CHAPTER 3

DESIGN, FABRICATION AND EXPERIMENTS

3.1 Design of Multi-Sectional Flow Fields

The cathode flow field design is aimed to feature uniform distribution, no flooding, low-pressure drop, and water recyclability. The flow field can be divided into three sections: (i) parallel channels for air flow and water collection, (ii) buffer channels, and (iii) water recycling channels and reservoir. Detailed description is as follows:

(I) Parallel Channels: This section is designed for uniform reactant distribution and water collection. The whole channel surface is made hydrophilic for stronger capillary attraction of water. It is expected that most of the water will be collected at the corners and walls of the channels. The rectangular channel width, length and rib width are fixed at 450μm, 2 cm, and 90μm, respectively. Furthermore, different channel depths with 250μm and 400μm are fabricated to test the channel depth effect on the micro-DMFC efficiency.

(II) Buffer Zone: Cross channel structure with hydrophilic surface is designed for this section to provide strong capillarity for water collection from the corners and walls of the upstream parallel channels. A semi-open slot on the top plate is used as the air outlet. Each solid pin has a dimension of 200μm × 200μm × 250μm (depth) or 200μm × 200μm × 400μm (depth).

(III) Water Recycling Channels and Reservoir: This part is designed as the water recycling channel, which will eventually connected to a recycling pump, which is not included in our current work. Instead, we use a reservoir filled with wick material, such as cotton wool, to provide capillary pumping force. The water recycling channels are designed with comparative narrow width than the above channels, say, 6mm (length) × 100μm (width) × 250 (or 400)μm (depth). The channel surface is also hydrophilic.

Detailed dimensions are shown in Fig. 3-1 and Fig. 3-2 shows the three-dimensional image of investigated flow field.

3.2 Fabrication of Silicon-Based Multi-Sectional Flow Field

Silicon wafer with 4 inch diameter and <100> type is used in this study. Two types of wafer thickness (~525μm and ~700μm) are adopted to fabricate different depths (250μm and 400μm).

The mask is drawn using *AutoCAD®* software and is exported on a plastic film (Mask precision=20,000dpi). The detailed fabrication process is as follows (Fig. 3-3):

I. Fabrication process of flow field:

1. Cleaning: Starting with H_2SO_4:H_2O_2=3:1 solution (Piranha Process) for about 15min.
2. Lithography:
   (1) Coating: AZ-4620 positive PR, the selectivity of Si and AZ-4620 is near 60:1, two different PR thicknesses, 5μm and 7μm, are coated for following ICP etching with 250μm and 400μm, respectively.
   (2) Soft Bake: 90 ^{o}C for 1minute

(3) Exposure

(4) Developing: AZ400K developer for 30seconds

(5) Hard Bake: 120 °C for 2minutes

3. ICP (Induced Coupled Plasma) Etching: ICP etching is a technique with high density low pressure (HDLP), it can yield a deep, high aspect ratio, strongly anisotropic structure and therefore will be adopted in this study.
4. PR Strip: Using H_2SO_4:H_2O_2=3:1 solution to remove the unwanted PR.
5. Drilling and Cutting: Using laser cutting system to drill hole for air inlet and cut wafer into several chips.
6. Cleaning: Rinse with acetone for 10 minutes to remove residual dust, then rinse with D.I. for 10 minutes to remove residual acetone. After rinsing, dry the chip with nitrogen stream.
7. Hydrophilic Treatment: Using O_2 plasma system to form native SiO_2 thin film on structure.

The completed silicon chip is shown in Fig. 3-4.

II. Fabrication of observation openings:

The through-channel structure is fabricated using laser micromachining. Considering the mechanical strength of the material, an alternating cut-through method with laser drilling is adopted. The completed flow field is shown in Fig. 3-5. From the enlarged pictures, some non-smooth area can be observed. These are melts and distorted structures caused by thermal stress during the high temperature fabrication process.

III. Surface treatment of flow field:

Three surface treatment methods are adopted for tests. For preliminary observation, structure surface is treated with hydrophilic or hydrophobic material. The treatment of hydrophilic material is to place the silicon chip into O_2 plasma chamber for 8 minutes to induce a thin native silicon dioxide, and the teflonization process is to soak the silicon chip into a 10% *Teflon*® solution (diluted with perfluoro-compound FC-75) for 10 minutes, before it is dried at 60 ^{o}C for 30 minutes and 300 ^{o}C for 15 minutes.

For actual DMFC, structure surface plated with chromium (Cr) and gold (Au) thin films to increase the electric conductivity is adopted. The Cr and Au film is plated using a DC sputter system. Cr layer is used as the primer, and the film thicknesses of Cr and Au are 0.1μm and 0.5μm, respectively.

3.3 Experimental Set-Up

3.3.1 Preliminary Observation of Water Wetting Process

Before the multi-sectional flow field is integrated into a DMFC, we first observe the water wetting process in the parallel channels and the buffer zone. The objective of this experiment is on the capillary attraction with and without air inlet. Hydrophilic surface (subjected to O_2 plasma treatment) and hydrophobic surface (subjected to *Teflon*® treatment) will be tested.

The experimental includes,

(1) Silicon-based multi-sectional channel structure, with a channel depth

of about 250μm.

(2) PMMA or glass covers, for hydrophobic or hydrophilic observations, respectively. The gap between chip and covers will be bonded with silica glue to prevent leakage.

(3) Micro flowmeter (*OMEGA Model FL-220* precision rotameter assemblies, glass float, nominal flow rate from 0.2 to 90*ml/min* for air). The rotameter is calibrated using a bubble meter, each fixed flow rate is averaged with three runs. The calibration curve is shown in Fig. 3-6.

(4) A syringe pump (*HARVARD APPARATUS Model 22*, syringe pump is powerd by step-motor) to provide precise and uniform flow.

(5) Image capture system:

- Digital video (*Sony, DCR PC330*) to record the flow process and capture images.
- Microscope (*Optem® zoom 125*): Ocular: 2X; Object lens: 0.6X ~ 6.5X.
- CCD camera (Chip size: 1/2”) to provide about 50X amplification factor in 14” monitor, with clear μm-level structure images.

3.3.2 Set-up for Single DMFC Testing

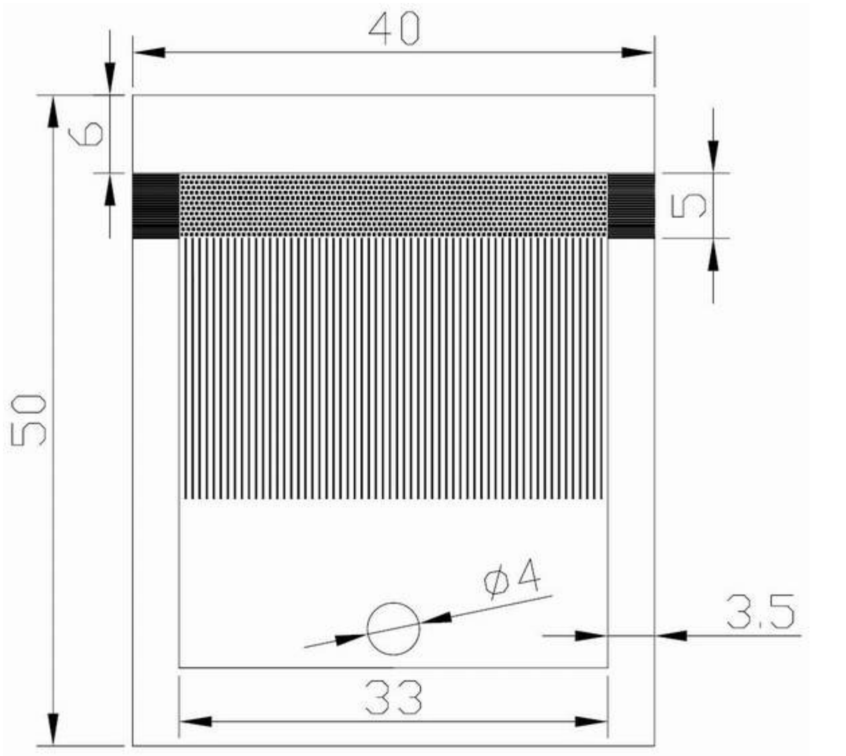
To observe the water-removing ability of our multi-sectional flow field, we integrate it into an actual single DMFC and test its performance. The set-up of DMFC testing system is shown in Fig. 3-7. Each component is described as follows:

The main body of the DMFC includes:

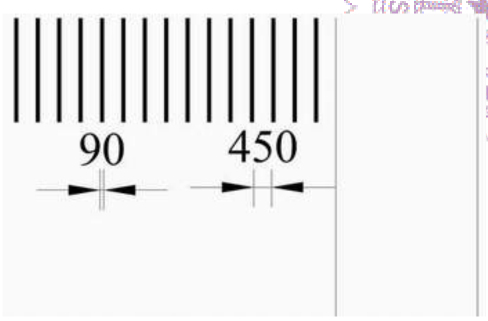
1. MEAs: Purchased from *DuPont™ Fuel Cells*. There are 3-layers and 5 layers (carbon cloth combined) configurations, appropriate catalyst coated *Nafion*® PFSA membrane.
2. Carbon paper: *Toray Industries, Inc.*, Unit: *TORAY™ TGP-H-030*, Non-wetting proof carbon paper. In this study, it will be teflonized to enhance the gas permeability and be the porous material for air outlet at the cathode side.
3. Spacer: a 1mm thick silicon rubber film to prevent leakage.
4. Gasket cover: Machined PMMA plate with eight M4 screws(Fig. 3-8)
5. Electric conductor: Three different materials are adopted, 100 μm thick stainless steel mesh, silver (Ag) coated wire and copper foil.

Testing devices include:

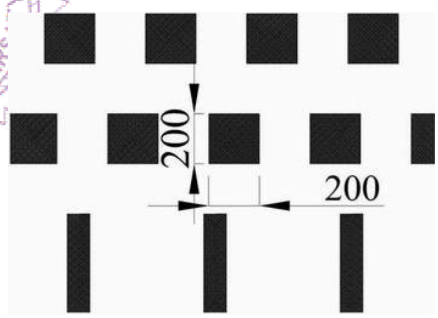
1. Micro flowmeter: *OMEGA Model FL-220*, to provide precision non-preheated and non-humidified airflow.
2. Syringe pump: *HARVARD APPARATUS Model 22*. For this study, the fluid is methanol, and its flow rate will be adjusted in accordance with the calculated result in Chapter 2.
3. Electronic load system: *PRODIGIT C3310*, used to measure the current variation with fixed voltage and the polarization curve.



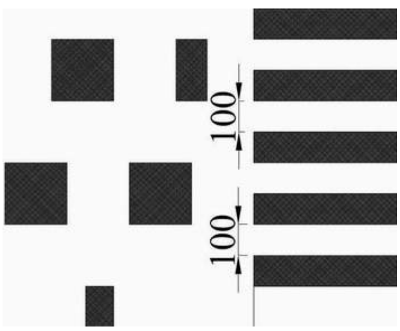
(a)Top View of Multi-section Flow Field



(b)Inlet Section



(c)Buffer Section



(d)Recycling Section

Unit: mm for (a)
um for (b)~(d)

Fig. 3-1 Marks of dimensions for multi-section flow field

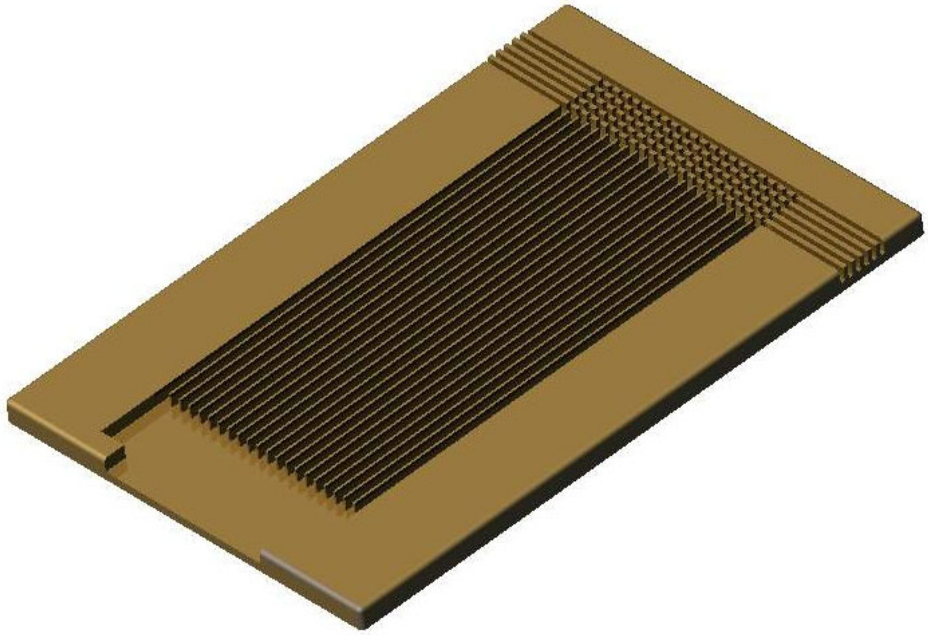


Fig. 3-2 Schematic of the silicon-based multi-section flow field (drawing not to scale)

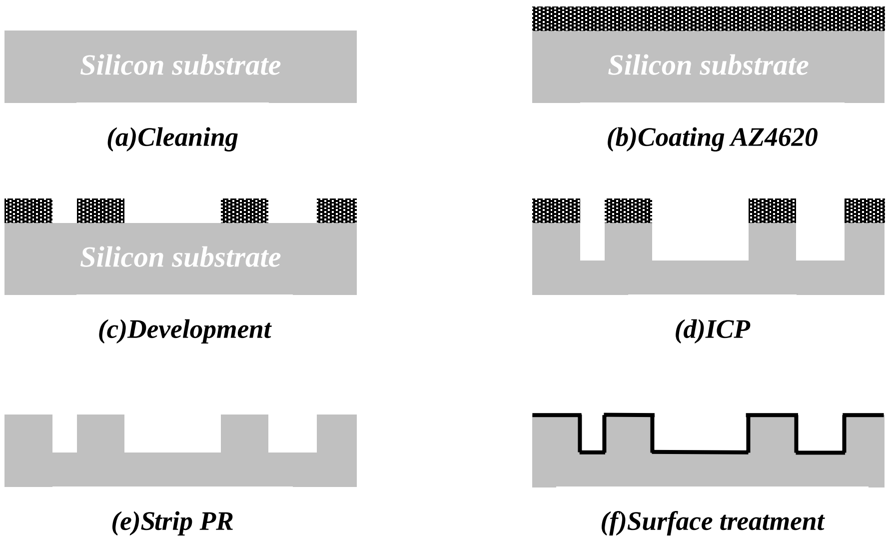


Fig. 3-3 Schematic of Si-based flow field fabrication process

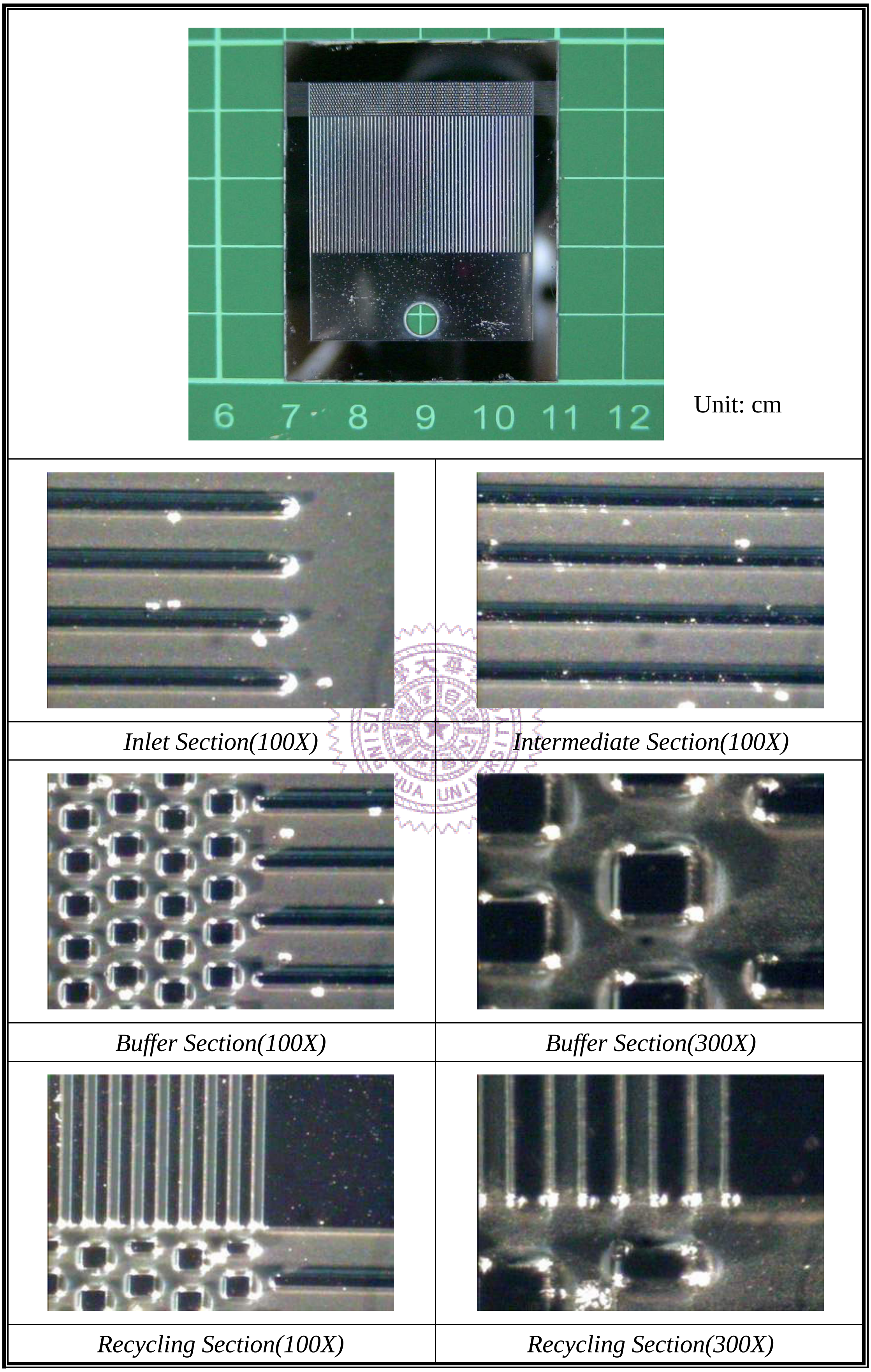


Fig. 3-4 Schematic of completed Si-based chip

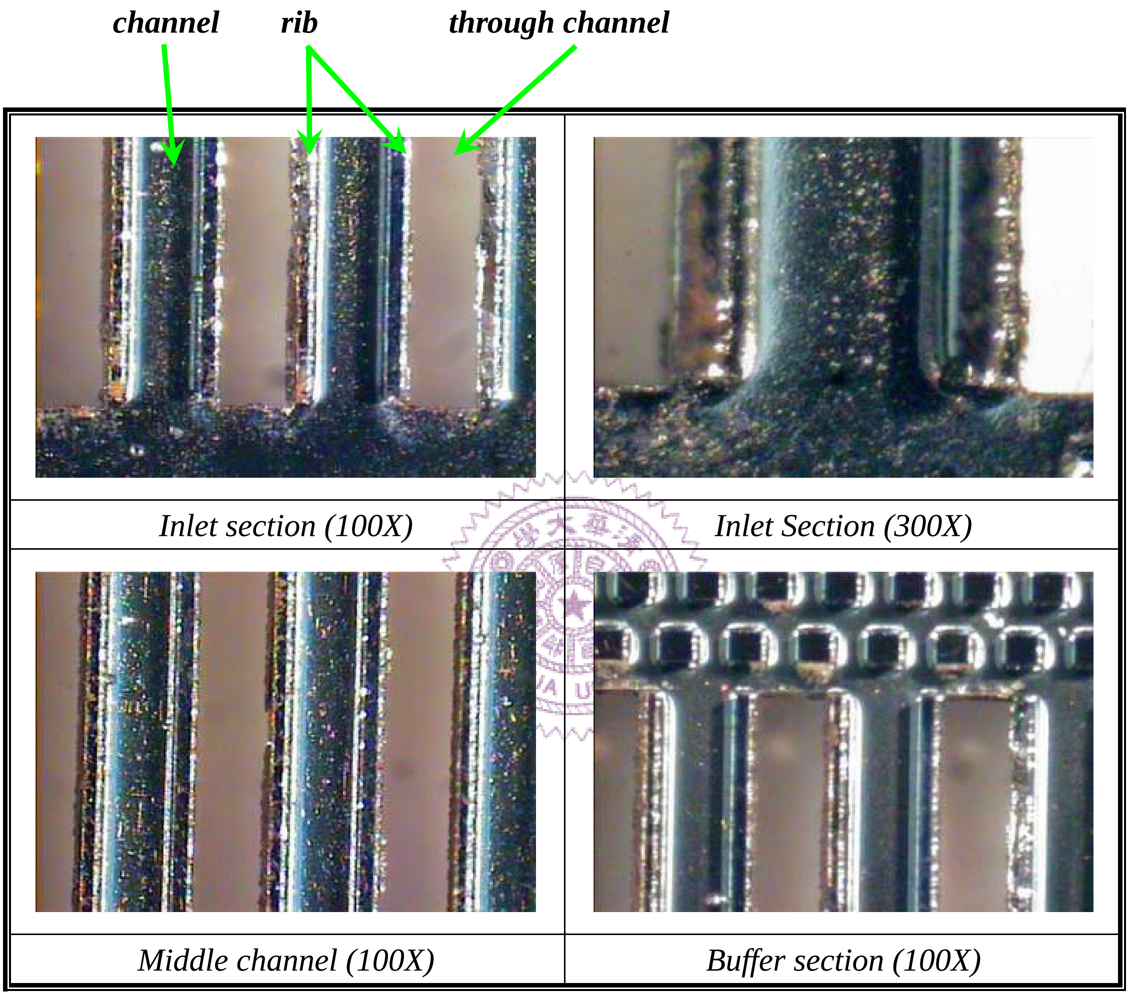
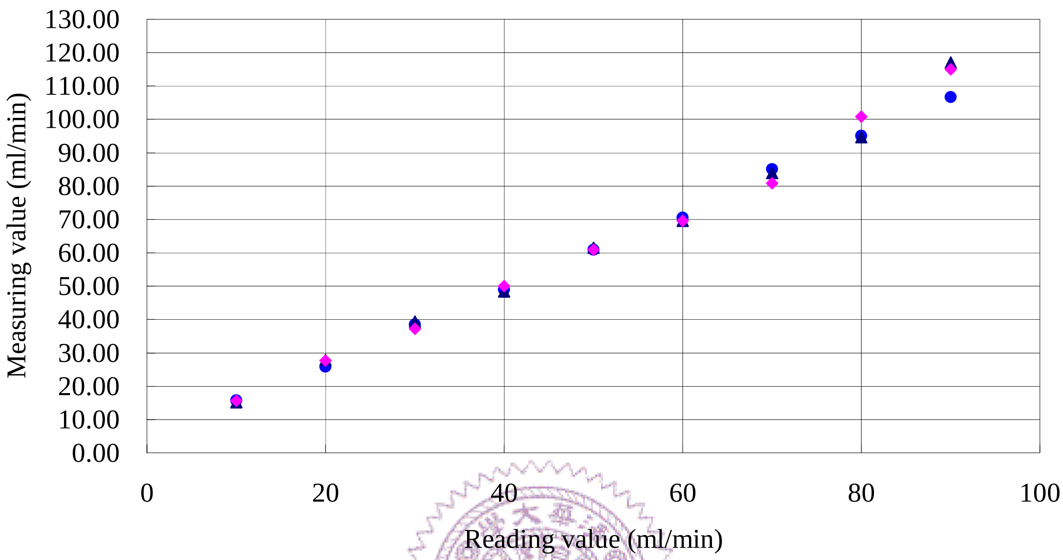


Fig. 3-5 Schematic of completed alternating through-channel silicon chip

Calibration curve of micro rotameter



Reading Value *(ml/min)*	**10**	**20**	**30**	**40**	**50**	**60**	**70**	**80**	**90**
Measuring Value I *(ml/min)*	*15.73*	*25.77*	*38.47*	*48.92*	*60.85*	*70.59*	*85.11*	*95.09*	*106.67*
Measuring Value II *(ml/min)*	*15.14*	*27.86*	*39.42*	*48.45*	*61.57*	*69.57*	*83.92*	*94.64*	*117.07*
Measuring Value III *(ml/min)*	*15.59*	*27.66*	*37.27*	*50.02*	*60.91*	*69.52*	*80.81*	*100.76*	*115.05*
Mean Value *(ml/min)*	***15.49***	***27.10***	***38.39***	***49.13***	***61.11***	***69.89***	***83.28***	***96.83***	***112.93***

Fig. 3-6 Calibration curve of micro rotameter

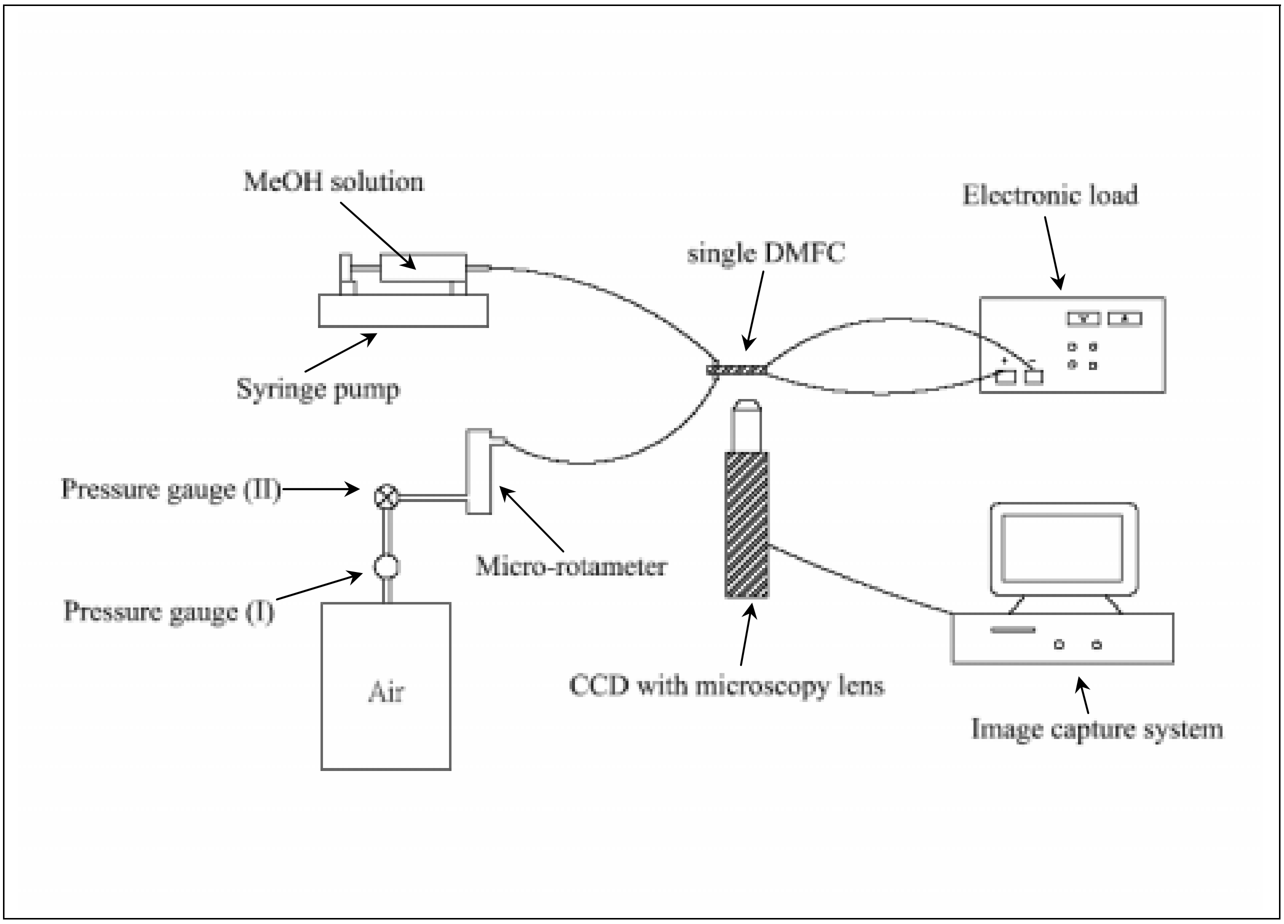


Fig. 3-7 Experimental set-up of DMFC testing system

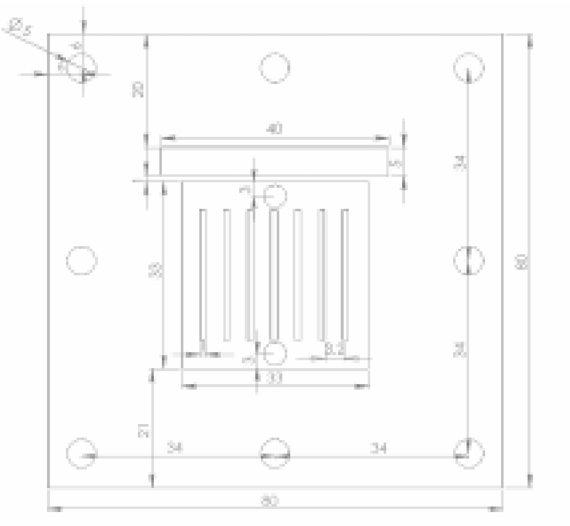
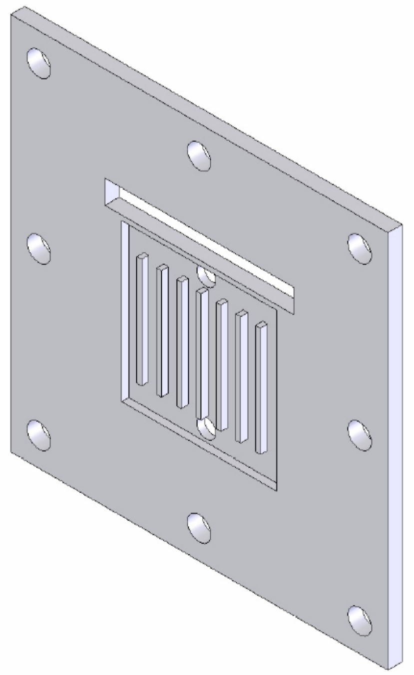
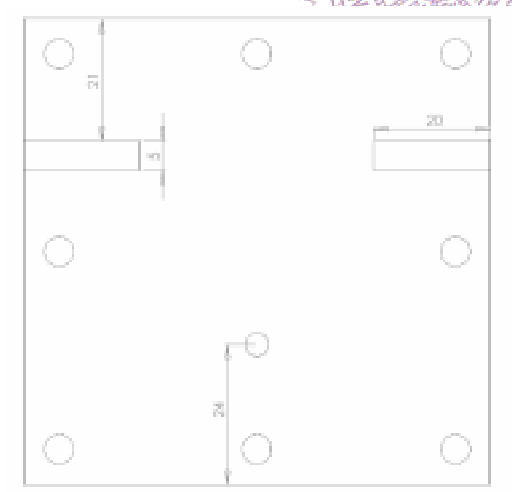
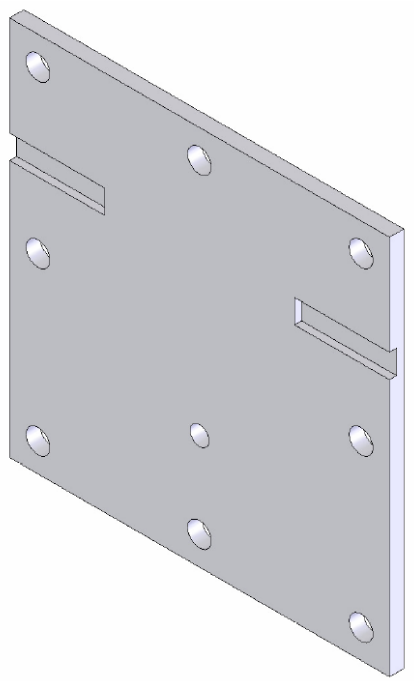
	Top view (Unit: mm)	**3D view (Thickness = 3mm)**
ANODE		
CATHODE		

Fig. 3-8 Schematic of machined PMMA plate