DELFT UNIVERSITY OF TECHNOLOGY

MASTER THESIS

An Electrowetting Valve with Air Channel

Jiahan Lu

An Electrowetting Valve with Air Channel

by

Jiahan Lu

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Supervisor:	Dr. ir. A. Bossche	
	Jeroen Bastemeijer	
Thesis committee:	Prof. Dr. P. J. French	EI/ME
	Dr. H. W. van Zeijl	EEMCS/EWI

ABSTRACT

In recent years, microfluidic systems are used in various fields. In important component is the valve. In a variety of valve principles, the electrowetting valve promises high reliability, good controllability, low voltage operation and high transmission speed characteristics. The micro-fabrication method to create a chip-level electrowetting valve will have lots of applications.

In this thesis, two designs of an electrowetting valve are presented that can be used to precisely control the flow of liquids. The hydrophobic air valves are used in the designs to allow liquid to flow forward when the valve is activated and to stop when it is deactivated. The design has different widths for the air channel to study its effect on the valve performance. Also, the after the design is completed, the design is translated into a mask set which is used to fabricate the chip in a cleanroom. Before fabrication, different types of materials with different thickness are simulated in Comsol Multiphysics to see what the respected expected saturation voltage are. Fabrication of the devices in the cleanroom involved silicon etching, TEOS deposition, metal deposition, insulating layer deposition and hydrophobic layer deposition and patterning. The depths of the channels were chosen as 20μm and 10 μm respectively. Subsequently measurement were performed to test the performance of the different designs and materials. Finally the conclusions and suggestions for the future work have been drawn.

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Jiahan Lu

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Chapter 1. Introduction

1.1 Introduction of micropumps

Over the past decade, microfluidic devices have been used in a variety of applications, from environmental monitoring to biochemical analysis (e.g. DNA testing, biomedical diagnostics). Most of these devices apply to a single measurement and the necessity to replace the microfluidic chip after each measurement is completed. However, very few devices can be used to perform multiple measurements over a longer period of time, which is necessary for online applications in industrial environments. Even then, automated sampling usually is applied to off-chip discrete components.

Lab-on-a-chip is an interdisciplinary field of integration of analytical chemistry, micro-electromechanical systems (MEMS), automatic control, electronics, materials science, biology and medicine. It is the use of micro-electromechanical system (MEMS) technology for sampling, sample pretreatment, sample separation, detection and other complex functions integrated on the chip. It has the advantages of reduced costs, faster results and reduced sample consumption, and can be applied to new drug research and development, genetic engineering, environmental monitoring and clinical disease testing.

The currently common technology used to create lab-on-a-chip is microfluidic technology and the most commonly used method to transport fluids on the chip is a micro pump. Based on piezoelectric, electrostatic, or thermal actuation, a small amount of solution is driven through a capillary to carry out the reaction or separation. The piezoelectric actuator mainly uses special deformable material. When the voltage is applied, the material will undergo extended deformation yielding a force to drive the liquid. The electrostatic actuators can be fully integrated in the silicon fabricating process in the form of parallel plate actuators. This kind of actuation offers operation frequencies up to several kHz and shows extremely low power consumption. However, the force/distance relation is nonlinear and the actuator stroke of about 5um at actuation voltage of 200V is relatively small. As for the heat-type actuator, there is a chamber opposite the primary pump chamber which comprises a secondary working fluid. Heating the secondary fluid, with for example a resistive heater, causes the fluid to expand and deflects the pump diaphragm. This kind of actuator can be integrated in the silicon processing. However, due to the heating and cooling of the working medium, it usually has a large time constant, hence the speed is low [1].

In all those microfluidic pumping devices, the surface tension is very important. Because at the micro-scale, the liquid surface tension and wall effect become the main factors affecting the flow. In fact, we can achieve the purpose of driving the fluid through the application by controlling the surface tension. The surface tension is dependent on the temperature. In general, the surface tension of the liquid and gas interface decreases as the temperature increases. The thermal resistance micro-driver utilizes this principle. The micro heater causes the liquid to produce a gradient in temperature along the capillary, so the surface tension also shows a gradient which can be used to drive the liquid flow. But through the heating method, it is likely to cause changes in the phase of the droplets or chemical changes in the liquid, or even have impact on the biochemical reaction. Therefore, the capacitive actuator is considered the most appropriate way to drive the fluid. Because it drives the liquid flow by changing the surface energy distribution by applying different voltages. In addition to faster speed and lower energy consumption, it also avoids the disadvantage of the micro heater caused by the temperature.

Based on the principle of capacitive actuator, the concept of electrowetting is proposed. It consists of electrodes, dielectric layers and hydrophobic layers. The dielectric layer serves to block the transmission of electrons and reduce the occurrence of hydrolysis. The hydrophobic layer causes the droplets to have a greater default contact angle on the surface. A voltage difference between electrode and liquid will change the surface charge and so the contact angle. This process is called the Electrowetting on Dielectric (EWOD). The details are presented in the next chapter. Using the concept of EWOD, the actuator can drive the fluid between solid and gas phase by the applied voltage. It is the biggest difference compared with the general driving device that the EWOD device can drive a discontinuous flow of fluid or only a single droplet.

On the other hand, this concept of EWOD can also be applied to the micro total analysis system. Figure 1.1 shows a schematic diagram of the concept of a movable microfluidic device [2]. MxN different reagents can be stored in the storage tank on the system and the droplet movement is controlled by the applied voltage. For example, single droplets are separated from M1 and N1, allowing them to fuse at the intersection. Thus, a variety of fusions or reactions can be made on one wafer. It is even possible to allow the various reactants to fuse and then react. This device can instantaneously and quickly synthesize the desired reactants each time. This can effectively reduce costs.

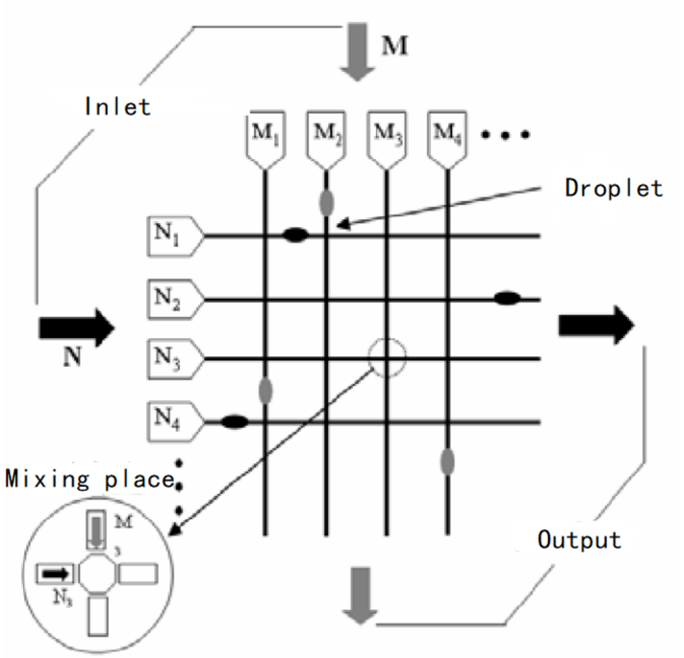


Figure 1.1 Mixing device of EWOD

It can be seen that EWOD has a wide range of applications. But many of the current studies are based on high voltages. This allows some studies such as medical research, implantable devices, etc. to receive a lot of restrictions. Research on low voltage driven droplet technology is a promising future direction.

1.2 Objectives

The study described in this thesis is to develop on-chip valving and metering components based on electrowetting of a (normally) hydrophobic channel segment, allowing automatic sample intake and manipulation. Figure 1.2 shows a schematic of such an electrically operated valve. When a small voltage is applied to the embedded control electrode the surface becomes hydrophilic and water will flow through the valve. When the electrode is deactivated, the water will evacuate the valve area due to the capillary pressure and the flow will stop. The hydrophobic air-vent channel allows air to flow in and out during switching. A series of such valve segments can be used to accurately separate a well-defined volume of fluid and transport it to the desired location on the chip for further processing (e.g. analysis or mixing).

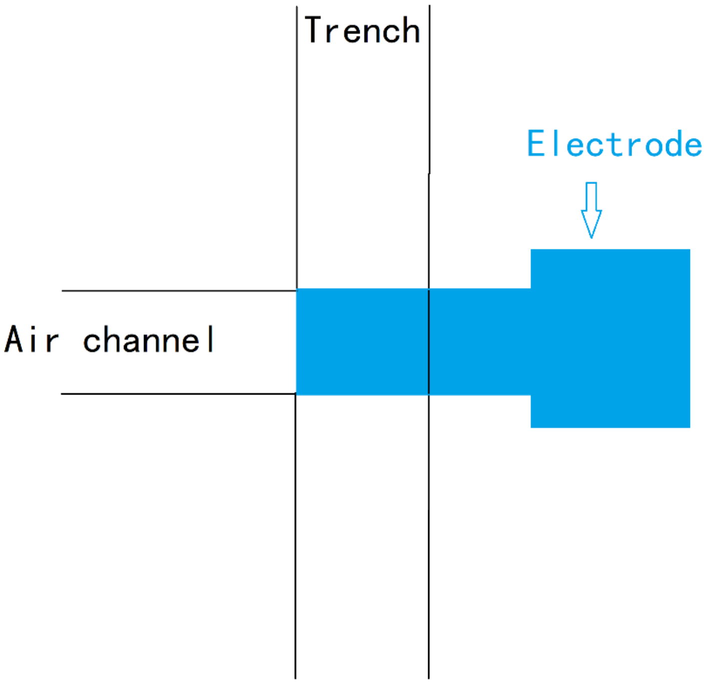


Figure 1.2 Schematic of electrowetting valve

1.3 Thesis outline

In chapter 2 the classification of the micropumps will be introduced. The theory of electrowetting on dielectric and the three phases in EWOD will be explained. The designs of the EWOD devices and the masks are explained in chapter 3. In chapter 4 the fabrication flow and some challenges are described. Chapter 5 describes the simulation of flat type and the channel type of EWOD devices and compares the outcomes with experimental results. Finally, chapter 6 present the conclusions and suggestions.

Reference

[1] Morsink, D. W. "Design and Feasibility Test of a Microfluidic MEMS for an Integrated Light and Scanning Electron Microscope." (2013).

[2] 微液滴驅動之研究與探討 (The study and discussion on Electrowetting) [D]. National Central University, 2006.

Chapter 2. Micropumps and Electrowetting on dielectric overview

2.1 Micropumps

Microelectromechanical systems (MEMS) are devices that integrate mechanical components with conventional electronic circuits on microchips. Due to the MEMS technology developing, a wide range of sensors and actuators with very small size (micron level) have been invented and can be used in both micro and macro world. Microfluidic device was the first MEMS device fabricated by the technology that was intended to fabricate the microelectronic chips. Over the past few decades, many microfluidic devices have been developed for flow sensors, pumps, valves, mixers and many other devices.

This section briefly describes several working principle of micropumps.

2.1.1 Micropump classification

Over the past few years, several review articles have been published to evaluate micropumps for their applications and performance. Some of them classify the micropumps according to their working principle and other articles classify the micropumps by their design or performance.

Nguyen et al. [1] have reviewed many publications on micropumps and divided them into mechanical and non-mechanical pumps. The mechanical pumps are divided into check-valve pump, peristaltic pump, valveless rectification pump, rotary pump and ultrasonic pump. Non-mechanical pumps are divided into electrohydrodynamic pump, electrokinetic pump, phase transfer pump, electrowetting pump, electrochemical pump and magnetohydrodynamic pumps.

It should be noted that the classification only considers the working principle. The design and performance of the same type of pump may be very different. Some designs are made in a single silicon substrate, while other designs consist of other materials or two substrates. In addition, the performance of the two pumps based on the same principle can be completely different. Pump performance is mainly due to the design of the actual geometry and size.

In the next paragraphs, some common designs will be introduced.

2.1.2 Check-valve pump

The check-valve is the first micropump to be realized. Its working principle is basically the same as the large pump. An actuator causes a stroke volume with two check valves that adjust the flow direction. When the pump chamber is inflated and the chamber is filled, the inlet valve opens. The outlet valve prevents the fluid from flowing back into the room. Then the pump chamber shrinks, the inlet valve closes and the outlet valve opens due to the overpressure in the chamber. See Figure 2.1 [2].

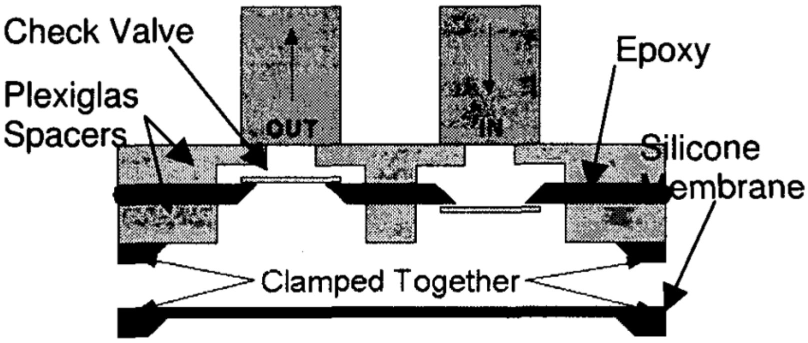


Figure 2.1 Check valve

Over the past few years, different valve designs have been reported. But all valves are only allowed to flow in one direction and prevent flow in the opposite direction. Some of the common designs of check valves are: ring diaphragms, flaps and cantilever beams. The performance of the valve depends to a large extent on the design of these things. The key features of the check valve are reverse flow, pressure drop and switching speed.

2.1.3 Peristaltic pumps

The peristaltic pumps runs in the absence of a check valve for flow rectification. The principle is based on the peristaltic motion of the pump chambers, which squeezes the fluid into the desired direction. The volume can be pumped in either direction by starting the chamber with a phase difference of 120° . The diaphragm of the chamber may also partially function as a check valve. Typically, the chamber pressure in the peristaltic pump is lower than the chamber pressure in the one way valve. Therefore, in the design of the peristaltic pump, the stroke volume and compression ratio become more important parameters. It shows in Figure 2.2 [3].

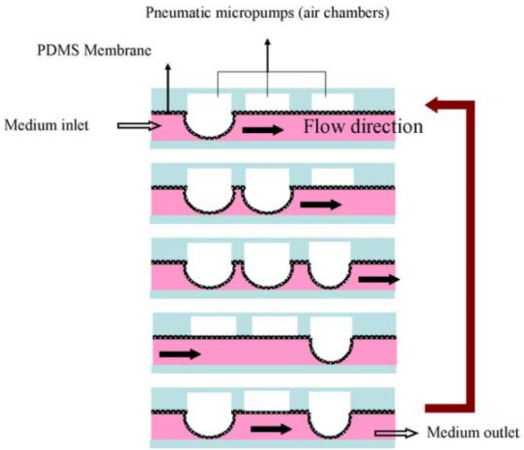


Figure 2.2 Peristaltic pump

2.1.4 Electrohydrodynamic pumps

The electrohydrodynamic (EHD) pump is based on the electrostatic force acting on the medium fluid. It can be divided into two main types: the EHD induction pump and the EHD injection pump. The EHD induction pump is based on the induced charge at the material interface. The electric field drags and pulls the induced charge along the wave direction. In the EHD injection pump, the Coulomb force is responsible for the ion transportation from one or two electrodes by electrochemical reaction. They are shown in Figure 2.3 [4].

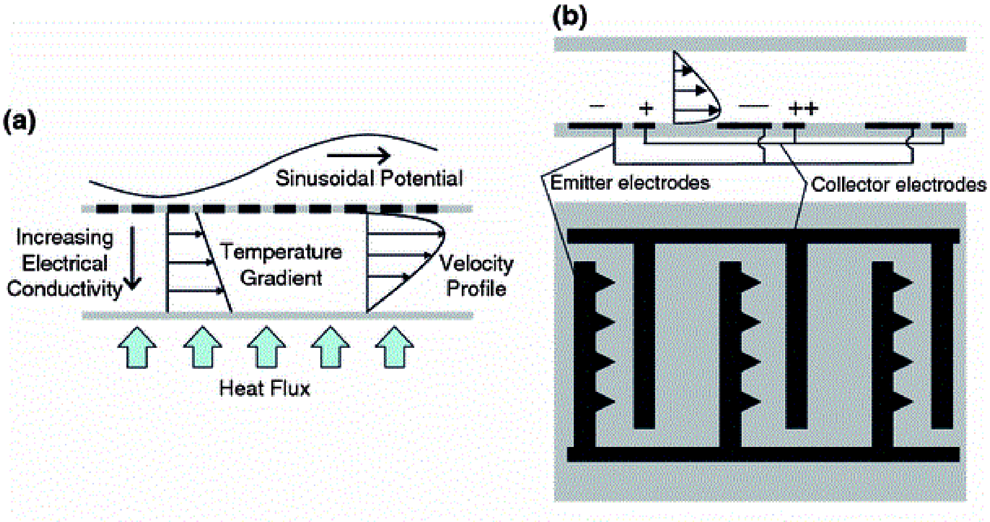


Figure 2.3 (a) induction type; (b) injection type

2.1.5 Electrowetting pump

The principle of the electrowetting pump uses the tension between the solid / liquid interface to remove the surface charge. This principle can be used for direct pumping. In recent years, more and more people have studied electrowetting related things, electrowetting pump is no exception. In the past decade, most people's research is based on several tens of volts to make the hydrophobic surface changed to hydrophilic surface. Such high voltage cannot be used in the MEMS technology. But recently the EWOD (Electrowetting on dielectric) based pump can drive the droplet at low voltage and fast transferring speed. So by studying the characteristics of electrowetting to reduce the driving voltage to make it a wider application is a trend.

2.2 Theory of electrowetting

In this section, the basic principles of electrowetting are introduced. There will be a theoretical description of electrowetting and the formulas' explanation. The three parts (dielectric layer, hydrophobic layer and conducting fluid) that make up the entire EWOD system will be introduced separately.

The standard EWOD device consists of electrodes covered with an insulating layer, conductive droplets and a hydrophobic layer (Figure 2.4). In most applications, the droplet's size is typically less than or equal to 1 mm of water or solution. The external environment is usually air or other liquid that is immiscible with the droplets, such as oil. In order to analyze the behavior of droplets, the Bond number β is considered. It represents the relative magnitude of the force due to gravity and interfacial tension:

$$\beta = \frac{\Delta\rho\, g\, R^2}{\gamma_{ci}} \qquad \text{(eq. 2.1)}$$

In the equation, Δρ represents the density difference between the droplet and the surrounding medium; g is the gravitational acceleration; R is the characteristic length of the droplet; and γ_{ci} is the interfacial tension between the conductive droplet and the insulating layer.

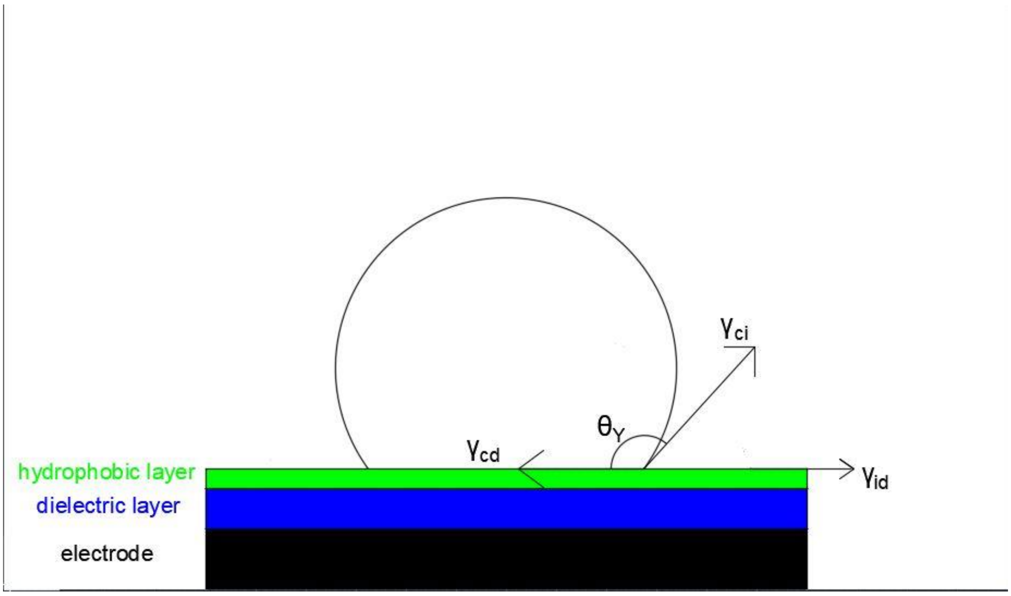


Figure 2.4 The classic schematic of EWOD (no applied voltage)

In a typical electrowetting system, especially when the droplets are in contact with the oil, β is much smaller than unity [5]. So the gravity can be ignored. Therefore, in the absence of externally applied voltage, the performance of the droplet will only be affected by the interfacial tension γ. The contact angle which is formed by the conducting liquid (c), the external insulating environment (i) and the dielectric substrate layer (d) is given by the Young's equation [6]:

$$\gamma_{id} = \gamma_{cd} + \gamma_{ci} \cos\theta_Y \tag{eq. 2.2}$$

θ_Y is the contact angle, it is also called the Young's angle. γ_{id}, γ_{cd} and γ_{ci} represent the interfacial energy, respectively (Figure 2.5).

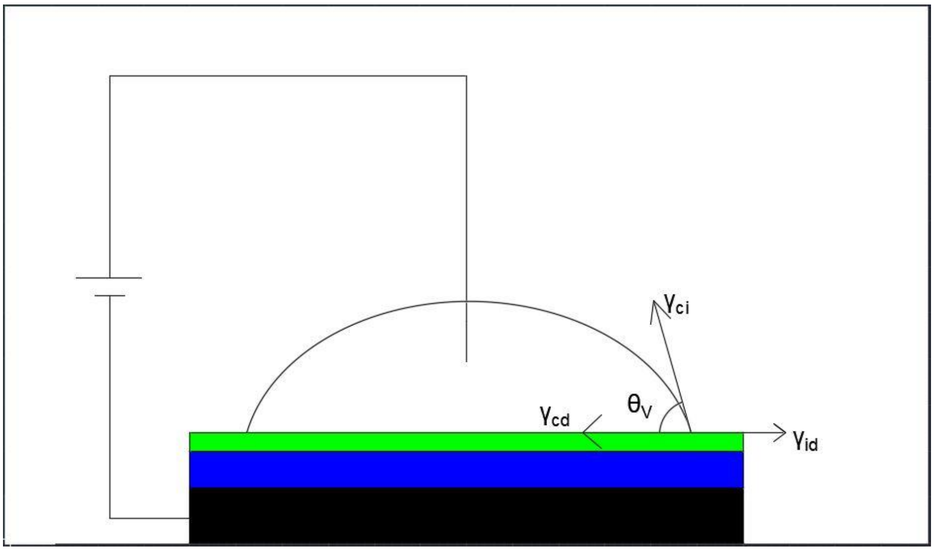


Figure 2.5 Balanced force distribution in an EWOD system with applied voltage

When an appropriate voltage is applied between the droplet and the bottom electrode (Figure 2.5), the charge from the liquid is attracted to the droplet / hydrophobic interface. Assuming that the liquid is a perfect conductor, its free charge density is zero. As can be seen from Figure 2.5, the force generated by the liquid / hydrophobic interface charge has a vertical component. It causes normal stress on the surface of the hydrophobic layer, which is compensated by elastic stress. However, as the contact line approaches, the surface charge appears on the interface of the liquid / hydrophobic layer due to sharp edge effects [7]. As a result, the electrostatic force F is generated in the horizontal direction. The expression consists of the vacuum dielectric constant (ε_0), the dielectric constant (ε_r) of the dielectric, the applied voltage (V) and the dielectric thickness (d):

$$F = \frac{\varepsilon_0\, \varepsilon_r\, V^2}{2\, d} \qquad \text{(eq. 2.3)}$$

This electrostatic force will have an attraction to the droplets, resulting in a decrease in the contact angle of the droplets formed on the hydrophobic layer. This contact angle is called the electrowetting angle (θ_V) in the electric field. From Figure 2.5, the force balance on the horizontal axis can be written as:

$$\gamma_{id} + F = \gamma_{cd} + \gamma_{ci} \cos\theta_V \qquad \text{(eq. 2.4)}$$

So the electrowetting angle can be obtained by combining equations 2.2, 2.3 and 2.4 as a function of the applied voltage change [8]:

$$\cos\theta_V = \cos\theta_Y + \frac{\varepsilon_0 \varepsilon_r V^2}{2\, d\, \gamma_{ci}} \qquad \text{(eq. 2.5)}$$

This is commonly referred to as Lippmann-Young's equation. As long as there is no leakage in the insulating layer, the droplet will return to its initial state when the voltage is removed.

In the derivation of the Lippmann-Young equation, the decrease in the contact angle is due to the force generated by the electric field that acts on the liquid. In addition to this interpretation, there are other different interpretations to describe the electrowetting effect. But all these explanations finally get the same expression. In thermodynamics, as the potential differences increases across the hydrophobic/droplet interface, charge builds up both at the solid electrode underlying the dielectric layer and at the droplet, causing a decrease in interfacial energy [9].

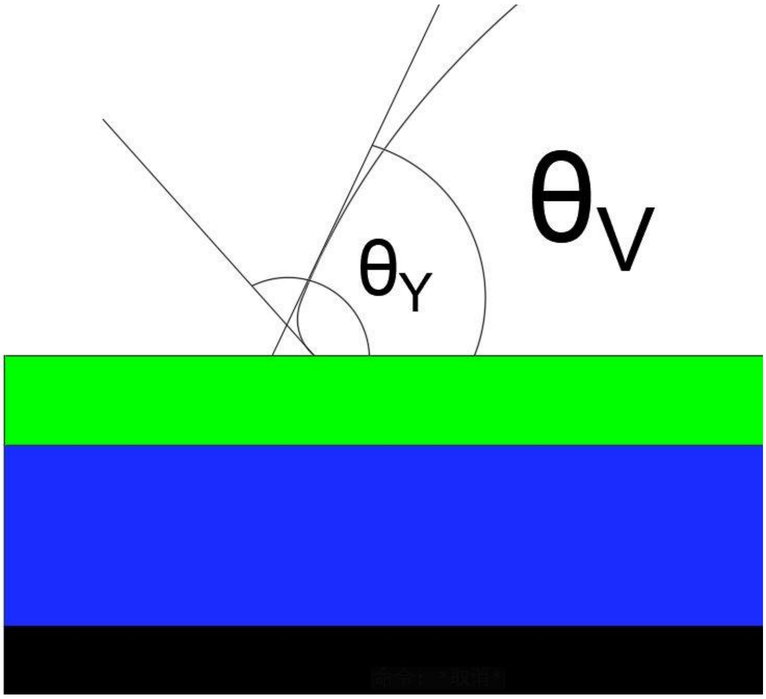


Figure 2.6 The contact angle and the Young's angle showing under the microscope

Nowadays, the 'electromechanical explanation' [10] has become to the most widely accepted explanation. As shown in Figure 2.6, Mugele et al. [11] observed that the microscopic contact angle is always maintained at the Young's angle, although the macroscopic angle has become hydrophilic. The theoretical prediction of small voltage is very consistent with the experimental results. However, when the macroscopic contact angle reaches a certain angle as the voltage rises, it does not change anymore. We call this angle that is no longer changing as a saturated contact angle.

Both DC and AC voltages can be used to drive the EWOD system. In the case where the applied voltage is slowly changed, the contact angle and the droplet shape can instantaneously change to a stable value. If the AC frequency is too high to exceed the fluid dynamic response time of the droplets (typically millimeter-level droplets at frequencies of several hundred hertz), the liquid response depends only on the time average of the applied voltage. This argument is correct, as long as the basic assumption of the Lippmann's equation is not violated: one of which is the liquid is a perfect conductor. However, when the frequency is increases, the droplet decomposes. While the dissolved ions can follow the applied field at moderate frequencies, they are not able to do so beyond a certain critical frequency ω_c. Far below ω_c, the liquid behaves as a perfect conductor; far above it behaves as a dielectric [5].

2.3 Three phases of electrowetting

As we talked about in section 2.1, a EWOD system consists of three phases: the conducting liquid, the external insulating environment and the dielectric substrate layer.

2.3.1 The conducting liquid

In the classical theory of electrowetting, liquid is considered as a perfect conductor. For aqueous salt solutions, this corresponds to the limits of high salt concentration or low frequency, as previously described. The requirements for the concentration and nature of charge carriers are not that strict. At low frequencies (f <1 kHz), even DI water shows significant electrowetting [12]. Typically, the salt, such as NaCl and sodium dodecylsulfate (SDS), concentration is 0.01-1 mol / L. Most reports indicate that there is no significant effect due to the type or concentration of the salt.

Ion surfactants have another advantage. In addition to providing the charge required for the electrowetting effect, it is possible to reduce the interfacial tension between the conductive droplet and the insulating fluid. And according to eq. 2.5, the voltage required to reach the saturation angle is reduced. However, Kuiper pointed out that if the reduction of γ_{ci} is too large, it will affect the switching speed, which is harmful to applications that need fast response [13].

2.3.2 The insulating environment

The insulating environment is generally composed of air or oil. The place where the gas environment is applied is not particularly large because there is not much maneuverability. But on the contrary, there are many applications using oil as an insulating environment.

2.3.3 The dielectric layer

For an EWOD system, the ideal situation is to get a large contact angle change using as low voltage as possible. In order to achieve this goal, there are basically two ways according to eq. 2.5: increase the Young’s angle or decrease the dielectric layer thickness.

In the EWOD system, aqueous solutions are the most commonly used conductive fluids. Therefore, to get a higher Young's contact angle, different hydrophobic materials can be used. Ideally, the dielectric itself is hydrophobic and pinhole free on its own (like PDMS, Parylene). Otherwise it is necessary to deposit an insulating layer without pinhole under the hydrophobic layer.

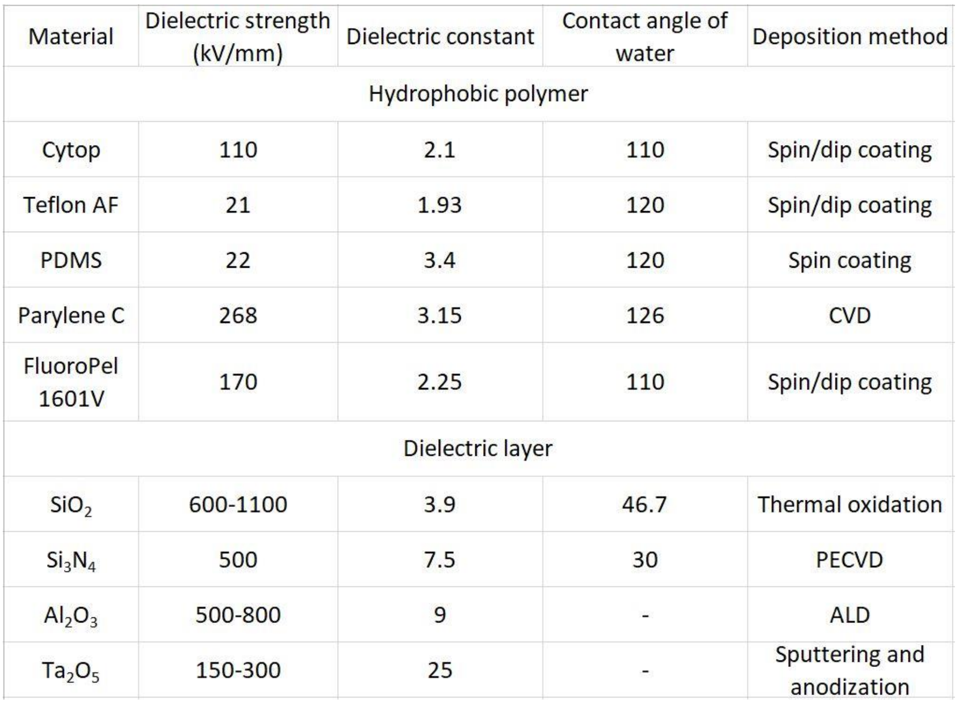
The second way is to reduce the thickness of dielectric layer. As we can see from eq.2.5, the thinner the insulating layer, the smaller the required voltage. But the thickness is limited by the breakdown strength of the insulating material. When the electric field in the insulating layer exceeds the breakdown strength, the molecular structure will be destroyed by the current generated in the material. The dielectric breakdown voltage (V_{bd}) is a function of its thickness (d) and the dielectric breakdown field strength (E_{bd}):

$$V_{bd}=E_{bd}\cdot d \qquad \text{(eq. 2.6)}$$

So, on the one hand, the thinner insulating layer can achieve the same contact angle change with a smaller voltage. But it also makes the breakdown voltage lower. According to eq.2.5, a higher relative permittivity could also reduce the voltage needed.

Commonly used hydrophobic layers for low voltage EWOD include Cytop, Fluoropel, Teflon AF and Parylene C. Wherein Cytop, Fluoropel and Teflon AF are amorphous fluoropolymer. They are deposited by spin coating or dip coating and usually only as a hydrophobic layer rather than as a main insulator. Parylene-C is a high quality pinhole-free polymer film produced by a vapor deposition process. Comparing with other kinds of hydrophobic polymer, Parylene C has high electrical insulating property and good mechanical strength. In order to increase the hydrophobicity of the system, fluoropolymer can also be added to Parylene C.

Table 2.1 characteristics of the common EWOD materials [7] [14]



Material	Dielectric strength (kV/mm)	Dielectric constant	Contact angle of water	Deposition method
Hydrophobic polymer				
Cytop	110	2.1	110	Spin/dip coating
Teflon AF	21	1.93	120	Spin/dip coating
PDMS	22	3.4	120	Spin coating
Parylene C	268	3.15	126	CVD
FluoroPel 1601V	170	2.25	110	Spin/dip coating
Dielectric layer				
SiO_2	600-1100	3.9	46.7	Thermal oxidation
Si_3N_4	500	7.5	30	PECVD
Al_2O_3	500-800	9	-	ALD
Ta_2O_5	150-300	25	-	Sputtering and anodization

Common insulating materials in EWOD include silicon dioxide, silicon nitride, aluminum oxide and tantalum pentoxide. Silicon dioxide (SiO2) is typically obtained by thermal oxidation of the silicon substrate. Silicon nitride (SiN) is prepared using PECVD. Aluminum oxide is made using the ALD (Atomic layer deposition) method. These are common materials in the microelectronics industry. Since they are all hydrophilic, they must be coated with one of the above fluoropolymers to achieve a hydrophobic effect. Most of these insulating materials have a high insulation breakdown field strength, which allows us to use thinner insulation to reduce the required voltage.

Al_2O_3 is one of the most widely studied materials due to its excellent dielectric properties, thermal stability and good adhesion to many surfaces [15]. In order to deposit pinhole free and very thin Al_2O_3 layer, atomic layer deposition (ALD) is one of the best methods. Especially the low-temperature Al_2O_3 ALD films exhibited very low surface roughness values and low leakage currents. The root-mean-squared surface roughness for a 1x1 um^2 area averaged 4 ± 1 Å [16]. So it is chosen as one of the dielectric materials for my low voltage EWOD application. The other insulating material used in the application is silicon nitride. It can be seen from Table 2.1 that silicon nitride also has a high dielectric constant and breakdown voltage. And the uniformity also can be well controlled during the process [17].

2.4 Droplet transportation by EWOD

As described in the previous section, the benefits of using an insulating layer between droplets and electrodes are very large. The presence of the dielectric layer not only allows the contact angle to change even more, but also can be a good solution to solve the problem that a higher voltage will cause fluid electrolysis. Because the number of the materials which can achieve the functions of both hydrophobic and insulating is limited. And all of them have a relatively low dielectric constant. If adding a special insulating layer with high dielectric constant, the constant of hydrophobic material can be ignored since the presence of the insulating layer prevent them from break through. According to e.q. 2.5, a larger dielectric constant results in a smaller voltage required when the contact angle stays the same. The hydrophobic layer is only used for hydrophobic. And also because of the pin-hole free insulating layer existing, there is no leaking current between the droplet and the electrode as long as the voltage is lower than its strength. This can protect the device in a good way and have a better performance.

By using this method, another innovation in EWOD is the use of patterned electrodes for droplet transportation which is shown in Figure 2.7.

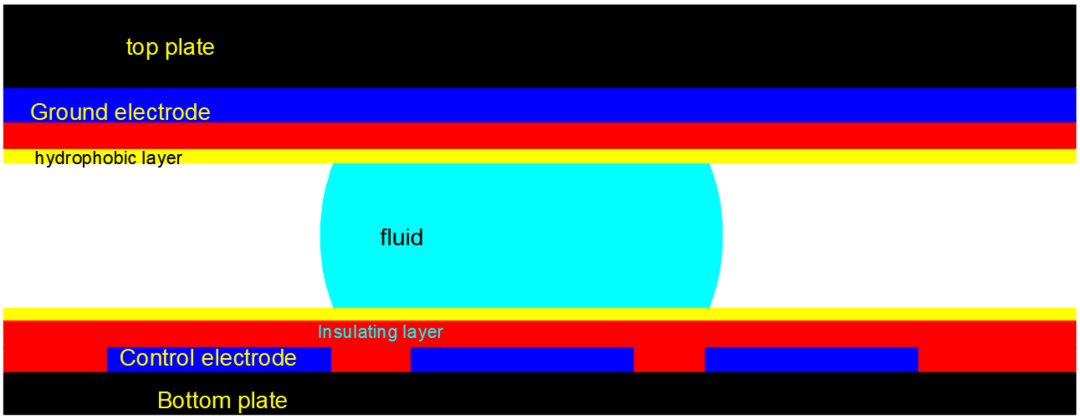


Figure 2.7 Schematic of EWOD transportation

Pollack et al. first used a few nanoliter to a few microliters of conductive droplets to show the droplet transmission [18]. These droplets are located between two glass plates. The bottom plate has regular electrodes covered by the dielectric and hydrophobic layers. The top plate has a continuous ground electrodes, as shown in Figure 2.7. The conductive droplet is initially placed in the center of the bottom electrode and sandwiched between the two hydrophobic layers. When proper voltage is applied between the left bottom electrode and the ground electrode, the contact angle on the left side of the droplet is changed according to eq. 2.5. When the applied voltage exceeds a certain threshold voltage, the droplet moves to the left electrode where the voltage is applied. Pollack's work was the first demonstration of digital microfluidic lab-on-chip systems that use droplets instead of the continuous microfluidic systems.

This process is shown in Figure 2.8 and Figure 2.9. The dotted line in the Figure 2.8 is the shape of the droplet when no voltage is applied. After the voltage is applied on the left electrode, the droplet shape changes to the solid one immediately. Figure 2.9, the filled droplet is the shape after it is transported.

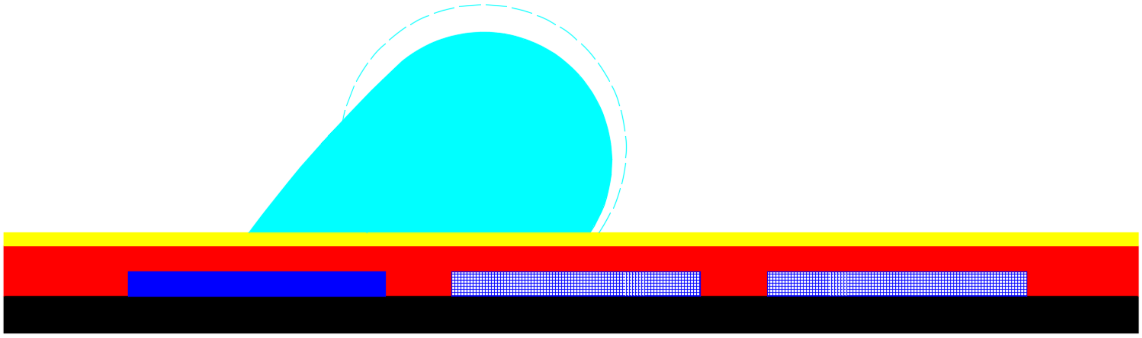


Figure 2.8 Immediately schematic of the drop when voltage applied on left electrode

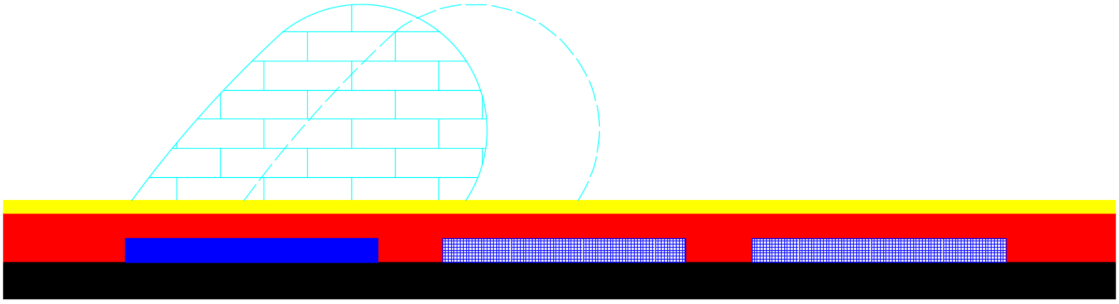


Figure 2.9 Schematic of droplet after transportation

2.5 EWOD Related Applications

Michael et al. presented a micro-actuator to quickly process discrete micro droplets [19]. Micro actuation is achieved by direct electrical control of surface tension of two opposing planar electrodes which are fabricated on glass. A simple device consisting of a linear array of seven electrodes at 1.5 mm pitch was fabricated and tested. Droplets of 0.7 μL were successfully transferred between electrodes at the voltage of 40-80 V. It has been shown that when the electrode switching rate is up to 20 Hz and the average velocities is 300 mm/s, the droplets can be transported repeatedly. Figure 2.10 shows the video frames of the moving droplet. The view is from the top looking through the ITO ground electrode. In the first picture, 80 V is applied on the electrode which is underneath the droplet. In the second and third pictures, 80 V potential is applied on the electrode on the right of the droplet and the droplet moves to that direction.

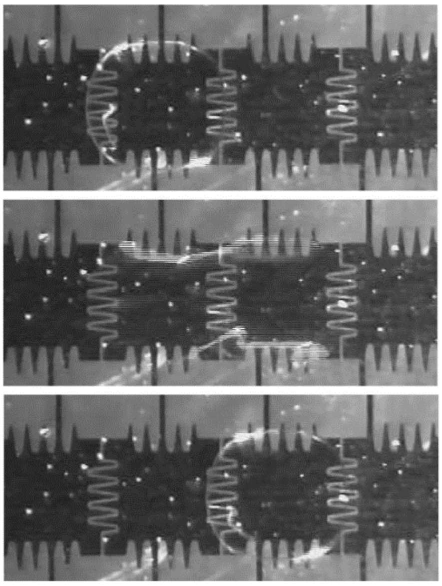


Figure 2.10 Video frames of the moving droplet

Banerjee et al. presented a continuous and discrete functions in a digital microfluidic platform in a programmed manner which focus on the ability of continuous microfluidics to process larger sample volumes at a higher throughput [20]. In his work, the parallel channels are formed and programmed to split into multiple droplets while the droplets are programmed to separate from one channel, but transfer and merge to another channel. Figure 2.11 shows the two channels. Chanel 1 is connected to the inlet and supplied by the syringe pump. Then three droplets were drawn from channel 1 by activating three electrodes underneath the gap between two channels. Subsequently, three droplets are then transported to channel 2 and merged with it.

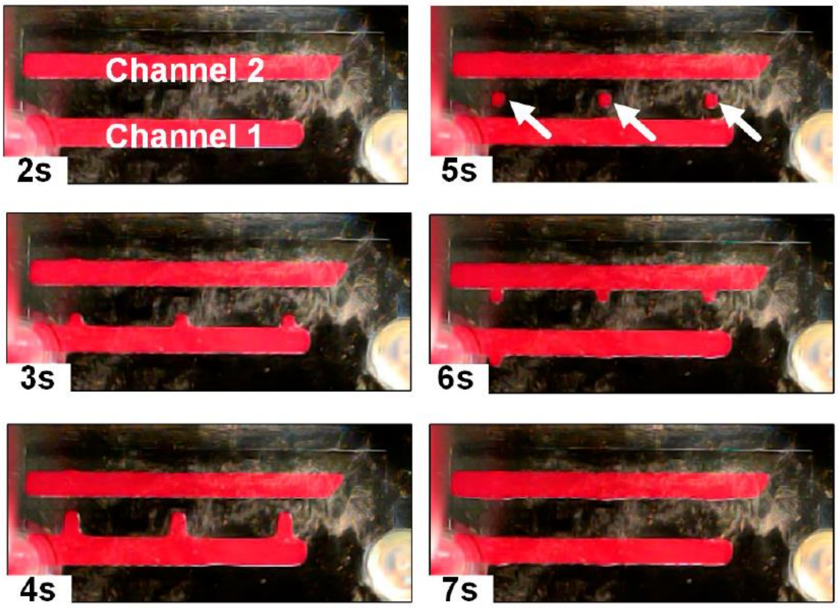


Figure 2.11 The transportation of three droplets between two channels

Microbubbles have been used in various microfluidic applications such as valve, pump and an actuator. Yang et al. introduced an electrolysis bubble actuated micropump that uses EWOD actuation to form an air valve in microchannel [21]. Figure 2.12 shows sequential images of micro fluid being pumped. When the first and second electrodes were turned on, the surface property of the electrodes changed from hydrophobic to hydrophilic. As a result, the microchannel that contains the EWOD electrodes was filled with fluid. When the first electrode was turned off, an air bubble was simultaneously trapped in the EWOD electrode, and the fluid inside the microchannel was then transported to the right by sequential electrolysis-bubble actuations. Because this EWOD device does not require mechanical moving parts, the pump eliminates the shortcomings of existing mechanical pumps, such as reduced performance and reliability due to long-term operational fatigue.

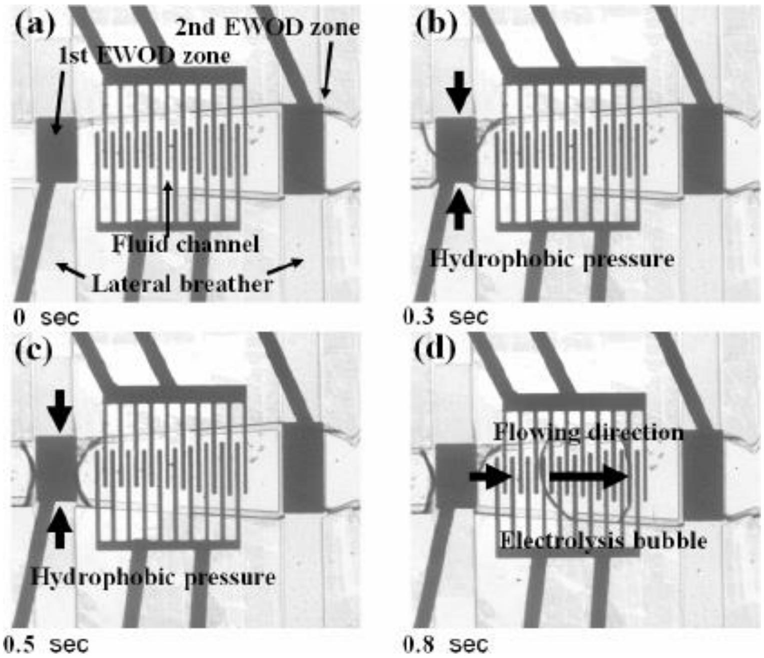


Figure 2.12 Time-sequential pictures. (a) The voltage difference is applied at first and second EWOD zones. (b) The voltage at first EWOD zone is turned off. (c) An air valve is formed. (d) Micro fluid is pumped without back flow

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Chapter 3. Design ideas

There are two different designs introduced in this chapter. One is a design with rectangular electrodes of different widths compared with the air channel, the other one is a design of 45° angled electrodes of the same size.

3.1 Different width electrodes and air channels

The main purpose of the entire design is to test whether the air vent valve based on electrowetting technology works. So the whole device is mainly composed of four parts: water storage part, water channel, air channel and electrodes. First the water in the tank will flow into the trench due to capillarity and stop in front of the hydrophobic area. When the electrode under this hydrophobic area is activated, the surface change into hydrophilic and the water flow continues to flow forward. When the electrode is deactivated, the surface becomes hydrophobic again. But due to the existing of the air channel, the surface tension is expected to force the liquid from the electrode area. The overall schematic diagram of the first design is shown in Figure 3.1.

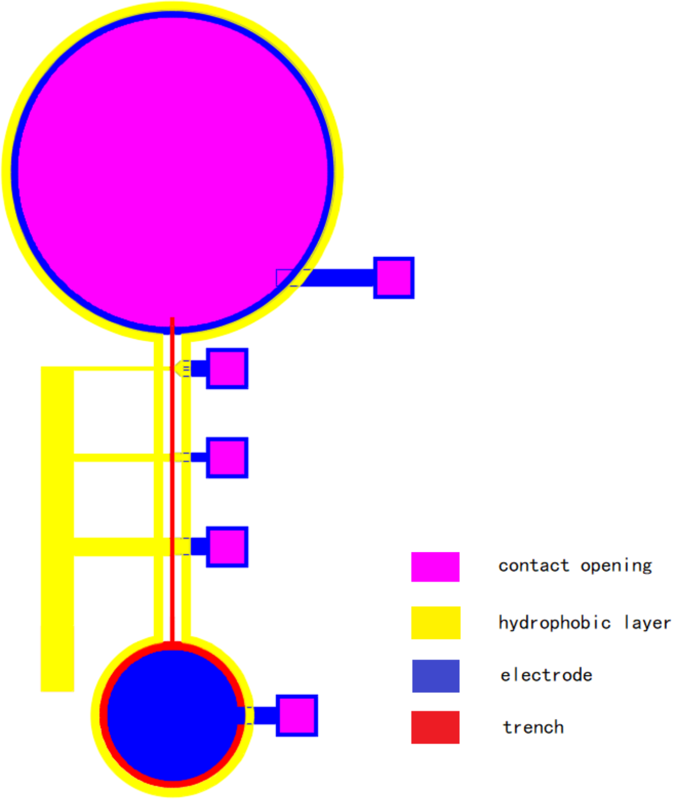


Figure 3.1 The schematic of the first design

As can be seen from Figure 3.1, there are two circular portions which are designed for water storage. They are both surrounded by a hydrophobic layer to prevent the droplets from spreading over the surface. The top one has a contact opening that is shown in the pink color because the water needs to be connected to the ground. The red channel that links the two water storage sections is the trench. As a precaution, it is surrounded by a hydrophobic layer. The width of the trench is 20um. The blue ones are the electrodes, they also have the pink contact openings where we can apply the voltage. Because the insulating layer is deposited on the entire wafer surface, there is no need for a specific design. The contact openings are in the pink color.

As we can see from Figure 3.2, the trench is marked in red and air channel is in green. In order to prevent the channel is blocked by some reason, a huge vertical (the vertical just looks from the Figure 3.2 in green color) channel on the left to increase the area of contact with air. And of course the whole air channel is covered by the hydrophobic layer to keep out the aqueous fluid. The electrodes are deposited on the trench and connected to the contacting area. The part deposited on the trench is covered by the insulating layer and hydrophobic material so the fluid flow can be controlled by applying the voltage.

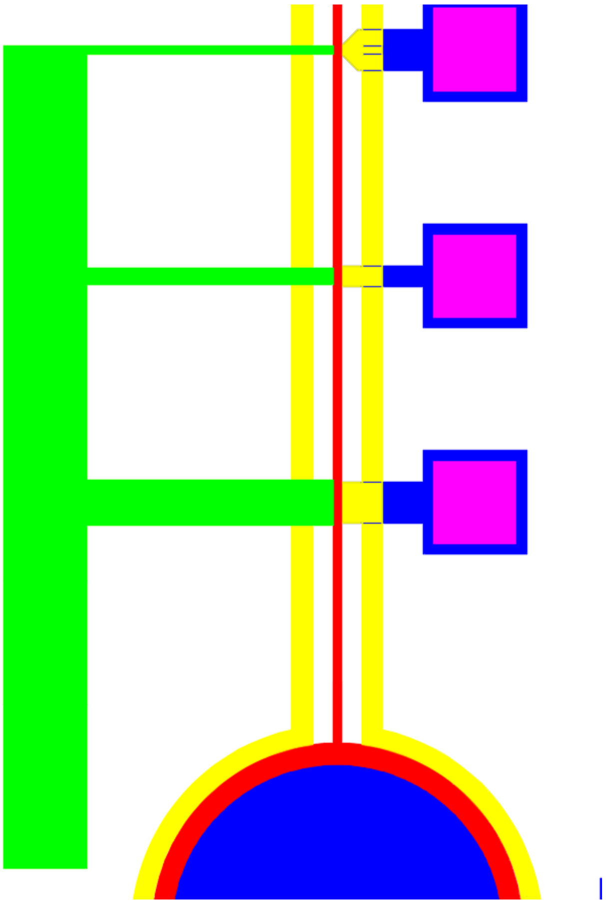


Figure 3.2 The specific design for trench and air channel

As mentioned before, the fluid should stop at the edge of the hydrophobic layer. When proper voltage is applied on the electrode, the hydrophobic surface changes to hydrophilic and the fluid continues to move forward. When the voltage is off, the fluid behind the electrode stops again. Because of the presence of the air channel, liquid will withdraw from the electrode area. The portion of the fluid which has already passed the electrode can continue to flow.

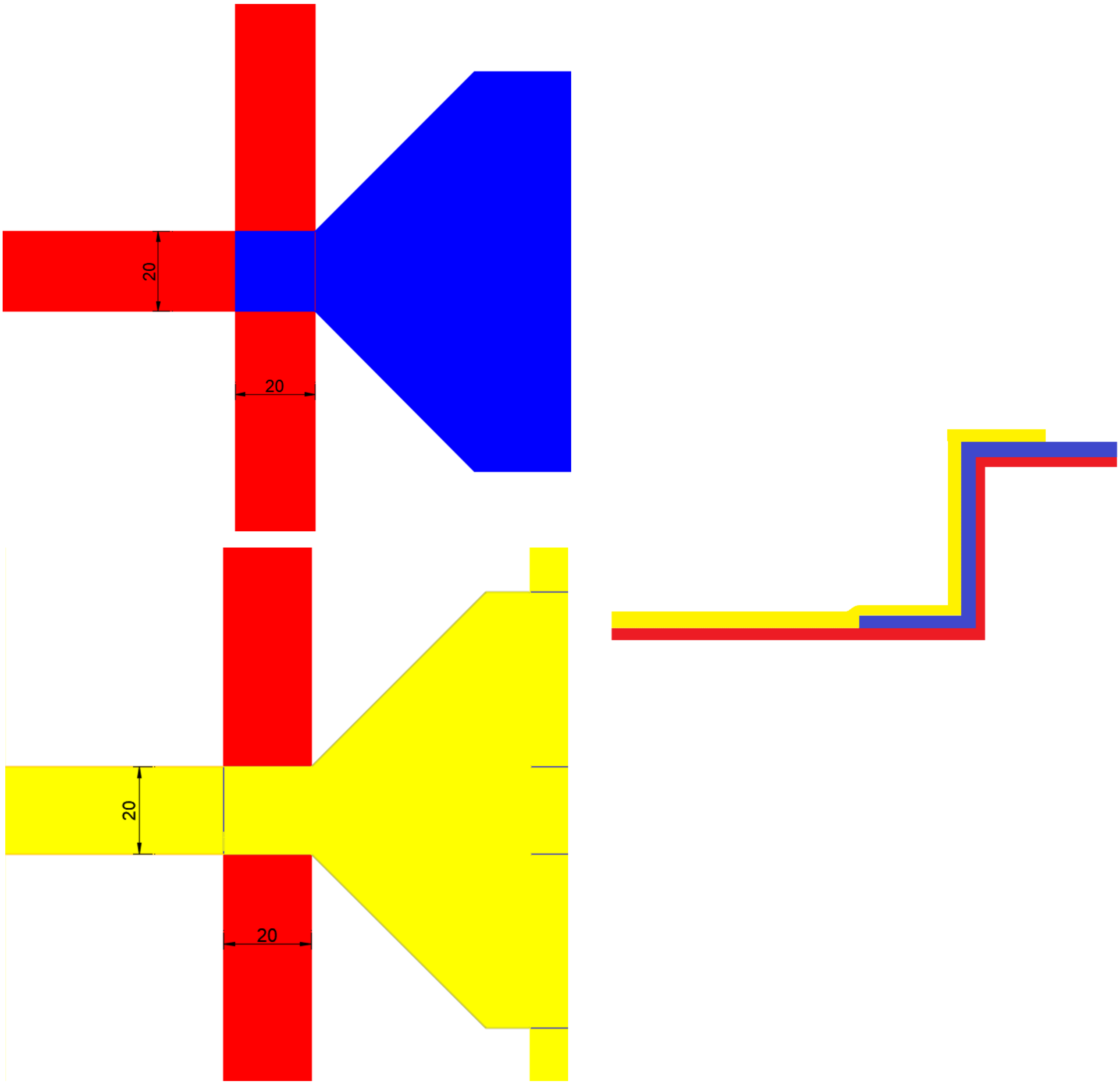


Figure 3.3 Specific design 1: trench (vertical red), air channel (horizontal red), electrode (blue) and hydrophobic layer (yellow) and the relationship between them

So, three different lengths of electrodes were designed to test the behavior of the fluid. On the left side of each electrode is an air channel covered with a hydrophobic layer. The width of the air channel is slightly shorter, longer or equal to the length of the electrodes. So that it is possible to observe whether the air channels of different lengths have an effect on the flow of the fluid. The specific design of the electrode and air channel section can be seen in Figure 3.3, Figure 3.4 and Figure 3.5. It should be noted that the vertical direction of the trench is continuous. That part is covered by the blue electrode and yellow hydrophobic material. All units are microns.

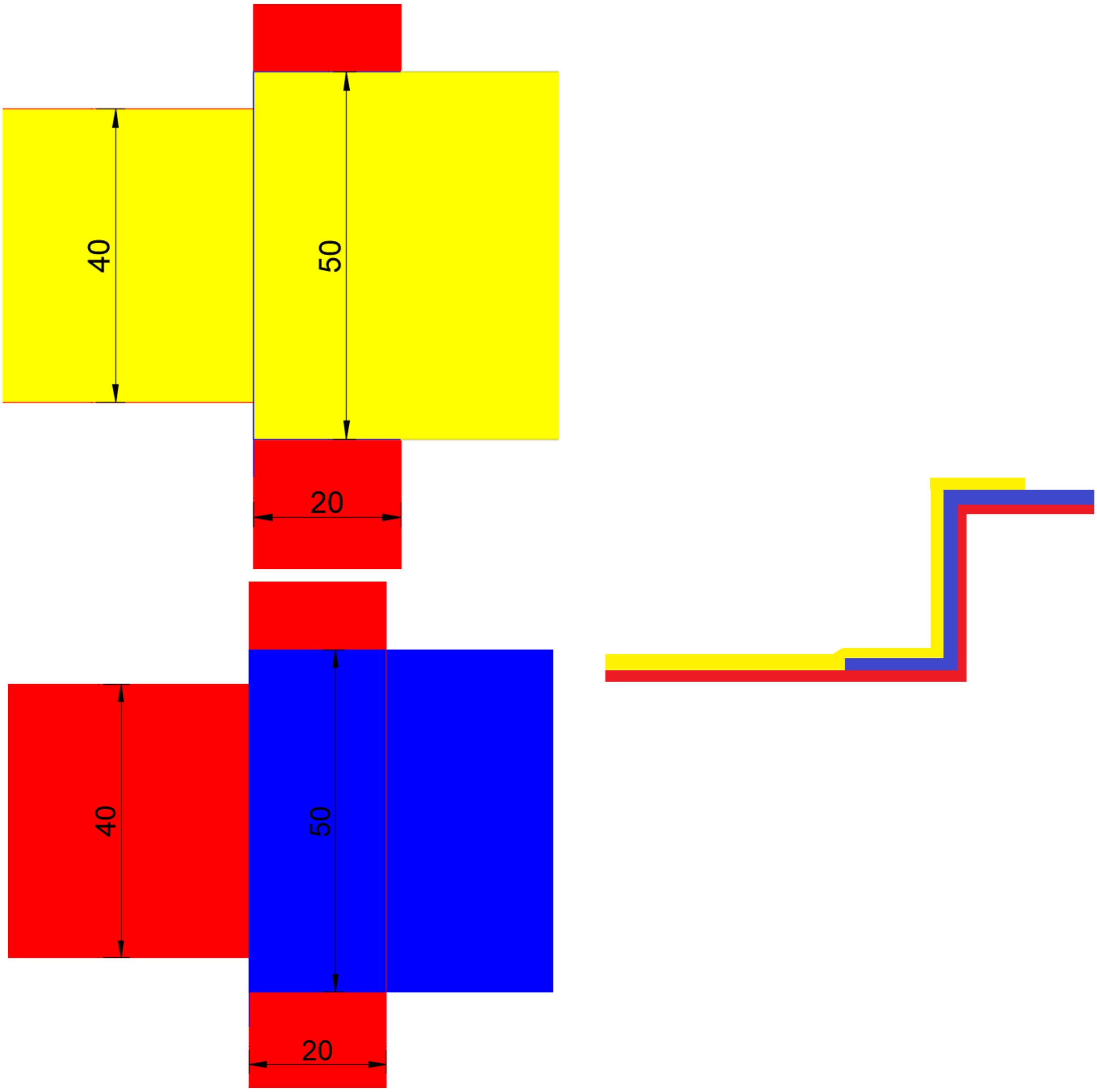


Figure 3.4 Specific design 2: trench (vertical red), air channel (horizontal red), electrode (blue) and hydrophobic layer (yellow) and the relationship between them

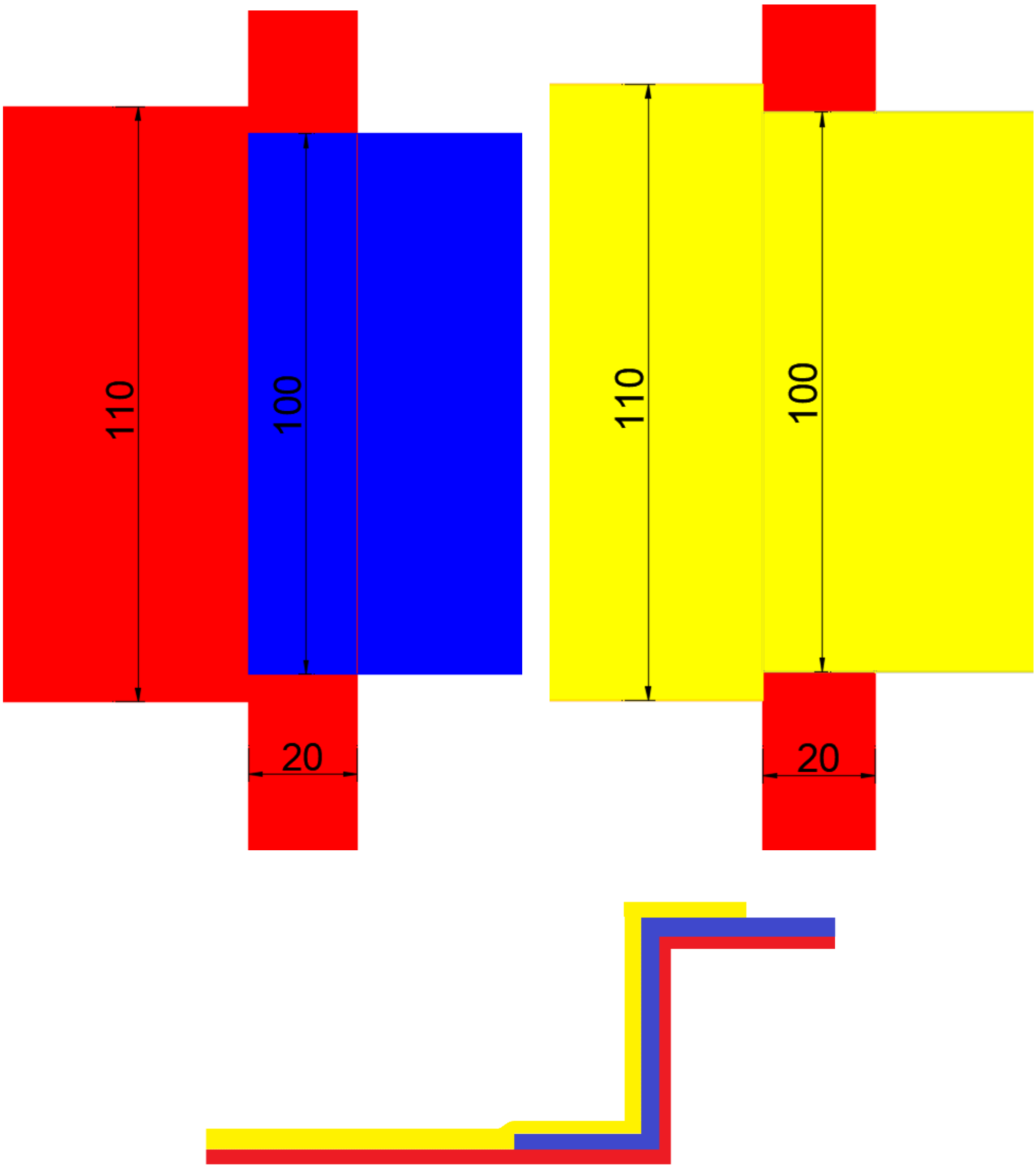


Figure 3.5 Specific design 3: trench (vertical red), air channel (horizontal red), electrode (blue) and hydrophobic layer (yellow) and the relationship between them

It is a special design that was made different length of electrodes and air channels. Because when the voltage disappears, the hydrophilic surface above the electrodes change to hydrophobic immediately. There is still water remaining on the surface. Since it is difficult to exactly predict how the fluid flow will behave during retreat from the electrodes, three different combinations of electrodes and air channels were designed. After the equipment is manufactured, tests will show how much the fluid remains on the electrodes after the voltage disappears.

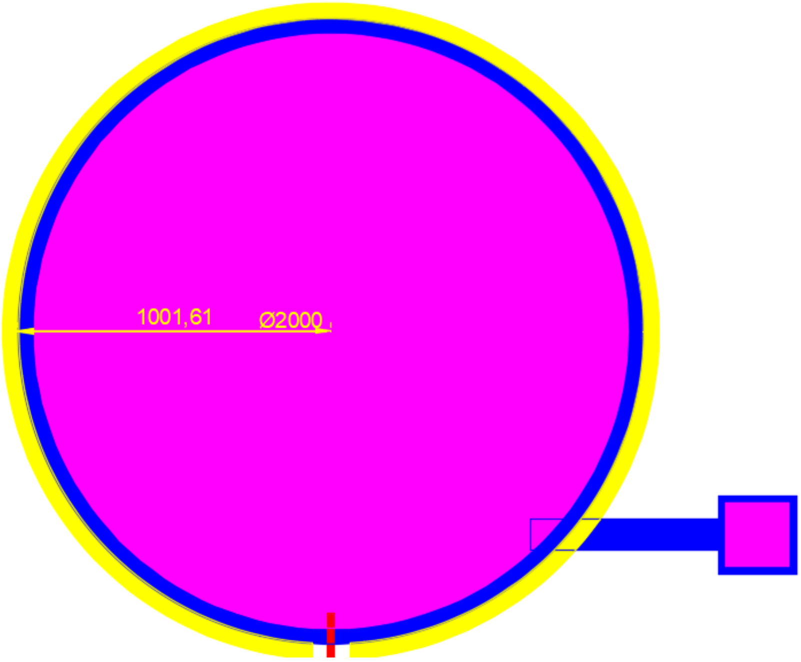


Figure 3.6 Design of the water storage part

As can be seen from Figure 3.6, the diameter of the electrode is 2000um. The outer periphery of the electrode is surrounded by a hydrophobic layer with a width of 50 microns to prevent liquid outflow. The upper layer of the electrode is etched with a contact opening. So the fluid in this area will have a good contact with the electrode that connects to the ground.

3.2 Oblique long electrodes

The main purpose of the entire design is to test whether the air vent valve based on electrowetting technology works. So the whole device is mainly composed of four parts: water storage part, water channel, air channels and electrodes. The overall schematic diagram is shown in Figure 3.7.

As can be seen from Figure 3.7, the overall structure design is roughly the same compared with design 1. Two water storage portions are both surrounded by a hydrophobic layer to prevent the droplets from spreading over the insulating hydrophilic surface. The difference compared with the first design is both of the storage portions have contact opening showing in the pink color. The reason is that this design would like to test two-way flow. The red channel that links the two water storage sections is the trench. As a precaution, it is also surrounded by a hydrophobic layer. The width of the trench is 100um. The blue areas are the electrodes. They also have the pink contact openings where we can apply the voltage. Green parts are the air channels. Details are underneath.

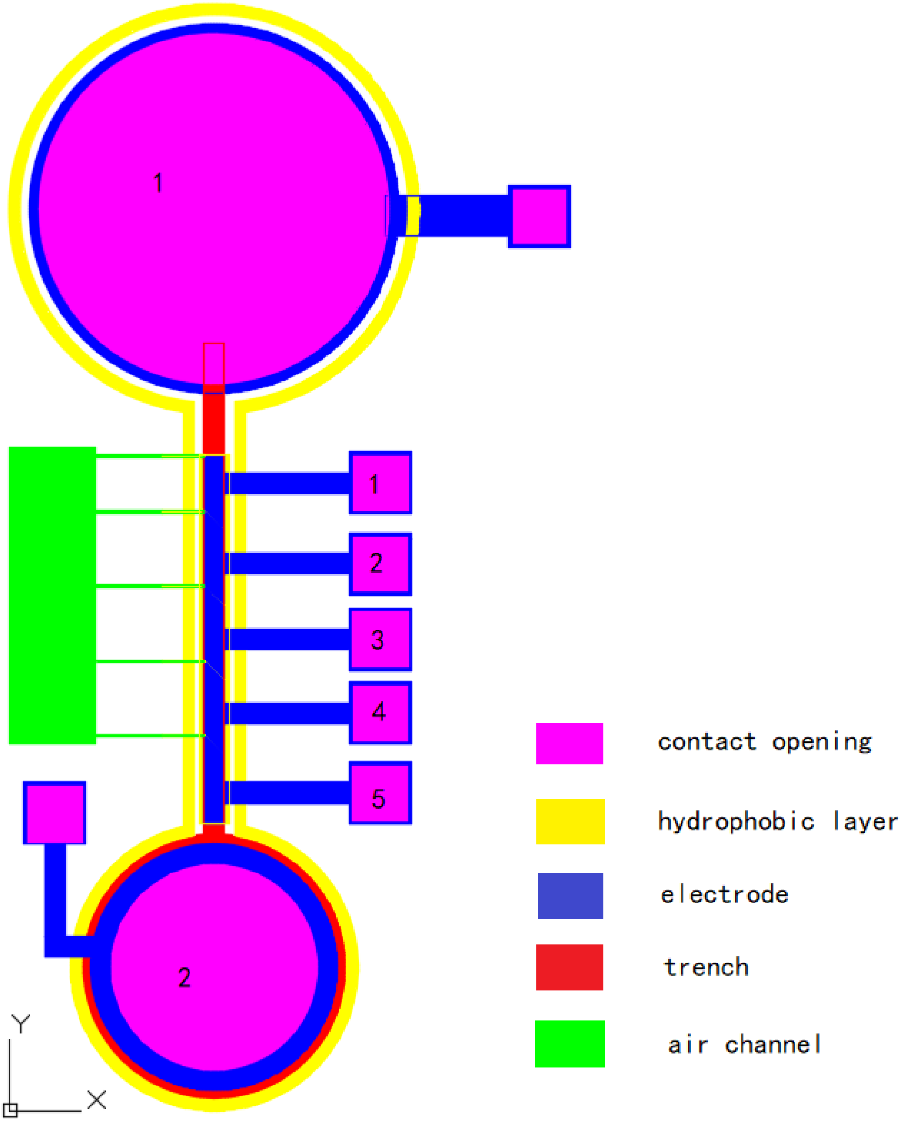


Figure 3.7 The schematic of design 2

As we can see from Figure 3.8, the trench is marked in red and air channel is in green. In order to prevent the channel from blocking, a huge vertical channel on the left to increase the area of contact with air. The air channel near the trench section is covered with a hydrophobic layer to prevent the fluid flowing in. The special designed oblique electrodes are deposited on the trench and connected with the contacting area. The first part also overlapped by the insulating layer and hydrophobic material to control the fluid flow by applying the voltage. Each air channel is designed in the upper left corner of the electrode.

As mentioned before, the fluid is meant to stop at the edge of the hydrophobic layer. In this case, the whole electrodes are all covered by the hydrophobic layer even the gap between the electrodes. When proper voltage is applied on the electrode, the hydrophobic surface changes to hydrophilic and the fluid continues to move forward. It stops at the edge of this electrode. When proper voltage is applied on the next electrode, the fluid continues to flow even when there is a gap.

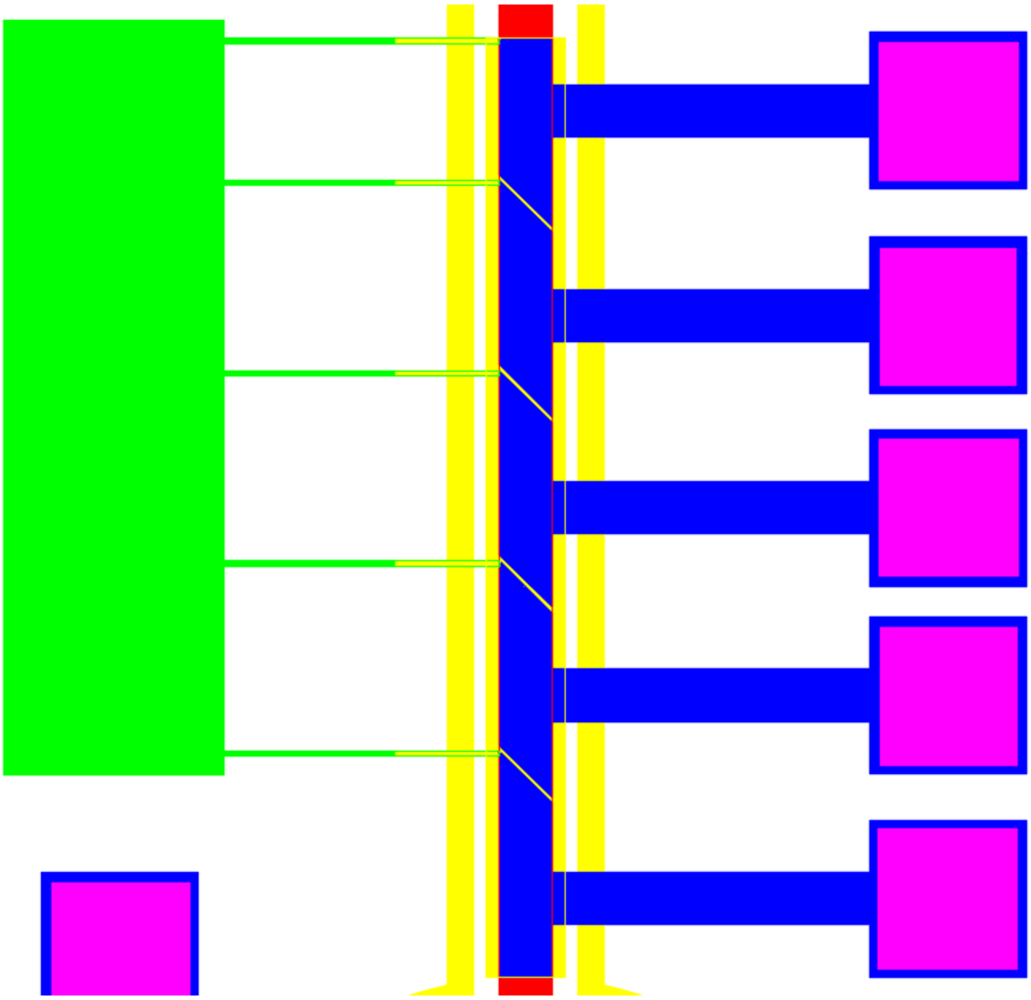


Figure 3.8 The schematic diagram of electrodes, hydrophobic layer and air channel

In Figure 3.9 and Figure 3.10, the design of all electrodes and air channels is presented. On the upper left corner of each electrode the air channel is connected. The width of all air channels are 10 micron. It is possible to continuously observe the motion of the fluid on each electrode after the voltage is switched off. It should be noted that the vertical direction (in the figures) of the trench is continuous. That part is covered by the blue electrodes. And according to the theory of electrowetting, all electrodes are covered by the insulating layer and yellow hydrophobic material. All dimensions are in microns.

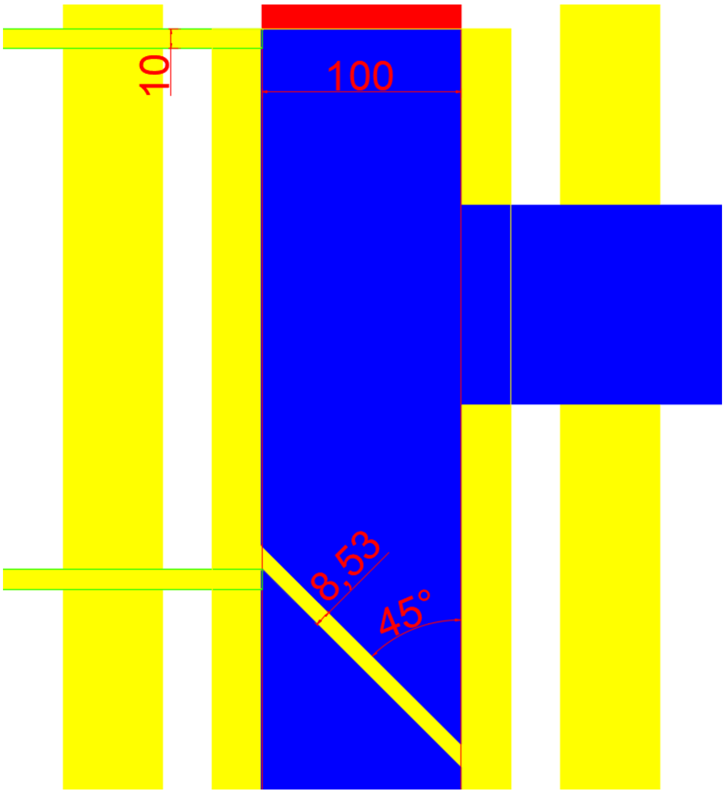


Figure 3.9 The specific design of electrodes, hydrophobic layer and air channel

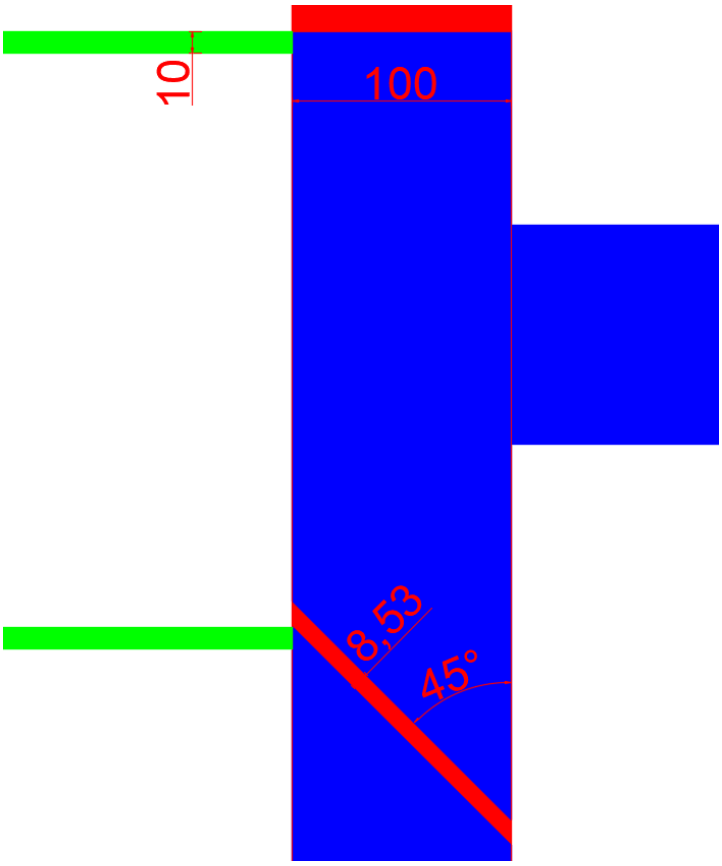


Figure 3.10 The specific design of electrodes and air channel

The water storage portion 2 also has the pink contact opening as shown in the Figure 3.7. This is because the fluid motion from water tank 2 to 1 is also something needs to know. After the electrode 5 is powered off, it will immediately become hydrophobic. But there is still water remaining on it. The portion of the fluid that has flowed through the electrode 5 will continue to flow forward. Because the air channel is hydrophobic and will provide the pressure the flowing needs. However, the design can function both as a valve and as a precise metering system, so the motion of the fluid remaining on the electrode is something needs to be find out. That's why several electrodes are designed to make sure what the measuring is accurate.

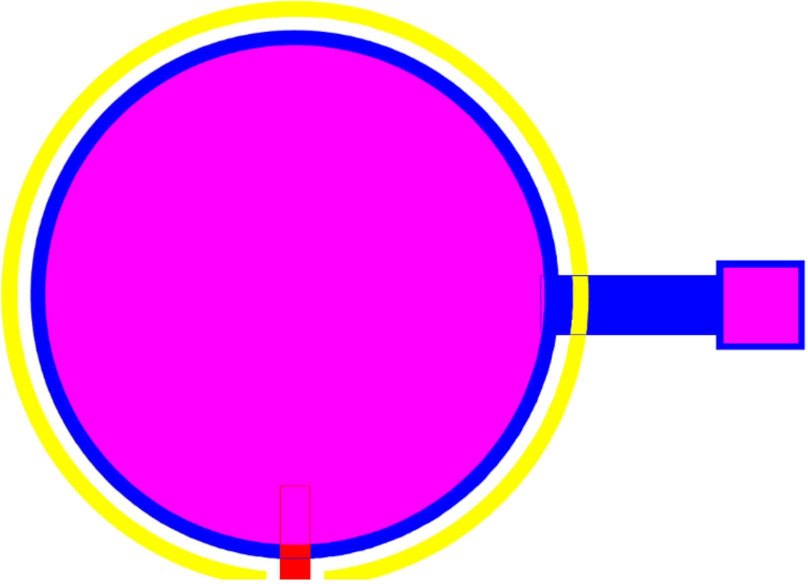


Figure 3.11 The specific design of top water storage portion and contact opening

From Figure 3.11, the diameter of the big circle electrode (blue one) is 900 micron. The outer periphery of the electrode is surrounded by a hydrophobic layer with a width of 50 microns to prevent liquid outflow on the hydrophilic surface. The upper layer of the electrode is etched with a contact opening (pink). So the fluid in this area will have a good contact with the electrode which connects to the ground. The opening is smaller than the designed electrode. This is to make sure the contact openings are all efficient ones.

3.3 Mask design

All the devices will be made on top of the silicon wafer. However, due to its small size, it is necessary to use a microfabrication process. So, one of the most efficient and reliable techniques to allow for very precise engraving of very small features is lithography. There will be a complete explanation in the manufacturing section (Chapter 4). It is important here to know that such a process requires the use of a mask through which the portion of the target is selectively exposed to ultraviolet light. However, the shape of the mask defines the contour to be printed. In addition, since the photoresist used in the cleanroom could be positive or negative according to the different processes, the part transferred into the mask is designed to be transparent or non-transparent. With this in mind, the masks are designed according to the different processes and solve the problems encountered.

There are different precision in the mask printing technology. In out cleanroom EKL, the minimum resolution is 5 micron. And in my design, the smallest gap showing in Figure 3.10 is 8.53 microns. So there is a safe margin and avoid any problems in this regard.

After determining the design and all the requirements in designing the mask, the designs need to be converted to the mask in order to fabricate the device in the cleanroom. The entire device consists of five layers, namely: trench layer, electrode layer, insulating layer, hydrophobic layer and contact opening. The trench layer includes channels for liquid flow and air channels. Because the entire device is fabricated on a silicon wafer, the micron-level depth of the channels are obtained by etching silicon. For the insulation layer, the silicon nitride or aluminum oxide is deposited on the entire wafer. So for this step, no mask is needed. In the contact opening step, the portion of the insulating layer where the voltage is applied to the electrode is etched away.

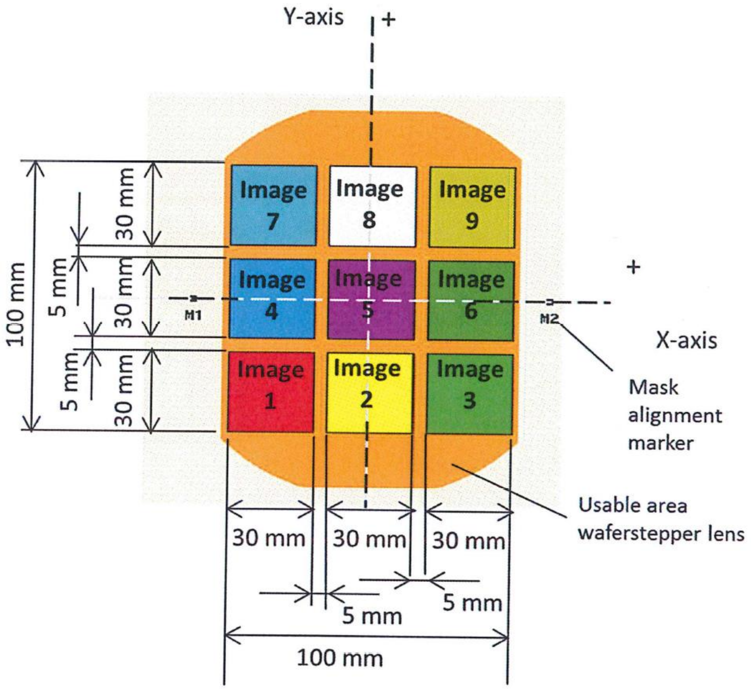


Figure 3.12 Mask layout, the dimensions are on the mask level

Figure 3.12 shows the mask layout. The dimension is in the mask level which is 5 times larger than the actual size in the designs. The reason why it is designed like this will be explained in the next chapter. From Figure 3.13, number 1-4 are the masks of design 2 and number 5-8 are the masks of design 1. The number 5 to 8 are trench, electrodes, contact opening and hydrophobic layer, respectively. The number 1 to 4 are also trench, electrodes, contact opening and hydrophobic layer, respectively. It should be noted that my project is in conjunction with someone else's project, so not all things in the picture are my design. And why I designed some of the parts solid and some parts designed into a hollow I will detail in chapter 5. This is a choice in fabrication.

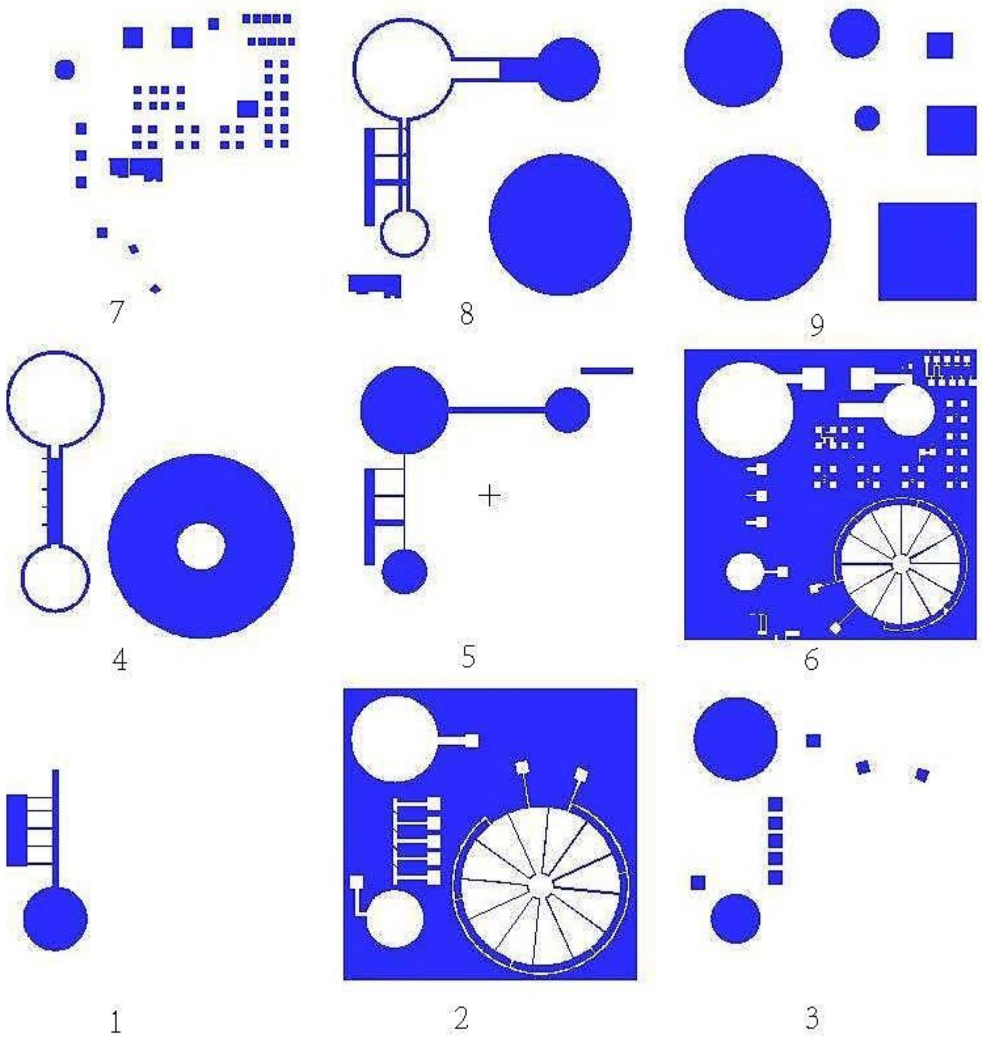


Figure 3.13 The overall diagram of mask

Chapter 4. Fabrication Process

In the previous chapters, the principle of electrowetting and design ideas are explained. In this chapter an overview of the silicon microfabrication technique and the fabrication flow are presented.

4.1 The fabrication method

In order to fabricate the device using silicon microfabrication techniques, there are some objectives:

- The fabrication of the micro channels must be accurate and the side walls should be vertical.
- The metal on the side walls must be uniformly deposited.
- The pattern of the metal inside the channel must be intact.
- The hydrophobic layer should have a uniform thickness, also on the sidewalls.

A schematic of the steps of the fabrication process is presented in Figure 4.1. First a 2000 nm thick silicon oxide layer is deposited on the silicon wafer. Then in the areas where the trenches and the air channel need to be fabricated, the silicon oxide layer is etched by plasma etching. After that, the trenches and the air channel are etched into the silicon using reactive ion etching (RIE). Meanwhile, the remaining silicon oxide layer is used as a mask layer to prevent the silicon outside the trenches and air channel to be etched. The mask layer of SiO_2 is then removed by wet etching using BHF leaving the trenches and the air channel on the wafer. In order to prevent the direct contacting of the silicon and the electrodes, a 500 nm insulating SiO_2 layer is added on the wafer. After that 200 nm thick metal is deposited and patterned on the SiO_2. Then the whole wafer is covered by an insulating layer of SiN_x or Al_2O_3. Then the contact openings are etched after which the hydrophobic layer is deposited and patterned.

The advantage of this microfabrication method is that the various features of the same portion in the design can be included in one single image of the mask to be fabricated. For example, the depth of trench depends on the time of etching but the shapes are all the same. So there is no need to design different masks for different depths of trenches.

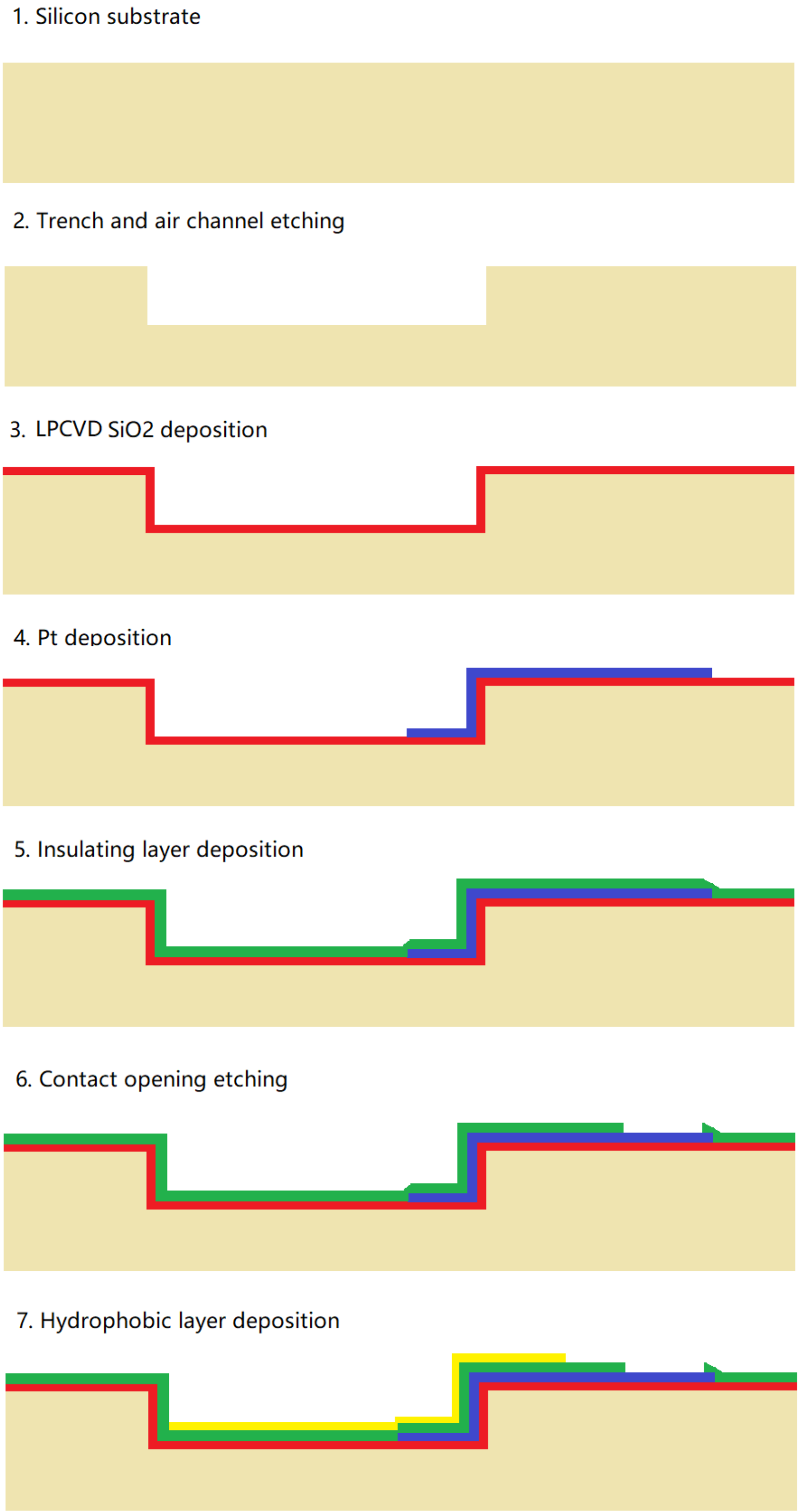


Figure 4.1 The fabrication process flow

4.2 Fabrication process flow

4.2.1 Cleaning Wafers

The very first step after opening a box carrier containing a new batch of silicon wafers, is cleaning the wafers before the start of the process. This to remove any contamination and to clean the wafer. Because the surface of the wafers could be contaminated or is not clean enough after being kept in the plastic box carrier for some time. A process named 'cleaning line' is needed for the cleaning. Figure 4.2 depicts an overview of the cleaning process.

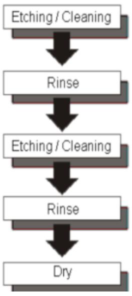


Figure 4.2
Cleaning step

4.2.2 Zero Layer

At the beginning of the whole process, the first processing step to a wafer is the zero layer. This includes the alignment marker and wafer ID. The alignment marker is a necessity during the photolithography process to make sure the wafer is exposed in the right position. Wafer ID is to ensure that each piece of wafer is numbered to prevent the mess.

4.2.3 PECVD Silicon Oxide Deposition

After the cleaning process, a layer of SiO_2 with a thickness of 2000 nm is deposited on the wafer. It is used as a mask layer for the process of silicon etching. The method chosen for SiO_2 was Plasma Enhanced Chemical Vapor Deposition (PECVD). At 400 °C the deposition parameters have been optimized to deposit a flat oxide layer without pinholes. The reason why choosing such a thickness is that it's important to protect the rest of the silicon from being etched during the trench etching process. The biggest depth of the trench is 50 μm and the selectivity between silicon and silicon oxide is 100: 1. To ensure that no silicon other than the trench is etched, the thickness is selected as 2000 nm. And the deposition time is 23.5 seconds. This is why the PECVD process is selected. Comparing to a few hours of LPCVD processing time, 23.5 seconds is much faster.

4.2.4 Coating, Lithography and Development

A layer of positive photoresist is deposited on the silicon oxide. After baking, the patterns defining the trenches are transferred from the photomask to the wafer with lithography. Photoresist is exposed to the patterns using a wafer stepper. The photoresist is then developed and the areas that were exposed are dissolved in the developer and removed while non-exposed areas remain and the pattern is transferred as can be seen in Figure 4.3.

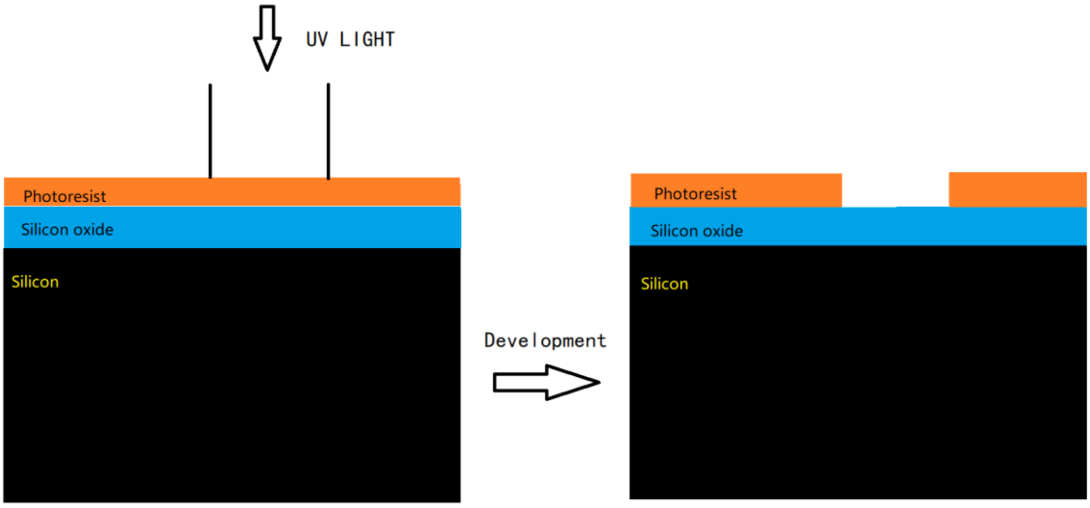


Figure 4.3 Wafer in lithography process and after development

Spin coating is a procedure used to deposit uniform thin films to flat substrates. Usually a small amount of coating material is applied on the center of the substrate, which is either spinning at low speed or not spinning at all. The substrate is then rotated at high speed in order to spread the coating material by centrifugal force. The coating and developing machine used in EKL cleanroom is EVG 120 coater and developer. The spinning process is shown in Figure 4.4.

Figure 4.4 Coating photoresist using EVG 120 [1]

Rotation is continued while the fluid spins off the edges of the substrate, until the desired thickness (usually 1-3 um) of the film is achieved. The applied solvent is usually volatile, and simultaneously evaporates. So, the higher the angular speed of spinning, the thinner the film. The thickness of the film also depends on the viscosity and concentration of the solution and the solvent. Usually, the thickness of photoresist should be taken into consideration because of the etching and exposure resolution. The smallest patterns were 5 um. The photoresist used could not be too thick as thicker layers will limit the resolution. And because of the development process, the photoresist cannot be too thin either. In this case a thickness of 2.1 μm was chosen.

After the prebaking, the photoresist is exposed to a pattern of intense light in the lithography machine. The exposure to light causes a chemical change that allows some of the photoresist to be removed by a special solution, called ‘developer’. Positive photoresist, the most common type, becomes soluble in the developer when exposed; with negative photoresist, unexposed regions are soluble in the developer. After the development process, the required pattern appears on the wafer. The whole process is shown in the first four steps of Figure 4.6.

The lithography machine used in the EKL cleanroom is ASML PAS5500/80 waferstepper. The photomask is located above a high resolution lens. The Cr-side of the mask is facing down to the lens and the features on the mask are imaged through the lens on to the wafer. Obviously, all images on the mask should be placed in the waferstepper’s available image area, which is 105*105 mm^2 on mask level. The reduction factor of the lens is 5, so the pattern on the mask is 5 times larger than the pattern on the wafer. In addition, due to the requirement of EKL, the images should be separated by a distance of 5 mm. This means that nine 30*30 mm^2 images can be accommodated in the waferstepper’s image area. See Figure 4.5 [2].

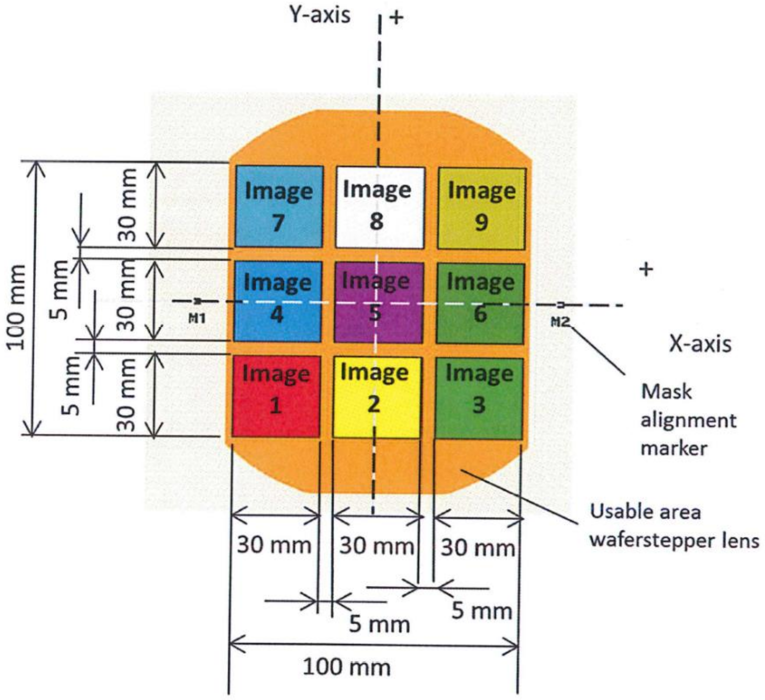


Figure 4.5 Mask layout

4.2.5 Plasma Etching Silicon Oxide

After the patterns are exposed and developed, the exposed areas of silicon oxide are plasma etched. The process pressure applied is 180 mTorr and the RF power is 300 W. The gasses & flows environment of C2F6/CHF3 is 36/144 sccm and the He pressure is 12 Torr. To ensure that all silicon oxide is etched away, overetch is required. That means that some of the silicon on the trench location is also etched. After the etching, the height difference between the remaining silicon oxide and the trench portion was measured. By comparing with the remaining thickness of the silicon oxide layer, it shows how much of the silicon in the trench is removed by the etching process. The average thickness of silicon oxide layer left is 1600 nm and the height difference is 1660 nm. So the silicon is etched about 60 nm which can be neglected compared to the compared to the 20 um depth of the trenches. The process is shown in the last 3 steps of Figure 4.6 [3].

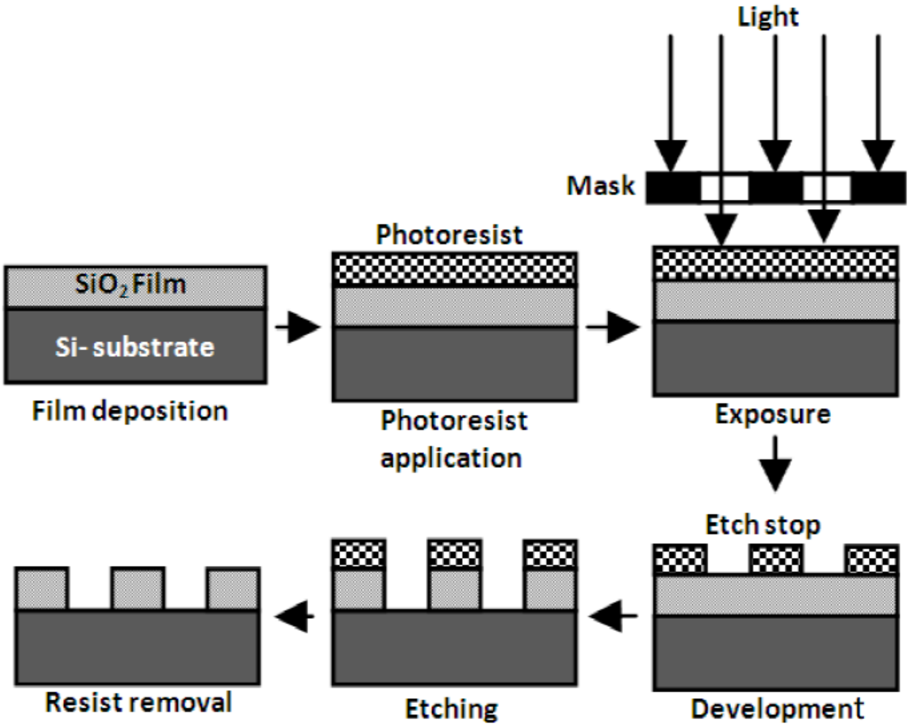


Figure 4.6 Coating, lithography, development and plasma etching process

4.2.6 Deep Reactive Ion Etching (DRIE)

After the silicon oxide is patterned, the exposed silicon is etched using plasma etching. The technology used is Deep Reactive Ion Etching (DRIE) using the Bosch process, where the SiO_2 layer serves as the mask layer. The selectivity between silicon and silicon oxide is high so there is no need to worry about the SiO2 layer being etched away.

Bosch Process: Deep etching with repeated cycles of etching and passivation. First the silicon is etched with a mix of SF6 and 10% O2, and then the sidewalls are passivated with C4F6. The cycle is then repeated. Longer cycles mean faster etchrate but bigger scallops on the sidewalls. The size of the opening in the mask directly influences the speed of DRIE. Very small openings in the mask result in a rapidly decreasing etch rate as the etch depth proceeds. In this case, the trench width is 100 μm and the depth is 20 μm. So there is no need to worry about this issue. The whole process is shown in Figure 4.7.

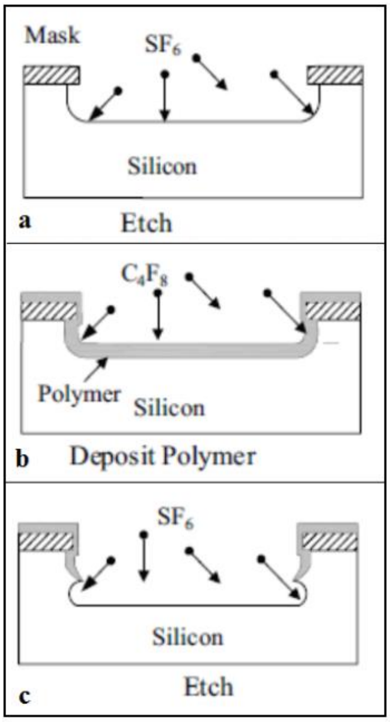


Figure 4.7 Bosch Process of DRIE [4]

4.2.7 TEOS Silicon Oxide Deposition

Before the metal deposition, a silicon oxide layer is deposited on the wafer to prevent the contact between metal and silicon. LPCVD TEOS is a common way of depositing silicon oxide. TEOS (tetraethylorthosilicate, $Si(OC_2H_5)_4$) is the source for silicon oxide and the reacting temperature is around 650°C to 750°C and it produces a relatively pure oxide [5].

The recipe used in the furnace of EKL is: TEOS bubbler (40℃); 250 mTorr pressure; 700 °C. After 1h 7min deposition, a 488 nm thick oxide layer was obtained.

4.2.8 Spray Coating

To pattern the metallization layer a lift-off process will be used. Which means that a photoresist and a lithography step have to be applied prior to metal deposition. Because of the existence of the trench, the wafer cannot be coated by spin coating. Otherwise this will lead to uneven distribution of photoresist in the trenches and cannot be fully developed. Instead of spin coating, spray coating is used. The spray method is to spray the photoresist from the edge of the wafer slowly to the middle. Meanwhile, the wafer is slowly rotating to make sure the photoresist is evenly coated on the surface of the wafer. The photoresist on the wafer is not as uniform as coated by spin coating, it is thinner at the edge and thicker at the center. However, this can be solved by over exposure during the photolithography process.

First the photoresist needs to be changed to the negative one because of the pattern in the mask. Then select recipe "HP-1000mbar-2mL-8 layers" on the user interface as shown in the Figure 4.8. After the pre-aligner falling down, put the wafer on the coat module and then the spraying module start to work. The recipe will form a 2-3 µm thickness photoresist layer after 8 times spray coating with 2 mL diluted nLOF 2070 negative photoresist. Once it is finished, the wafer needs a 15 minutes bake at 100°C. The next steps are photolithography and development. Because there is no existing recipe for spray coating with trenches, manual development is needed. The wafer is treated with "322 developer" in a beaker and shaken slowly. The photoresist gradually disappeared from the edge to center.

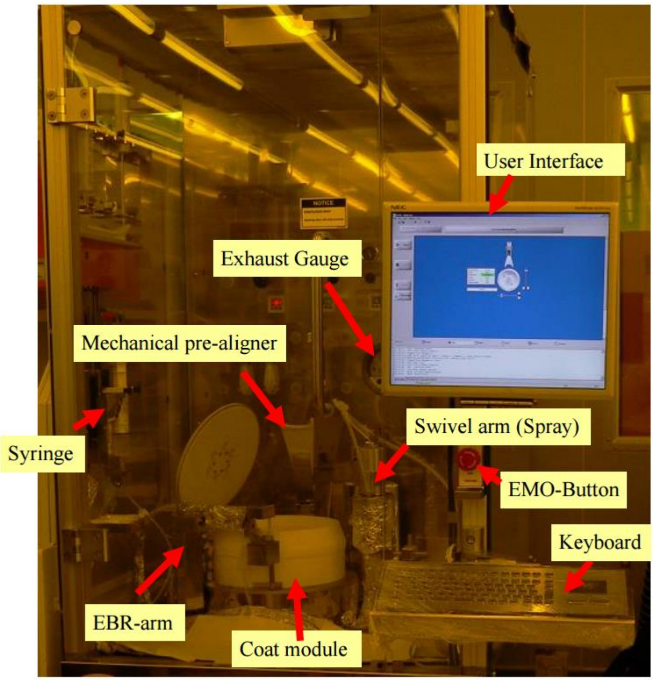


Figure 4.8 Spray Coater [6]

During the spray coating process, different thicknesses were tested. A triple deposition according the aforementioned standard recipe showed hard to develop. The structures on the edge disappeared during the development process while the photoresist in the middle was not removed. But during the process, it was found that 1 layer of spray coated photoresist is relatively uniform and pin-hole free. After development all structures were perfectly preserved.

4.2.9 Metal Evaporation

After definition of the metal pattern in photoresist, the metal is deposited on the wafer. The technology used in this process is metal evaporating deposition and the machine is CHA Evaporator. The CHA Evaporator is an electron beam metal evaporator with the capability of coating several wafers at the same time. It can control the metal deposition automatically with angstrom level accuracy.

Figure 4.9 and Figure 4.10[7] show the inside of the evaporator. The wafers are placed face down on the tray and secured. Once the program is started, it becomes highly vacuum. Then the source is heated and evaporation starts. The tray will be rotating to ensure uniform deposition. Because there is still photoresist on the wafer, the temperature inside the machine cannot be too high otherwise it will destabilize the photoresist on the wafer. So once the temperature reaches 80°C, the machine needs to be stopped by hand and cooled down to 60°C.

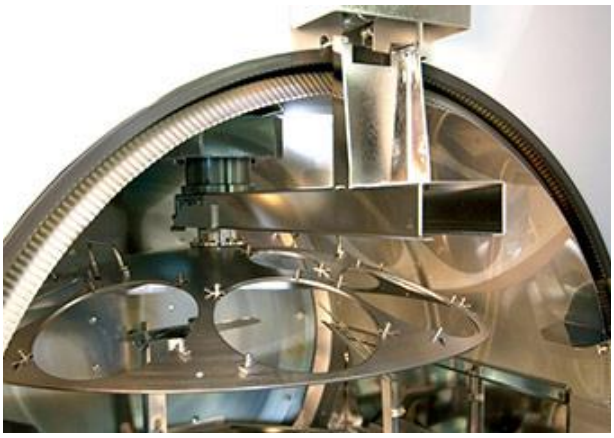


Figure 4.9 Wafer tray in CHA (face down)

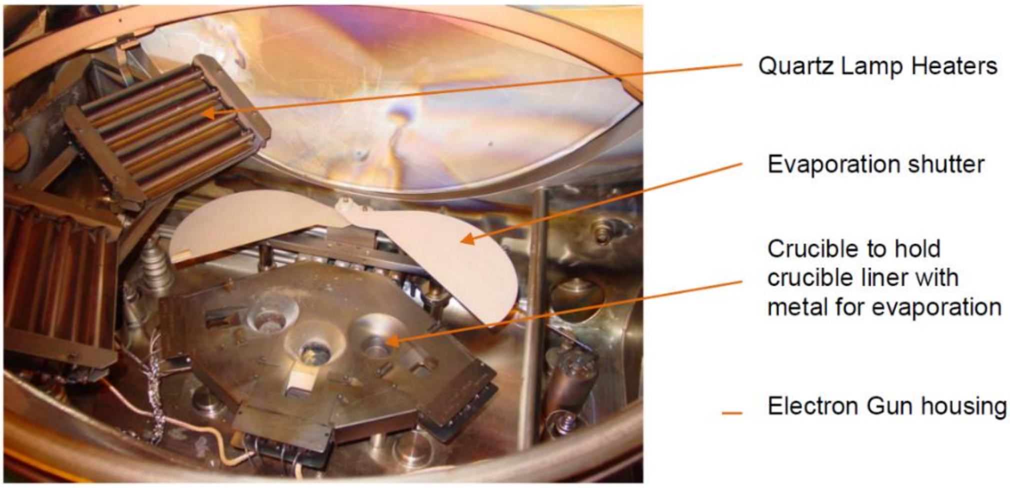


Figure 4.10 Source of metal and electron gun

The recipe for metal deposition is 20 nm Ta and 180 nm Pt. The adhesion of Pt to silicon oxide is not good enough, therefore, an intermediate adhesion layer is required. First Ti was used, but it showed to have insufficient adhesion leading to loss of metal pattern after lift-off, as shown in Figure 4.12. A 20 nm Ta adhesion layer under 180 nm Pt showed better performance. For the lift-off process the wafer was submersed in a beaker with 70°C NMP (a developer to dissolve the photoresist) until the photoresist was fully dissolved (see Figure 4.11). Then the wafer was dried and put into the Tepla machine to have an oxygen plasma treatment. After the metal deposition process, the wafer is contaminated and needs to put it into a special box.

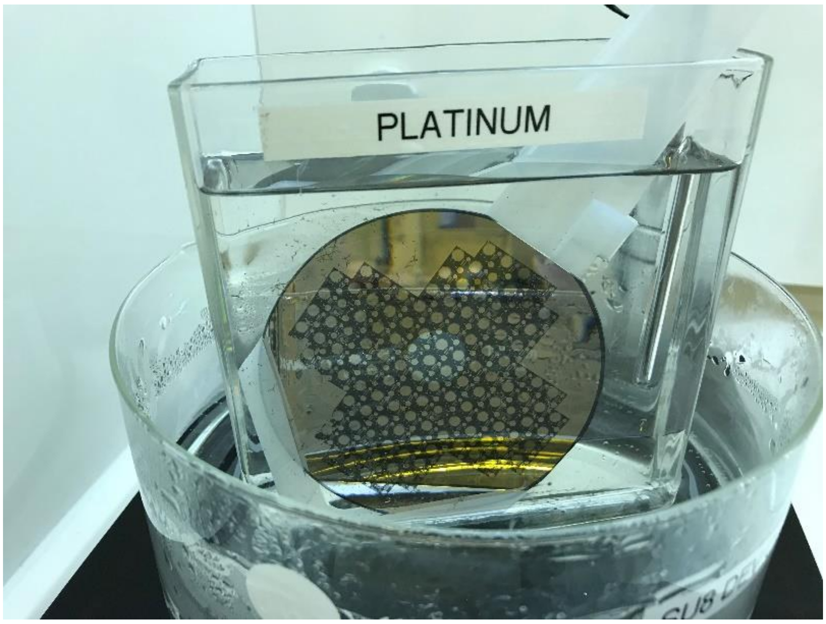


Figure 4.11 Lift-off process

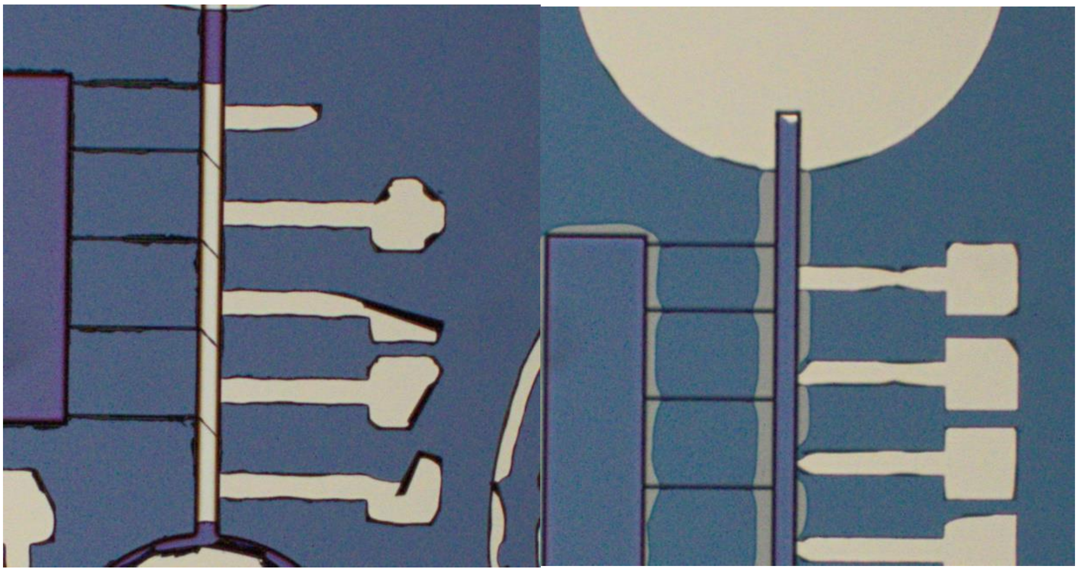


Figure 4.12 Loss of metal pattern after lift-off due to bad Ti adhesion.

4.2.10 PECVD Silicon Nitride

In this step, a thin silicon nitride layer is deposited on the wafer using PECVD. The purpose of this layer is as an insulating layer for electrowetting. The thickness is determined according to the experiment and the only difference for different thickness is the depositing time. At 400°C, the deposition parameters have been optimized to deposit a uniform silicon nitride layer without pin-holes. The recipe we use is as follows:

Gasses & flows: $N_2/SiH_4/NH_3$=100/280/1800 sccm.
Pressure: 2.8 Torr
HF power: 320W
LF power: 480W

The depositing rate is 30 nm/s which is known from the previous experiments. After calculation, if 100nm silicon nitride is needed, the deposition time is 3.2 seconds. By measuring the thickness of silicon nitride on the test wafer, the thickness of silicon nitride on the wafer which have structures on it is known and the thickness is 102 nm.

4.2.11 Hydrophobic Layer Deposition

Two hydrophobic materials are considered to create the hydrophobic top-layer: Parylene and FluoroPel.

Parylene is a common used hydrophobic material. It is deposited by vapor deposition in EKL MEMS lab. The machine shows in Figure 4.13. In order to get a thin layer, 1 gram Parylene is used and the thickness realized is 588 nm. This is the minimum thickness that can be achieved in EKL. Because the ideal thickness for the electrowetting device is less than 100 nm, it needs to be etched back by oxygen plasma etching.

Figure 4.13 Parylene deposition

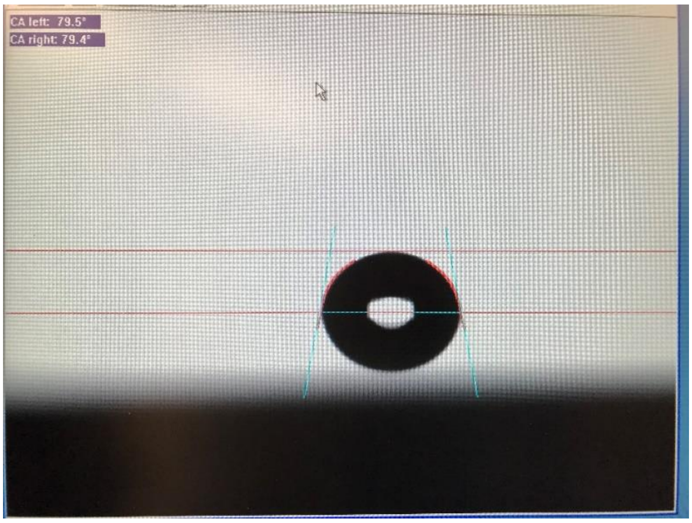


Figure 4.14 Contact angle of Parylene after deposition.

The contact angle before etching is shown in Figure 4.14. After the plasma etching, the surface of Parylene is ionized and loses its hydrophobicity with the contact angle of 79°. In fact it has become super hydrophilic and it is almost impossible to measure the contact angle. Then a special chemical of thiol end organic compound is mixed with Isopropyl Alcohol at the ratio of 1:100. The purpose is to form a molecular film on the surface of Parylene. But the result is not very satisfactory. Despite the improved hydrophobicity, the contact angle is still only 31 degrees. The result shows in Figure 4.15 and Figure 4.16. So the result of this experiment shows that a proper hydrophobic Parylene layer can only be achieved without back etching.

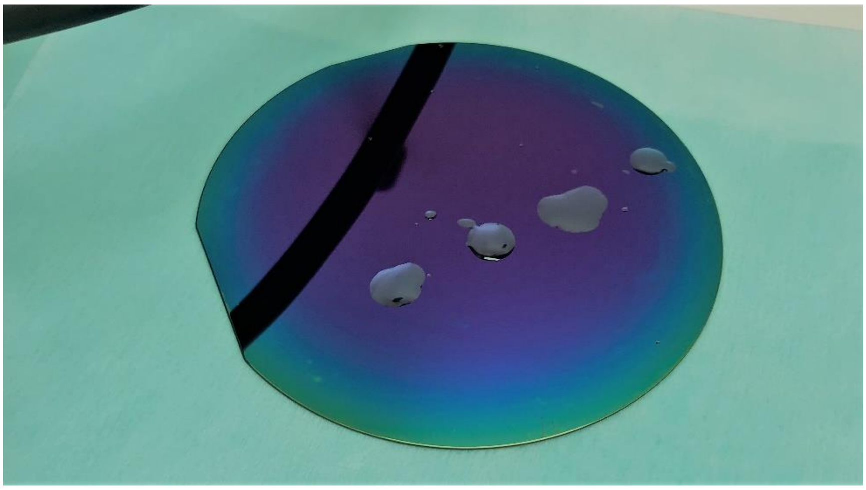


Figure 4.15 Droplet on the wafer after the treatment

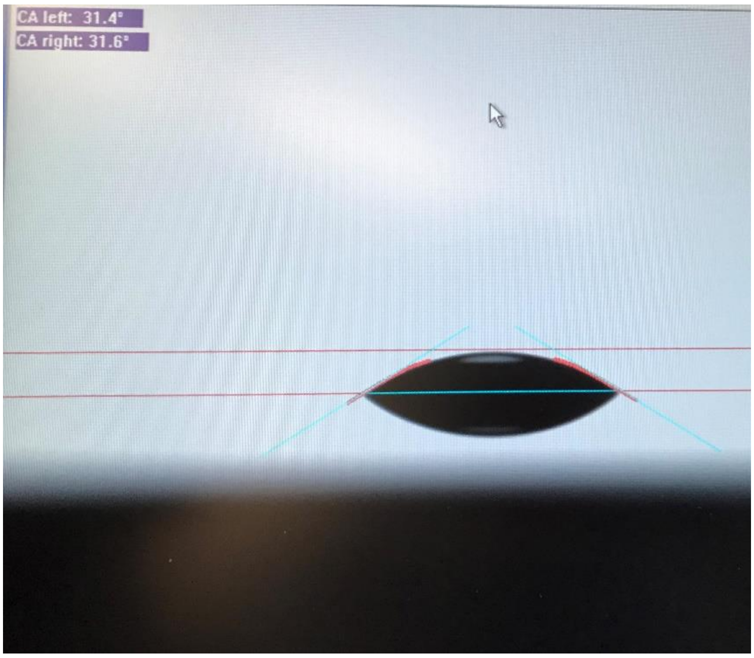


Figure 4.16 Contact angle after the treatment

Because what is needed is a hydrophobic layer, so another method is tried to make the surface hydrophobic again. From literature [8], a plasma treatment with CF_4 can result in a lager contact angle even if the surface has already changed into hydrophilic. The recipe is as follows: 20sccm CF_4; 20 W; 10 seconds. After the treatment, the contact angle change into 108° which is shown in Figure 4.17.

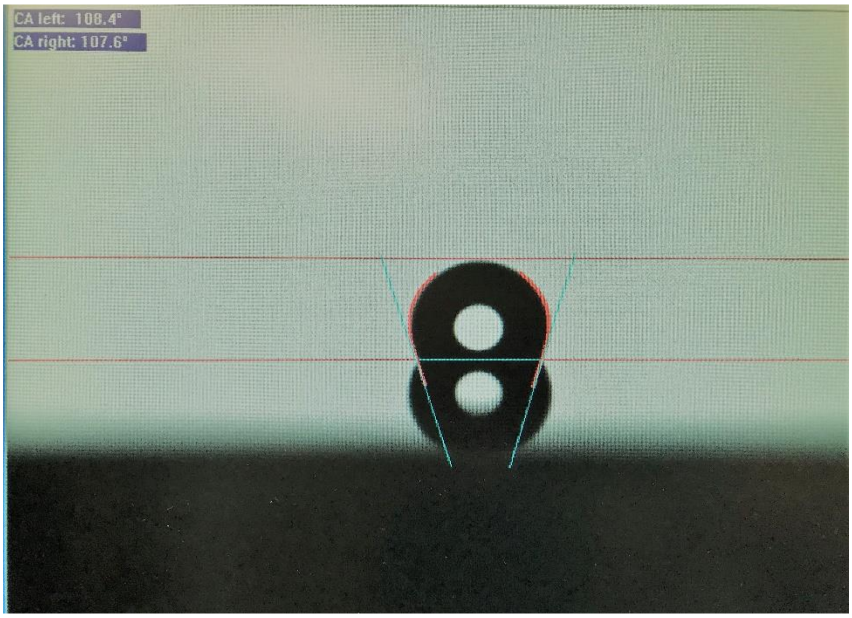


Figure 4.17 Contact angle of Parylene after CF4 treatment

Fluoropel is a new hydrophobic material fabricated by CYTONIX. It comes as a one percent fluoropolymer solution in a fluorosolvent (BP=160°C). The contact angle to water is 110 degree as shown in Figure 4.18. This is an ideally hydrophobic material for the experiment. The method of coating it on a wafer is: first let the wafer rotate slowly and drip the Fluoropel on the wafer; accelerate the rotation speed to 3000 rpm for 1 minute; put the wafer on the hot plate at 80 °C for 10 minutes and then 10 minutes at 160°C. The thin film dries in 4-6 hours at room temperature to a surface energy of about 16 dynes/cm. Heating up to 160°C for 30 minutes could optimize adhesion and reduce the surface energy to about 10 dynes/cm.

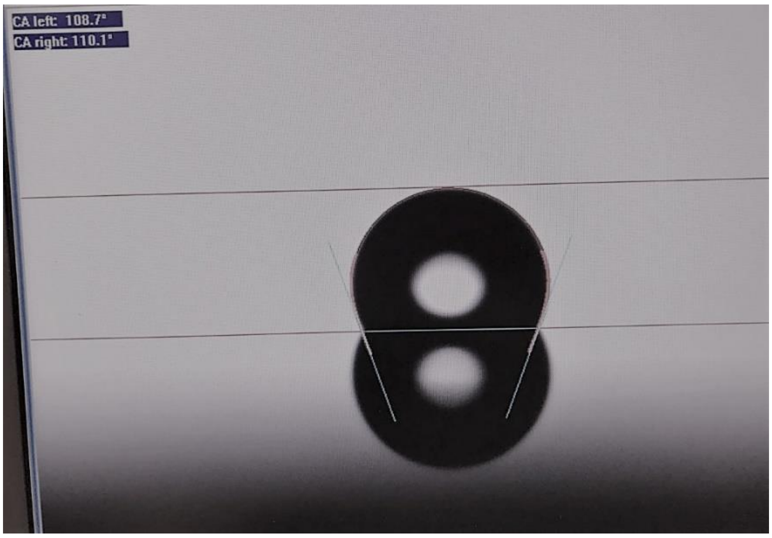


Figure 4.18 Contact angle to water

After the spin coating of Fluoropel, the wafer needs to be coated by negative photoresist. First, a standard spin coating process is tried and it turns out that there is no photoresist on the wafer because the Fluoropel is super hydrophobic. The negative photoresist does not stick to it. Then the spray coating negative photoresist method is tried. After coating with the recipe of "HP-1000mbar-2ml-8layers", the wafer is checked under a microscope. It is found out that there are a lot of pin-holes on the wafer as shown in the Figure 4.19. After 3 times of spray coating, the pin holes have basically disappeared except at the center. The wafer then went through photolithography and manual development to achieve the patterns that can be plasma etched.

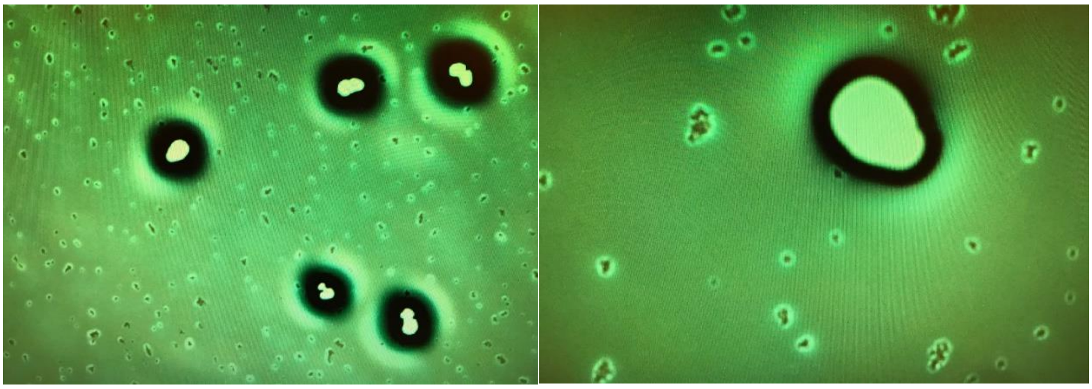


Figure 4.19 Surface of the wafer after one time spray coating

The machine used for plasma etching is Alcatel GIR300 F etcher. And the recipe is as follows:

O2: 70 sccm
Power: 60 W

It is an oxygen plasma etching. Because the other materials like SiN_x and Pt are not etched by this method, the wafer can stand over-etching to remove the Fluoropel completely. Then the rest of the photoresist on the wafer is removed by acetone. The remaining part, as can be seen from the Figure 4.20, is still hydrophobic. A lot of experiments were done before finding a feasible solution, but the results were not satisfactory. This is the best way that can be achieved in EKL using the spray coater and the result seems good.

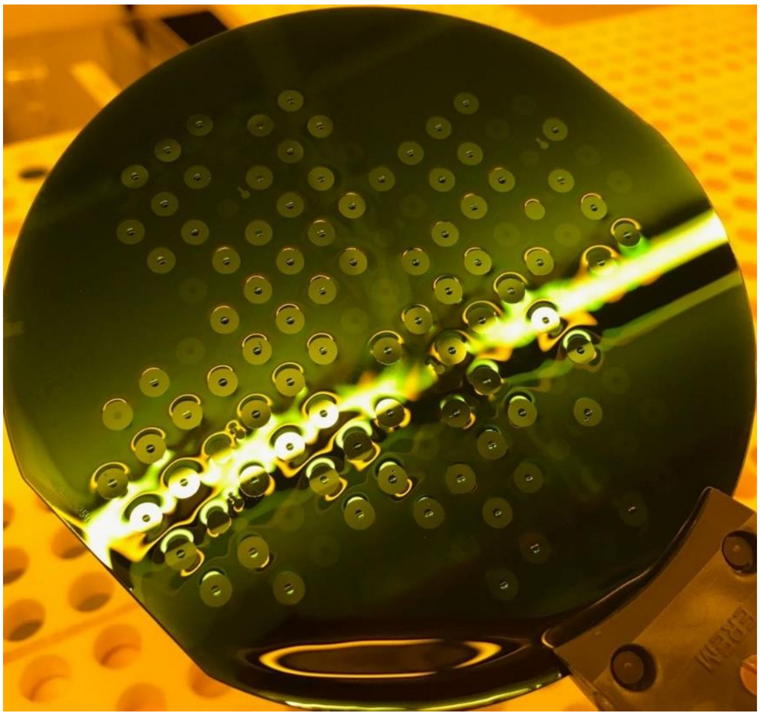
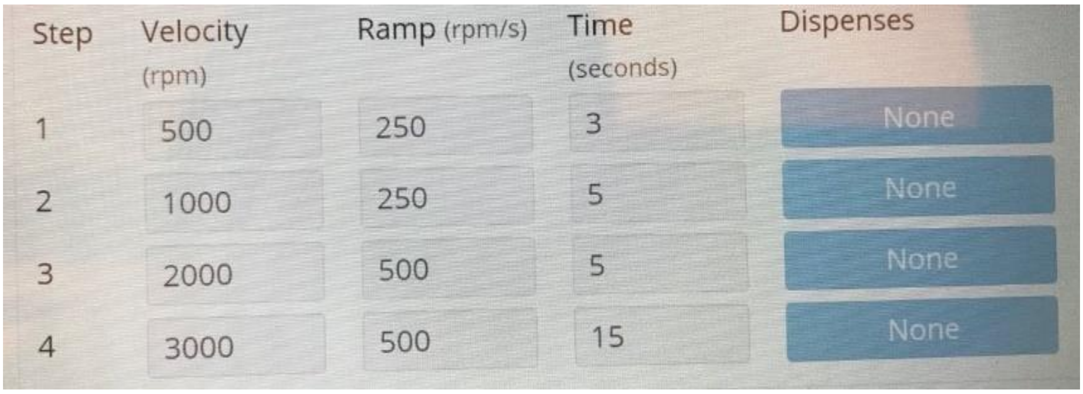


Figure 4.20 Hydrophobicity after oxygen plasma etching

The reason why the result of coating is not good enough is because the photoresist is not sticky enough. When the spin speed is high, the photoresist is thrown out rather than stuck on the wafer. So the Nlof 2070 is used which is super sticky. First fully coat the wafer with Nlof 2070 and then use a low accelerate rate to reach the spinning speed of 3000 rpm. The recipe shows in Figure 4.21. After coating, bake the wafer for 2 minutes at 100°C then go to the photolithography. In this way, the wafer is well patterned and can be developed well.



Step	Velocity (rpm)	Ramp (rpm/s)	Time (seconds)	Dispenses
1	500	250	3	None
2	1000	250	5	None
3	2000	500	5	None
4	3000	500	15	None

Figure 4.21 The recipe of coating Nlof 2070 on FluoroPel

Other methods to pattern the Fluoropel, like lift-off techniques, have been researched as well. However none of them showed satisfactory results.

4.2.12 Dicing and Bonding

The last two steps for the chip are dicing and bonding. First the wafer should be put on a foil. The dicing used is a 40 μm blade with high pressure water gun to scour the residue from dicing. After alignment, the dicing is done automatically. Figure 4.22 shows the wafer after dicing.

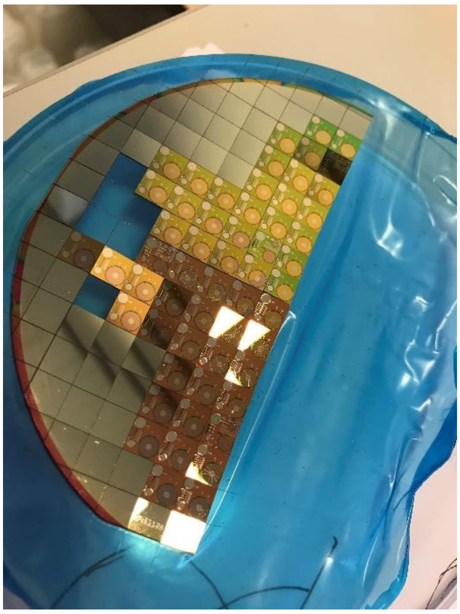


Figure 4.22 The wafer after dicing

After pick some good chips from the diced wafer, they need to be bonded with gold lines. The bonding process is to connect the contact pads of the chip to the substrate in order to easier the testing process. Figure 4.23 shows the chips after bonding and Figure 4.24 shows the two bonding methods of two designs.

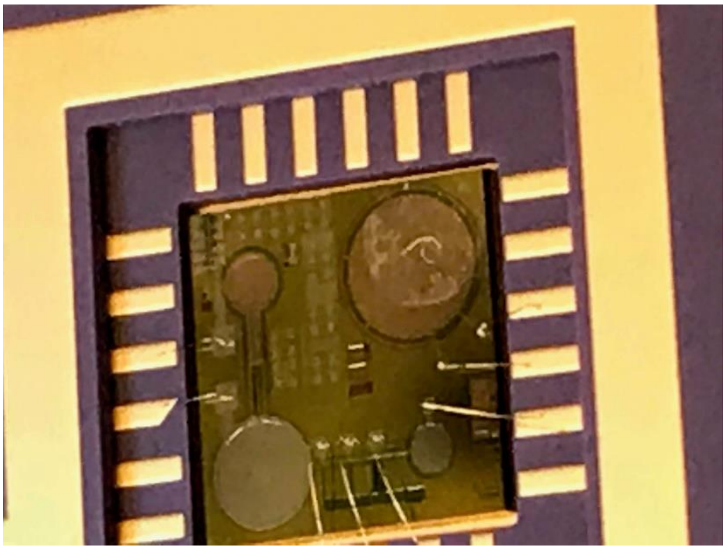


Figure 4.23 The chips after bonding

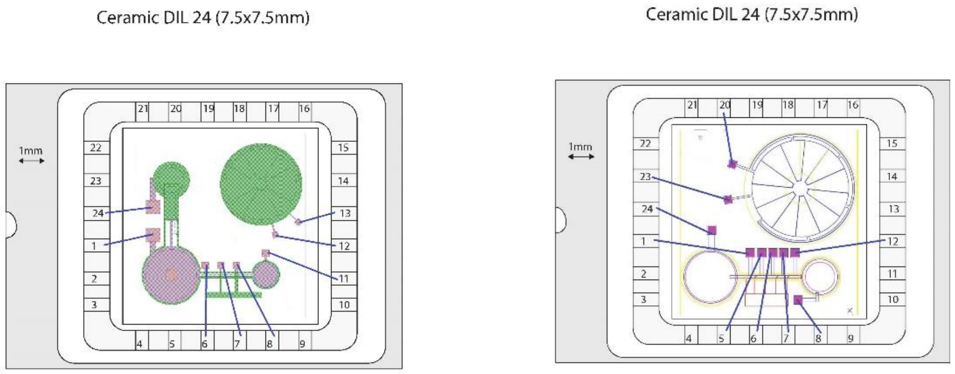


Figure 4.24 Bonding methods of design 1 (Left) and design 2 (Right)

Reference

[1] https://www.youtube.com/watch?v=4FCeKf4deiU

[2] Multi image mask exposure job description - Henk Van Zeijl (2016)

[3] Kiani, Amirkianoosh, et al. "Maskless lithography using silicon oxide etch-stop layer induced by megahertz repetition femtosecond laser pulses." Optics express 19.11 (2011): 10834-10842.

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[5] Cale, T. S., and G. B. Raupp. "A unified line-of-sight model of deposition in rectangular trenches." Journal of Vacuum Science & Technology B: Microelectronics Processing and Phenomena 8.6 (1990): 1242-1248.

[6] http://mfz140.ust.hk/Eq_manual/EVG%20101%20Spray%20Coater%20Operation%20Manual.pdf

[7] https://www.utdallas.edu/~rar011300/CHAevap/CHAmanual.pdf

[8] Yeh, Fu-Wen, et al. "The arrowed surface ratchets with hydrophobic parylene for droplet transportation." Nano/Micro Engineered and Molecular Systems, 2009. NEMS 2009. 4th IEEE International Conference on. IEEE, 2009.

Chapter 5. Simulations and Measurements

Comsol Multiphysics simulation is used to estimate the performance and assist in the design of the device before fabrication. It can give us a rough idea of the experimental process, including how much voltage will result in contact angle saturation. Then the measurements performed on the fabricated devices are described.

5.1 Simulations

The purpose of this simulation is to look at the relationship between the thickness of the insulating layer and the voltage. According to the theory of electrowetting, the contact angle of water will change when there is voltage applied. But the contact angle does not diminish indefinitely, it will have a saturation angle. By simulation, varying voltages can be applied to the electrode under the insulating layer to follow the change in contact angle. Multiple simulations can be run for different insulating materials and layer thicknesses. In this way, the relationship between the thickness and composition of the insulating layer and the value of voltage to reach the saturation contact angle can be found.

5.1.1 Simulation of the Classic Model

The simulation on a flat type wafer is to verify the feasibility of electrowetting and how much the angle of change will be given a certain voltage applied in the experiment. The first method used to simulate is the level set method which is also the most common one. The basic illustration is shown in Figure 5.1. This is a basic structure of electrowetting which consists of electrodes, a droplet, a hydrophobic layer and an insulating layer.

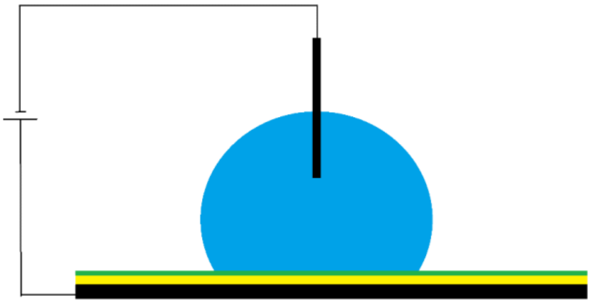


Figure 5.1 A schematic of the electrowetting geometry

This model aims to simulate the movement of the interface between two immiscible phases by using the level-set method with Navier-Stokes equations [1]. The behavior of the interface is controlled by the surface tension of the two phases. Also, the wetting properties of the two fluids can be described by calculating for the contact angle. In this case, the parameters in this simulation can be changed easily, like the applied voltage, the thickness of insulating layer, etc.

In this simulation, once the parameters of the material and the voltage is set up, the shape changing of the droplet with time can be simulated by Comsol which used a voltage sweep of 1V, 2V, 3V to get some more results in one simulation. Figure 5.2 shows the output of the simulation with 3V. For the insulating material, $\mathrm{Si_3N_4}$ is chose with the dielectric constant of 7.5 and a thickness of 80 nm. The total time of simulation is 0.2s.

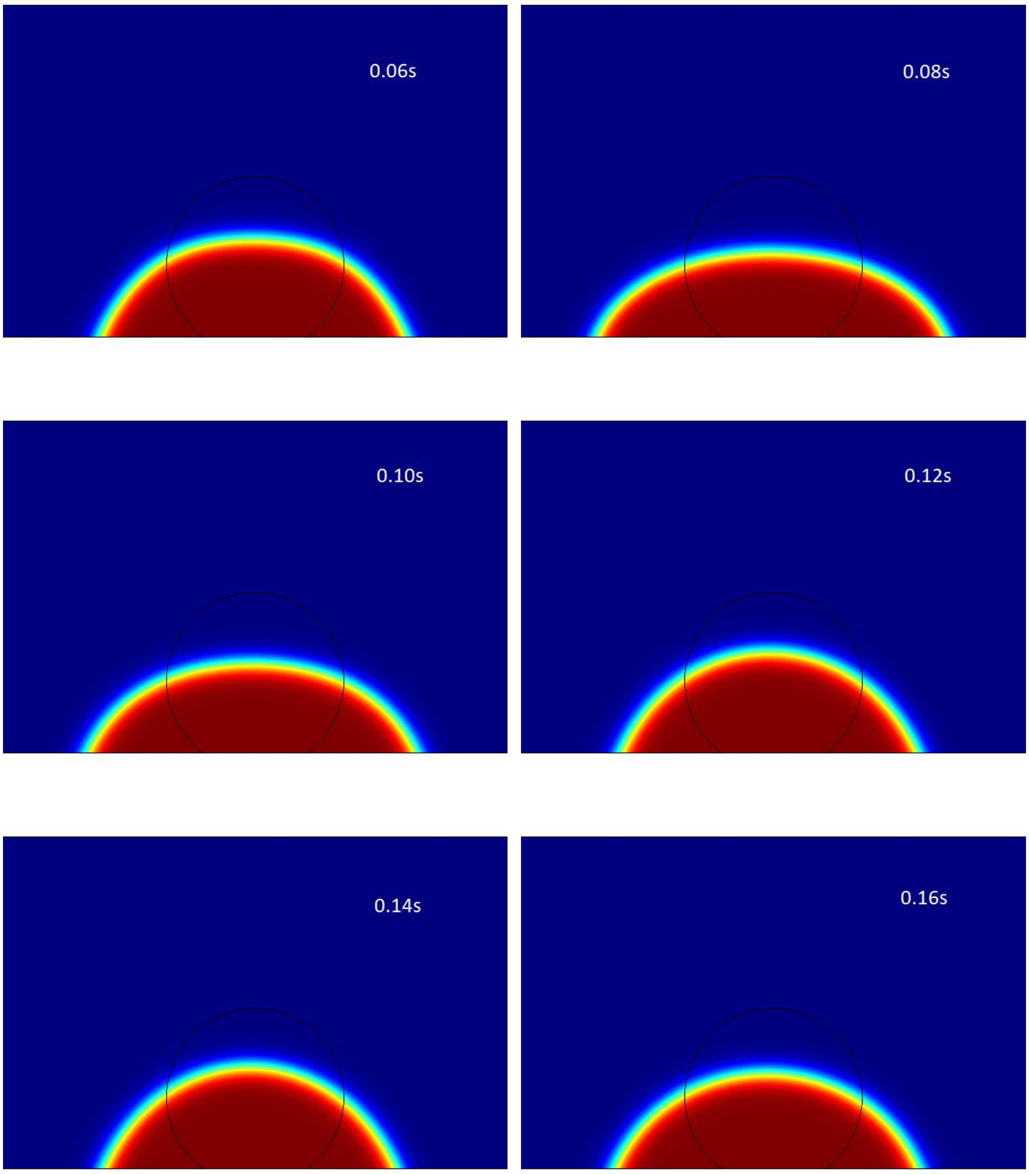


Figure 5.2 Simulation results over time

The initial droplet is shaped with a black line. When the voltage is applied, the surface changes to hydrophilic and the contact angle is obviously less than 90°. From this simulation, the shape changing of the droplet can be seen very clearly. When the time reach to 0.12s, the shape changing basically stopped and after that it only has some small modification. The angle change is really fast even when the applied voltage is smaller than 3V which will be shown below. For the applied voltages 1V, 2V, 3V, the times for the droplet to reach the saturation angle are all 0.16s, see Figure 5.3.

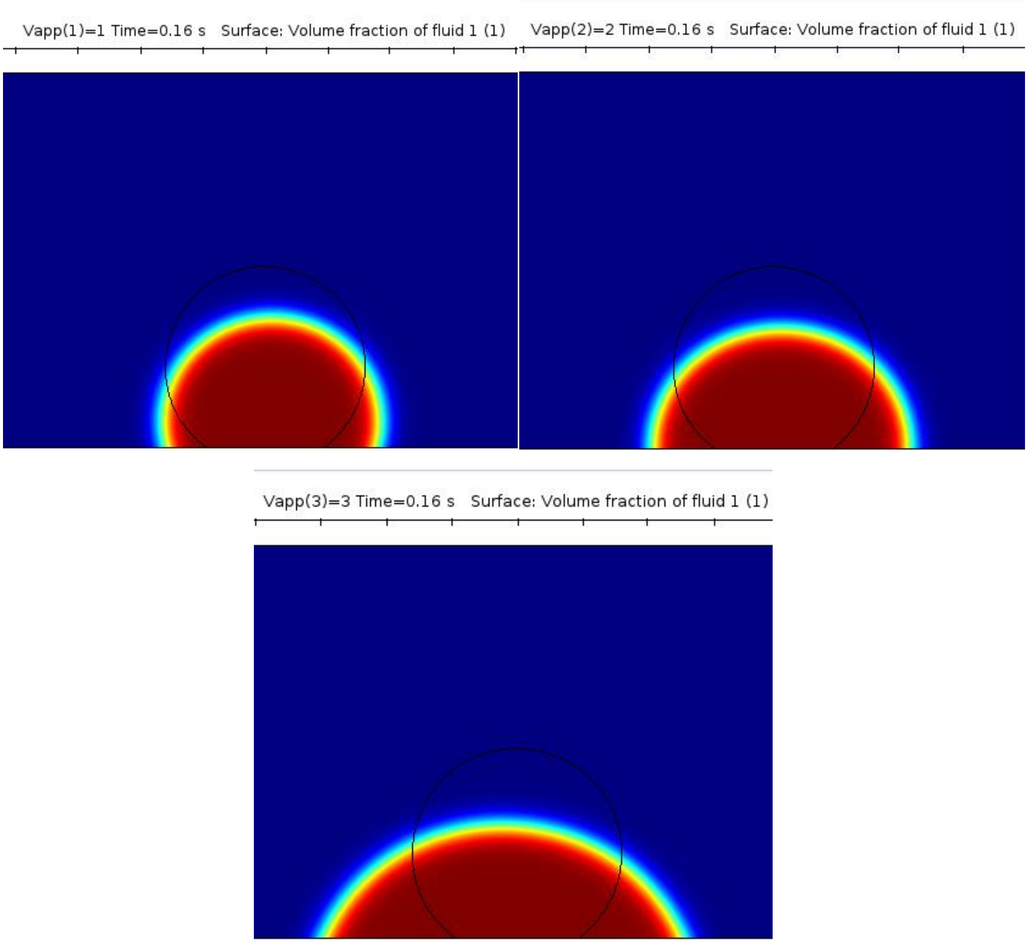
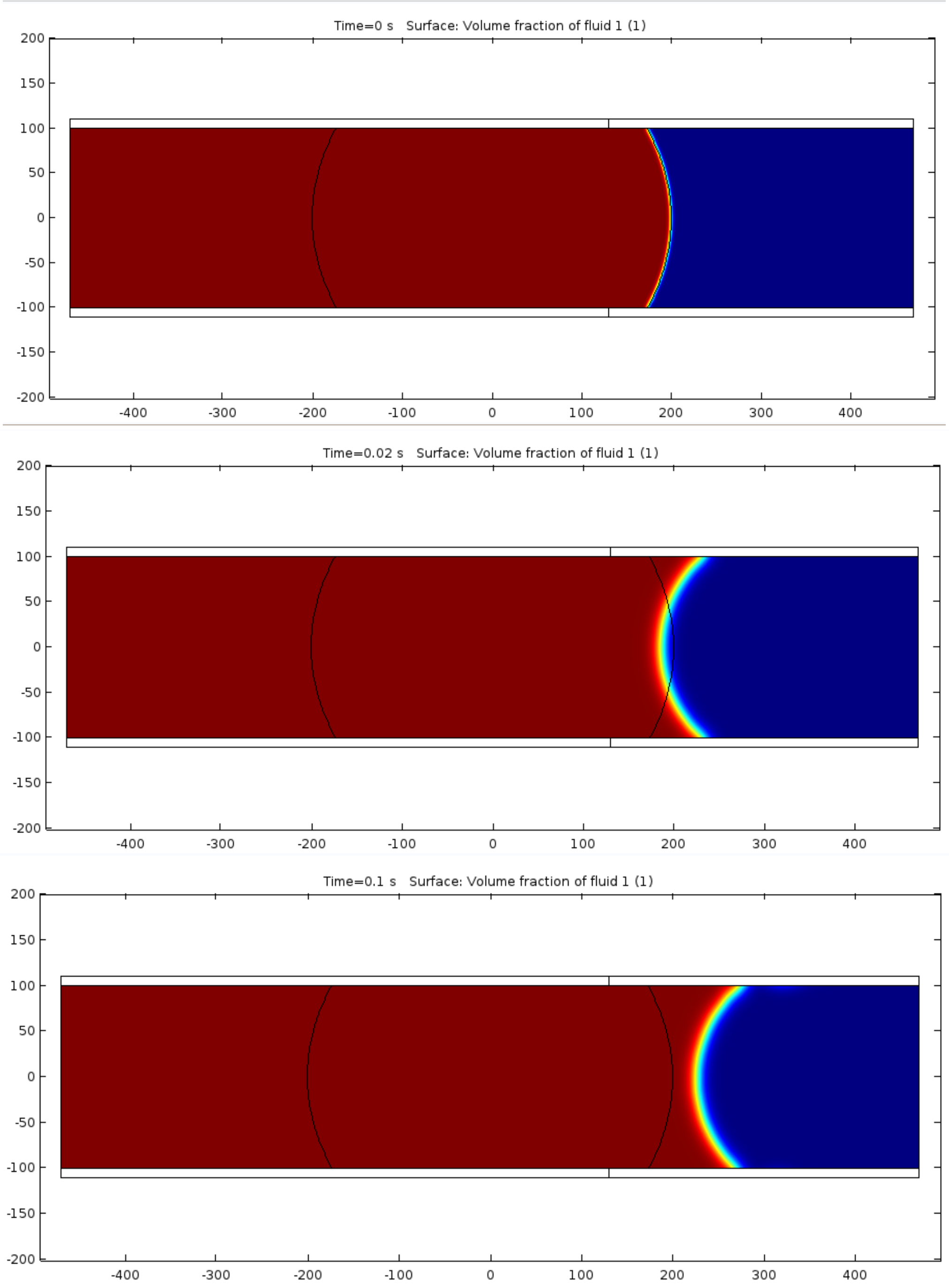


Figure 5.3 Time required of droplet reaches the saturation angle

5.1.2 Simulation of the Channel Model

The purpose of this project is to control the flow of fluid through a channel by switching it on and off. So the simulation of the fluid in a channel is needed. In this simulation, there is no voltage applied on the electrode at first and the surface is hydrophobic. Once the electrode is energized, the contact angle will be reduced to hydrophilic. And because of the charge attraction, water will continue to flow to the edge of the energized electrode. The initial contact angle is 140° and the dielectric layer thickness is 50 nm with the dielectric constant of 9. The hydrophobic layer is set as

50 nm with the dielectric constant of 2.7. With the applied voltage of 10V on both sides of the 200 µm width channel, the simulation process is shown in Figure 5.4. The angle changing is really fast within 0.02s and the fluid starts to flow forward after that. It can be seen from this phenomenon that the water in the channel can be driven forward by applying proper voltage.


Time=0 s Surface: Volume fraction of fluid 1 (1)

Time=0.02 s Surface: Volume fraction of fluid 1 (1)

Time=0.1 s Surface: Volume fraction of fluid 1 (1)

Figure 5.4 Simulation of the channel model

5.2 Measurements

5.2.1 Measurement on Flat Type Wafer

The measurement on the flat type wafer aims to test whether the theory of electrowetting works. The setup of this experiment is very simple, and basically the same as the classic electrowetting, composed of droplet, electrodes, hydrophobic layer and insulating layer as shown in Figure 5.1. The insulating layer is 102 nm or 155 nm silicon nitride and the bare silicon substrate is used as the electrode. The whole wafer is covered with a 27 nm thick FluoroPel hydrophobic layer.

The initial contact angle of DI water on the FluoroPel layer is 108° as shown in the Figure 5.5. The first measurement used a 155 nm silicon nitride layer. The needle is inserted in the droplet as a ground electrode as shown in the Figure 5.6 which is the initial state of this experiment. When the voltage rises to DC 10V, the droplet starts to change and the contact angle changes to around 90° when the voltage reaches 12V. At a voltage of 25V there will be a sudden great change that the contact angle has a big change, probably because the shape of the tip is asymmetric. Once the droplet leaves the non-uniform part of the tip, there will be a large change. The smallest contact angle happens when the voltage reaches approximately 30V as shown in Figure 5.7. If the applied voltage changes to 32V, the insulating layer is broken through and gas bubbles can be seen inside the droplet.

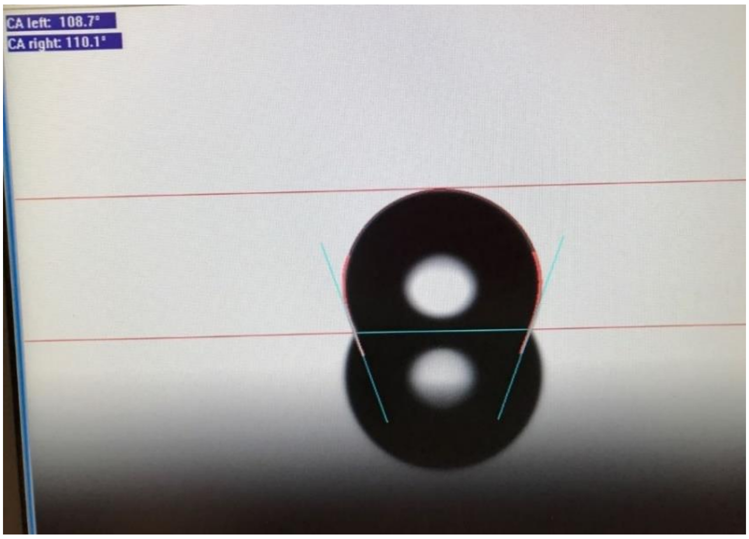


Figure 5.5 The contact angle of DI water droplet on FluoroPel layer

Figure 5.6 Initial state of the droplet

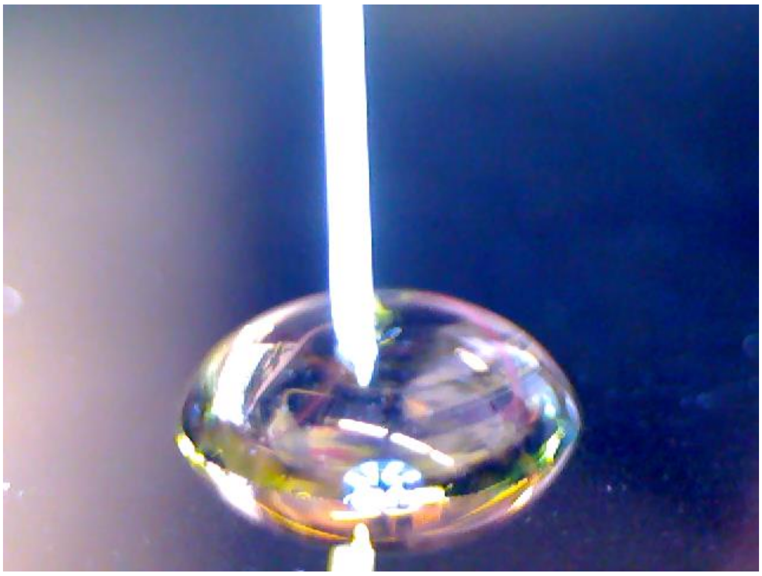


Figure 5.7 The shape of the droplet at 30V

The second measurement happens on the wafer with 102nm silicon nitride. The initial contact angle of DI water to FluoroPel is the same of 108° which can be seen from Figure 5.8. But due to the thinner insulating layer, the driving voltage should also be lower either. In this experiment, the droplet starts to have some shape changing when the voltage is 6V. The significant shape changing happens at about 15V (Figure 5.9) and the maximum angle is observed at 18V.

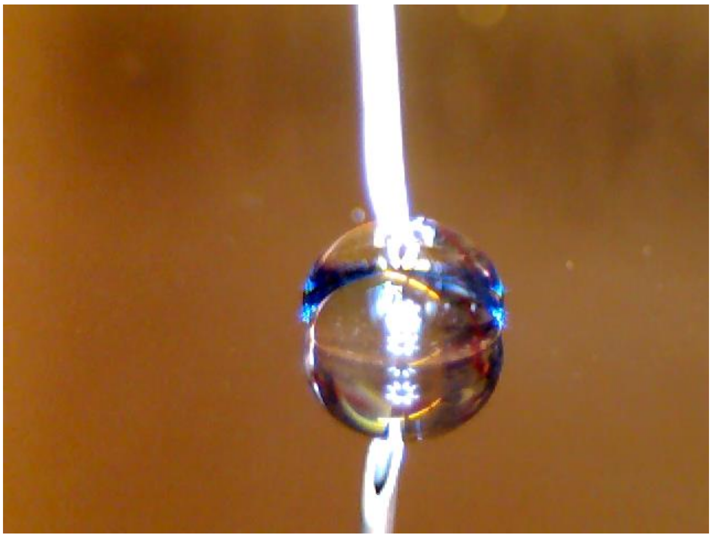


Figure 5.8 Initial state of the droplet with 102 nm SiN

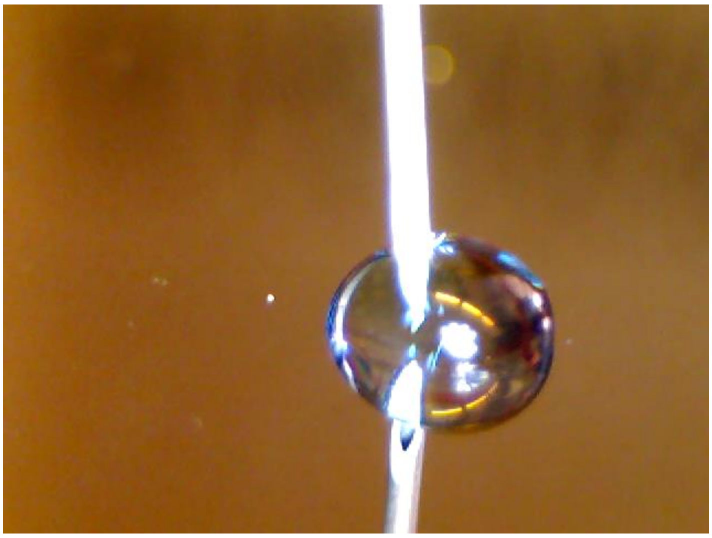


Figure 5.9 The shape of droplet at 15V

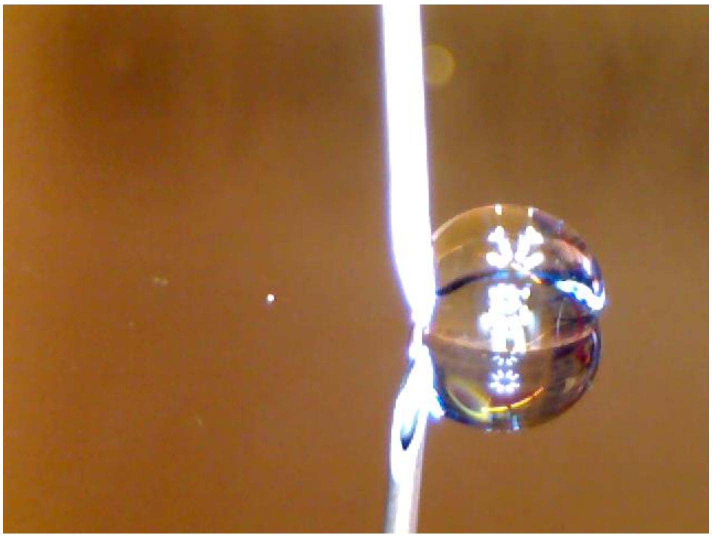


Figure 5.10 The droplet jumps away at 18V

When the voltage is larger than 18V, the droplet jumps away from the needle and loses the contact with the potential as shown in Figure 5.10. Then it re-form to the initial shape within 3 seconds. Because of this performance, the saturation state of electrowetting on this wafer cannot be observed. But the contact angle of 18V is pretty small around 40°. According to the experiment result on the 155nm silicon nitride wafer, it can be estimated that the contact angle at 18V is basically the saturation angle. Because the droplet jumps away, there is no breakthrough to be observed on this wafer.

5.2.2 Measurement on the Chip

The silicon wafer is diced into separate chips, each having the designed electrowetting structures on it. After packaging and bonding, the chip can be tested on a printed circuit board to which the external test voltages can be applied, as shown in Figure 5.11. The first experiment is to test whether the electrowetting theory works on the designed structure with parylene as hydrophobic layer. For this a special circular test structure with interdigitated electrodes was used. A tiny droplet of DI water was dropped on the chip and the droplet stays in a hydrophobic state. After applying voltage on the electrodes, the shape of the droplet starts to change but not as much as expected, see Figure 5.12. After applying a DC voltage of 35V on the electrodes, the droplet spreads out a little bit but not very obvious. During the measurement, the parylene seems to lose its hydrophobicity. This also could be one of the reason that the shape changing is not obvious. If the voltage goes any higher, the insulating layer is breaks down and gas bubbles become visible inside the droplet.

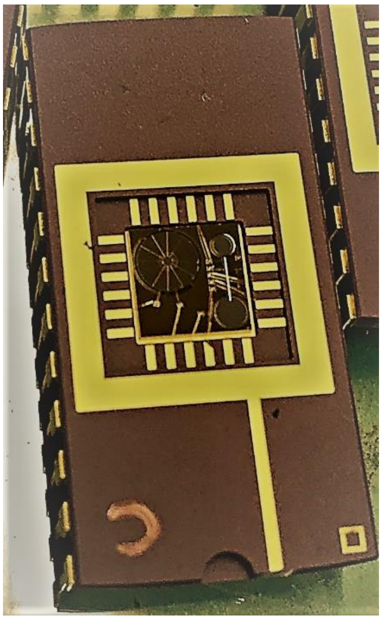


Figure 5.11 Bonded chip

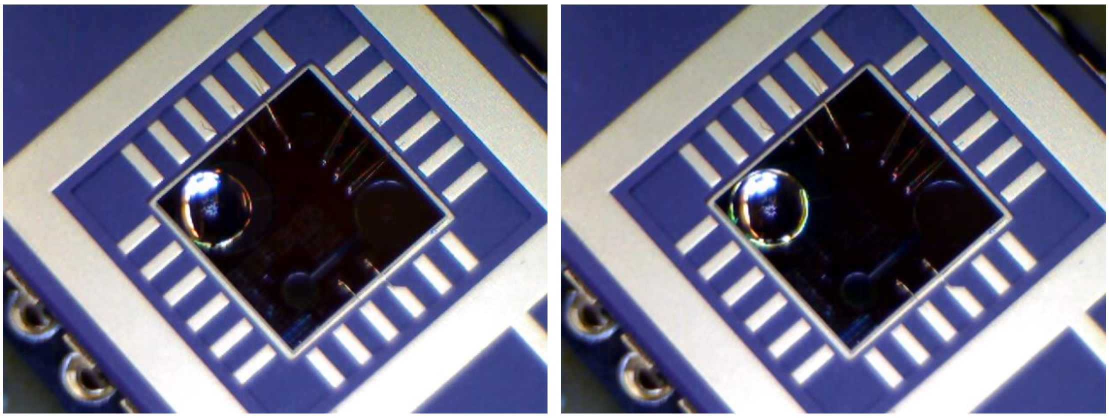


Figure 5.12 No voltage (left); 35V (right)

5.2.3 Measurement on the Probe Station

Since the dicing and bonding process really takes time, the measurements were performed on the whole wafer on a probe station. This has the advantages of greatly reducing the preparation time of the experiment while measurements can be done on any chip on the wafer. The setup of this probe station is shown in Figure 5.13 which use vacuum suction to stabilize the wafer and use probes needles to apply voltage on the contact pads of the chips.

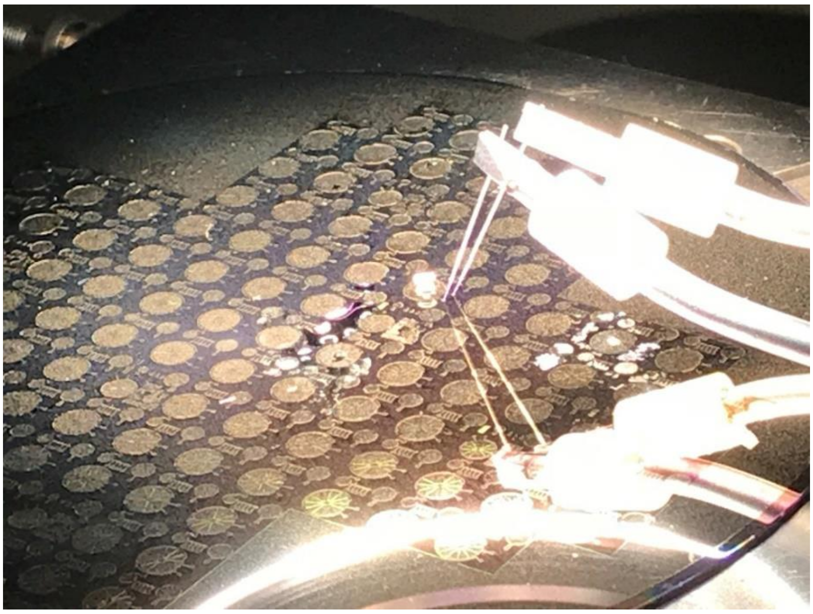


Figure 5.13 Probe station setup

First the wafer with 88nm silicon nitride as the insulating layer and FluoroPel as the hydrophobic layer is measured. The hydrophobicity of FluoroPel is good and the initial contact angle is 108°. When the voltage is slowly applied on the contact pad, the shape of the droplet starts to change.

Figure 5.14 shows the shape changing of the droplet from which can be seen that there is small change on the edge of the droplet. When the voltage goes higher than 23V, the insulating layer breaks through and gas bubbles appear.

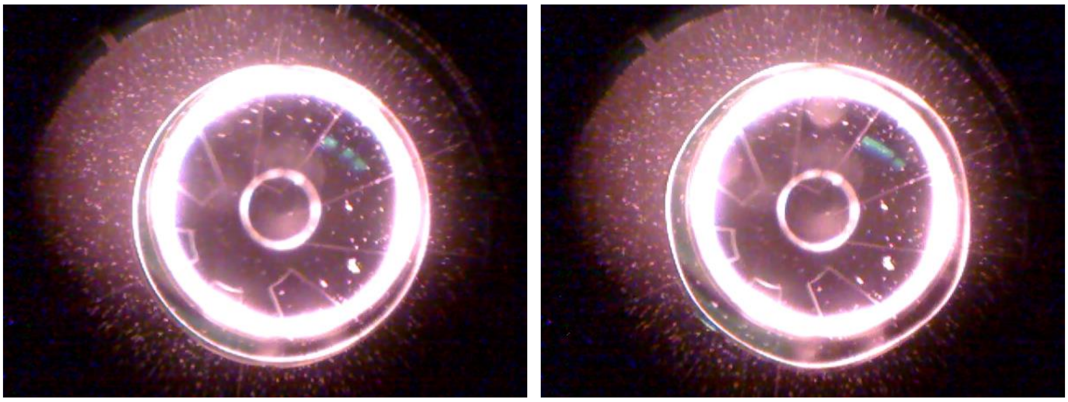


Figure 5.14 The shape changing from 0V (left) to 20V (right)

Then the wafer with 30nm Al_2O_3 as the insulating layer is used to observe the shape changing under the microscope. First the DI water with NaCl is used. When the DC voltage is used, it easily breaks through with a low voltage. There is a small changing with 10V AC voltage but the voltage cannot go any higher. Then the pure DI water is tried. With AC voltage, there is no big changing when it goes to the maximum of 10V. But when the DC voltage rises to about 17V, the shape starts to change until the insulating layer breaks down at 22V. The shape changes a little during the potential rising but not as much as expected.

The reason the droplet shape does not change much could be the big surface tension. So 1% SDS solution is used to reduce the surface energy. First the 100Hz AC voltage is tried and which shows a small change at 10V. Since the AC source cannot supply higher voltage, DC voltage is applied on the two electrodes. With this combination, the shape starts to change at 7V and get the biggest changing on 23V as shown in Figure 5.15. From the right picture below can be seen that the shape changing is not circular. It shows a regular deformation consistent with the electrode structure. Since the droplet change is very small compared to the tests on the flat wafer it can be concluded that the interdigitated electrode structure is not very suitable for electrowetting tests.

When the voltage reaches 25V, the insulating layer breaks down. This damage is permanent and doesn’t recover when the voltage is removed.

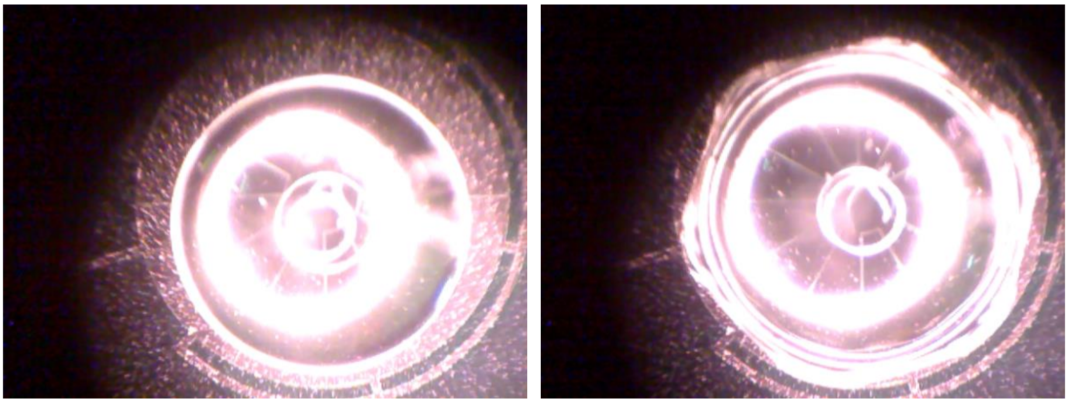


Figure 5.15 Shape changing from 0V (left) to 23V (right)

From these experiments, it can be seen that the reliability and stability of FluoroPel is much higher than that of Parylene. Although the Parylene becomes hydrophobic again after the CF_4 plasma treatment, but it is really not stable. In contrast, FluoroPel retains its initial hydrophobicity after many different tests. What's more, the contact angle changing on the top-bottom contact structure is bigger than that on this interdigitated structure. One of the reason is this structure requires twice the voltage of the top-bottom structure to achieve the same angular variation due to the special design without any contact between droplet and the electrodes.

5.2.4 Measurement on the Channel

At first, the water does not flow into the channel as designed. The water stops at where it was dropped and never flows into the channel. Figure 5.16 shows the situation of the design and the droplet. Once the water is dropped on the device, it does not move any further even there is a channel existing. The initial guess was that probably some parylene residue was left at the edges of the channel which stopped the fluid from flowing into the channel. Another guess was that the liquid would not flow into the channel because there is no hydrophilic cover to the channel. So different experiments have been done on these two conjectures respectively.

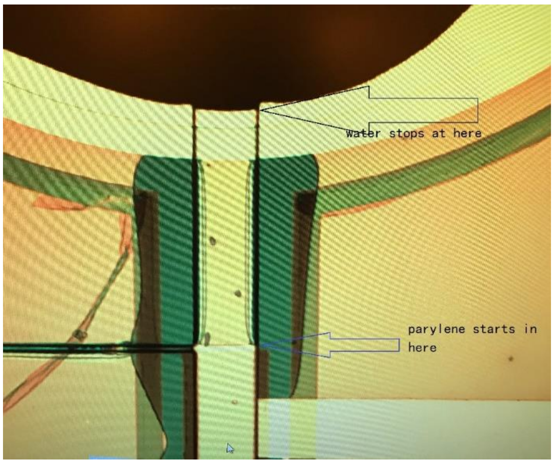


Figure 5.16 Water does not flow into the channel

The first attempt is to reduce the surface tension of the droplet. So 1% SDS solution is used and dropped on the inlet of the channel. From Figure 5.17, it can be seen that the fluid flows into the channel without any top cover and stops at the hydrophobic part. Unfortunately the parylene loses its hydrophobicity over time (within one day) which made it impossible to repeat this experiment.

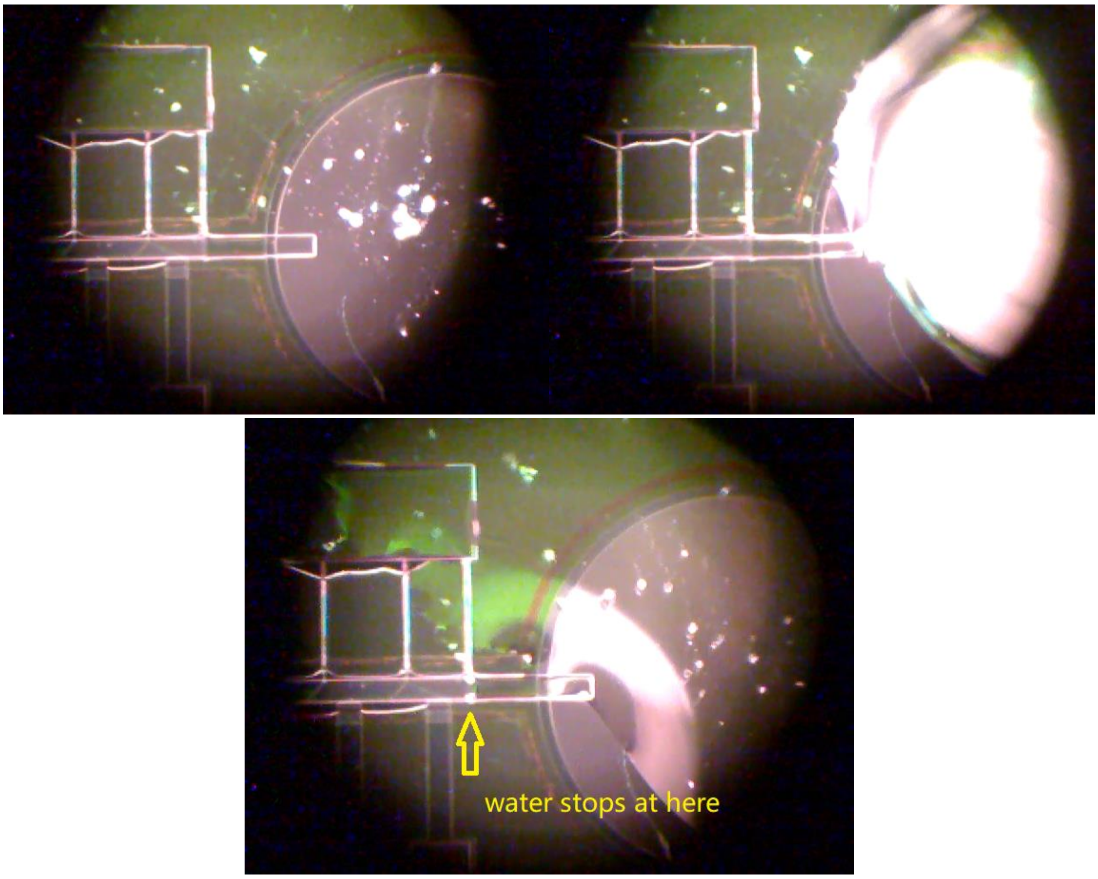


Figure 5.17 SDS solution flows into the channel and stops at the Parylene edge

The second attempt is to observe the flow of water in the case of a hydrophilic tape as a cover. The reason for using a hydrophilic tape is that it is hoped that it will direct the water into the channel and then stops on the edge of the parylene. After that, when a voltage is applied to the electrode, the water-transporting material becomes hydrophilic and the water flow will continue to flow forward due to the guidance of the hydrophilic tape. However, as Figure 5.18 shows, water flows into the channel faster than the previous one without cover, but does not stop at the edge of parylene. It can be observed from the reflection in the channel. This may be because parylene once again loses its hydrophobicity within one day after CF_4 treatment. The contact angle of water again reduced to about 40 degrees. So it will not stop but continue to flow forward due to the more hydrophilic cover.

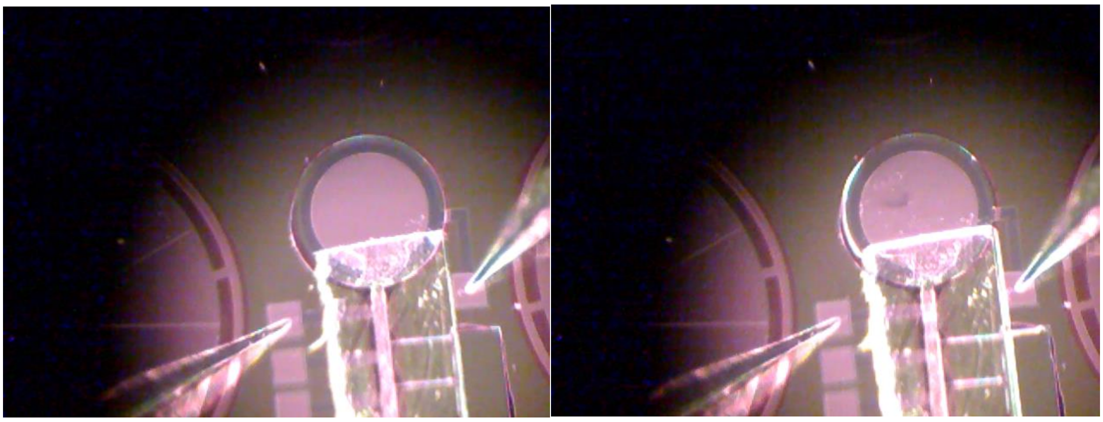


Figure 5.18 Water flows into the channel

5.2.5 FluoroPel Long-term Stability Test

The FluoroPel is a rather new material and there is no literature documenting its stability. So for this material, the stability test is very important. The set up for this test is shown in Figure 5.19. It consists of an aluminum block, silicone oil, the FluoroPel wafer with 102nm silicon nitride and the needle to apply voltage to the droplet. The silicone oil was used to prevent the water from evaporation and the aluminum block is used to confine the oil. An O-ring is placed between the aluminum block and the wafer to prevent the oil from leaking away. The needle is stuck into the droplet to provide the connection to the ground. At first, only one needle is used and the droplet jumps away after about 1 hour. So then two needles were used as the ground electrode and both of them stick into the droplet. In this way, the droplet remains in position in between the two needles. In this experiment, a step like changing voltage with values of 0V, 10V and 20V is applied to the wafer substrate. From Figure 5.20, the shape changing of the droplet is shown. At 10V, the wafer still acts as a hydrophobic surface. When the voltage is 20V, the shape changed but not big. And at 25V, the contact angle reached its maximum change. The FluoroPel lost its hydrophobicity during the test after one day and there was no way to make it hydrophobic again. Since the FluoroPel showed before to be stable at air for more than three months and survived multiple electrowetting experiments it is assumed that a chemical reaction with silicone oil changed the surface properties of the FluoroPel in an irreversible way. So it was not possible to show the long term stability of FluoroPel under active electrowetting conditions for more than one day.

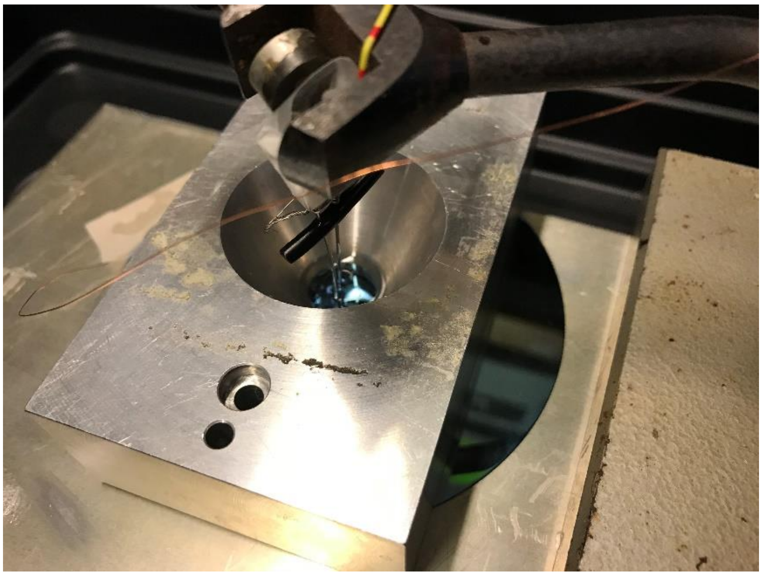


Figure 5.19 The experimental setup for long-term testing of FluoroPel

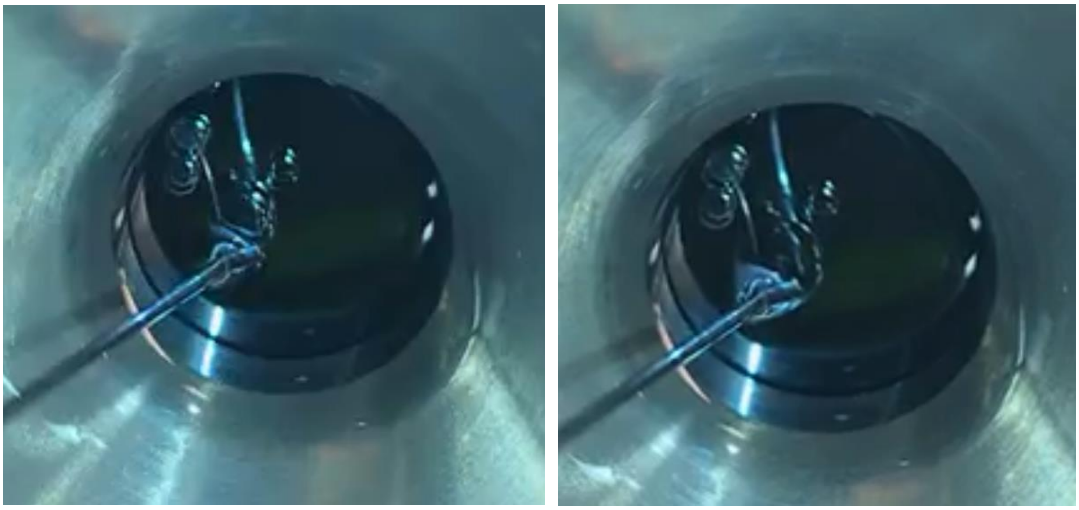


Figure 5.20 The droplet state at 0V, 20V

Reference

[1] Kuo, Jason S., et al. "Electrowetting-induced droplet movement in an immiscible medium." *Langmuir* 19.2 (2003): 250-255.

Chapter 6. Conclusions and future work

6.1 Conclusions

In this thesis, a voltage controlled electrowetting valve with air channel has been developed. Unfortunately the device was not completely functional due to several processing problems. The main challenge was to create a hydrophobic layer of uniform thickness to establish a good relation between thickness and activation voltage.

During the fabrication process, the metal lift-off and spray coating showed to be difficult steps as well as the patterning of the hydrophobic material.

Metal platinum lift-off is an unstable process. Although 20 nm Ta is used as the adhesion layer, the desired platinum pattern was damaged during lift-off. Even with the use of ultrasonic cleaners, the pattern is disturbed due to the poor adhesion between Ta and the insulating layer. The solution to this is to immerse the wafer in 80-degree NMP for a long time without motion. In this case the metal was nicely patterned. A cotton swab was used to gently remove any residue.

The non-uniform thickness of the spray coated resist also gave some problems, especially at the edges of the trenches. Hence, the development process needed to be done manually. Once the photoresist in the center is developed, the parts that should stay on the edge of the wafer may have disappeared due to over-development. During this process, the disappearance of the photoresist in the center of the wafer should be observed very carefully. Once disappeared, the wafer is immediately submerged in water to stop the development. This will minimize the damage to the structure.

Since the hydrophobic material Parylene is too hard to pattern, an oxygen plasma etch for 10 seconds is used to make the surface hydrophilic, which makes the photoresist better stick to the surface. After patterning, the CF_4 treatment to Parylene makes it hydrophobic again. However, the effect of this treatment is not lasting. In the experiment, the hydrophobic surface became hydrophilic again after only one day. In experiments, FluoroPel kept its hydrophobicity. But it is too hydrophobic to coat a uniform photoresist layer on it. After many tests, Nlof2070 under the specific coating recipe will form a relatively thick uniform layer on the FluoroPel. However, this method is only suitable for spin coating and so not for the wafer with trenches.

In the test process, the initial test is to bond the chips after dicing. But this process is time-consuming and it would be a waste if some of the chips failed. Then the probe station is used to do the measurement on the whole wafer. In this case any structure on the wafer could be chosen for testing, which saved the time and costs for packaging. Another advantage is that the droplet changes can be observed by the microscope during the test.

The aim of this project is to control the fluid flow by applying the voltage. During the measurement on the chip, the 1% SDS solution flows into the channel and stops at the edge of hydrophobic area. But due to the parylene losing its hydrophobicity after a day, there was no chance to control the contact angle by potential. But during the experiment on the FluoroPel device, the angle change is pretty big and could be observed even by eyes. So controlling the fluid flow by applying proper voltage should be achievable at least on FluoroPel. Right now, there is no good ways to deposit a uniform FluoroPel layer on a wafer with trenches. So this is a problem that needs to be focused on in the future.

The design of this project also has some shortcomings. In this project, for simplicity, an open channel structure (without cover) was chosen. This leads to problems in getting the water to flow into the channel in the tests. Closing channels with hydrophilic tape solved this problem, i.e. water was able to flow into the channel. The location of contact openings is also worth reconsidering. The position of the contact pads were now too close to the trench to properly cover the channel and simultaneously apply good electrical contact. Designing them more to the edge of the chip, away from the channel, would make them better accessible.

6.2 Future Work

Improvement on the fabrication process and the design of a transparent hydrophilic cover could be the main work that needs to be done in the future work. Suggestions on this project are summarized as follows:

- Find a new way to deposit hydrophobic material more uniformly on a wafer with trenches. The material should be able to be patterned and have a treatment to make it hydrophobic again or it shouldn't lose its hydrophobicity at all.
- It is better to have a roof on the channel for a good fluid flow. It is best to process two wafers and then stick the two wafers together. One of them should be transparent glass to have a good view of the liquid flow. In this way, there are electrodes on both top and bottom

of the channel that control the flow of liquid. And all sides of the channel have hydrophobic material coverage.

- Optimize the manufacturing process. Find a better way to avoid the problems described above. A more sticky photoresist which can be used on the spray coater to pattern the hydrophobic material without pin-holes would be a great benefit.
- Optimize the design and the fabrication. It is better to design and manufacture the device which can be controlled simultaneously from top and bottom. Otherwise, make a transparent sheet resist as a cover on the top.
- Move the contact pads to the edge of the chip. Since there is trench with water flowing in it, this is a better choice for both bonding and safety.

Appendix 1. Flowchart

1. CLEANING PROCEDURE: HNO3 100% and 65% (Si) (optional)

Cleaning	10 minutes in fuming nitric acid (Merck: HNO3 100% selectipur) at ambient temperature. Use wet bench "HNO3 (100%)" and the carrier with the red dot.
QDR	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.
Cleaning	10 minutes in concentrated nitric acid (Merck: HNO3 65% selectipur) at 110 °C. Use wet bench "HNO3 (65%)" and the carrier with the red dot.
QDR	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.
Drying	Use the Ultra pure-6 Spin Rinse Dryer with the standard program, and the white carrier with a red dot.

2. COATING AND BAKING

Use the coater station of the EVG120 system to coat the wafers with photoresist. The process consists of a treatment with HMDS (hexamethyldisilazane) vapor, with nitrogen as a carrier gas, spin coating of Shipley SPR3012 positive resist, dispensed by a pump, a soft bake at 95 °C for 90 seconds, and an automatic edge bead removal with a solvent.

Always check the relative humidity (48 ± 2 %) in the room before coating, and follow the instructions for this equipment.

Use program "Co - 3012 - zero layer". There will be a larger edge bead removal.

3. ALIGNMENT AND EXPOSURE on the PAS5500/80 waferstepper: ZEROLAYERS • Sep 2008

Tool: PAS5500/80 waferstepper

Location: Class 100 Litho room

Manual: *<location>*

User mask name: COMURK

Mask ID: COMURK

Exposure job: EPI0.0

Layer ID: LAYER-1

Focus: 0

Energy: 150

Alignment strategy: NONE

Settings: no additional user settings are required.

Notes: The chuck-side of the wafers should be free of photoresist and other polymers.

4. WRITE WAFER ID

The lot will be split in the next process flow. Therefore it is important that the wafers are **clearly** numbered. Use a quartz pen to write the wafer numbers in the photoresist. Number the wafers as follows: EI2220-1……

5. DEVELOPMENT

Use the developer station of the EVG120 system to develop the wafers. The process consists of a post-exposure bake at 115 °C for 90 seconds, developing with Shipley MF322 with a single puddle process, and a hard bake at 100 °C for 90 seconds.

Always follow the instructions for this equipment.

Use program "Dev - SP".

6. INSPECTION: LINEWIDTH AND OVERLAY

Visually inspect the wafers through a microscope, and check linewidth and overlay.

7. DRY ETCHING: URK_NPD

Use the Trikon Ωmega 201 plasma etcher.

Follow the operating instructions from the manual when using this machine.

Use sequence URK_NPD (with a platen temperature to 20 °C) to etch 120 nm deep structures into the Si .

Process conditions of chamber recipe URK_NPD:						
Step	Gasses & flows	Pressure	Platen RF	ICP RF	Platen temp.	Etch time
1. breakthrough	CF4/O2 = 40/20 sccm	5 mTorr	60 W	500 W	20 °C	0'10"
2. bulk etch	Cl2/HBr = 80/40 sccm	60 mTorr	20 W	500 W	20 °C	0'40"

8. RESIST REMOVAL: Tepla

Use the Tepla plasma system to remove the photo resist in an oxygen plasma. Follow the instructions specified for the Tepla stripper, and use the quartz carrier. Use program 1

9. CLEANING PROCEDURE: HNO3 100% and 65% (Si) (optional)

Cleaning	10 minutes in fuming nitric acid (Merck: HNO3 100% selectipur) at ambient temperature. Use wet bench "HNO3 (100%)" and the carrier with the red dot.
QDR	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.
Cleaning	10 minutes in concentrated nitric acid (Merck: HNO3 65% selectipur) at 110 °C. Use wet bench “HNO3 (65%)” and the carrier with the red dot.
QDR	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.

10. DEPOSITION PECVD OXIDE: 2000nm ● Jan 1995

Tool: NOVELLUS

Location: Class 100 tunnel 3

Manual: *<location>*

Recipe name: XXXSTDOXIDE

Settings: Check the selection switch on the gas box (see the tool manual).

Set the station deposition time (SDT), nominal value is ca 30 sec, check recent depositions for the actual value

Process conditions for recipe STD OXIDE					
Gasses & flows	Pressure	HF power	LF power	Temperature	Time
$N_2/SiH_4/N_2O$ = 3150/205/6000 sccm	2.2 Torr	1000 W	0 W	400 °C	variable $^{sec}/_{station}$

Notes: Change only the SDT in this recipe, leave other parameters unchanged.

11. MEASUREMENT: Oxide thickness ● Oct 1996

Tool: Leitz MPV-SP

Location: Class 100 tunnel 3

Manual: *<location>*

Recipe name: Th. SiO2 on Si, >50nm auto5pts

Settings:

12. CLEANING PROCEDURE: HNO3 100% and 65% (Si) (optional if the wafers are stored until the next day)

Cleaning	10 minutes in fuming nitric acid (Merck: HNO3 100% selectipur) at ambient temperature. Use wet bench "HNO3 (100%)" and the carrier with the red dot.
QDR	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.
Cleaning	10 minutes in concentrated nitric acid (Merck: HNO3 65% selectipur) at 110 °C. Use wet bench "HNO3 (65%)" and the carrier with the red dot.
QDR	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.
Drying	Use the Ultra pure-6 Spin Rinse Dryer with the standard program, and the white carrier with a red dot.

13. COATING ● Apr 2008

Use the coater station of the EVG120 system to coat the wafers with photoresist. The process consists of:

- a treatment with HMDS (hexamethyldisilazane) vapor, with nitrogen as a carrier gas
- spin coating of Shipley SPR3012 positive resist, dispensed by a pump. The approximate spinspeed is 1575 rpm.
- a Soft Bake (SB) at 95 °C for 90 seconds
- an automatic Edge Bead Removal (EBR) with a solvent

Always check the relative humidity (48 ± 2 %) in the room before coating, and follow the instructions for this equipment.

Use program "1-**Co - 3012 – 2,1µm**".

14. ALIGNMENT AND EXPOSURE-Trench

Tool: PAS5500/80 waferstepper

Location: Class 100 Litho room

Manual: *<location>*

User mask name: EI2200

Mask ID: 3X3

Exposure job: DIE6X6_9IMG

Layer ID: IMG_5

Focus: 0

Energy: 150

Alignment strategy: "GLOBAL"

Settings: no additional user settings are required.

Notes: The chuck-side of the wafers should be free of photoresist and other polymers.

15. DEVELOPMENT

Use the developer station of the EVG120 system to develop the wafers. The process consists of a post-exposure bake at 115 °C for 90 seconds, developing with Shipley MF322 with a single puddle process, and a hard bake at 100 °C for 90 seconds.

Always follow the instructions for this equipment.

Use program "**Dev - SP**".

16. INSPECTION: LINEWIDTH AND OVERLAY

Visually inspect the wafers through a microscope, and check linewidth and overlay.

17. PLASMA ETCHING: 2000 nm oxide ● Jun 2004

Use the Drytek Triode 384T plasma etcher.

Follow the operating instructions from the manual when using this machine.

It is **not** allowed to change the process conditions from the etch recipe, except for the etch time!

Use recipe **STDOXIDE** to etch the oxide layer. **Set the etch time to ????**".

Process conditions from recipe STDOXIDE:					
Step	Gasses & flows	Pressure	RF power	He pressure	Etch time
1. bulk etch (RIE)	C_2F_6/CHF_3 = 36/144 sccm	180 mTorr	300 W	12 Torr	variable

18. **INSPECTION**:

No Oxide residues are allowed.

19. MEASUREMENT: Oxide thickness ● Oct 1996

Tool: Leitz MPV-SP

Location: Class 100 tunnel 3

Manual: *<location>*

Recipe name: Th. SiO2 on Si, <50nm auto5pts

20. DRIE PLASMA ETCHING SILICON: Depth: 10, 20, 50 or 100um ● May 2014

Use the STPS RAPIER plasma etcher.

Follow the operating instructions from the manual when using this machine.

It is **not** allowed to change the process conditions and times from the etch recipe!

Use sequence through wafer (with a platen temperature of 10°**C**) to etch the Si layer and landing on SiO_2.

Total etch time: ca1h 20min/wafer.

21. Layer stripping Photo resist)

Strip resist Use the Tepla Plasma 300 system to remove the photoresist in an oxygen plasma.

Follow the instructions specified for the Tepla stripper, and use the quartz carrier.

Use **program 4**: 1000 watts power for 15 minutes.

22. CLEANING: HNO_3 99% and 69.5% ● Nov 2013

Clean 10 minutes in fuming nitric acid at ambient temperature. This will dissolve organic materials.

Use wet bench "HNO_3 99% (Si)" and the carrier with the red dot.

Rinse Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.

Clean 10 minutes in concentrated nitric acid at 110 °C. This will dissolve metal particles.

Use wet bench "HNO_3 69,5% 110C (Si)" and the carrier with the red dot.

Rinse	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.
Dry	Use the Avenger Ultra pure-6 "rinser/dryer" with the standard program, and the white carrier with a red dot.

23. WET ETCHING: Oxide strip ● Jan 1995

Moisten	Rinse for 1 minute in wet bench "H2O/Triton X-100 tbv BHF 1:7". Use the carrier with the blue dot. The bath contains 1 ml Triton X-100 per 5000 ml deionized water.
Etch	Use wet bench "BHF 1:7 (SiO2-ets)" at ambient temperature, and the carrier with the blue dot. The bath contains a buffered HF solution.
Time	Etch until the front side of the wafer(s) is hydrophobic, plus an extra 30 seconds. The required etch time depends on the layer thickness and composition. The etch rate of thermally grown oxide is 1.3 ± 0.2 nm/s at 20 C.
Rinse	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 M.
Dry	Use the Ultra-pure-6 "rinser/dryer" with the standard program, and the orange carrier with a red dot.
Inspection	Visually, through a microscope. The wafer should be hydrophobic test may be applied.

24. INSPECTION: oxide removal ● July 2016

Visually inspect the wafers.

The front and back side of the wafer should be clean of any oxide residues.

Hydrophobic test

25. CLEANING: HNO_3 99% and 69.5% ● Nov 2013

Clean	10 minutes in fuming nitric acid at ambient temperature. This will dissolve organic materials. Use wet bench "HNO_3 99% (Si)" and the carrier with the red dot.
Rinse	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.
Clean	10 minutes in concentrated nitric acid at 110 °C. This will dissolve metal particles. Use wet bench "HNO_3 69,5% 110C (Si)" and the carrier with the red dot.
Rinse	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.
Dry	Use the Avenger Ultra pure-6 "rinser/dryer" with the standard program, and the white carrier with a red dot.

26. LPCVD DEPOSITION: 500 nm TEOS ● Jul 2003

Furnace tube: E1 Program name: NEWTEOS1

Follow the instructions for the LPCVD furnace when using this equipment.

Process conditions from recipe NEWTEOS1:			
Gasses & flows	Pressure	Temperature	Time
TEOS bubbler (40 °C)	250 mTorr	700 °C	**variable** **command**

27. MEASUREMENT: TEOS oxide thickness ● Apr 2002

Use the Leitz MPV-SP measurement system for layer thickness measurements.

Follow the operating instructions from the manual when using this equipment.

⇨ Program: TEOS

TEOS thickness: nm ⇨ on a bare silicon wafer

NEXT SECTION OF THIDS FLOWCHART IS UNDER DEVELOPMENT (2017/10/4)

28. Option: LPCVD Silicon Nitride

LPCVD DEPOSITION: 500nm low stress Silicon nitride

Furnace tube: E2 Program name: 4INCHST (waits for operator)

Follow the instructions for the LPCVD furnace when using this equipment.

Process conditions from recipe 4INCHST:			
Gasses & flows	Pressure	Temperature	Time
SiH_2Cl_2 / NH_3 = 169.5 / 30.5 sccm	150 mTorr	850 °C	**variable command**

Note: ▪ The layer thickness depends on the deposition time, which can be calculated from the average deposition rate during recent furnace usage.

▪ An extra test wafer can be deposited for measurements and etch tests.

29. MEASUREMENT: Silicon Nitride thickness

Use the Leitz MPV-SP measurement system for layer thickness measurements.

Follow the operating instructions from the manual when using this equipment.

⇨ Program: SiN 4inch

Nitride thickness: ± 10 nm ⇨ on a bare silicon wafer

30. PECVD DEPOSITION: variable thickness oxide deposition ● Jan 1995

Use the Novellus Concept One PECVD reactor.

Follow the operating instructions from the manual when using this machine.

It is **not** allowed to change the process conditions and time from the deposition recipe!

Use macro **0XXX_OXIDE_var nm** (recipe XXXnmSiO2) to deposit a **var** nm thick SiO_2 layer.

Process conditions from recipe 600nmSiO2:					
Gasses & flows	Pressure	HF power	LF power	Temperature	Time
$N_2/SiH_4/N_2O$ = 3150/205/6000 sccm	2.2 Torr	1000 W	0 W	400 °C	Var $^{sec}/_{station}$

Note: The deposition time is subject to minor changes, in order to obtain the correct film thickness.

31. MEASUREMENT: Oxide thickness ● Oct 1996

Use the Leitz MPV-SP measurement system for layer thickness measurements.

Follow the operating instructions from the manual when using this equipment.

⇨ Program: Th. SiO2 on Si, >50nm auto5pts

Oxide thickness: nm ⇨ after oxidizing bare silicon wafers (without implantations)

END of Option: LPCVD Silicon Nitride

32. Spray coat XXXX photoresist ● Nov 2008

Tool: EVG110 Spray coater/

Manual: *<location>*

Recipe name: --------

Settings: use manual flat alignment to align the flat edge of the wafers.

Process conditions		
Layer 1	Photoresist name and dilution	Pressure, nozzle settings and rotation speed etc.
Bake	Hotplate ---	Temperature 95 °C time 90 s proximity … µm

Notes: Always check the relative humidity (48 ± 2 %) in the room before coating, and follow the instructions for this equipment.

The backside of the wafers should be free of photoresist.

NOTE: Use several test wafers to find the optimum illumination energy VS development time

33. ALIGNMENT AND EXPOSURE on the PAS5500/80 waferstepper: METAL ● Sep 2008

Tool: PAS5500/80 waferstepper

Location: Class 100 Litho room

Manual: *<location>*

User mask name: EI2200

Mask ID: 3X3

Exposure job: DIE6X6_9IMG

Layer ID: IMG_6

Focus: 0

Energy: 150

Alignment strategy: ”GLOBAL”

Settings: no additional user settings are required.

Notes: The chuck-side of the wafers should be free of photoresist and other polymers.

34. X-LINK BAKE ● Apr 2008

Use the developer station of the EVG120 system to cross link the exposed AZ Nlof 2000 resist. The process consists of:

- A cross-link bake 115 °C for 90 seconds

Always follow the instructions for this equipment.

Use program "**Only – X-link bake**".?????

35. DEVELOPING MANUAL ● Apr 2008

Use the developer area red room

Developing with the Diluted AZ400K: H2O = 1 : 2 XXX developer for ????s.

Do not use to much developer and rinsing fluid (mostly water) during the developing..

Note: Dispose the developer into the right container if it is not water based.

NOTE: When finished always turn of the DI-water.

36. INSPECTION: Linewidth and overlay ● Feb 1996

Visually inspect the wafers through a microscope, and check the line width and overlay. No “ resist” residues are allowed.

37. TEPLA FLASH: Photoresist residues ● Jul 1997

Strip resist Use the Tepla Plasma 300 system for an oxygen plasma flash.

Follow the instructions specified for the Tepla stripper, and use the quartz carrier.

Use **program 2**: Etch for 1 minute at 600 watts. This will not remove a photoresist masking layer

Note: be sure that the chamber is at room temperature.

38. METALLIZATION: 200 nm Pt

Tool:	CHA (?)
Location:	CR10000
Manual:	*<location>*
Recipe name:	---
Settings:	use manual flat alignment to align the flat edge of the wafers

Evaporation of ?? nm Ta ???Å/s and xxx nm Pt 3Å/s in one run with an evaporation starting power of 45%.

Maximum number of wafers 7. Evaporation pressure is < 3.0 E-6 mbar.

The Temperature should not become higher than 85°C.

Remark:

Stop the process on the Inficon IC/5 controller reach 85°C with the stop button.

Continue processing with the the Inficon IC/5 controller start button if the temperature is cooled down to < 65°C.

Use evaporation recipe 11. Only change thickness value of the Ta and Pt layers

39. INSPECTION: Pattern quality, linewidth and overlay ● July 2016

Visually inspect the wafers.

40. LIFT-OFF MANUAL: NMP ● Jun 2015

Use the NMP in SAL at 70°C in fume hood to lift-off the metal layer.

Use special glass container with Pt on the sticker.

Heat up the NMP in a beaker filled with water to 70°C in a fume hood.

Put the NMP in an ultrasonic bath. Put the wafers in a holder in the NMP with ultra-sonic on.

Help the lift off process with a Clean tips swabs..

Do not use too much NMP.

Note: There are special bottles for used NMP that could be reused for the lift off process.

NOTE: When finished always turn of the DI-water.

41. INSPECTION: Linewidth and overlay ● Jun 2015

Visually inspect the wafers through a microscope, No Pt metal residues are allowed.

42. MANUAL COATING: Membranes and Metal Contaminated wafers ● Apr 2

Use the spray coater to coat resist.

The process consists of:

- Cover the inside of the coating station with aluminium foil
- Use the membrane chuck for **Contaminated** wafers
- spray coating of XXXX, dispensed by manual or out a syrince

Use program "HP-2ml-1000mbar 8layers".

Note: It is not allowed to change parameters of set spin recipes.

43. EXTRA RESIST BAKE ● Nov 1998

Use the Memmert oven in tunnel 1 to perform an extra resist bake:

⇨ bake the wafers at 140 °C for 30 minutes

44. WET ETCHING: Oxide etch, SAL ● Jan 1995

USE Special Glass beaker with Pt

Moisten Rinse for 1 minute in wet bench "H_2O/Triton X-100 tbv BHF 1:7". Use the carrier with the blue dot.

The bath contains 1 ml Triton X-100 per 5000 ml deionized water.

Etch Use wet bench "BHF 1:7 (SiO_2-ets)" at ambient temperature,.

The bath contains a buffered HF solution.

Time Etch until the **backside** of the wafer(s) is hydrophobic, plus an extra 30 seconds.

The required etch time depends on the layer thickness and composition.

The etch rate of thermally grown oxide is 1.3 ± 0.2 nm/s at 20 °C.

45. CLEANING

Rinse	Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.
Dry	Use the Ultra-pure-6 "rinser/dryer" with the standard program, and the orange carrier with a red dot.
Inspection	Visually, through a microscope. All the windows must be open and the hydrophobic test may be applied.

46. REMOVE RESIST WITH FRESH NMP ● Jun 2015

Use the NMP in SAL at 70°C in fume hood to lift-off the metal layer.

Use special glass container with Pt on the sticker.

Heat up the NMP in a beaker filled with water to 70°C in a fume hood.

Put the NMP in an ultrasonic bath. Put the wafers in a holder in the NMP with ultra-sonic on.

Help the lift off process with a Clean tips swabs..

Do not use too much NMP.

Note: There are special bottles for used NMP that could be reused for the lift off process.

NOTE: When finished always turn of the DI-water.

47. INSPECTION: ● Jun 2015

Visually inspect the wafers through a microscope, No resist residues are allowed.

48. LAYER STRIPPING: Photoresist ● Feb 1999

Strip resist	Use the Tepla Plasma 300 system to remove the photoresist in an oxygen plasma. Follow the instructions specified for the Tepla stripper, and use the quartz carrier.
Use	Special quarts carrier with Pt on it Use **program 4**: 1000 watts power for 15 minutes.

49. PECVD DEPOSITION: 50, 100 or 150 nm Silicon nitride ● Jul 1995

Use the Novellus Concept One PECVD reactor.

Follow the operating instructions from the manual when using this machine.

It is **not** allowed to change the process conditions and time from the deposition recipe!

Use macro **XXXnmSiNstd** (recipe xxxnmSiNstd) to deposit a chosen thick SiN layer.

Process conditions from recipe 800nmSiN:

Gasses & flows	Pressure	HF power	LF power	Temperature	Time
$N_2/SiH_4/NH_3$ = 1000/280/1800 sccm	2.8 Torr	320 W	480 W	400 °C	var $^{sec}/_{station}$

Note: The deposition time is subject to minor changes, in order to obtain the correct film thickness.

Note Use bare silicon wafer for measurement.

50. MEASUREMENT: Silicon Nitride thickness

Use the Leitz MPV-SP measurement system for layer thickness measurements.

Follow the operating instructions from the manual when using this equipment.

⇨ Program: SiN 4inch

Nitride thickness: ± 10 nm ⇨ on a bare silicon wafer

Option: ALD Al2O3

51. ALD Al2O3 ● Jan 1995

Tool:

Location:

Manual: *<location>*

Recipe name:

Settings: Check the selection switch on the gas box (see the tool manual).

Set the station deposition time (SDT), nominal value is2 sec, check recent depositions for the actual value

Notes: Change only the SDT in this recipe, leave other parameters unchanged.

52. MEASUREMENT: AL2O3 thickness ● Oct 1996

Tool: Leitz MPV-SP

Location: Class 100 tunnel 3

Manual: *<location>*

Recipe name: Th. Al2O3 on Si, >50nm auto5pts

Settings:

EN of Option: ALD Al2O3

NOTE: Use several test wafers to find the optimum illumination energy VS development time test with grey ND filter

53. Spray coat XXXX photoresist ● Nov 2008

Tool: EVG110 Spray coater/

Manual: *<location>*

Recipe name: --------

Settings: use manual flat alignment to align the flat edge of the wafers.

Process conditions		
Layer 1	Photoresist name and dilution	Pressure, nozzle settings and rotation speed etc.
Bake	Hotplate ---	Temperature 95 °C time 90 s proximity … µm

Notes: Always check the relative humidity (48 ± 2 %) in the room before coating, and follow the instructions for this equipment.

The backside of the wafers should be free of photoresist.

54. ALIGNMENT AND EXPOSURE on the PAS5500/80 waferstepper; MASK VIA ● Sep 2008

Tool: PAS5500/80 waferstepper

Location: Class 100 Litho room

Manual: *<location>*

User mask name: EI2200

Mask ID: 3X3

Exposure job: DIE6X6_9IMG

Layer ID: IMG_7

Focus: 0

Energy: 150

Alignment strategy: “GLOBAL”

Settings: no additional user settings are required.

Notes: The chuck-side of the wafers should be free of photoresist and other polymers.

55. DEVELOPING MANUAL ● Apr 2008

Use the developer area red room

Use special beakers with Pt

Developing with the AZ400K developer for YYYs.

Do not use to much developer and rinsing fluid (mostly water) during the developing..

Note: Dispose the developer into the right container if it is not water based.

NOTE: When finished always turn of the DI-water.

56. INSPECTION: Linewidth and overlay ● Feb 1996

Visually inspect the wafers through a microscope, and check the line width and overlay. No “ resist” residues are allowed.

57. PLASMA ETCHING: 50, 100 or 150 nm Silicon nitride ● Aug 1995

Use the Alcatel GIR300 Fluorine plasma etcher.

Follow the operating instructions from the manual when using this machine.

It is **not** allowed to change the process conditions from the etch recipe, except for the etch time!

Use the process from the table, and **set the etch time to ■'■■**".

Process conditions:					
Step	Gasses & flows	Pressure	RF power	He pressure	Etch time
1. bulk etch (RIE)	CF_4/SF6/O_2 = 70/10/10 sccm	0.05 mBar	60 W	not available	**variable**

58. **INSPECTION**: No residues are allowed.

SAL Option: ALD Al2O3

59. EXTRA RESIST BAKE ● Nov 1998

Use the Memmert oven in tunnel 1 to perform an extra resist bake:

⇨ bake the wafers at 120 °C for 30 minutes

60. WET ETCHING: Al_2O_3 etch: SAL ● Jun 2007

Etch Use wetbench "0.55% HF for metals", and the carrier with a red and black dot.

Time 1 minute. Al_2O_3 will only remain on the capacitance areas. 30 nm of aluminium will be lost.

Rinse Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.

Dry Use the Ultra-pure-6 "rinser/dryer" with the standard program, and the orange carrier with a black dot.

Note **Always apply an "OXYGEN PLASMA FLASH" step to your wafers prior to etching!**

61. WET ETCHING: Aluminium etch: SAL ● Jan 1995

Bake **If SPR3012 resist is being used: If the time between developing and wet etching exceeds 4 hours, an extra oven bake of 1 hour at 120 °C must be performed !**

If SPR3017M resist is being used: Always bake the wafers for 1 hour at 130 °C before wet etching!

Moisten Rinse for 1 minute in wet bench "H_2O/Triton X-100 tbv Al. Ets". Use the carrier with the yellow dot.

The bath contains 1 ml Triton X-100 per 5000 ml deionized water.

Etch Use wet bench "Al. ets 35°C", and the carrier with the yellow dot.

1 liter buffered aluminium etch fluid contains:

770 ml concentrated phosphorus acid (H3PO4, 85%), 19 ml concentrated nitric acid (HNO_3, 65 %),

140 ml concentrated acetic acid (CH3COOH, 100%) and 71 ml deionized water

Time Etch until the pattern is **clearly visible** over the whole wafer.

The required etch time depends on the layer thickness and composition.

The etch rate at 35 °C is ± 150 nm/min.

62. CLEANING

Rinse Rinse in the Quick Dump Rinser with the standard program until the resistivity is 5 MΩ.

Dry Use the Ultra-pure-6 "rinser/dryer" with the standard program, and the orange carrier with a **black** dot.

Inspection Visually, through a microscope. Cross-links between patterns are not allowed.

Note If the aluminium layer contains 1% silicon, then a "**POLY SILICON DIP ETCH**" step (30 seconds) **must** be performed after the wet etching, to remove the poly-Si grains that will deposit on the wafer surface.

63. REMOVE RESIST WITH FRESH NMP SAL ● Jun 2015

Use the NMP in SAL at 70°C in fume hood to lift-off the metal layer.

Use special glass container with Pt on the sticker.

Heat up the NMP in a beaker filled with water to 70°C in a fume hood.

Put the NMP in an ultrasonic bath. Put the wafers in a holder in the NMP with ultra-sonic on.

Help the lift off process with a Clean tips swabs..

Do not use too much NMP.

Note: There are special bottles for used NMP that could be reused for the lift off process.

NOTE: When finished always turn of the DI-water.

64. INSPECTION: Linewidth and overlay ● Jun 2015

Visually inspect the wafers through a microscope, No resist residues are allowed.

END Option: ALD Al2O3

65. LAYER STRIPPING: Photoresist ● Feb 1999

Strip resist Use the Tepla Plasma 300 system to remove the photoresist in an oxygen plasma.

Follow the instructions specified for the Tepla stripper, and use the quartz carrier.

Use Special quarts carrier with Pt on it

Use **program 4**: 1000 watts power for 15 minutes.

66. INSPECTION: resist removal ● July 2016

Visually inspect the wafers.

The front and back side of the wafer should be clean of any photoresist residues.

67. Spray coat XXXX photoresist ● Nov 2008

Tool: EVG110 Spray coater/

Manual: *<location>*

Recipe name: --------

Settings: use manual flat alignment to align the flat edge of the wafers.

Process conditions		
Layer 1	Photoresist name and dilution	Pressure, nozzle settings and rotation speed etc.
Bake	Hotplate ---	Temperature 95 °C time 90 s proximity … µm

Notes: Always check the relative humidity (48 ± 2 %) in the room before coating, and follow the instructions for this equipment.

The backside of the wafers should be free of photoresist.

68. ALIGNMENT AND EXPOSURE on the PAS5500/80 waferstepper: MASK PARYLENE ● Sep 2008

Tool: PAS5500/80 waferstepper

Location: Class 100 Litho room

Manual: *<location>*

User mask name: EI2200

Mask ID: 3X3

Exposure job: DIE6X6_9IMG

Layer ID: IMG_8

Focus: 0

Energy: 150

Alignment strategy: “GLOBAL”

Settings: no additional user settings are required.

Notes: The chuck-side of the wafers should be free of photoresist and other polymers.

69. DEVELOP with Single Puddle process● Apr 2008

Tool: EVG120 coater/developer

Location: Class 100 Litho room

Manual: *<location>*

Recipe name: 1-DEV-SP

Settings: use manual flat alignment to align the flat edge of the wafers.

Process conditions		
Post exposure bake		Temperature 115 °C time 90 s proximity … µm

develop	MF322	Dispense at … rpm, Puddle at 0 rpm Time 56 s,
Rinse/dry	DI water	rinse with DI water, spin-dry at … rpm.
Hard bake		Temperature 100 °C time 90 s proximity … µm

70. INSPECTION: Pattern quality, linewidth and overlay ● July 2016

Visually inspect the wafers through a microscope.

For positive photoresists, the exposed areas should be clean and free of any photoresist residues, for negative photoresists, the unexposed areas should be clean and free of any photoresist residues. The resist areas should show a smooth surface, clean, free of bubbles and particles.

Measure the linewidth using SEM or optical microscope.

Linewidth measurements (delete or add more tables when needed)

Linewidth in micrometer

Check the overlay with verniers or box-in-box structures when available

Overlay measurements (delete or add more tables when needed)

Overlay in micrometer

71. PARYLENE DEPOSITION ● …

Tool: --
Location: MEMS lab
Manual: *<location>*
Recipe name: --
Settings: --, check recent depositions for the actual value
Notes: .

72. MEASUREMENT: Parlene thickness ● …

Tool: Leitz MPV-SP
Location: Class 100 tunnel 3
Manual: *<location>*
Recipe name: --
Settings:

73. Parylene Etching ● …

Tool: Alkatel
Location: CR100
Manual: *<location>*
Recipe name: ----
Settings: no user settings required
Notes:

74. INSPECTION: ● July 2016

Visually inspect the wafers.

Appendix 2. List of Figures

Appendix 3. List of Tables