

3 Design and Fabrication of Bubble Separator

3.1 Bubble Separator Design

The main idea in the design to achieve bubble removal is by capillary force in the bubble, caused by coating hydrophobic/hydrophilic materials on proper parts of the microstructures. Furthermore, the separator for a portable μ DMFC should be able to exhaust bubbles in more than one direction. Therefore, multi-directional (x,z) design could comply with the mobility of portable μ DMFC. In addition, fuel recycle capability is another requirement for an ideal μ DMFC separator.

Fig 3.1 is the 3-D sketch of a test sample of bubble separator. The array of hydrophobic microholes on the plate is to exhaust CO₂ bubbles in the direction perpendicular to the plate. The alternating hydrophobic gas microchannels and hydrophilic liquid microchannels are used to exhaust bubbles and recycle the fuel liquid in the lateral direction. In the test samples, the microholes have a diameter of 100 μ m and a depth of 250 μ m. The liquid microchannels have a width of 25 μ m and a depth of 250 μ m and the gas microchannels have a

width of $100\text{ }\mu\text{m}$ and a depth of $250\text{ }\mu\text{m}$. The hydrophilic surface was made with SiO_2 , while the hydrophobic surface was made with a Teflon® coating. With the design including the array of microholes along with the microchannels, multi-directional bubble exhausting capability is achieved. The test sample, as shown in Fig. 3.1 will be covered with a glass plate so that experimental observation can be made.

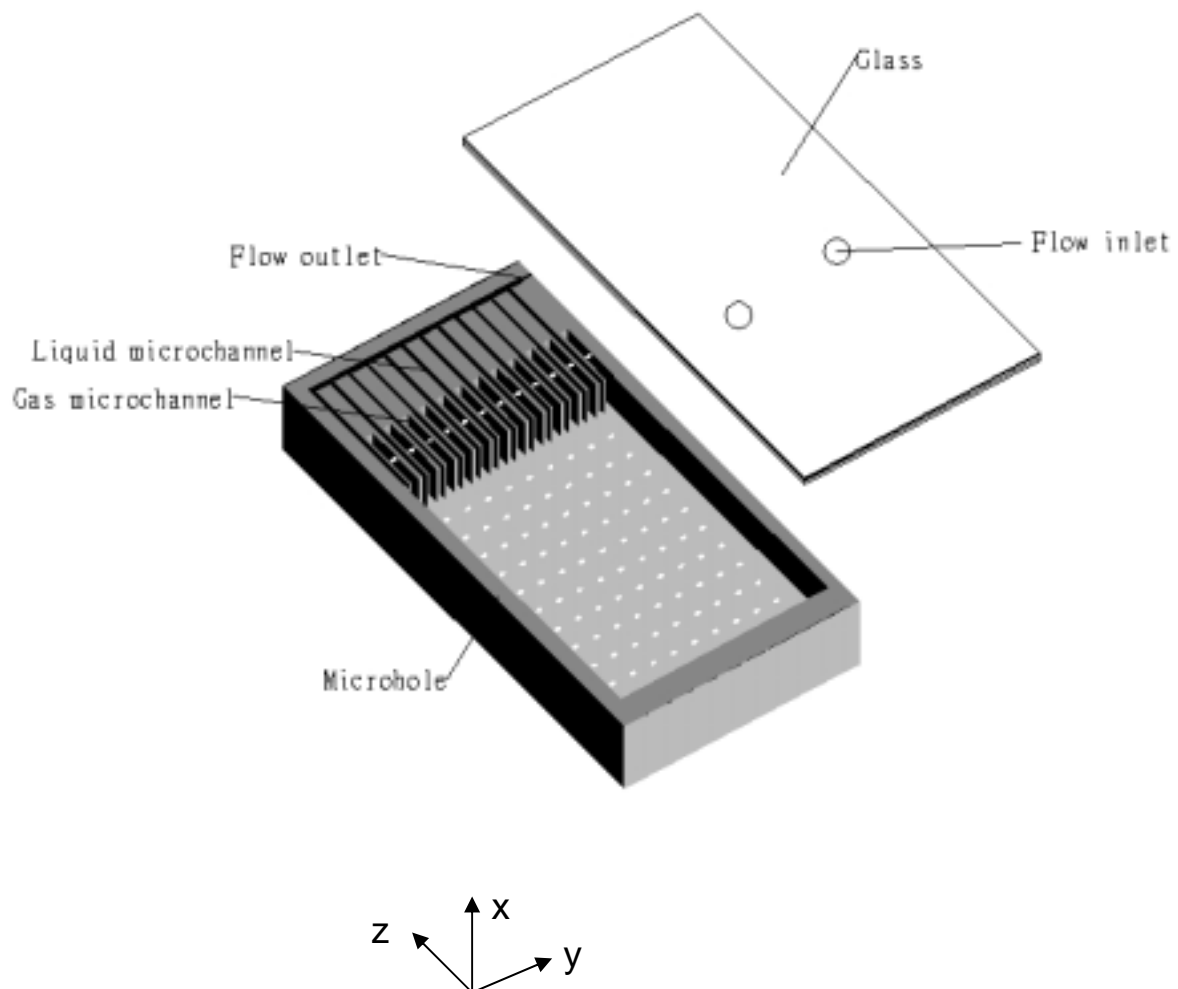


Fig. 3.1 The separator design for μDMFC

3.2 Fabrication Process

The bubble separator for μ DMFC is fabricated by MEMS technology. The detailed fabrication process is described in Fig. 3.2. A wafer (110) is first cleaned by using the standard RCA clean process before depositing Si_3N_4 (2000-Å thick) on its both sides by low pressure chemical vapor deposition (LPCVD), whose recipe is shown in Table 3.1. After the photolithography with mask #1, the unwanted Si_3N_4 will be removed by reactive ion etching (RIE), whose recipe is shown in Table 3.2. Then, the deep reactive ion etching (DRIE), will be used to create the microholes before the PR-removing process. In order to generate 250 μm deep microholes, thick photoresist AZ4620 with 3 μm is coated.

After photolithography with mask #2 on the reversed side of the already dry-etched wafer, the unwanted Si_3N_4 are removed by RIE, whose recipe is shown in Table 3.2, before the PR-removing process. The wafer with remaining Si_3N_4 is etched by the KOH solution to generate microchannels with a length of 10mm, and a depth of 250 μm . The fuel chamber has a total area of 3X3 cm^2 . During the wet etching process, a fixture is used to protect the shape of holes. By now, the structure of the separator has been fabricated. Subsequently, the surface characteristics will be changed by hydrophilic or hydrophobic materials.

Native oxide rendering the etched wafer hydrophilic is induced on the entire sample by immersing it into an acid solution ($\text{H}_2\text{SO}_4+\text{H}_2\text{O}_2$). Afterwards, to make the microholes hydrophobic, 1% Teflon® solution will be sprayed on the back surface of the plate. The inner surface of the microholes will be

wetted by the solution as a result of capillary attraction. Since the gas microchannels are hydrophilic at this time, the 1% Teflon® solution was sucked from the holes in the wider gas microchannels and spread over the gas microchannels by the capillary effect. Afterwards, the sample was heated on a hot plate at 135 °C for 10 minutes. After the heating, the thinner FC75 solvent evaporated to leave hydrophobic Teflon® on the channel surface. The hydrophobic regions include the inner surfaces of the gas microchannels, the inner surface of the microholes, and the whole back of the sample plate.

The finished sample was then covered with glass for performance test.



Table 3.1 : LPCVD recipe of Silicon Nitride

Gas Flow Rate		Pressure	Temperature	Thickness	Process Time
NH ₃ (sccm)	SiHCl (sccm)	mTorr	(°C)	(Å)	(min)
105	35	140	800	2000	32

Table 3.2 RIE recipe of LPCVD Nitride

Gas Flow Rate		Pressure	RF Power	Etching Time
SF ₆ (sccm)	CHF ₃ (sccm)	mTorr	(W)	(sec)
30	10	50	100	90

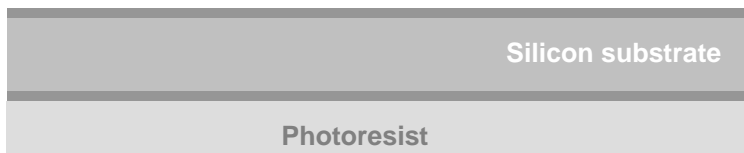


Silicon substrate



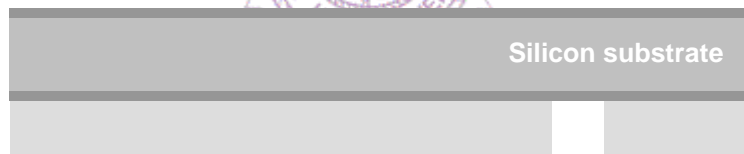
Silicon substrate

Deposit Si_3N_4 on double-sides

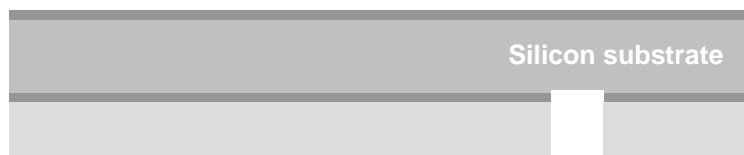


Silicon substrate

Photoresist



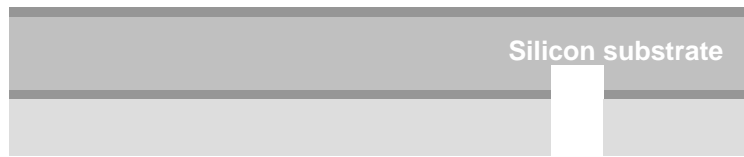
Silicon substrate



Silicon substrate

Using RIE to open a window

Fig. 3.2 Fabrication Process (continued)



Using DRIE to create micro-holes

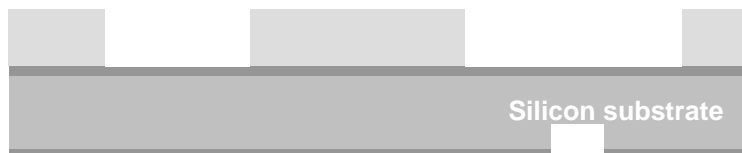
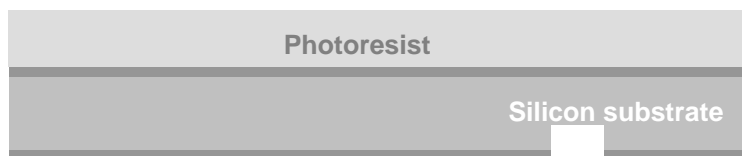


Fig. 3.2 Fabrication Process (continued)



Wet-etching to create microchannels and chamber



Immersing in $\text{H}_2\text{O}_2 + \text{H}_2\text{SO}_4$ (1:3) solution to induce native SiO_2



Coating Teflon® on microholes and gas microchannels

Fig. 3.2 Fabrication Process