explained here, the size of the uniform silicon micropore may further be reduced using thermal oxidation process to achieve nano range size pores for the silicon-based planar microfiltration system [28].

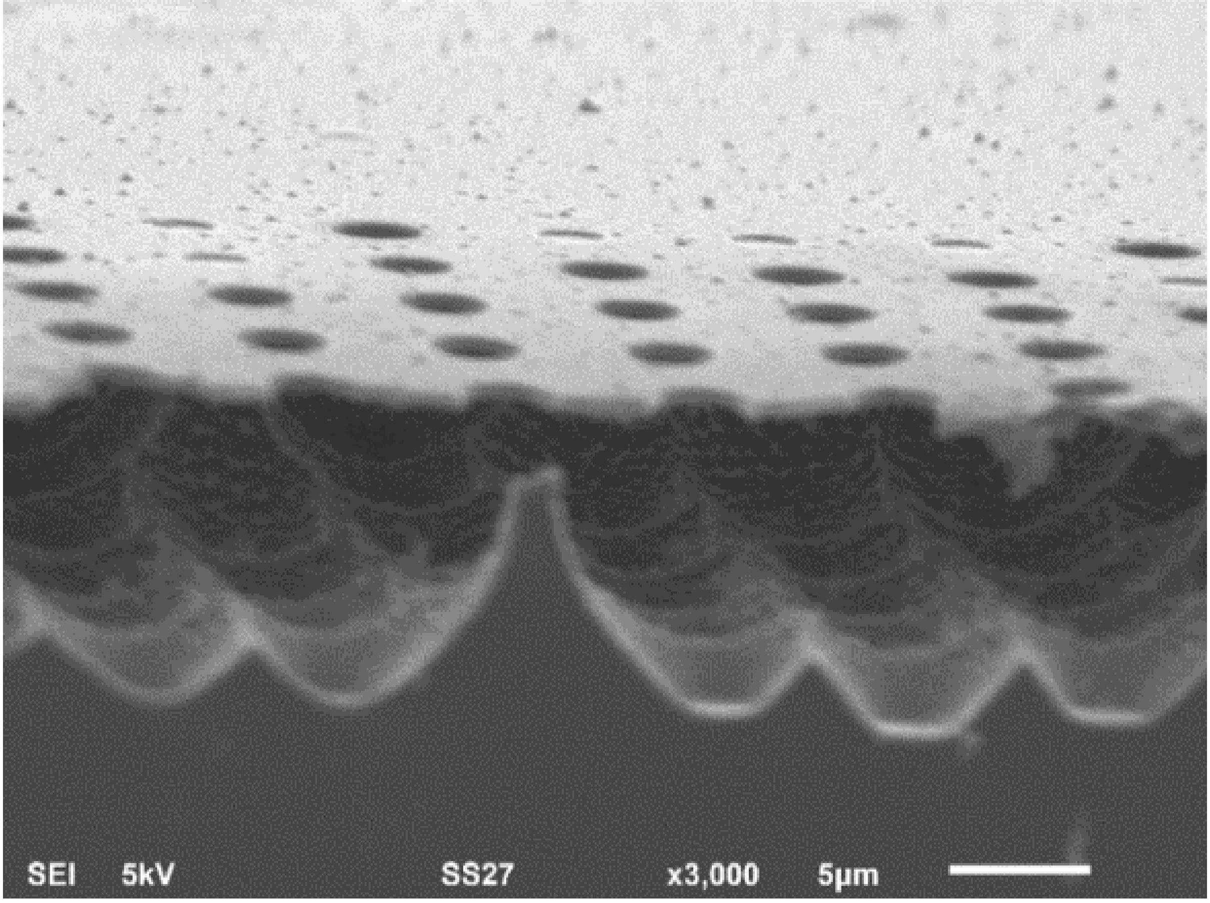


Figure 5. SEM cross-section of Al uniform pore filtration membrane with underneath fluid passageway chamber.

4. CONCLUSION

This paper has discussed a planar silicon-based microfiltration system that comprises of a chamber working as the fluid trapping chamber, a fluid inlet, and fluid exits on a planar surface. The microfiltration system is fabricated on a single side of the silicon substrate where molecules with a size that is smaller than the uniform micropores flow through the openings in the filtration membrane into the silicon-based chambers. Using optimised parameters in preparing the uniform micropores, the desired 1.8 μm could be achieved. PDMS layer and Tygon tubes complete the silicon-based planar microfiltration system by providing openings for fluid inlet, retained fluid exit and filtered fluid exit. The study benefits in providing a simple alternative way in fabricating the microfiltration system compared to a vertical type system that reduces the complexity of the bulk micromachining process step. By manipulating parameters in the DRIE process to get large lateral etching, the etched silicon chamber as the fluid trapping chamber is obtained to realise a silicon-based planar microfiltration system. The larger the lateral etch in the DRIE process, the better the structure of the fluid trapping chamber will be.

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