A Low-Leakage 3-Way Silicon Microvalve

by

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BARKER
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ABSTRACT

This thesis presents an electrostatically actuated silicon microvalve designed for use in a miniature gas chromatography system for sample preparation and injection. In contrast to prior art, this design combines an integrated low voltage electrostatic microactuator as well as tight sealing capability. The device uses only silicon and silicon dioxide, which allows it to be used in a gas chromatography system for a wide range of chemically compatible gases. Using a 3-way design, the valve can switch an input gas flow to either of two output ports, which helps reducing wafer space consumption if used in a valve array, by combining two 2-way on/off valves into one device. After a thorough discussion of prior art in microvalve technology, the design and modeling of the microvalve is presented. A detailed discussion of the microfabrication issues is given, along with a final process plan. One-sided prototype valves were fabricated and their performance was characterized. Leakage rates on the order of $10^{-6}$ atm-cc/sec have been measured. The prototype valves are capable of switching inlet pressures of typically 8 psi with open flow rates of 8 sccm Nitrogen at an operation voltage of 23 V.

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Recent efforts in microsystem design and microfluidics have led to the development of Micro Total Analysis Systems (uTAS), where different chemical or physical processes are carried out on a very small scale, essentially on the surface of a microchip. One branch of these types of systems is miniature gas chromatographs. In gas chromatography systems, a small amount of sample gas is first isolated from the main stream and then a carrier gas is used to push the sample gas through a small capillary, called the separation column. There are many different types of separations columns, as shown in Figure 1.1. Usually the inside of the column is lined or filled with a material that has a temperature dependent affinity/solubility to the different molecules of the sample gas. During chromatography, the components of the sample gas are adsorbed into the column lining with different intensities, which causes each component to be delayed and thus require a unique travel time through the column. These travel times are dependent on temperature and often, the column assembly of a GC system is equipped with a heater. If the sample gas contains components that have very short as well as very long travel times, the heater can be used to speed up the analysis after the fast traveling components have left the column.

At the end of the separation column, a sensor is waiting to detect each component that leaves the column. There are several different detector technologies with different ranges and sensitivities. The detectors used by the sponsor company, ABB, are the FID (flame ionization detector) and the TCD (thermal conductivity detector). The FID works by
burning the gas that exits the separation column. Hydrogen is supplied to help burn the sample gas and to maintain a pilot flame when the detector is idling. Burning the gas generates ions that cause a measurable current. If a burst of a specific gas leaves the column, a change in current can be measured.

The TCD uses a thin electric wire that is suspended in the gas flow that emerges from the column. It’s resistance is dependent on the thermal conductivity and therefore it can sense changes in the composition of the gas that leaves the column.

Gas Chromatography only works with prior calibration, meaning that the detector can not sense what kind of gas it is detecting, but it gives an indication if there is a gas different from the carrier gas. Because of the different travel times, the gas mixture that leaves the column is carrier gas, interrupted by small bursts of the individual gas components. Each time the detector senses a different gas, a peak in the chromatogram is created. The flats between the peaks indicate the time when only carrier gas has passed the detector. Together with calibration data obtained prior to the actual measurement, the travel time indicates the type of gas component present in the gas mixture. Figure 1.2 shows a typical
Gas chromatogram, where the intensity of the detector output is plotted versus time. Each peak represents a different gas component.

Gas chromatography systems usually require a plurality of valves and fluid channels for sample preparation and to connect the different system components. An example of a layout of a gas chromatography system is shown in Figure 1.3. An array of valves is used to route the gas flows. The elements labeled as V1 through V10 are one way on/off valves.

There are basically two modes that this system operates in. In idle mode, all even numbered valves are open and all the odd number valves are closed. As a result the gas flow follows the solid lines in the figure. The sample gas flows through the sample loop, the carrier gas flows through the column backwards, which is called "backflush", because it is done to clean the column from residual sample gas from previous analyses. Also, carrier gas is led into the detectors for baseline calibration.
In injection mode, the even numbered valves are closed, and the odd numbered valves are opened so that the gas streams follow the dashed lines. The sample gas is now bypassed around the system and flows to "sample out". The amount of sample gas still trapped in the sample loop (from running in idle mode) is now pushed into the column by the carrier gas via valves V3 and V8. In the column, separation by travel time occurs. The separated gas is then pushed out of the column to the detectors via valve V5 to complete the analysis.

The valves described here are designed to work with such a system. Considerable emphasis is put on a low leakage rate as this is a key requirement to prevent sample contamination.

**Figure 1.3** Example of the layout of a gas chromatography system by ABB
Chapter 2

DISCUSSION OF PRIOR ART

Countless different microvalve designs have been reported and patented. After a thorough literature search, approximately 30 journal papers and more than 50 patents have been found as summarized below.

Microvalves have been designed in many different ways. There are, for example, passive valves that act as check valves, allowing fluid to flow only in one direction. Such valves have been reported by Hoek et. al. [1], Trah [2], Smith et. al. [3], Barth [4], Ulrich et. al. [5], Koch et. al. [6], Oosterbroek [7], and Evans et. al. [8]. These kinds of valves are often used in combination with micropumps, where a check valve is needed at the inlet as well as at the outlet of, for example, a membrane pump in order to allow flow into one direction and block it into the backwards direction. For medical applications, the check valves used in a design by Van Lintel [9], [10] are equipped with electrodes for the detection of malfunctions.

Active valves that are equipped with an actuator can open or close against a fluid flow. In some cases, an external actuator consisting of conventionally manufactured parts is attached to a silicon micromachined valve body, which includes the inlet and outlet channels as well as the valve seat and the closing member. Valves of this kind have been reported by Giachino et. al. [11], [12], [13], [14], and Bergstrom [15] as part of an automotive fuel injector system, Reinicke [16] and Pourahmadi [17] use externally attached electromagnetic solenoids to actuate their microfabricated valves. Meckes et. al.
[18] use a permanent magnet attached to the backside of the wafer, and a gold plated coil on top of a membrane exerts an actuation force on the membrane when a current is passed through. Yanagisawa and Hirano et. al. [19], [20] have presented a microvalve with a membrane made from a sputtered NiFe layer. An external coil provides the magnetic field to move the membrane for opening and closing the valve. The valve also claims a very low leakage rate due to the tight conforming of valve seat and closing member.

A valve that utilizes an externally attached piezo stack has been presented by Shoji et. al. [21]. Normally open, normally closed, and 3-way valve designs are proposed. The valve consists of a polymer membrane spun over an inlet and an outlet port. The membrane is moved by an attached piezo stack assembly. A similar principle is used by Chakraborty et. al. [22] for the design of a valve for space applications. Ernst et. al. [23] use a tri-morph piezo bending actuator which is externally attached by using an epoxy based adhesive. An external piezoelectrically actuated valve that advertises a low leakage rate has been published by Yang et. al. [24]. Low leakage is achieved by concentric valve seat rings as found in labyrinth seals, and a pre-loaded closing member.

Other externally actuated valves, sometimes made entirely by using conventional non-microfabrication methods, use an externally pressurized control fluid (e.g. pressurized air) that is used to deflect a membrane structure which in turn closes against a valve seat, such as reported by Sittler et. al. [25], Mehaffy et. al. [26], Wylie et. al. [27], Levenson et. al. [28], Sim et. al. [29], [30], Vicider et. al. [31], and Takao et. al. [32], [33]. The disadvantage of such valves is that a mostly larger scale external pilot valve is needed to control the pressurized control fluid, which might not be an efficient design if overall system miniaturization is the desired goal.

An interesting microvalve design incorporating an integrated pilot valve stage has been described by Carr [34]. It is a pressure balanced design where both sides of a closing member membrane are pressurized with the working fluid. A small pilot valve in the
membrane backside chamber is used to influence the pressure differential and thereby moving the membrane which closes or opens the main valve.

Valves that are entirely made in silicon technology use many different actuator principles, such as electrostatic, electromagnetic, thermal, thermopneumatic, piezo electric, and shape memory alloy (SMA). Even a thermally driven paraffin actuator and a diffusion driven hydrogel actuator have been utilized to actuate a microvalve structure. The following paragraphs will discuss the prior art of each kind.

A valve design incorporating an SMA actuator has been published by Hahm et. al. [35]. A layer of TiNi, which acts as the SMA membrane is pre-loaded by a spring, which makes the valve normally closed. Local heating of the SMA opens the valve by deforming the spring, which pushes the SMA layer back after cool down.

A paraffin actuated microvalve has been reported by Carlen et. al. [36]. A trapped amount of micro structured paraffin is melted with a microheater. The volumetric expansion due to the melting process deforms a membrane, which presses against a valve seat to close a normally open valve.

Hydrogel actuation has been reported by Baldi et. al. [37] and Liu et. al. [38], where the swelling of trapped amount of hydrogel, due to changes in concentration of specific chemical species in an external liquid environment, is used to actuate a rubber membrane, which in turn drives a membrane that engages in a valve seat.

Another exotic type of actuator is the thermopneumatic kind where an actuation pressure is created by heating a small amount of trapped fluid, as reported by Zdeblick et. al. [39], [40], [41], [42], [43], and in a similar design by Rich et. al. [44], [45]. The evaporation pressure of the boiling fluid deforms a membrane which closes against a valve seat. The thermopneumatic valves published by Evans et. al. [46] and Papavasiliou et. al. [47] use a bubble generated by a heater to push a set of fins lateral to the wafer surface against a spring, which opens the normally closed valve. To close the valve, the bubble is bypassed.
to the other side of the fins by a second micro heater creating a vapor bypass channel. If no external power is applied, the bubble stays in its current position, which makes the valve bistable.

Most thermally actuated microvalves work by having membranes that are suited with micro heaters. Membranes are either used with uniform thickness or with a central boss that closes or opens against a valve seat. Turning on the micro heaters causes thermal expansion of the membrane, which will then buckle under the compressive stress and thereby move away from or towards the valve seat, depending on whether the valve is a normally closed or normally open design. Valves with this fundamental principle have been presented by Jerman [48], [49], Gordon et. al. [50], Beatty et. al. [51], Barth et. al. [52], Lisec et. al. [53], Franz et. al. [54], Popescu et. al. [55], and Tsai [56].

Electromagnetically actuated valves are often made by having parts of the actuator, either the coil or a permanent magnet, externally attached to the microvalve. There are some electrostatic microvalves, however, that are entirely microfabricated. Sadler et. al. [57] have made a normally closed microvalve where a silicon membrane is pre-loaded over two adjacent valve seats. An inductor made from electroplated coils induces a magnetic field, which in turn exerts a force on a Ni/Fe pad electroplated on top of the membrane. Mikkor [58] has proposed a fuel injector valve that uses a similar principle, with a permanent magnet attached to a membrane which is pushed or pulled by the magnetic field of an inductor coil fabricated into the substrate.

An unusual electromagnetic valve design has been proposed by Schutten et. al. [59]. Two electrically conductive layers sandwiched on top of each other are separated by an insulating layer. Each layer has holes that are not aligned with each other, so that when the layers are touching, fluid can not pass through the holes. The electromagnetic interaction of currents passed through each layer causes the layers to repel from one another, opening a fluid passage through the valve.
An electromagnetic microvalve that also implements bistability has been reported by Capanu et. al. [60]. The closing member consists of a cantilever with a rigid part and a flexible part. The flexible part allows rotation of the rigid part about the edge of the valve seat, and it is the elastic force in the flexible part that keeps the valve normally closed. A microfabricated coil is used to either increase or decrease the flux of a small pad of Ni/Fe soft magnetic alloy, which causes the valve to toggle between the open and closed state.

Piezoelectric actuators are known for their high force and relatively small strokes. Roberts et. al. [61] and Li et. al. [62] have designed a microvalve where a drive piston with different areas is used as a force-stroke converter in order to increase the stroke on a valve membrane by utilizing the abundant force of a piezoelectric element.

The prior art on electrostatically actuated microvalves is most relevant for this work and shall therefore be discussed in more detail where necessary.

A "latched" microvalve design that uses pneumatic action with subsequent electrostatic holding has been claimed by Pan et. al. [63], [64]. It uses membranes that are initially actuated with a primary force, i.e. external pneumatic pressure. Then, the membrane is held by applying an electrostatic charge between the membrane and the substrate.

Another electrostatically actuated microvalve has been published by Huff et. al. [65], [66], [67], Mettner et. al. [68], [69], and Kober [70]. Figure 2.1 shows a schematic. As a countermeasure to the well known weakness of electrostatic force in microsystems, the valve incorporates a pressure balanced design. A sealed cavity contains the electrostatic actuator. The fluid pressure that acts on the closing member also acts on the bottom membrane, which cancels the fluidic forces completely, so that the electrostatic actuator only has to provide enough force to deform the bottom membrane. Thus, the electrostatic actuator can be decoupled and isolated from the fluid and the fluid pressure.

Another electrostatic valve design has been suggested by Bonne and Ohnstein et. al.[71], [72], [73], [74], [75], [76], [77]. Several different incarnations of a similar valve design
are described. Some valves have doubly clamped beams, while other designs have cantilevers. The valves are surface micromachined with buried electrodes in the beams and substrates. Energizing the electrodes causes the beams to pull in onto the substrate, which blocks the fluid ports.

Electrostatic valves that use a traveling wave actuator have been reported by Shikida and Sato et. al. [78], [79], [80], [81], [82], [83], [84], [85], [86], [87], [88] and in a similar way by Biegelsen et. al. [89], [90], and Cabuz et. al. [91]. A conductive film is confined between plates in a single or double S-shape configuration. Electrodes embedded in the confinement plates can be energized to move the "wrinkle" in the film to either side. Holes in the plates are used as fluid vias, which are either open or closed, depending on where the wrinkle in the film is located.
Another way to overcome the limited force capability of electrostatic actuators is to use a zipper type actuator as proposed by Aine et al. [92] and Haji-Babaei [93]. An initially curled leaf spring member is pulled onto a substrate containing a port hole, closing the fluid flow through the same. Branebjerg et al. [94] have reported a zipper design incorporating an initially upwardly bowed membrane that is clamped around the perimeter. An electrode underneath the membrane slowly pulls in the membrane from the edges to the center. A boss in the center of the membrane can be used to close against a valve seat. Yobas et al. [95] utilize the zipper effect in a normally closed microvalve to attract a closing membrane that has been deformed by fluid pressure.

An interesting technique to incorporate bistability into an electrostatically actuated microvalve design has been reported by Wagner et al. [96], [97] as shown in Figure 2.2.

An electrically conductive silicon membrane is spun over two cavities with electrodes. The cavities are pneumatically connected to each other by vent vias. The membranes operate in counteraction. If one electrode is pulled down electrostatically, the other is pushed up pneumatically and vice versa. An interesting aspect of the microfabrication
process is that grey tone lithography is used to create the dome shaped indentations for the electrodes.

Mettner [98] has claimed a valve design with a slider suspended by flexures. The slider has fluid passages and it is sandwiched between two plates, which have fluid passages as well. If the slider is moved, the holes can either be lined up or offset, so that the valve is either open or closed. The valve is similar to a conventional solenoid driven piston valve, where different pressure, drain, and work ports can be selectively connected to one another depending on the axial position of a piston and the design of the housing.

Another electrostatic valve design has been claimed by Wise et. al. [101], where a valve is formed by a series of surface micromachining steps. The valve consists of a cantilevered closing member that is pulled in electrostatically onto a valve seat.

A very common way of making a microvalve structure is to suspend a membrane over a recess to create a sealed cavity. The membrane is in most cases electrically conductive and it can be actuated in combination with electrodes embedded in the substrate. Microvalves that are made this way usually show the most compact design and are usually the easiest to manufacture with current microfabrication techniques. The deformation of the membrane transmits a displacement action into the cavity while maintaining a sealed and closed system. These valves usually have an inlet and an outlet port fluidically connected to the sealed cavity. The position of the membrane then determines when the valve is open and closed.

Ohtaka [99], [100] has claimed a microvalve design where a membrane is formed using electroplating on a sacrificial layer covering a valve seat, as shown in Figure 2.3. The valve seat has the shape of a nozzle and the sacrificial layer covers the valve seat shape evenly. After the electroplating, the sacrificial layer is dissolved and the membrane has a shape that conforms very closely to the valve seat shape. Electrostatic actuation is done with the conductive membrane and electrodes embedded into the substrate.
Another membrane based microvalve design has been claimed by O'Connor [102]. As shown in Figure 2.4, a membrane is formed over a substrate with a valve seat and port holes. The substrate itself and a conductive pad on top of the membrane can be electrically energized to close the normally open valve.

A microvalve with a diaphragm made from polyimide is presented by Goll et al. [103]. As shown in Figure 2.5, it uses a buckled polyimide diaphragm with a soft silicone platelet which closes against an inlet orifice. The membrane is mechanically bistable and a resistive heater together with a small orifice is used to provide the pressure bursts by thermal expansion which move the membrane between states. A short burst in heating current will increase the temperature of the air trapped inside the actuation chamber very
quickly. Due to the ideal gas law, the pressure also increases very quickly. The orifice in the bottom of the actuator chamber is designed small enough, so that the air does not get a chance to escape before it has pushed the diaphragm up into the closed valve state (as shown in the picture). After a certain time has passed, the excess hot air has entirely escaped from the chamber through the orifice, and the chamber pressure is atmospheric (while still hot). The return stroke is done by stopping the heating current, which causes
the air in the chamber to cool down. The resulting drop in pressure pulls the diaphragm away from the orifice towards the actuation chamber into the open valve state. Again, the size of the orifice provides enough resistance so that the inflow of air from the outside is slow, giving the vacuum enough time to pull back the membrane into the other mechanically stable state.

A microvalve with an open diaphragm has been reported by Vandelli et al. [104]. Instead of having a closed cavity with both, the inlet and the outlet holes going through the substrate of the wafer, it uses a surface micromachined diaphragm which is open to the sides. When the valve is open, fluid flowing in from the inlet port in the substrate flows around the diaphragm and out through the top as schematically shown in Figure 2.6.

![Figure 2.6 Surface micromachined diaphragm valves by Vandelli et al.](image)

Additional packaging is needed to confine the inflow and outflow.

A similar type of valve has been reported by Dubois et al. [105], which uses a deformable doubly clamped diaphragm made from Ta-Si-N that is suspended over an inlet hole in the
substrate, as shown in Figure 2.7. A special geometry of the membrane includes a side tab

![Diagram of microvalve design by Dubois et al.](image)

Figure 2.7 Microvalve design by Dubois et al.

(not shown in Figure) which reduces the pull-in voltage to values as low as a few volts. Pulse width modulation of the actuation voltage is used to create a proportional flow control. The membrane is made by sputtering the Ta-Si-N alloy onto a sacrificial Aluminum layer. Silicon dioxide is used as an insulator. For higher flow rates, a number of valves are arranged in an parallel array.

There are also electrostatically actuated valves where an elastically suspended rigid valve plate is used instead of a deformable membrane. The elastic deformation is confined to specific regions, best described as elastic tethers, whereas the valve plate, which is the closing member that engages into the valve seat, is kept rigid and does not deform during operation. Such a valve design, for example, has been claimed by Doering et al. [106] as shown in Figure 2.8. The design uses three layers, with the outer layers having an inlet as well as an outlet port. The center layer has a moveable member that is held by flexible silicon strips. Electrostatic actuation moves the center piece up and down so that the fluidic connection between the port holes can be changed. The image shows a configuration where the valve is used to control the fluid exchange between two fluid lines running at different pressure levels.
Another tether based microvalve design has been published by Messner et al. [107] as shown in Figure 2.9. A silicon bulk micromachined 3-way microvalve is described which is made of three bonded layers. The valve plate is held by a flexible suspension and is electrostatically actuated to achieve two different fluid communication configurations between an inlet, and outlet, and an exhaust port. The valve is claimed to be useful for industrial pneumatic applications, where it can be used as a 3-port 2-position control valve. The fabrication requires a plurality of KOH etch steps and about 10 masks are necessary for the lithography. The valve is normally closed by having a pre-loaded valve plate, which determines the maximum inlet pressure against which the valve can seal. The amount of pre-load is limited because it has to be overcome by the electrostatic actuator. Hence, the maximum switchable pressure is limited as well.

**Conclusions:**

The overwhelming number of prior microvalve designs is a clear indication that there is no single best all-round microvalve. Each one of these designs has their strengths and
weaknesses. The functional requirements of the target application have to be considered in
detail when choosing a particular valve design. Going back to the requirements for
microvalves useful for gas chromatographs, the most important performance parameter is
the leakage rate when the valve is closed to prevent cross-contamination within the system
which can skew the chromatogram. Therefore, from all the microvalves reported in the
literature, only the ones where low leakage values have been observed shall be taken here
for a final comparison. Unfortunately, different research groups use different units for
pressure, flow rate, leak rate etc. Additionally, the specific conditions (i.e. temperature
and pressure) at which gas flow rate values are given are not always explicitly and
consistently reported, which makes a detailed comparison difficult. However, an attempt
has been made here to convert all data into common units. Furthermore, leak rates in the
literature have been measured under different valve inlet pressures. To account for this,
we have divided the leak rate values by the inlet pressure at which the leak rate was
measured, which yields a value that may be interpreted as a "leak conductance". This
value represents the volumetric flow rate at 1 Pa reference pressure per Pa of inlet pressure. Figure 2.10 shows a comparison table of the most relevant references where

<table>
<thead>
<tr>
<th>Ref.</th>
<th>Pressure units</th>
<th>Open Flow Rate (test gas)</th>
<th>Leakage conductance</th>
<th>Type of actuator</th>
<th>Voltage [V]</th>
<th>Power [mW]</th>
<th>Materials used</th>
</tr>
</thead>
<tbody>
<tr>
<td>[103]</td>
<td>0.04</td>
<td>3.3·10^{-5} (N_{2})</td>
<td>3.33·10^{-12}</td>
<td>TH</td>
<td>)*</td>
<td>)*</td>
<td>PMMA, polyimide</td>
</tr>
<tr>
<td>[20]</td>
<td>0.1</td>
<td>3.2·10^{-4} (He)</td>
<td>5.8·10^{-15}</td>
<td>EM/EXT</td>
<td>30</td>
<td>)*</td>
<td>Si, SiN, NiFe</td>
</tr>
<tr>
<td>[36]</td>
<td>0.107</td>
<td>1.67·10^{-4} (N_{2})</td>
<td>7.83·10^{-12}</td>
<td>TH</td>
<td>)*</td>
<td>100</td>
<td>Paraffin, parylene, Au, Cr, SiO_{2}, glass</td>
</tr>
<tr>
<td>[24]</td>
<td>2.07</td>
<td>0.0868 (He)</td>
<td>1.21·10^{-14}</td>
<td>PZ/EXT</td>
<td>30</td>
<td>3</td>
<td>Si, SiO_{2}, metal</td>
</tr>
</tbody>
</table>

**NOTE:** Most values obtained after unit conversion

*) = not reported; **) = concluded from paper, not explicitly given

Actuator type abbreviations:

"EM" = electromagnetic, "TH" = thermal, "PZ" = piezo, "EXT" = external actuator

Figure 2.10 Microvalve Comparison Table

After conversion to common units, a leakage conductance on the order of 10^{-12} Pa·m^{3}/s/Pa or less have been found. To our knowledge, the lowest leakage rates for silicon microvalves were reported by Hirano [20] and Yang [24]. Both valves achieve respectable leakage rates, but both need an externally attached actuator. The lowest leakage rates for microvalves with integrated micro actuation were reported by Goll [103] and Carlen [36]. However, these valves use materials that are not a good choice for gas chromatography applications due to their insufficient thermal, chemical, and/or physical performance.

The mechanisms to achieve low leakage in these valves are quite different. Goll [103] uses silicone, a very compliant polymer to form an elastic seal against an orifice. Hirano [20] and Carlen [36] use a process that will produce mechanical members with very complementary surfaces. This is done by depositing a sacrificial layer on top of a valve seat surface. This layer then serves as a substrate for depositing the closing member layer. If the sacrificial layer is very uniform in thickness, it will leave a very uniform gap and
hence very complementary surfaces between the valve seat and the closing member after it is dissolved or etched away.

Yang [24] uses a labyrinth seal combined with a large positive sealing force. The valve seat consists of several concentric rings, all covered by a flat valve plate. The valve plate is held by relatively thick tethers. Due to the geometry of the valve parts, the valve is normally closed and pre-loaded after assembly. The pre-load force that keeps the valve closed is quite large due to the thickness of the tethers, which helps sealing against large pressures. A piezo stack is used to pull the valve plate off the seat to open the valve.

From these observations we have chosen to use the principle of complementary surfaces for the sealing mechanism. Using compliant polymers to form a seal would limit the operating temperature of the valves and would furthermore impose significant restrictions on the thermal budget during a microfabrication process. We also wanted to avoid using an external actuator since the labor cost of manual assembly time defeats the economic advantages of batch fabrication. Our fundamental philosophy is that complementary surfaces are very economically available by simply utilizing the polished surfaces of standard wafers, which are already flat to typically 5 nm. The cost of such wafers is essentially negligible compared to, for example, cost of cleanroom processing. The following chapters will explain, how we developed a fabrication process to incorporate these principles into a microvalve design.
Chapter 3

DESIGN AND MODELING

3.1 Introduction

The fundamental purpose of a valve is to control the flow of a fluid, either proportionally or in an on-off mode. Therefore, every valve represents a boundary between a higher pressure and a lower pressure region of a fluid. The pressure drop always causes forces on certain parts of the valve, which either have to be overcome by the valve actuator, or with a pressure balanced design. The latter often increases the design complexity since additional counteracting pressure cavities are needed. This chapter will describe various valve design strategies and concepts and their operating physics.

3.2 Principal Strategies

Figure 3.1 illustrates different valve design strategies that create a barrier for the flow of a fluid through a port. Figure 3.1a shows a valve where the closing member is lifted normal to a valve seat surface. Figure 3.1b shows a sliding valve member. Figure 3.1c shows the closing member being pivoted inside the port to reduce the covered projected area. Another valve principle is the peeling lift off of the closing member as shown in Figure 3.1d. And yet another way of restricting flow is by squeezing a compliant duct as indicated in Figure 3.1e. All of these principles can be incorporated in various valve design concepts, either separately or in combination. The following section will describe
several different valve concepts and discuss their implementation from the point of view of microfabrication constraints.

### 3.3 Valve Design Concepts

The different strategies described in Section 3.2 can be embodied into different concepts to meet the system's functional requirements. An important part of concept evaluation is fabrication complexity and the anticipated sealing capability i.e. the quality of the sealing surfaces of which the valve seat and the closing member will be made. Figure 3.2 shows a linear gate valve. A slab of material is inserted into the fluid duct. A linear actuator drives the slab in and out of the duct, restricting the flow. The advantages of such a valve is that there is virtually no dead volume used for the valve itself. The stroke required from the actuator is the same as the diameter of the channel. For good sealing it will also be required that the shape of the gate and that of the duct will be extremely accurate, otherwise leakage is expected. Furthermore, if the gate and duct are implemented as shown above, friction between the flap and the sidewall can cause wear.
In another implementation, the valve can have a rotating gate as opposed to a linearly moving one as shown in Figure 3.3. Instead of a linear actuator, a rotary actuator is required for controlling this type of valve. Similar to the linear gate valve, the accuracy requirements on the duct surfaces and the gate shape are very high in order to achieve good sealing. Both, the linear and the rotary gate valve need a seal where the gate penetrates the duct in order to prevent fluid from leaking out into the environment. In addition, sealing in the corners could be extremely difficult.
Figure 3.4 shows a linear aperture valve. It is similar in function to the linear gate valve in that it has a gate that moves linearly to control flow. The difference is that it has a fixed wall with a fixed hole pattern. The gate has the same hole pattern, but offset by one pitch length. The valve is open when the hole pattern in the gate coincides with the hole pattern in the flap and it is closed when the gate moves by half a pitch. This means that this valve can be designed for a very small actuator stroke, which is necessary for most MEMS actuators. On the other hand, the area for the flow in the open state is at most only half that of the total duct area. The gate must be preloaded against the stationary wall to ensure sealing and during gate movement, friction may be generated. Depending on which side of the flow the gate is located, the pressure drop can increase this frictional force considerably, requiring large actuator forces.

Figure 3.5 shows a rotary version of the aperture valve. The functionality, and the limitations and advantages are similar to the linear aperture valve. Instead of a linearly sliding bearing, a rotary bearing or pin joint is needed in the center of the stationary plate.

Yet another valve design is shown in Figure 3.6. A rigid plate with an orifice constitutes the valve body and a compliant plate is used as a closing member. In this design, the closing plate is pulled down onto the substrate starting at one end. The motion in this
actuator is similar to that of a zipper on clothing. It can be a challenge to fabricate the normally curled closing plate because the residual stresses have to be controlled very carefully.

Figure 3.7 shows a switch valve. In contrast to all the other valve designs described above, this valve has one inlet and two outlets. Without the second outlet, it could also be designed into a simple on/off valve. The challenge here is actuation as well as the sealing of the sliding flap on the top and the bottom surfaces and in the corners, which pose potential leakage points.

A very common type of microvalve is depicted in Figure 3.8. A shallow cavity with two ports is covered by a membrane. The membrane can be actuated for example
electrostatically, pneumatically, or thermally. The reason why it is so common is that the geometry is favorable for microfabrication. It is very easy to fabricate a shallow recess
into a silicon substrate, and it is easy to create a membrane that can be actuated electrostatically. One of the disadvantages of this design is that there is a fairly large dead volume inside the valve cavity, which comes from the requirement that the lateral dimension of the membrane must be much larger than the deflection it allows. This fact also dictates that the membrane consumes a large amount of lateral design space compared to the actual valve port area. The biggest advantage, however, is that a leakage free sealed cavity can be formed with the compliant membrane. Challenges are to fabricate the membrane stress-free (or with tensile stresses so it is self-stretching). Compressive residual stresses will cause it to buckle. The sealing capability of such a valve is dependent on the surface quality of the valve seat and the membrane underside as well as the deformation of the membrane in the valve seat area due to the elastic deflection.

Another valve design that can be either a 2- or 3-way valve is shown in Figure 3.9. A centrally clamped plate is deformed such that the edges touch down on the bottom or top surfaces of a cylinder shaped valve cavity. An inlet located on a central layer delivers the fluid which can then be routed through outlet A or B depending on which way the centrally clamped plate is deflected.
A valve that takes advantage of the elastic deformation of a fluid duct is shown in Figure 3.10. These kinds of valves are commonly referred to as "pinch valves". The figure shows a normally open as well as a normally closed version of a pinch valve. This kind of valve has been implemented in microfabrication by means of soft lithography, where a silicon master is microfabricated to serve as a mold and then PDMS is poured over the mold and after solidification, small microchannels are formed. Microchannels cross each other and pneumatic pressure can expand one channel, pinching off the other [108]. From a gas chromatography point of view, compliant channels made from organic materials are less preferable since the structure can retain small amounts of sample gas, release them later over time and thus contaminate and skew subsequent analyses. Materials for GC applications also have high requirements on chemical inertness and temperature stability.

3.4 Functional Requirements

The target application of the microvalve described here is gas chromatography. The underlying efforts of the sponsor company was to create a micro version of a gas chromatograph, fabricated on a silicon chip. Apart from the valves, several other system components are needed, such as separation columns and detectors. According to this application, the functional requirements of the microvalves have been defined as:
- good sealing capability (10^{-8} \text{ atm-cc/sec} \text{ at 1 bar pressure drop});
- operating voltage less than 100 V;
- durability (20 x 10^6 cycles);
- low wafer space consumption;
- low cost;
- chemically inert (resistent to CxHy, CO2, CO, O2, H2O, H2S, H2, NH3);
- compact packaging of valve array;
- temperature range of 0 to 120 degrees C;
- switching pressures of several atmospheres;
- switching time less than 20 ms;
- no retaining of sample molecules (e.g. avoid use of polymers);
- suitable for the existing system of fluid channel cross sections of 100 x 30 microns.

The most critical part of a valve are the surfaces that are involved in the sealing action when the valve is closed. Since a very low leakage rate has to be achieved, the requirements for those surfaces are quite high. In macro scale valve designs, low leakage rates of fluid valves are achieved by squeezing a compliant material (i.e. rubber) between two rigid members, which causes perfect surface contact between the rigid and the compliant material. The sealing capability is then only limited by diffusion of the fluid molecules through the valve materials. For the gas chromatography microvalves, however, compliant materials are not tolerable, since they can retain some of the sample fluid that is being analyzed and then skew the results for subsequent analyses. Furthermore, some GC devices work in temperature ranges of up to several hundred degrees C, in which the valves have to survive (e.g. if a liquid has to be analyzed, a vaporizer heats up the sample to beyond the boiling point or the columns are heated so that the analysis times for different compounds can be shortened). This rules out most organic compounds or polymers as sealing materials. Suitable materials that are typically available in silicon microfabrication include silicon, silicon dioxide, silicon nitride and some metals.
3.5 Variations of the Membrane Valve Design Concept

A series of valve designs have been conceived that could achieve the functional requirements. The valve was to be microfabricated in silicon technology, which was assumed to be the most cost effective method since a large number of small devices can be fabricated in a batch. However, microfabrication also comes with very tight design constraints in terms of achievable geometries and processable materials. This must be kept in mind when designing a MEMS device, in addition to other fabrication limits such as thermal budget, contamination budget, and achievable accuracy.

From all the previously described valve concepts, a membrane type valve was chosen because of the possibility to use it as a 3-way switch valve as well as an on/off valve. Dead volume can be made acceptable by minimizing cavity depth. An initial valve design, as shown in Figure 3.11, consisted of a centrally clamped membrane inside a cylindrical cavity having a lateral inlet, and outlet fluid paths on the top and the bottom surfaces. With the fluid entering the valve cavity from the side, bending the membrane towards the top causes the fluid would flow through the bottom, and vice versa. The contact between the
Variations of the Membrane Valve Design Concept

valve plate and the substrate will be a circular line. Any deviations of the circumference of the valve plate from a perfect circle can result in a leakage gap. Furthermore, if the valve is closed and the pressure inside the chamber increases, this chamber pressure would act on the entire valve plate, pressing it down onto the bottom surface of the valve chamber. As will be analyzed in more detail later in this chapter, on the size scales estimated for this valve, the electrostatic forces are 100 times smaller than fluidic forces, so an electrostatic actuator will have a difficult time pulling the center membrane back up off the bottom cavity surface if the valve is to be opened.

In the previously described valve design, much of the actuation energy is used to deform the central plate to get the valve to switch. Another version of the valve would be a free floating plate inside a valve chamber, which is pulled up and down to block the ports, but without the need to deform the valve plate. Figure 3.12 shows this design. The fluidic forces can be controlled by the size of the valve seat. The free floating valve plate could

Figure 3.12 Free floating valve plate valve
cause fabrication problems in terms of getting lost during wafer handling, or it could defeat the benefit of batch fabrication if each valve plate had to be assembled manually.

These considerations lead to another version of the clamped membrane design where the valve plate is held by small tethers as shown in Figure 3.13. These tethers can be designed to provide enough force to pull the membrane off the valve seat, and an electrostatic actuator can be integrated by using the tethers as electric leads. Furthermore, the tethers prevent the valve plate from getting lost during manufacturing, considering, for example, wet processes for cleaning, etching etc.

3.6 Bench Level Experiments / Prototypes

In order to get a feel for the functionality of the different valve designs, scaled up prototypes were built and tested in terms of sealing function and relative actuation effort. Figure 3.14 and Figure 3.15 show the bench level prototype of a valve with a centrally clamped valve plate. In the actual microvalve design, this valve is supposed to be double sided with a lateral inlet and outlets on both sides of the valve cavity. A one-sided
Figure 3.14  centrally clamped membrane valve

Figure 3.15  Plexiglas prototype of the centrally clamped membrane valve
prototype, however, has enough detail to show the basic functionality. The material used for the prototype body was Plexiglas. The central plate was made of a thin polycarbonate sheet and a piston inserted from the top simulates an actuator bending the valve plate to one side to either open or close the valve for the gas flow. Pressurized shop air was used as a fluid with this prototype valve. In order to visualize the open flow rate and the leak rate, the valve prototype was submerged in a tank of water and then manually actuated by pushing the piston. Figure 3.16 shows a photograph of the submerged valve. A bubble test

![Figure 3.16 Bubble test of bench level prototype, open (left) and closed (right)](image)

is a very good method of visualizing even small leaks. In the open position, very large bubbles came out of the valve outlet. Then, the piston was pushed down manually until the perimeter of the valve plate touched the bottom of the valve cavity to simulate the closed condition of the valve (circular line contact). A fairly large leak rate was observed, which
decreased only when the piston was pushed very hard, which made an entire circumferential region of the valve plate touch down (circular area contact). The leak rate could be reduced further after applying a self adhesive compliant material around the perimeter of the valve plate.

The prototype showed that in order to form a good seal in line contact, the perimeter of the valve plate would have to be extremely accurate in terms of circularity if only the perimeter of the valve plate is in contact. Else, if area contact has to be achieved, large force requirements from the valve actuator have to be expected. These sealing problems and the anticipated difficulties of fabricating the centrally clamped membrane prevented this concept from being pursued any further. It was determined that a different design had to be found with a more compliant valve plate structure.

3.7 Final Valve Design

The most promising valve design was determined to be the tether suspended valve. A solid model of the design can be seen in Figure 3.17, which shows a cross section of a 3-way valve having one lateral inlet and two outlets. For further clarity, Figure 3.18 shows an isometric view with flow schematic. The microvalve consists of a silicon substrate made from an SOI wafer. The device layer of this SOI wafer is etched to form the circular valve cavity, and the buried oxide layer is patterned to form the valve seat, the valve plate landing pads, and the tether support pads. A through hole etched in the SOI handle wafer forms the valve outlet. The valve plate layer is bonded on top of the SOI wafer and it is etched to create the inlet channel and the circular valve plate with the suspending tethers.

For prototyping, we chose to build only a one sided valve instead of a two sided switch valve. Therefore, a pyrex wafer with a relief recess is bonded on top of the structure to form a cover that seals the valve against the inlet pressure. If a pressure is applied to the inlet channel, the gas enters the valve cavity through the inlet. A rigid central valve plate can be used and the stiffness of the tether suspension can be finely tuned by modifying the tether geometry on the etch mask in order to adjust the valve to different pressure and flow
rate levels. The valve plate remains rigid and undeformed and will engage with a flat valve seat. If the plate and the seat have polished surface quality, good sealing can be expected. The valve plate layer can be electrically insulated from the bottom and top layers to form an electrostatic actuator by using intermediate layers of silicon dioxide. For the double sided switch valve version, identical parts can be used for the top and the bottom half to simplify fabrication. If several valves are to be used in an array, the valve plate layer can be used for routing the fluid as well as the electrical connections between individual valves. Figure 3.19 shows a cross section of the bottom half of the valve with approximate dimensions to give an idea about the relative size of different features. On the wafer mask, some dimensions are slightly changed in order to examine the influence of certain geometrical parameters, for example valve plate size, valve seat diameter etc.
Figure 3.20 shows the electrical diagram of the actuator circuit. The handle wafer of the SOI wafer is used as the ground pole, the valve plate layer is energized with an electrical charge which flows to the valve plate through the tethers. This causes an attractive force pulling in the valve plate towards the valve seat and thereby closing the valve. If the voltage is turned off, the elastic energy stored in the tethers will lift the valve plate off the valve seat back into its initial position. The SOI buried oxide layer can be used to separate the charges and to prevent an electrical short. It is described in Chapter 4 of this thesis how an additional oxide layer "coating" of the entire valve structure at the end of the fabrication sequence is needed to prevent sudden spark discharge between the plates of the electrostatic actuator. A properly insulated electrostatic microactuator will consume very little power since there is only leakage current flowing between the poles.
Figure 3.19 Typical valve dimensions

Figure 3.21 shows a schematic of the functionality of a double-sided 3-way switch valve. Fluid enters the valve cavity sideways through a channel etched into the valve plate layer. Depending on the electrical conditions between the layers, the valve plate can either be pulled up or down and the fluid will exit either through port A or B.

3.8 Mathematical Model

3.8.1 Overview

The mathematical model of the switch valve has many design parameters. Figure 3.22 shows in a flow chart the different stages of how the valves performance is modeled. It is initially designed for an existing mini gas chromatograph prototype set up by the sponsor company ABB. An existing separation column, microfabricated in silicon technology, was used to constrain the size of the flow area when the valve is open. The flow channel in the column has a cross sectional area of 100 microns x 30 microns. In order to prevent the valves from becoming restrictors for the flow within the mini GC system, the valve
Figure 3.20 Electrical diagram of the actuator circuit

geometry was chosen such that in the open state, the cross sectional area for the flow in all critical places was at least the same as in the existing mini GC column of 3000 μm². This determines, for example, that outlet port holes should have a diameter of at least

\[
d_{\text{hole}} = \sqrt{\frac{4 \cdot A_{\text{channel}}}{\pi}} = \sqrt{\frac{4 \cdot 3000}{\pi}} \mu m = 62 \mu m ,
\]

where \(d_{\text{hole}}\) is the outlet hole diameter, and \(A_{\text{channel}}\) is the cross sectional area of the flow channel.

With this hole diameter and the required flow area, the minimum stroke of the valve plate can be computed. One can imagine that in the open state, the fluid has to flow radially inwards to the port hole. The smallest area that the fluid has to flow through is a
Mathematical Model

$cylinderical\ area\ with\ the\ diameter\ of\ the\ port\ hole\ and\ a\ height\ determined\ by\ the$

maximum\ valve\ plate\ stroke.\ The\ maximum\ stroke\ $s_{\text{max}}$\ can\ then\ be\ found\ with

$$s_{\text{max}} = \frac{A_{\text{channel}}}{\pi \cdot d_{\text{hole}}} = \frac{3000}{\pi \cdot 62} \mu m = 15 \mu m \quad (3.2)$$

The\ maximum\ stroke\ $s_{\text{max}}$\ is\ then\ equal\ to\ twice\ the\ travel\ of\ the\ valve\ plate.\ The\ actual

stroke\ was\ conservatively\ chosen\ to\ be\ 20\ microns\ with\ a\ valve\ plate\ travel\ of\ 10\ microns\ to\ either\ side.\ This\ will\ later\ be\ the\ thickness\ of\ the\ device\ layer\ of\ the\ valve\ substrate\ SOI\ wafer.
The next given valve parameter is the maximum leak rate. The sponsor company has determined this valve to be on the order of $10^{-8}$ atm-cc/sec. Given the maximum valve pressure of about 2 bars, the minimum outer diameter of the valve seat can be determined using equations that will be described in Section 3.8.4. From this seat outer diameter and the seat inner diameter (which is the same as the fluid port hole diameter $d_{\text{hole}}$), the pressure force of the valve plate when the valve is closed can be estimated. From this pressure force, the necessary stiffness of the tethers can be computed so that valve can...
open against the pressure force. The tethers are the main driving force for pulling the valve plate off the valve seat when it is pressurized. The tether stiffness together with the maximum allowable operation voltage determines the minimum electrode area that is needed to operate the spring tethers.

The individual components of this mathematical model and their physics will be discussed in the following sections.

3.8.2 Electrostatic Actuator

Electrostatic actuators are very attractive for microsystem designers due to their low power consumption (ideally zero with no leakage current) and relative design simplicity. However, disadvantages such as low force and stroke, sensitivity to contamination, charge accumulation at dielectric interfaces which can lead to stiction, and the danger of fatal spark discharge at high voltages and small gaps also have to be considered. Electrostatic actuation for this valve has been chosen because of the aforementioned advantages, and how the detailed design avoids the potential problems shall be discussed along with a few fundamental insights.

Electrostatic actuator with an electrode suspended by a linear spring

The fundamental principle of an electrostatic actuator is shown in Figure 3.23. Two electrostatically charged conductive plates exert an attractive force towards each other. One of the plates is attached to mechanical ground, whereas the other one is suspended by a spring with spring constant $k_{spring}$. The mechanical force follows a linear relationship with respect to the displacement $x$ from the initial position,

$$ F_{mech} = k_{spring} \cdot x, \quad (3.3) $$

where $F_{mech}$ is the mechanical spring force, $k_{spring}$ is the spring constant, and $x$ is the displacement of the spring. The electrostatic attraction follows the well known Coulomb formula
Figure 3.23  Fundamental principle of a parallel plate electrostatic MEMS actuator

$$F_{el} = \frac{\varepsilon_1 \cdot A_{electrode} \cdot V^2}{2 \cdot (x_0 - x)^2},$$  \hspace{1cm} (3.4)

where $F_{el}$ is the electrostatic attraction, $\varepsilon_1$ is the absolute dielectric constant of the medium between the electrodes, $A_{electrode}$ is the electrode surface area, $V$ is the applied voltage, and $x_0$ is the initial gap between the electrodes. Figure 3.24 shows a typical plot of these force functions. The location of the electrostatic curve is dependent on the applied voltage. For voltages where the electrostatic curve has a segment that lies below the mechanical curve, there are 2 points where the curves intersect, i.e. where the forces balance. At each point, the actuator has a different behavior [109]. The lower point is a stable equilibrium, the upper point is unstable. At the stable point, the moveable capacitor plate will stably hover above the grounded plate. A way to explain it is to imagine a small perturbation from this position: if the plate is displaced further, the mechanical force will be larger than the electrostatic force, pushing the plate back to the stable point. A perturbation in the other direction will cause the electrostatic force to be larger than the mechanical force and, again, the plate will be pushed back to the stable point. The other intersection point, however, is an unstable equilibrium. A small perturbation will cause the
Figure 3.24 Behavior of the electrostatic and the mechanical force in an electrostatic actuator

electrostatic actuator to collapse since the electrostatic force increases at a faster rate than the mechanical force.

If the voltage on an electrostatic actuator is steadily increased, the curve for the electrostatic force as shown in Figure 3.24 will move upwards in the chart. Initially, the lower stable equilibrium point will be to the left of the pull-in point. Increasing the voltage will cause this intersection point to move upwards along the mechanical force line. At the same time, the unstable equilibrium point will move downwards toward the pull-in point. When the voltage reaches the pull-in voltage, the stable and the unstable equilibrium points come together. At this point, the actuator will collapse and the two capacitor plates will pull into contact.
In order to compute the displacement at pull-in, two conditions must be met. First, the slope of the electrostatic curve must be equal to the slope of the mechanical spring curve, which can be found by simply equating the derivatives of both curves with respect to the displacement. Secondly, the mechanical and the electrical force must be equal. Using Equations 3.3 and 3.4 and their derivatives, the pull-in displacement can be calculated to be [109]

\[ x_{pull-in} = \frac{x_0}{3}, \]  

(3.5)

which is interestingly enough always one third of the initial electrostatic gap, independent of any of the other actuator design parameters. The pull-in voltage can be found by substituting the pull-in displacement into the electrostatic actuator equation and solving for the voltage [109]

\[ V_{pull-in} = \frac{8 \cdot k_{spring} \cdot x_0^3}{27 \cdot \varepsilon_0 \cdot A_{electrode}} \]  

(3.6)

This is the nature of an electrostatic actuator and the reason why the electrostatic plate can not be gradually pulled towards the ground plate for more than one third of the total travel distance. This could be changed by having a non-linear spring, which typically require a much more complex mechanical design.

**Influence of Dielectric Materials**

If a slab of dielectric material is inserted into an electric field, its molecules will polarize, i.e. positive and negative charges will be pulled away from each other inside the dielectric. This charge separation creates an inherent electric field inside the dielectric material which counteracts the externally applied electric field, reducing its strength within the dielectric material.

If a dielectric layer is introduced into the air gap of an electrostatic actuator as shown in Figure 3.25, the force acting on the electrodes will increase, but on the downside, the total
actuator travel $x_1$ will decrease if the total distance between electrodes $x_0$ is kept constant. Therefore, actuators with the same amount of travel are compared here, i.e. where the initial air gap $x_1$ will remain constant, and $x_0$ will increase by the thickness of the dielectric layer $x_2$ according to $x_0 = x_1 + x_2$.

In order to compute the force, the capacitor can be modeled as two capacitors in series, which yields a total system capacitance of

$$C_{total} = \frac{C_1 \cdot C_2}{C_1 + C_2} = \frac{\varepsilon_1 \varepsilon_2 A_{electrode}}{\varepsilon_1 x_2 + \varepsilon_2 x_1}. \quad (3.7)$$

It is assumed here that when the actuator moves, only the dimension $x_1$ changes and the thickness $x_2$ of the dielectric material remains constant. Therefore, by using the quantity $x_1$ as the derivation variable, the force of the actuator can be computed to

$$|F_{el}(x_1)| = \frac{1}{2} \cdot V^2 \cdot \frac{dC_{total}}{dx_1} = \frac{\varepsilon_1 \varepsilon_2^2 A_{electrode} V^2}{2 \cdot (\varepsilon_1 x_2 + \varepsilon_2 x_1)^2}. \quad (3.8)$$

Note that a directional minus sign has been omitted from Equation 3.8 since only the magnitude of the force is of importance here.
In order to evaluate how a dielectric layer changes the force characteristics, we use the force equation for a capacitor where there is only air in the gap \( x_2 = 0 \). This force is

\[
F_{el,0}(x_1) = \frac{\varepsilon_1 \cdot A_{electrode} \cdot V^2}{2 \cdot x_1^2} \quad (3.9)
\]

Dividing Equation 3.8 by Equation 3.9 yields

\[
\left| \frac{F_{el}}{F_{el,0}} \right| = \left( \frac{\varepsilon_1}{\varepsilon_2} \cdot \frac{x_2}{x_1} + 1 \right). \quad (3.10)
\]

This is the factor by which the force of an electrostatic actuator is reduced when a dielectric material is present for the same displacement. Figure 3.26 shows a plot of this factor as a function of the thickness ratio \( x_2/x_1 \) for different dielectric ratios \( \varepsilon_1/\varepsilon_2 \). It can

![Figure 3.26](image)

**Figure 3.26**  Force reduction factor as a result of the presence of a dielectric layer
be readily seen that the presence of a dielectric material always reduces the actuator force, given that the actuator travel $x_1$ is kept constant. This can be further clarified by the following explanation: if a dielectric material is put between the gap, then the capacitor plates have to be moved apart from each other by the thickness of the dielectric layer in order to keep the actuator travel $x_1$ constant. This increase in total gap $x_0$ naturally decreases the actuator force.

The microvalve design described here uses a thin layer of silicon dioxide in order to electrically insulate the poles of the electrostatic actuator from another, especially after the plate has pulled in and would otherwise be touching. As described earlier, the pull-in happens after 1/3 of the initial gap has been traveled. With an initial gap of 10 microns, this will leave a gap of about 6.7 microns at pull-in. If a silicon dioxide layer of 0.1um is used, and the relative dielectric constant of silicon dioxide is about 3.9, we find by using Equation 3.10

$$\frac{F_{el}}{F_{el,0}} = \frac{1}{\left(\frac{1}{3.9} \cdot \frac{0.1}{6.7} + 1\right)} = 0.996. \tag{3.11}$$

This means that the force in the electrostatic valve actuator at pull-in is reduced by the negligible amount of only about 0.38%, which does not change the pull-in voltage significantly.

**Electrical Breakdown**

If two electrostatically charged electrodes are brought in close proximity, there is a maximum voltage that can be sustained before the medium between them, usually air, breaks down. In this case, an ionized electrical conduit is formed that supports a current. In a MEMS device, this current can cause local heating to the melting point of the electrode material and therefore can cause great damage by fusing parts of the actuator together or by vaporizing them.
The literature [110], [111] reports on different mechanisms and theories that govern the onset of sudden spark discharge. The *Townsend avalanche criterion* is often used to explain the phenomena of electrical gas discharge. A number of initially free electrons in the gap between the electrodes are being accelerated by the electric field. After reaching a critical velocity, collision events with gas molecules create positive ions and more free electrons, which are accelerated again to create more ions. This exponentially increasing electron avalanche leads to a spark discharge and is characterized by the well-known *Paschen curve*. In Paschen curves, the breakdown voltage is plotted versus the pressure-gap product $p \cdot d$. Figure 3.27 shows a Paschen curve for air at atmospheric pressure where

![Paschen curve](image)

**Figure 3.27** Paschen curve for air at atmospheric pressure [111]

the breakdown voltage is plotted as a function of the gap spacing. The Paschen curve suggests that there is a minimum breakdown voltage of about 360 V for gaps of about 5 microns. At gaps of less than 5 microns, the breakdown voltage increases dramatically, which is consistent with gas ionization theorems, because at smaller gaps, the mean free
path becomes too short for free electrons to gain enough energy to initiate the ionization process. However, in practical MEMS devices where gaps are in fact on the order of a few microns, this phenomenon is not confirmed, as the practical breakdown voltages can be significantly smaller than what the Pashen curve suggests [112].

In order to understand this discrepancy, one has to realize that the Paschen curve data was taken for the gap between two large spheres. In most MEMS devices, however, the electrode geometry is quite different. Especially sharp corners can lead to locally higher field line concentrations with much higher field strength. These spikes in field intensity decrease the necessary acceleration distance for electrons to initiate the avalanche ionization.

Furthermore, at small gaps and large field strengths, electrons can get pulled off the surface of the electrode and create a current path. This is the process used, for example, in vacuum transistor tubes and is called field emission. These effects are captured in the modified Paschen curve [113]. Figure 3.28 shows a modified Paschen curve for air at atmospheric pressure. This curve is much less optimistic about the electrical breakdown voltage. After a small plateau at about 5 microns, the breakdown voltage drops dramatically to zero as the gap size approaches zero.

This deviation of the breakdown behavior from the Paschen curve has been observed with microvalve prototypes as well. The geometry of the valves dictated that there would be a separation gap between the electrodes of 0.5 microns after the valve plate has pulled in. The original Paschen curve would indicate that for a gap of this size, the breakdown voltage would be hundreds of volts. However, when the electrostatically actuated valve plate pulled in, which occurred at around 60 Volts, a fatal spark discharge would occur every time. Only after a final oxidation step had been added to the process, could sparks be prevented.
3.8.3 Tether design

The tethers that suspend the valve plate are located around the valve plate perimeter. For the design and optimization of the electrostatic actuator it is important to know the spring constant, because it directly influences the pull-in voltage that is needed. In general, due to the weak force of the electrostatic actuator, the difficulty has always been to design tethers that are soft enough so that the pull-in voltage would stay below 100 V.

The shape of the tethers in the wafer plane is dependent on the thickness of the wafer that is used to form the valve plate layer. Due to the evolution of the fabrication process, the thickness of the valve plate layer has increased from initial valve designs to the final versions (see Chapter 5). This increase in thickness has brought about a need to decrease the tether stiffness by reducing their width and increasing their effective length, from the point of view of the mask layout.

Figure 3.28  modified Paschen curve for air at atmospheric pressure [111]
Due to the need for complete mechanical constraint of the valve plate for robustness in manufacturing as well as the need to keep the stiffness low, the minimum and maximum number of tethers that should be used is three. If the tethers are designed circularly around the perimeter of the valve plate, as shown in Figure 3.29a, the maximum wrapping angle is 120 degrees, less some room for anchoring. For tethers where the necessary length exceeds 120 degrees, a spiral design was chosen as shown in Figure 3.29b. This design allows for very compact and compliant tethers. Also, spirally shaped tethers have a very smooth line and therefore avoid sudden changes in cross section from which fatigue cracks can initiate.

**Circular tether design**

Figure 3.30 shows a cantilevered beam which is curved from the top view with a constant radius $R$. One end is rigidly clamped, the other end is free to move up and down with the condition that the bending slope and the twisting angle must be zero. This is the clamping
situation for each of the three tethers if used, for example, as shown in Figure 3.29a. The zero-slope-zero-angle condition implies that there be a bending moment $M_B^0$ and a twisting moment $M_T^0$ applied to the end of the beam which keeps both, the bending slope as well as the twisting angle at zero as the beam is deformed.

In order to find the displacement of the beam, one must first find a relationship for the bending and the twisting moment inside the beam as a function of a length coordinate. The total moment inside the beam at an arbitrary angular position alpha is comprised of the vectorial components of the end-moments $M_B^0$ and $M_T^0$, as well as the moment exerted by the force $F$ applied at the moveable end of the beam. Due to the beam’s curvature, the force $F$ contributes to the bending moment as well as the twisting moment through the bending leverage $r_b$ and the twisting leverage $r_t$, respectively. Geometrical considerations yield the equations for the bending and twisting lever arms as

$$r_b = R \cdot \sin(\alpha_0 - \alpha),$$

(3.12)
\[ r_t = R \cdot [1 - \cos(\alpha_0 - \alpha)] \quad (3.13) \]

The bending moment and the twisting moment inside the beam can be written by evaluating the moment balance for the beam cross section at an arbitrary angle alpha in bending and twist direction as

\[ M_B(\alpha) = -F \cdot r_b + M_B^0 \cdot \cos(\alpha_0 - \alpha) + M_T^0 \cdot \sin(\alpha_0 - \alpha) \quad (3.14) \]

\[ M_T(\alpha) = F \cdot r_t - M_B^0 \cdot \sin(\alpha_0 - \alpha) + M_T^0 \cdot \cos(\alpha_0 - \alpha) \quad (3.15) \]

Assuming a slender beam where the length is much greater than the thickness, the Newton-Euler equation can be used to predict the bending part of the beam deformation \( y \). The variable \( x \) is used as a coordinate running along the length of the beam. This is expressed as

\[ \frac{d^2 y}{dx^2} = \frac{1}{E \cdot I_b} \cdot M_B(x) \quad (3.16) \]

with the following boundary conditions:

\[ y'(x= 0) = 0 \] (slope is zero at the clamped end), \quad (3.17)

\[ y'(x= R \cdot \alpha_0) = 0 \] (slope is zero at the moveable end), \quad (3.18)

\[ y(x= 0) = 0 \] (displacement is zero at the clamped end) \quad (3.19)

The twisting component of the beam deformation can be described using the formula

\[ \frac{d\phi}{dx} = \frac{1}{G \cdot I_p} \cdot M_T(x), \quad (3.20) \]

where \( \phi \) is the twisting angle in the beam at each position along the length. The total deflection at the tip due to the twisting effect is then the sum (or the integral, actually) of the twisting angle in each small element \( dx \) together with the leverage \( r_t \), computed by
\[ du = r_t(x) \cdot d\phi \]  

(3.21)

Using Equation 3.21 in Equation 3.20 yields

\[ d\phi = \frac{1}{G \cdot I_p} \cdot M_T(x) \cdot dx , \] 

(3.22)

with the boundary conditions

\[ \phi(x = 0) = 0 \text{ (no twist at the root of the cantilever)} \] 

(3.23)

\[ \phi(x = R_\alpha_0) = 0 \text{ (no twist at the tip of the cantilever)} \] 

(3.24)

which can be used to integrate along the length of the beam to find the displacement component \( y_b \) at the tip due to twisting load.

Equations 3.12 to 3.15 are given as a function of the angle \( \alpha \), but in order to carry out the integration with respect to \( x \), one must first convert the angle \( \alpha \) to the variable \( x \) (which is the arc length variable along the beam). For a curved beam where the radius \( R \) is constant, this can easily be done by replacing \( \alpha \) with

\[ \alpha = \frac{x}{R} \] 

(3.25)

Substituting Equation 3.12 into Equation 3.14 as well as Equation 3.13 into Equation 3.15 and using Equation 3.25 to convert the integration variable from \( \alpha \) to \( x \), one obtains

\[ M_B(x) = (M_T^0 - FR) \cdot \sin\left(\alpha_0 - \frac{x}{R}\right) + M_B^0 \cdot \cos\left(\alpha_0 - \frac{x}{R}\right) \] 

(3.26)

\[ M_T(x) = (M_T^0 - FR) \cdot \cos\left(\alpha_0 - \frac{x}{R}\right) - M_B^0 \cdot \sin\left(\alpha_0 - \frac{x}{R}\right) + FR . \] 

(3.27)

Now we first solve the bending part by using Equation 3.26 together with Equation 3.16. After integrating twice and using the boundary conditions 3.17 through 3.19 we are left with two equations, namely the bending line equation...
Mathematical Model

\[ y(x) = \frac{R^2}{E \cdot I_b} \left[ (FR - M_T^0) \cdot \left( \sin \left( \alpha_0 - \frac{x}{R} \right) - \sin \alpha_0 + \frac{x}{R} \right) \right. \]
\[ + M_B^0 \cdot \left( \cos \alpha_0 - \cos \left( \alpha_0 - \frac{x}{R} \right) \right) \]

(3.28)

and a first relationship between \( M_B^0 \) and \( M_T^0 \)

\[(M_T^0 - FR) \cdot (\cos \alpha_0 - 1) - M_B^0 \cdot \sin \alpha_0 = 0 \] (3.29)

which we will use a little later. What we are interested in is the deflection at the tip of the beam, which is from the bending Equation 3.28 evaluated at \( x = R \cdot a_0 \), Thus

\[ y_b(x = R \alpha_0) = \frac{R^2}{E I_b} \cdot [M_T^0 \cdot (\sin \alpha_0 - \alpha_0) + M_B^0 \cdot (\cos \alpha_0 - 1) + FR \cdot (\alpha_0 - \sin \alpha_0)] \] (3.30)

Turning now back to the twisting part of the deformation, we integrate Equation 3.20 and use the boundary conditions 3.23 and 3.24. This yields the twisting angle as a function of the length

\[ \phi(x) = \frac{R}{G I_p} \cdot \left[ FR \cdot \left( \sin \left( \alpha_0 - \frac{x}{R} \right) - \alpha_0 \right) + M_B^0 \cdot \left( 1 - \cos \left( \alpha_0 - \frac{x}{R} \right) \right) \right. \]
\[ - M_T^0 \cdot \sin \left( \alpha_0 - \frac{x}{R} \right) + Fx \] (3.31)

as well as a second relationship between \( M_B^0 \) and \( M_T^0 \)

\[(FR - M_T^0) \cdot \sin \alpha_0 + M_B^0 \cdot (1 - \cos \alpha_0) - FR \alpha_0 = 0 . \] (3.32)

Using Equation 3.22 and 3.27 we can find the displacement component at the tip due to twisting deformation by integrating from \( x = 0 \) to \( x = R \cdot a_0 \) and find
\[ y_{t,\text{tip}} = \frac{R^2}{GI_p} \left[ FR \left( \frac{3}{2} \alpha_0 - 2 \sin \alpha_0 + \frac{1}{4} \sin(2\alpha_0) \right) + \frac{M_B^0}{4} \left( \cos \alpha_0 - \cos \alpha_0 - \frac{3}{4} \right) \right. \]
\[ \left. + \frac{M_T^0}{4} \left( \sin \alpha_0 - \frac{1}{4} \sin(2\alpha_0) - \frac{\alpha_0}{2} \right) \right] \quad (3.33) \]

Equations 3.29 and 3.32 can now be used to find the constants \( M_B^0 \) and \( M_T^0 \), which are
\[ M_B^0 = FR \cdot \frac{\alpha_0}{2} \quad (3.34) \]
\[ M_T^0 = FR \left[ 1 - \frac{\alpha_0 \cdot \sin \alpha_0}{2 - 2 \sin \alpha_0} \right] \quad (3.35) \]

Finally, by applying the principle of linear superposition, the total displacement of the tip of the beam can be found by adding the bending component (Equation 3.30) and the twisting component (Equation 3.33) to find the final solution
\[ y_{\text{tip, total}} = FR^3 \cdot \frac{\alpha_0}{2} \left( \frac{\sin \alpha_0}{1 - \cos \alpha_0} \right) \left( \alpha_0 - \sin \alpha_0 \right) + \cos \alpha_0 - 1 \]
\[ EI_b \]
\[ \left[ 1 - \frac{\alpha_0 \cdot \sin \alpha_0}{2 - 2 \sin \alpha_0} \right] \]
\[ + \frac{4 \cdot \sin \alpha_0 \cdot \cos \alpha_0 + (\alpha_0^2 - 4) \cdot \sin \alpha_0 + \alpha_0 \cdot (\sin \alpha_0)^2}{4(1 - \cos \alpha_0)} \]
\[ \frac{G \cdot I_p}{4} \]
\[ (3.36) \]

**Straight tethers approximation**

If the tethers are short and the diameter is large, the tethers can be approximated by straight beams with one clamped and one rotationally constrained end as shown in Figure 3.31. The spring constant of such a flexure can be simply calculated by realizing that it merely consists of two clamped cantilevers joined at the free end. The stiffness can then be computed with
where $E$ is the materials Young’s Modulus, $I$ is the bending moment of inertia, $n$ is the number of tethers used, and $L$ is the total length of the tether.

In order to understand some of the tether design problems, some words about the manufacturing process must be said here. Initially, the valve plate was designed to be 20 microns thick. This design bonded an SOI wafer with a 20 micron thick device layer on top of a substrate wafer with valve seats and then removed the handle wafer from the SOI wafer back side, leaving the 20 micron thick device layer behind. This layer was supposed to be etched in order to form the tethers and the valve plate. However, a reliable method of removing the sacrificial handle wafer from the SOI wafer could not be found, so that alternatively, a single thinned wafer of 100 micron thickness was used instead. Due to availability issues from the vendors, 110 micron thick wafers were used for the last prototype wafers that were fabricated. This method was very advantageous because a number of fabrication steps were eliminated, however, the minimum thickness of the valve plate wafer that can be used is limited due to handling issues. A wafer of less than
100 microns in thickness can break very easily during, for example, wet cleaning steps that are necessary for bonding preparation.

However, in terms of the tether design, using a valve plate layer with a multiple of the original thickness also means that the tethers have to be a multiple in length, or wrapping angle, respectively. If the necessary wrapping angle is less than 120 degrees, circular tethers can be used. For wrapping angles greater than 120 degrees, spirally shaped tethers must be used since a spiral shape allows for having a very space efficient nested design. The latter is necessary here.

**Spiral tether design**

A closed form solution to the spiral tether design equations is no longer possible, because the radius is now a function of the sweeping angle. The integration of the equations can only be done numerically. Therefore, Finite Element Analysis was chosen to determine the geometrical parameters of the spiral shaped tethers. In an FEA design study, the length of the spirally shaped tethers was varied in increments, and from the numerical results the appropriate tether dimension was picked. Figure 3.32 shows a typical FEA result.

### 3.8.4 Fluidmechanical Considerations

The fluid-mechanical considerations include two aspects of the valve design, the sealing capability of the valve seat and valve plate when the valve is closed, and the equilibrium of the valve plate when the valve is open.

**Sealing Capability**

One of the main features of the microvalve is the low leakage rate when the valve is closed. This is achieved by utilizing polished wafer surfaces. Standard wafers can be bought relatively cheaply with a surfaces roughness on the order of 5 nm. The wafers are polished so they can be bonded to one another by silicon direct bonding or fusion bonding. The microvalve design takes advantage of the flatness by utilizing these surfaces for fabricating the valve seat and the closing member. If the valve seat and the closing
The valve seat is made of silicon dioxide, about 0.5 micron thick, which is thermally grown on a polished wafer surface. The closing member is made of a thinned, double side polished wafer, bonded onto the valve seat substrate wafer.

The different flow regimes of gas dynamics

In order to estimate the leak rate, one has to realize that the gap between the valve seat and the valve plate in the closed condition is extremely small, on the order of a only few nm. Standard fluid mechanical equations do not apply when the size of the gap becomes
comparable to the mean free path of the gas. The different regimes of gas flow dynamics are characterized by the Knudsen number, which is defined as [114]

\[ Kn = \frac{\lambda}{L}, \]  

(3.38)

where \( \lambda \) is the mean free path of the gas, and \( L \) is a characteristic length of the flow. For flow in small gaps, \( L \) would correspond to the gap width. If the Knudsen number is smaller than \( 10^{-2} \), the flow can be assumed to be a continuum and Navier Stokes equation can be used. For \( 10^{-2} < Kn < 10^{-1} \), the non-slip boundary conditions start to fail, and a finite fluid velocity at the walls has to be assumed to correct the continuum equation. This is called the slip-flow regime. If the Knudsen number is \( Kn > 10^{-1} \), the fluid can no longer be regarded as a continuum, and Navier-Stokes Equation can no longer be used. Instead, the Knudsen equation for rarefied gas dynamics has to be used.

For the valve described in this thesis, the gap between the two surfaces (valve seat and closing member) of the closed valve seat is smaller than the mean free path of the gas and thus, rarefied gas dynamics equations apply.

**Rarefied gas dynamics**

The mass flow rate in rarefied gas dynamics (i.e. where the mean free path of the gas molecules is comparable with the fluid channel cross sectional dimensions) is described by the following Knudsen equation [114]:

\[ \dot{m} = \frac{p_1 - p_2}{\sqrt{\rho_1} \cdot W}, \]  

(3.39)

where \( W \) is a form factor given by

\[ W = \frac{3}{8} \cdot \frac{\pi}{2} \cdot \int_0^L \frac{Q}{A^2} dl. \]  

(3.40)
The parameter $O$ is the wetted channel circumference, and $A$ is the cross sectional area. For a radial flow towards a central hole, the form factor $W$ is

$$W = \frac{3}{8} \sqrt{\frac{\pi}{2}} \cdot \frac{1}{\pi h^2} \cdot \ln \left( \frac{r_o}{r_i} \right). \tag{3.41}$$

The density $\sqrt{\rho_1}$ can be computed with the ideal gas law

$$\sqrt{\rho_1} = \sqrt{\frac{M}{R_0 \cdot T}}, \tag{3.42}$$

where $M$ is the molecular weight in mols, $R_0$ is the universal gas constant, and $T$ is the temperature in Kelvin.

In order to obtain the volumetric flow rate $Q$, the mass flow rate has to be converted using the ideal gas law according to

$$Q = \dot{m} \cdot \frac{R_0 \cdot T}{M \cdot p_{ref}}, \tag{3.43}$$

where $M$ is the molecular mass of the gas, $R_0$ is the universal gas constant, and $p_{ref}$ is the reference pressure at which the flow rate is to be computed.

Hence, the volumetric flow rate through the valve seat for molecular flow can be modeled as:

$$Q_{Knudsen} = \frac{\Delta p}{p_{ref}} \cdot \frac{8}{3} \cdot \frac{h^2}{\ln \left( \frac{r_o}{r_i} \right)} \cdot \sqrt{\frac{2 \cdot \pi \cdot R_0 \cdot T}{M}}, \tag{3.44}$$

where $\Delta p$ is the pressure drop across the closed valve seat, $p_{ref}$ is the reference pressure (to compute the volumetric flow rate), $h$ is the gap, $r_o$ and $r_i$ are the outer and the inner valve seat radius, $R_0$ is the universal gas constant, $T$ is the temperature in Kelvin, and $M$ is the molecular mass of the gas that is being used.
For the design of the valve prototypes, the Navier Stokes equation was used initially to determine the inner and outer radius of the valve seat. However, since the gap between the valve plate and the valve seat is only on the order of a few nanometers when the valve is closed, Navier Stokes equation becomes invalid for this domain. Therefore, Equation 3.44 was used to verify the experimental results.

### 3.8.5 Valve Plate Equilibrium for the Open Valve

Initially, the valves were designed statically, where in the closed state, the spring constant of the tethers was determined such that for a given inlet pressure the valve plate would lift off the pressurized valve seat after the electrostatic actuator was turned off. After obtaining the first experimental results, however, the more accurate model described in this section was derived in order to describe the observed behavior. When the valve is open, fluid enters laterally through inlet channel in the valve plate layer and flows towards the outlet hole in the center of the valve recess. In order to avoid a pressure gradient across the plate that could cause the valve to close like a check valve, the valve plate is perforated with small "vent holes" (Figure 3.17 and Figure 3.18). In the center region of the plate near the valve seat, however, where there are no vent holes, the fluid flow still causes a pressure gradient and a net force, pulling the plate down towards the valve seat. Figure 3.33 shows a schematic of the open flow model that was used to capture this behavior. The Navier-Stokes Equation according to [115] is used to calculate the flow rate, pressure gradient and the resulting forces. Reduced to the significant terms, Navier Stoke’s equation written in cylindrical coordinates is

\[
0 = -\frac{1}{\rho} \cdot \frac{dp}{dr} + \nu \cdot \frac{d^2 \nu_r}{dz^2},
\]

where \( z \) is the coordinate used for the flow equations, pointing into the same direction as the coordinate \( x \) in Figure 3.33. This equation is also known as the Poiseuille flow equation. The non-slip boundary conditions for integration are
Figure 3.33  Schematic of the valve without actuation voltage applied

\[ v_r(z = 0) = 0 \quad \text{and} \quad v_r(z = h) = 0 \]  

(3.46)

Solving for the velocity gradient and applying the boundary conditions upon double integration yields a relationship for the velocity profile

\[ v_r(z) = \frac{1}{2\mu} \cdot \frac{dp}{dr} \cdot z \cdot (z - h). \]  

(3.47)

Assuming radial flow to or from a central fluid port leads to the equation for the flow rate according to

\[ Q = \int_{0}^{h} 2\pi r \cdot v_r(z)dz \]
After integration, the flow rate can be found to be

\[ Q = \frac{\pi rh^3}{6\mu} \cdot \frac{dp}{dr} \]  

(3.48)

A further integration can be done by separating the variable \( r \) and \( p \) such as

\[ \int_{p_1}^{p_2} dp = \frac{6\mu Q}{\pi h^3} \cdot \int_{r_i}^{r_o} dr \]  

(3.49)

which yields

\[ Q = \frac{\pi h^3 \cdot (p_2 - p_1)}{6\mu \cdot \ln\left(\frac{r_o}{r_i}\right)} \]  

(3.50)

Using Equation 3.48 and integrating between the limits of \( r_i \) and an arbitrary radius \( r \) yields the pressure function across the plate

\[ p(r) = \frac{6 \cdot \mu \cdot Q}{\pi \cdot h^3} \cdot \ln\left(\frac{r}{r_i}\right) + p_1. \]  

(3.51)

Substituting Equation 3.50 for the flow rate \( Q \) yields

\[ p(r) = (p_2 - p_1) \cdot \frac{\ln\left(\frac{r}{r_i}\right)}{\ln\left(\frac{r_o}{r_i}\right)} + p_1. \]  

(3.52)

The force exerted on the valve plate by the fluid can be found by using

\[ F_{fluid} = p_2 \cdot \pi \cdot r_o^2 + \int_{r_o}^{r_i} p(r) dA - p_1 \cdot \pi \cdot r_i^2, \]  

(3.53)

which yields after integration
\[ F_{\text{fluid}} = \frac{\pi \cdot (r_o^2 - r_i^2)}{2 \cdot \ln \left( \frac{r_o}{r_i} \right)} \cdot (p_2 - p_1). \] (3.54)

Since the fluid force balances the spring force (assuming that the valve is not actuated, electrostatic force is zero), the equilibrium position of the valve plate can be found by using

\[ F_{\text{fluid}} = F_{\text{spring}} = k_{\text{spring}} \cdot x \] (3.55)

In order to simplify the following equation, three geometrical constants shall be introduced here, namely

\[ A = \frac{\pi \cdot (r_o^2 - r_i^2)}{2 \cdot \ln \left( \frac{r_o}{r_i} \right)}, \] (3.56)

\[ B = \frac{\pi}{6 \cdot \mu \cdot \ln \left( \frac{r_o}{r_i} \right)}, \] (3.57)

\[ R_h = \frac{8 \cdot \mu \cdot L}{\pi \cdot r_i^4}. \] (3.58)

Equations 3.50 and 3.54 can then be rewritten as

\[ F_{\text{fluid}} = A \cdot (p_2 - p_1) \] (3.59)

\[ Q = B \cdot (x_0 - x)^3 \cdot (p_2 - p_1). \] (3.60)

The pressure \( p_1 \) at the entrance of the port hole is higher than at the outlet because of the fluid resistance \( R_h \) of the port hole given by its geometry and can be found using

\[ p_1 = R_h \cdot Q \] (3.61)
Executing Equation 3.55 by using Equation 3.59 and solving for x, then substituting x into Equation 3.60, and at last substituting Equation 3.61 into Equation 3.60 yields an equation for the flow rate though the system as a function of pressure and the constants introduced above

$$Q = B \cdot \left( x_0 - \frac{A}{k_{spring}}(p_2 - R_h \cdot Q) \right) \cdot (p_2 - R_h \cdot Q)$$  \hspace{1cm} (3.62)

This polynomial was solved numerically using Matlab. The result is shown in Figure 3.34

Figure 3.34 Flow Rate through the unactuated valve as a function of pressure
together with measured data. The experimental setup used to obtain the data is described in Chapter 5. The parameters that were used to obtain the theoretical curve in Figure 3.34
were adjusted to reflect the manufacturing inaccuracies in the valve geometry. In order to measure all the necessary geometrical dimensions, it is necessary to perform destructive tests, i.e. section cuts with the die saw. Because of low yield on the prototype wafer, the device that produced useful experimental data was never measured this way in order to avoid losing them. Therefore, the geometrical adjustments were made by examining the surrounding valves that did not function properly.

The geometrical values used to obtain Figure 3.34 and Figure 3.36 were

Length of port hole (i.e. thickness of bottom SOI wafer): \( L = 525 \) microns

Inner radius of port hole: \( r_i = 17.7 \) microns

Radius of central area without vent holes: \( r_0 = 185 \) microns

Diameter of the electrode: \( d_{\text{electrode}} = 2.0 \) mm

Initial electrode distance: \( x_0 = 9.3 \) microns

Tethers stiffness: \( k_{\text{spring}} = 316.8 \) N/m

**Valve with the actuator turned on**

The fluid force exerted on the valve plate changes the pull-in voltage of the valve at different inlet pressures. The influence will be rather advantageous, however, since the fluid force will actually assist the electrostatic actuator to close the valve. Figure 3.35 shows a schematic of the valve when a voltage is applied on the actuator. It is the same situation as in the unactuated case, except that now an electrostatic force must be added to the model. Specifically, it has to be added to the force balance of Equation 3.55, which now becomes

\[
F_{\text{spring}} = F_{\text{fluid}} + F_{\text{electric}}
\]  

(3.63)

with

\[
F_{\text{electric}} = C \cdot \frac{V^2}{(x_0 - x)^2}
\]

(3.64)
Tethers modeled as a spring

Valve Plate

Vent holes

Figure 3.35  Schematic of the microvalve with electrostatic actuation

utilizing a constant $C$ with

$$ C = \frac{\varepsilon \cdot \pi \cdot d_{\text{electrode}}^2}{8} , $$

(3.65)

where $\varepsilon$ is the dielectric constant and $d_{\text{electrode}}$ is the diameter of the actuator electrode.

Substituting the formula for each of the forces in Equation 3.63 and eliminating the variable $x$ by using Equation 3.60 and 3.61 yields

$$ 0 = A(p_2 - R_h Q) + C \cdot y^2 \cdot \left( \frac{Q}{B(p_2 - R_h Q)} \right)^{2/3} - k_s \left[ x_0 - \left( \frac{Q}{B(p_2 - R_h Q)} \right)^{1/3} \right] $$

(3.66)
This polynomial can only be solved numerically and therefore, Matlab was used to obtain solutions. Figure 3.36 shows the numerical result compared to the measurements described in Chapter 5. The geometrical variables used for the theoretical curves were the same as the ones used for Figure 3.34. The pull-in voltage actually does decrease with increasing inlet pressure. This "pressure assisted pull-in effect" is due to the fact the pressure force adds to the electrostatic force and helps to pull-in the actuator plate. By increasing the stiffness of the tethers, the valve could be designed for much higher inlet pressures while keeping the pull-in voltage well within the limits of dielectric breakdown.

Figure 3.36 Flow rate vs. actuation voltage at different inlet pressure levels
Chapter 4

VALVE MICROFABRICATION

4.1 Introduction

The microfabrication of the valve was performed in the Microsystems Technology Lab (MTL) at MIT. The MTL provides microfabrication processing equipment located in three labs with different levels of cleanliness: class 10 (ICL), class 100 (TRL) and class 1000 (EML). The majority of the valve fabrication was done in the class 100 lab (TRL).

The fundamental fabrication challenge is the fact that two ultra smooth surfaces are needed in the valve structure, where one surface would be located at the bottom of a recess. Figure 4.1 illustrates this problem, showing half of a switch valve structure. One of

![Figure 4.1 Fundamental Problem](image-url)
the smooth surfaces is needed for silicon direct bonding so that the valve plate layer can be attached to the substrate layer. Silicon direct bonding is a process that is very unforgiving to surface defects or contamination and it requires surfaces smooth and flat to a few nanometer. The other surface with high quality requirements is located at the bottom of the valve recess, roughly 10 microns deeper than the surface used for silicon direct bonding. This surface is used to place the valve seats and it forms a seal by mating with the actuatable valve plate surface.

There are two ways of creating such a recess, either with additive or subtractive fabrication processes. An additive process would be used to build up the surface around the valve recess, which means that the top layer of the added material would be used for silicon direct bonding. However, the general literature indicates that the surface roughness of a 10 micron additive process is likely not sufficiently smooth for silicon direct bonding. Furthermore, a 10 micron deposited layer usually creates a significant amount of surface stress, which can lead to surface cracks and substrate deformations (bow).

A subtractive processes, such as dry or wet etching, would be used to etch the circular recess into the substrate. Given that this surface would then be used for making the valve seats, the question becomes how smooth of a surface this process leaves behind. Since the valve has very high requirements in terms of leakage when closed, the smoothness of these surfaces is critical for the valve performance.

In terms of fabrication cost, subtractive processes tend to be cheaper, especially the wet etching processes, such as KOH, TMAH, or isotropic silicon etch.

Initially, several different fabrication plans were conceived that could achieve the desired valve geometry. Before the actual fabrication was carried out, however, the most critical fabrication steps were identified and experiments were designed in order to quickly determine the critical issues and the probability of success for these process steps.
4.2 Fabrication of the valve recess

4.2.1 Introduction

The fundamental challenge of this fabrication step is to etch a shallow recess into a silicon substrate and maintaining the silicon direct bonding grade surface quality on the bottom of the recess. Wet etching techniques, such as KOH or TMAH were considered and tested, especially in light of the surface roughness that such a wet etch would leave behind. While wet etching techniques are more economical, a more expensive but also more reliable method was chosen for the prototype valves that uses a silicon on insulator (SOI) wafer, with custom manufactured device layer thickness.

4.2.2 Valve recess etched by KOH and TMAH

A study was carried out in order to quantify the surface roughness of wet etched surfaces. Figure 4.2 shows the simple fabrication plan used to etch the recesses that were then

![Figure 4.2 Wet etching surface roughness experiment](image-url)
characterized using an optical surface profilometer. The fabrication of the experiment starts with a plain silicon wafer with a <100> surface normal (1). Anisotriopic etchants will create a flat bottom surface with 54.7 degree angled sidewalls. Nitride of 2000A in thickness is grown in a tube furnace (2). The nitride is then structured by defining a photoresist mask (3, 4, 5) and then etching the nitride in a CF4 plasma etcher (6). The photoresist is then stripped in piranha acid (7) and then the wafer is etched in KOH or TMAH, respectively to create the recesses (8). Both, KOH and TMAH are used at a temperature of 85 degree C, where KOH etches at a rate of 1 micron/min and TMAH at approximately 0.5 microns/min. Figure 4.3 shows an image of the photomask as well as the etched wafer. After the recess is etched, the wafer is taken to an optical profilometer to measure the surface roughness in the bottom of the trench.

4.2.3 Wet etching results with KOH and TMAH

A typical image of the KOH etched surface topography can be seen in Figure 4.4. The most distinguishable phenomena observed here are small "pot holes" with various depths distributed all over the surface. These micro pits can be observed on every surface that has seen the KOH etching solution. The formation of these holes could have to do with

![Figure 4.3 The photomask (left) and the etched wafer (right)](image-url)
localized imperfections within the silicon crystal lattice where silicon atoms can be extracted by the etchant from the crystal lattice more easily than at other places.

Figure 4.5 shows a more close up view of two of these pit surface defects. As indicated in the 2D cross sectional plot, the pit defects are quite shallow, with 100 microns wide and only about 0.1 microns deep.

In terms of the usefulness of this etching method for the microvalve fabrication, it has to be realized that the depth of the recess relies on a timed etch, which is not the most accurate method of fabricating a desired depth. The problem to be mentioned here is that the design of the electrostatic actuator is dependent on an accurate depth of this recess, which defines the initial gap. The force of the actuator has a square dependency on the size of this gap, which makes the margin for error quite small. Furthermore, since valve seats are to be located on this recessed surface, the pit shaped defects can impair the surface shape and quality of a valve seat and thereby introduce leakage when the valve is closed.

The surface generated by a TMAH etch is better as the phenomenon of pot holes was not observed here. Figure 4.6 shows a surface plot of a TMAH etched surface. The data has
Figure 4.5 Close-up of a typical pit defect on a KOH etched surface

Figure 4.6 Roughness of a TMAH etched surface
been taken over the entire diameter of a TMAH etched valve recess. There are no surface
defects that can be observed with this etchant.

As with KOH, the depth of the recess etched with TMAH is also dependent on a timed etch, but the surface topology is definitely superior and TMAH is the etchant of choice if a smooth etch surface is to be generated.

As for the masking materials, both, silicon nitride and silicon oxide have been tried. However, it has been found that KOH will eventually eat through an oxide mask (on the order of 1 hour for 1 micron of oxide). Hence for KOH, a nitride mask is the preferred choice. While for TMAH both, nitride and oxide can be used, the selectivity to silicon oxide is much better so that in most cases, an oxide mask will suffice. The disadvantages of a nitride mask as opposed to an oxide mask is that the stripping process for a nitride mask is more difficult after the etch. For stripping nitride, hot phosphoric acid at 140 degree C must be used, whereas an oxide mask can be stripped in a simple BOE etch at room temperature at a rate of approximately 0.1 microns/min.

4.2.4 Valve recess created by using SOI wafer

The previously mentioned problems with wet etches, such as difficult depth control, inferior surface quality and often poor uniformity have lead to the use of SOI wafers for the valve prototypes since the device layer can be custom ordered to precise thickness specifications. Device layer thickness specifications of 10 microns +/- 1 micron are standard. Furthermore, a DRIE plasma etch can be used to create a recess into the device layer, which can then be stopped at the buried oxide layer (BOX). This creates valve recesses with precise depths and excellent uniformity across the wafer.

There is also the possibility that the buried oxide layer can be used for making the valve seats. The issue here is that the DRIE plasma might attack and roughen the oxide surface, which is not desired. The surface roughening can occur as a by-product of the non-
uniformity of the DRIE plasma etcher, i.e. some oxide is exposed already while other recesses are still etching.

**Oxide roughening by DRIE**

In order to quantify this surface roughening effect, an experiment was carried out where a layer of silicon oxide was exposed to the DRIE plasma for a varying amount of time. Figure 4.7 shows the process plan along with the mask layout. Silicon oxide of 5000A in

![Figure 4.7 DRIE attack process plan and layout](image-url)
thickness is grown on a plain silicon wafer (1, 2). Then, a photoresist mask is created with small openings (3) of about 5mm X 5mm in size. The openings are distributed in clusters of 9 each, one cluster located in the center of the wafer, and 4 clusters around the wafer perimeter, one at each quadrant. Then, the wafer is exposed to the DRIE plasma (4) for a certain amount of time (10 seconds). After 10 s, the DRIE etch is aborted, the wafer is removed from the plasma chamber, and all openings labeled with #1 are patched up using photoresist and a Q-tip and then baked for about 15min to cure the patch of resist (5). The wafer is then put back into the DRIE etcher and exposed for an additional amount of time (6). Steps (5) and (6) are repeated 9 times, until the last opening is patched. At the end, the photoresist mask is removed by cleaning the wafer in the asher (oxygen plasma clean).

This process creates distinguished areas of silicon oxide that are exposed for various time to the DRIE plasma, and surface roughness of the oxide as a function of exposure time and wafer location can be obtained. The table in Figure 4.7 lists the additional time for each consecutive etch, the total exposure time for each opening, and an equivalent etch depth, if pure silicon instead of silicon oxide was exposed to the etch. This number is relevant considering the non-uniformity of the DRIE etcher: when the SOI wafer is etched later to create the valve recesses and the buried oxide is used as an etch stop, at the position of the wafer area where the etch rate is the highest, the plasma will first reach the oxide. At the position where the etch rate is slowest, the most amount of silicon will be left to etch. This amount can be expressed in microns, and using the table in Figure 4.7, the maximum exposure time of the oxide surface at the position of the highest etch rate can be estimated (worst case). Together with the results from the oxide exposure experiment, the added surface roughness can be determined.

The results are shown in Figure 4.8. The plot shows the surface roughness of the oxide up to a maximum exposure time of 1800s, or 30min. The plot shows no considerable change in roughness, the roughness band stays between 2.5 and 4.5 nm. The fact that the overall path of the curves doesn't change (even though the exposure has been done over an
extensive period of time) indicates that the variation in roughness is mainly due to the measurement uncertainty rather than a real change in surface roughness.

This experiment shows that the buried oxide can be used as an etch stop first, and then for valve seats later, because the oxide surface quality should not suffer from DRIE plasma exposure.

4.3 Fabrication of the valve seats

4.3.1 Valve seats made with metal deposition and liftoff

After the recess is fabricated, the next challenge is to fabricated valve seats with a surface quality of a few nanometers. In a first approach, a metal liftoff deposition process has been tried. Figure 4.9 shows the process plan. A layer of silicon nitride (2) is deposited on a plain wafer (1). An isotropic etch (3) produces "dummy" valve recesses for metal
deposition experiments. Then, image reversal resist is spun on and structured (4). A Platinum layer is PVD deposited in an e-beam evaporator (5). The wafer is then submerged and soaked in acetone for a few hours. The acetone dissolves the resist, and metal that has been deposited on top of resist is washed away, whereas metal that has deposited directly on the silicon surface remains (6).

Two problems were discovered with this process. First, many of the features that were supposed to stay attached to the wafer were washed away during the acetone soak, indicating a metal to silicon adhesion problem. Second, a severe edge burr was discovered around all the metal features. Figure 4.10 shows the edge of a metal deposited feature. The higher surface is the metal, the lower surface is the silicon. A burr around the perimeter is not tolerable. If the valve seats were made this way, a burr would prevent the valve plate from touching down flat and create leakage paths. Figure 4.11 shows SEM images of the deposited metal features on the bottom of the anisotropically etched recess. The top left shows how some of the metal features have washed away during the acetone soak. Upon closer inspection, it seems that the metal features are left with a fair amount of residual stress after the PVD process. Some of them appear to be bent up, which could be the result of a combination of high residual stress gradient and poor surface adhesion. The bottom
right also shows the burrs around the perimeter that have been discovered earlier, in the image obtained by the optical profilometer.

### 4.3.2 Making Valve seats by using the BOX of an SOI wafer

The final choice for fabricating the valve seats was to use the buried oxide layer of an SOI wafer that has been structured as described in Section 4.2.4. The challenge is to spin photoresist onto a surface into which the valve recesses have been etched. What comes to the rescue here is the fact that the valve recesses are quite shallow, with a depth of 10 microns and a diameter of 2.5 to 3 mm. The part of the process used here is shown in Figure 4.12. It starts with an SOI wafer with valve recesses etched into the device layer (1). Resist is then spun onto the surface (2). It has been found that the resist coverage after the spin-on was fairly uniform and the subsequent expose, develop, and post bake steps resulted in sharply defined resist features. After the lithography step, the oxide is etched using the AME5000 plasma etcher with CF4 used as the etching gas (3). Later, the etching method was changed to a wet BOE etch, which worked equally well. A BOE wet etch is
Fabrication of the valve seats

Figure 4.11 SEM images of the metal deposited valve seat features

Figure 4.12 Fabrication plan for the BOX valve seats

also more cost effective. The resist is stripped in piranha acid and the oxide valve seat features are inspected (4).
Figure 4.13 shows an SEM image of the first oxide valve seats made with this method. On this particular wafer, the back side port hole has been etched already, which is described in more detail in Section 4.4. Also, the valve seat is supposed to look like a "donut" shaped feature, but in the SEM image, the inner and the outer perimeter are very wavy. The reason is that for this experiment, inexpensive transparency photo plot masks were used with a resolution of 5080 dpi, which corresponds to a dot size of 5 microns. The scale on the image shows that the ripples in the valve seat perimeter are approximately of that size.
4.4 Fabrication of the backside port holes

In order for the fluid to flow out of the valve cavity, port holes have to be etched into the substrate silicon wafer. The port hole etch turned out to be difficult. The problem has to be seen in conjunction with the picture of the entire valve die: access holes for an electrode as well as the inlet hole are being etched into the valve wafer in the same step. The problem is, that the outlet port hole is much smaller in diameter, 60 microns, whereas the inlet hole and the electrode access hole are 500 microns and 1500 microns, respectively. Large diameter holes etch much faster in the DRIE plasma etcher than do smaller holes. Considerable overetching of the larger holes causes backside blowout and damage to the bonding surface. Initial etches have shown a difference of up to 1 hour in etching time between the small and the large holes.

In order to compensate for this effect, the etch on the larger holes must be slowed down to approximately match the etch rate on the smaller holes. This is usually done by using a halo technique as indicated schematically in Figure 4.14. Instead of etching the entire area

![Figure 4.14 Halo etch mask to adjust the etching speed of differently sized holes.](image)

of the large holes, only a halo shaped outline is etched, which slows down the etch
sufficiently to make the etch fronts of the small holes and the large holes arrive at the backside of the wafer within minutes from each other.

As an aside, it is impossible for the metrology tools available in the lab (microscope, optical profilometer) to detect the depth of a hole 60 microns in diameter, when the depth is near the end of the etch through the 500um wafer. The method used here was to target mount the wafer onto a transparent quartz glass wafer and then use a microscope with a back light to see when the light stated shining through the small hole. This method worked very reliably.

4.5 Fabrication of the membrane/valve plate layer

4.5.1 Using a second SOI wafer to form the valve plate

Suspending a valve plate layer with tethers over the valve recess was the most difficult part of the fabrication process. At first, a second SOI wafer with appropriate device layer thickness was bonded face down onto the valve seat wafer and then the handle wafer of this SOI was removed to leave behind a thin valve plate layer. Figure 4.15 shows the fabrication process steps. The fabrication starts with a valve seat wafer having the valve recess, the port holes, and the actual valve seats already fabricated (1). Then, an SOI wafer with appropriate device layer thickness is bonded on top using silicon fusion bonding that includes an annealing step where the bonded stack is heated to 1000 degree C and held at this temperature for 1 hour (2). Then, the "sacrificial" handle wafer of the second SOI wafer is completely removed in a silicon etch, such as KOH or TMAH for about 9 hours until the buried oxide layer is exposed (4). This buried oxide layer will act as an etch stop. A short BOE etch of approximately. 15 min will remove the buried oxide layer of about 1 micron in thickness (5). After removing the back side protection layer (6), a photoresist masking step is carried out to define the tether geometry (7). A short etch in the DRIE etcher (3-4min) defines the tethers by etching through the valve plate layer into the valve cavity.
Wet etch the back side

A lot of time was spent finding a method to protect the back side of the valve wafers during the long term wet etch (4). The protection is necessary because only the "sacrificial" handle wafer is to be etched, not the back side where the valve port holes are located. Heated KOH and TMAH etchants have been used, but few materials can hold up to these etchants, especially at 85 degree C.

Two basically different methods have been tested. In one method, certain compounds or adhesive tapes have been used to cover up the back side of the wafer, in other methods, compounds have been used to glue a protective wafer (e.g. with a nitride layer) onto the back side. Figure 4.16 shows the two different methods in comparison. A series of
different compounds and tapes were used with mainly negative results. The compounds included 5-min epoxy, black wax, SU-8, and several different grades of silicone. The tapes, which usually failed more quickly than any of the compounds, included teflon tape, die saw tape, and different all-purpose tapes. None of these methods provided perfect back side protection against the hot anisotropic etchants. While every method failed one way or another, back side protection with SU-8 delivered results with the least catastrophic failures. SU-8 can be considered as back side protection when a short time, shallow etch is needed. SU-8 can be easily removed by a piranha clean.

**Sacrificial handle wafer removal by backside grinding / polishing**

After the tremendous problems encountered with the handle wafer removal using a wet etch, a different approach was tried. After bonding the second SOI wafer on top of the valve seat substrate wafer, the stack was sent out to a polishing company, Aptek Industries in San Jose, CA, to have most of the sacrificial handle wafer removed by grinding and polishing. It had to be assured, that the polishing process would not cut into the buried oxide layer and thus damage the valve plate layer. The way the polishing process works is that the amount of material removed can be monitored very accurately in situ. The thicknesses of the backside handle wafer is 525 +/- 25 microns. This means, that in the
worst case scenario, only 500 microns need to be removed until the polisher would cut into the buried oxide layer. In order to be on the safe side, a total material removal of 475 microns was specified to the polishing company. This also means, that at most 50 microns of silicon are left on top of the buried oxide layer. The remaining silicon can be etched off using the DRIE plasma etcher with a plain SF6 etch recipe (no polymer passivation). This will bring the wafer stack to the fabrication level equivalent to step (4) in Figure 4.15, after which the fabrication process can continue as specified there, with the wet BOE etch and the DRIE tether etch.

The results of this polishing procedure were good in terms of the handle wafer removal, however, the polishing slurry also contaminated the inside of the valve cavities. Figure 4.17 shows the standard procedure used by the polishing company. At first, the wafers were mounted to a chuck by a bed of wax. This is done in order to create a stress free mount, which usually prevents the wafers from breaking after polishing. After the polishing procedure, the wafer is dismounted by dissolving the wax in solvents. It is presumed that this very cleaning procedure washed dissolved wax into the valve cavities. During the subsequent tether etch, the DRIE plasma encountered the wax residue and showed a lot of contamination under the microscope. Since the valve cavities are mostly enclosed, the only way to look inside is by using an infrared microscope. Figure 4.18 shows a typical image obtained by this microscope. The image was taken right after the wafers had returned from the polishing company. It clearly shows that wax residue leaked in through the port hole in the center. This wax residue disturbed the tether etch and also caused short circuits when the valves were tested electrically.

4.5.2 Using a thinned wafer to form the valve plate

A way to avoid all problems with removing the sacrificial handle wafer is to use a thinned wafer instead of an SOI wafer. The limitations in thickness are much higher, since thinner wafers pose a handling problem during fabrication. Wet processing steps, spin dryers, vacuum clamps and other steps can break a tinned wafer quite easily, so that special care
and planning has to be done when thinned wafers are used in a process. Thinned wafers of 100 and 110 microns in thickness were used successfully in this valve fabrication, and they have survived all necessary handling. With the previously described method of using an SOI wafer with a sacrificial handle wafer, much thinner membrane layers can be created because the device layer is being held by the handle wafer until after it is bonded to the valve seat substrate wafer and thereby protected against breakage. Nevertheless, using a thinned wafer eliminated the sacrificial handle wafer removal problems and made it possible to create cleanly etched valve plate and valve plate tethers.
If polished correctly, thinned wafers are free of residual stresses and can be bent to very small radii. Figure 4.19 shows a picture of a thinned wafer being bent after a back-
Fabrication of the membrane/valve plate layer

grinding and polishing procedure [Aptek Industries]. It has been found that thinned wafers survive batch wet processes better if in the wafer cassette, a regular thickness wafer is put into the two adjacent slots. This way the thinned wafer will not get hit directly by the water spray nozzles that can be found in dump rinsers or spin dryers.

Figure 4.20 shows the altered fabrication plan where a thinned wafer is used to form the valve plate layer as opposed to an SOI wafer. All backside protection problems discussed in Section 4.5.1 were eliminated, together with a number of processing steps; compare Figure 4.15 and Figure 4.20. Figure 4.21 shows an SEM image of a single valve die from a completed valve wafer with a 100 micron thin wafer as the valve plate layer. The bonding procedure as well as the tether etch worked very well.

The use of a thinned wafer with its minimum thickness of 100 microns has an impact on the way the tethers are designed. As it can be seen in the SEM image, there are three tethers boxed into each other by using a spiral shape. Since the valve plate layer is relatively thick, the tethers had to have a wrapping angle greater than 120 degrees. Thus, the spiral shape was necessary to accommodate all three tethers into the design space. The tether analysis was described in more detail in Section 3.8.3.
4.6 Electrical Contacts

For testing the valve actuator after fabrication, electrical contact has to be established with each of the two electrically separated poles of the valve wafer: the bottom handle wafer and the valve plate layer. A low resistance connection to silicon can be established with metal needle probes if a low resistivity, highly doped p-type silicon wafer is used as a substrate. The junction of metal and silicon usually creates a diode effect, where a certain threshold voltage is necessary to drive a current through the connection. Figure 4.22 shows the test setup that has been used to verify the contact resistance to different resistivity wafers. Two standard 3-axes micro manipulator stages are used to press two electrical contact needles onto the wafer surface at a distance of a few mm from each
Figure 4.22 electrical contact test setup

An isolator pad must be used to insulate the wafer from the metal base plate. A high voltage source forces a current through the silicon, which is measured by the ammeter. Figure 4.23 and Figure 4.24 show the current as a function of voltage for a high and a low resistivity wafer, respectively. For the high resistivity, low dopant concentration wafer, about 60 Volts are needed before a noticeable current starts to flow. The low resistivity wafer, however, is much better as it takes only about 2-3 Volts to overcome the diode effect. Therefore, low resistivity p-type wafers were chosen as substrates for the valve devices.
Final Valve Fabrication Plan and Fabrication Results

4.7 Final Valve Fabrication Plan and Fabrication Results

After many iterations and changes, a final fabrication plan was selected. The fabrication of the valve prototypes is based on SOI wafers, which are more expensive, but they allow for more reliable fabrication of the valve cavities. Wet etching techniques as described in
Section 4.2.2 have the potential of replacing the SOI wafers if more cost effective mass produced valve devices are desired. However, more research is needed to solve the problems of precise and uniform depth control.

Figure 4.25 shows the final fabrication plan. An SOI wafer with low resistivity handle wafer is used as the substrate (1). A first lithography step defines the valve recess (2), which is then etched in a DRIE plasma etcher (3). Then, a second lithography step is carried out to define the valve seats and the landing pads (4), which are then etched in a wet BOE etch (5). A third lithography step on the backside defines the outlet port hole, the
inlet port hole, and the electrode access hole (6). These features are etched in the DRIE plasma etcher (7) using a microscope with a backlight to detect the point when the holes have etched through. A thinned wafer is bonded on top of the valve seat wafer and then annealed for 1 h at 1000 degree C (8). We have used thinned wafers of 100 and 110 microns in thickness. The fourth lithography step defines the valve plate tethers (9), which are then etched in the DRIE plasma etcher (10).

After this, there are two possibilities for further processing as shown in Figure 4.26. One possibility is to take another identical (but mirror imaged) valve seat wafer from step (7) and bond it on top of the fabricated wafer from step (10). This creates a switch valve that can direct the flow of a gas from an inlet into one of two possible directions. The other possibility is to fabricate a pyrex glass wafer which can be anodically bonded on top of the wafer from step (10) to provide a pressure housing. This was actually done here for testing purposes. The fabrication of the pyrex lid also involves several steps. A blank pyrex wafer is first cleaned with piranha acid (3:1 hydrogen peroxide and sulfuric acid) and then further cleaned in an oxygen plasma asher (A). Then, a 200 Angstrom thick adhesion layer of chromium is deposited by e-beam evaporation followed by a 1000 Angstrom layer of gold. Both layers have to be deposited on both sides of the wafer (B). Then, one side of the wafer is structured with a photoresist mask. The gold is then wet etched using Aqua Regia, and the chromium is etched with CR-7 chromium mask etchant using the same self aligning mask (C). The wafer is then submerged into diluted 2:1 H2O:HF acid to etch the recess (D). The etching time is 16 min which results in a depth of approximately 10 microns. The recess prevents the valve plate from bonding to the pyrex wafer during the anodic bonding process. After this, the gold is stripped off in aqua regia, and the chromium is stripped off in CR-7 (E). The last step is to anodically bond the pyrex wafer on top of the valve wafer to create packaged valve chips (F). The glass lid makes it possible to die saw the wafer into individual valve chips because it prevents the die saw slurry from entering the valve cavity and destroying the valves.
Figure 4.26 Further processing of the valve wafer

Figure 4.27 shows an SEM image of a cross section of a fabricated valve after cutting through the center using the die saw. Since the valve plate was only held by the small micro tethers during the sawing operation, the cut is not very smooth. However, the basic features such as the valve plate with the vent holes, the tethers and the outlet hole can be easily recognized. Figure 4.28 provides a better view of the inlet channel and the inlet hole, which is located further upstream. The arrows indicate the gas flow direction during valve operation.
Figure 4.27 SEM image of a sectioned microvalve
Figure 4.28  SEM of the inlet hole and the inlet channel
Chapter 5

VALVE PERFORMANCE

5.1 Introduction

In order to completely characterize the microvalve, several different tests were carried out. The objective of the first tests after microfabrication was to verify the proper function of the electrostatic actuator. Secondly, the sealing capability needed to be measured, and at last, a measurement was done to find the dependency of the flow rate on the applied pressure drop and actuation voltage.

5.2 Packaging

Valve dies have been tested at different stages of the fabrication process. The first tests were done to see how well the electrostatic actuator by itself would work. For this, it was not necessary to provide a fully diced and fluidically packaged microvalve, so the actuator tests were carried out on the wafer level. A valve top cover was also omitted for optical access to the valve plate so the valve plate displacement as a function of the actuation voltage could be directly measured using a ZYGO optical profilometer system. Electrical connections were established using standard electrical needle probes with 3-axes positioners. Figure 5.1 shows a photograph of the experiment.

Figure 5.2 illustrates how basic leakage tests were performed on the wafer level without having a full package. PEEK tubing is connected to the backside port hole of the valve and
Figure 5.1 Electrical test setup

Figure 5.2 Wafer level leakage tests
attached to the He leakage tester. From a He bottle, a spray nozzle is used to displace the air and create a cloud of Helium around the closed valve. Any Helium that leaks past the valve plate and the valve seat and reaches the port hole is detected by the He-leakage tester. Since the valve is uncapped and open to the environment, the pressure drop across the closed valve can be at most 1 atmosphere, which is the vacuum generated by the leakage tester.

For the fluid flow tests, a positive pressure must be applied across the valve seat. Therefore it was necessary to package the valve on the die level so the inlet can be pressurized. The valve itself was capped with a Pyrex glass wafer, then diced, and then put into a specially made package that uses O-rings to provide the interface between the microvalve and the external pressure source. Figure 5.3 shows an exploded view of the package design. A base plate is made of Plexiglas and contains holes and channels that connect to the inlet and the outlet ports of the valve, and is sealed with O-rings at the Plexiglas silicon interface. A set of dowel pins is used for alignment, and a valve die alignment plate ensures that the valve die comes to rest at the correct position so the vias for fluid and electrical connections line up properly. The die alignment plate is a little thinner than the thickness of the valve die, so that when the top cover is bolted on, the clamping force is available to squeeze the O-rings to form a proper seal. Pogo pins, which have a spring loaded test needle, are used to provide the electrical connections.

5.3 Electrical Tests

Measurement method and results

The first component that could be tested in the course of fabricating the valves was the electrostatic actuator, for which the setup in Figure 5.1 was used. A ZYGO optical profilometer was used to measure the displacement of the valve plate as an electric potential was applied. The objectives of the profilometer generate fringe patterns on a surface as soon as it comes into focus. A displacement of this surface normal to the objective axis can be visualized by observing the fringes moving sideways. Figure 5.4
shows a typical view through an objective lens by the CCD camera. When the valve plate is at rest (and level with the surrounding structures surface, the fringe patterns go straight through the valve plate. After pull-in, the valve plate is displaced and the fringe patterns move sideways (out of the field of view of the CCD camera, but still captured by the measurement).

Figure 5.5 shows a measurement result obtained by this method. Shown is the valve plate displacement as a function of the input voltage. Three different theoretical predictions are shown. The tethers for this particular valve were circular in shape with a constant radius and a designed width of 20 microns. Since the radius of the circular tethers was large and
the tether angle small, the first order model assumes the tethers to act like straight, double clamped beams (Equation 3.37). The second order model (Equation 3.36) takes the
curvature into account, and shows a better match with the theory. Measurements of the as-fabricated tethers showed that imperfections in the fabrication process caused the tethers to actually be 16 microns in width rather that the designed 20 microns. The measured width of 16 microns was used in the second order model and yielded an excellent match with the experimental results. The tether thickness in this case was 20 microns (i.e. the thickness of the valve plate layer), the tether radius was $R=1380$ microns, and the wrapping angle was $\alpha_0 = 26.4^\circ$.

**Electrical breakdown and solution to prevent the same**

During the first electrical tests, breakdown was observed on a regular basis. This was due to the fact that the valve plate was not sufficiently isolated from the substrate. The oxide standoffs that were provided to prevent the valve plate from touching down turned out to be insufficient. While the standoffs did prevent the two surfaces from physically touching, the mere proximity of only 0.5 microns caused sudden spark discharges, initiated at sharp corners where electrical field lines accumulate. Figure 5.6 shows a victim of such a failure where a piece of tether has even been ejected out of the valve. If the field between the valve plate and the substrate breaks down, a relatively large current is passed though the tethers. Due to their electrical resistance, this current causes Joule heating beyond the melting point of silicon. The SEM image shows how the tips of the ejected tether are melted into small pin head shapes.

The method that was used to prevent this failure was to grow a dry thermal oxide of approximately 1000 A in thickness as a last step in the fabrication process. This oxide encapsulates the entire structure of the valve before it is packaged. This proved to be very reliable and robust, and spark discharge failures were not observed again. Silicon dioxide is an excellent insulator and its dielectric strength dictates that for each volt of electricity applied to an electrostatic actuator, approximately 1 nm of oxide is necessary to prevent breakdown.
5.4 Leakage Tests

The leakage tests were carried out in two different settings. The first tests were done on the wafer level as described in Section 5.2. A special technique was used to connect PEEK tubing to the outlet of the valves on the wafer. Figure 5.7 shows a photograph and a schematic of the packaged wafer. First, a metal ferrule is attached with a minimal amount of epoxy. Then, a first layer of epoxy is applied to keep the ferrule in place so that the PEEK tubing can be inserted. Finally, the PEEK tubing is held in place by a second layer of epoxy. A perforated plate made of Plexiglas was glued in place with the second layer of epoxy. This Plexiglas plate provided a structural link between all the ferrule connections so that the entire structure has more rigidity and single ferrule connections are less prone to breaking off.
A He-leakage tester was connected to the free end of the PEEK tubing using Swedgelock connectors. After evacuation of the leakage tester, Helium was sprayed through a nozzle around the vicinity of the valve to be tested (see Figure 5.2). This was repeated for each working valve on the wafer. The results of these tests can be seen in Figure 5.8. The lowest measured leak rates were on the order of $10^{-9}$ atm-cc/sec. These values, however, stopped being repeatable after a few tests. The lowest, more reliable and repeatable values obtained were on the order of $10^{-7}$ atm-cc/sec. This, and the large number of valves with
higher leakage rate can be explained by the fact that the valves were uncapped and open to the environment. The tests did not take place in a controlled clean room and therefore, particles were likely caught between the valve plate and the valve seat, causing the valve to not close completely. Leakage values are very sensitive to the effective gap height for gaps smaller than about 5 nm (see Figure 5.9).

Additional leakage tests were carried out by using the fully packaged valves as shown in Figure 5.3. The package baseline was measured by assembling the Plexiglass package with the O-rings in place, but without the valve die and without the alignment plate. The baseline for this type of package was $3 \cdot 10^{-7}$ atm-cc/sec, most likely because of the permeability of the Buna-N rubber O-ring seals. The valve leakage rate was measured after flushing the system to make sure that the inside of the valve and the supply lines were filled only with Helium. The leakage rate was repeatedly measured to be $5.8 \cdot 10^{-6}$ atm-cc/sec. The units used here are the ones that are shown on the display of the leakage tests.
Leakage Tests

tester, a Varian 979 portable helium leak detector made by Varian, Inc., Palo Alto, California, USA.

The leakage measurements obtained here can be compared to the values found in the literature in Figure 2.10 by converting into leakage conductance. After this conversion, for the wafer level leak tests we obtained between $1.8 \cdot 10^{-15}$ Pa-m$^3$/s/Pa and $4.6 \cdot 10^{-12}$ Pa-m$^3$/s/Pa. For the tests using the Plexiglass package we obtained $5.8 \cdot 10^{-12}$ Pa-m$^3$/s/Pa.

From the Knudsen equation for molecular flow introduced in Chapter 3 (Equation 3.44), one can plot the leakage flow rate through the closed valve as a function of the gap between the valve seat and the closing member, as shown in Figure 5.9. It is assumed that

![Figure 5.9 Molecular flow rate as a function of the gap](image)

the gap height $h$ is on the order of the roughness of the two touching surfaces. The values used for this plot are the temperature $T=293K$, the universal gas constant $R_0=8.3143$ J/mol-K, the molecular mass for Helium $M_{He}=0.004$ kg/mol, the inner valve seat radius $r_i=31$ microns, and the outer valve seat radius $r_o=80$ microns. The pressure drop $\Delta p$ and
the reference pressure $p_{ref}$ were both 1 atmosphere. As mentioned earlier, we have not performed destructive tests on working microvalves due to low yield, so it is difficult to relate the actually measured leakage rate to the gap between the valve seat and the closing member when the valve is closed. However, we have examined the backside of valve plates from valves that were built on earlier wafers that underwent the same microfabrication plan. Figure 5.10 shows that in the SEM image, a discoloration can be observed near the area on the valve plate backside that is facing the valve seat. We have used an optical profilometer to image this part of the surface and we have found that the discoloration represents a bump of appx. 18 nanometers in height. Assuming that the height of the bump dictates the sealing gap for a closed valve, a leakage value on the order
of $10^{-6}$ atm-cc/sec should be expected, which is indeed a typical value measured in valves from the final built that were tested in the plexiglass package. Due to the fact that the bump occurred near the valve seat and port hole, a possible explanation could be preferential oxide growth, because the oxygen in the tube furnace has better access to the underside of the valve plate through the port hole on the backside. This could have happened during the final oxidation step that was discussed in Section 5.3 to prevent electrical breakdown. Since a total thickness of 100 nm of oxide is grown in this step, a non-uniformity of 18 nm sounds plausible. Work should be done in the future to avoid this phenomenon. If the surface roughness can be kept at 5 nm, which is a value typical for an unmachined wafer surface, an order of magnitude better sealing capability can be expected.

### 5.5 Flow Tests

The flow tests were carried out using the package of Figure 5.3. Two different characteristics of the valve were tested. First, the flow rate as a function of the pressure drop without any actuation voltage, then, the flow rate through the valve as a function of actuation voltage for different inlet pressure levels. Figure 5.11 shows the test setup and Figure 5.12 provides a schematic. A Nitrogen bottle with a coarse pressure regulator was used to provide testing gas at about 20 psi pressure. The gas was then passed through a finer pressure regulator where the valve test pressure could be dialed in. Subsequently, a mass flow meter MKS Type 179A in combination with an MKS Type 247D Four-Channel Readout was used to measure the gas flow rate. The flow meter was calibrated for Nitrogen, so no further scaling adjustments were necessary. After the flow meter, a pressure gauge Type Ashcroft 0-30 psi was used to measure the gas pressure just before the microvalve. Having the pressure gauge as close to the microvalve inlet as possible ensures that pressure drops across previous fluid elements in the line do not influence the pressure reading. After the pressure gauge, the testing gas flows through a short length of silicone tubing into the microvalve package. At the outlet of the package, the gas flows through another short length of the same tubing into a bubble indicator. The bubble
indicator consists of a jar filled with water where the end of the gas tubing is submerged under the waterline. This was used for easy visualization of the state of the microvalve, because when the valve is open, the out flowing gas creates bubbles rising in the water. To drive the valve, a voltage source was used and the voltage across the power leads was measured using a voltmeter.

The flow test results and a discussion about the model can be found in Chapter 3, Section 3.8.
Figure 5.12 Schematic of the flow test setup
An introduction to gas chromatography systems has been given in Chapter 1 and it has been explained that valves are needed in a gas chromatography system for directing the sample and carrier gas. An extensive literature review of publications on existing microvalves was provided in Chapter 2, which comprised more than 30 journal papers and 50 patents. A comparison parameter, the "leakage conductance" has been introduced in order to be able to compare the leakage values given in the publications, which are given by the different research groups in different units and for different inlet pressures. Four publications with the lowest leakage rates have been identified and compared. It was concluded that none of the existing valves would meet the requirements of a gas chromatography system, and that a design approach with complementary surfaces for valve seat and closing member would be the most promising. Chapter 3 then described how a design was chosen with a tether suspended valve plate that closes against a flat valve seat. In Sections 3.82 through 3.84, equations were provided to capture the behavior of the electrostatic actuator, the suspension tethers, the fluid flow when the valve is open, and the leakage rate when the valve is closed. The equations were then combined in Section 3.85 and a theoretical solution was found that is capable of predicting the behavior of the valve, which is shown in Figures 3.34 and 3.36. Chapter 4 described the different microfabrication techniques that were experimented with in order to achieve the valve recess, the valve seats, the backside port holes, as well as the valve plate layer. It was determined that for the prototype valves, an SOI wafer should be used for the valve
substrate that contains the valve recess, the valve seats, and the port hole, and a thinned wafer should be used to make up the valve plate layer that contains the valve plate and the tethers. The final fabrication plan was described in Section 4.7. The final size of the valve is about 3mm in diameter. The packaging and test of the valve was reported in Chapter 5. The first pull-in tests with the electrostatic actuator showed very good correlation between the theoretical formulas and the measured data (Section 5.3). However, these tests also revealed a weakness in the design, which led to repeated failure due to electrical short circuits and the resulting current overheating immediately after pull-in. The problem was solved by growing a protective layer of approximately 1000 Angstrom of thermal silicon dioxide on the entire valve structure as a final fabrication step. The experimental setup used for the flow tests was described in Section 5.5, where a plexiglass package assembly with O-rings was used to interface the test equipment with the valve dies. Typical values for the open flow rate were 8 sccm of Nitrogen at 8 psi inlet pressure, with a pull-in voltage of about 23 Volts.

The most amount of time in this work was invested into the fabrication process. Many fabrication techniques have been tried unsuccessfully to fabricate the valve geometry. A large amount of time was spent finding an appropriate technique to create valve seats inside a valve recess with surface roughness of less than 10 Angstrom. And another major problem has been to accomplish the valve plate and the tethers. The most important breakthroughs were the use of SOI wafers to fabricate the valve substrates containing the valve seat and port hole, and the use of thinned wafers to fabricate the valve plate layer. By using SOI wafers it was possible to take advantage of polished wafer surfaces for fabricating the valve seats. By using thinned wafers it was possible to replace an earlier process where a second SOI wafer was bonded on top of the valve seat SOI wafer and the handle wafer of this second SOI wafer was removed by a KOH or TMAH wet etch to leave only the device layer of the second handle wafer. This process had unresolvable problems with protecting the backside of the valve seat SOI wafer during the hour long wet etching step. No materials could be identified that were able to first hold up against the acid, and then be easily removed afterwards without damage to the rest of the
structure. Using a thinned wafer instead has solved an entire series of problems at once and yielded very nice fabrication results.

There are several aspects where recommendations for future work can be given. The first improvement should be in the yield. The valve wafer is quite rugged during fabrication up to the point where the tethers are etched using DRIE. After etching the tethers, it is necessary to grow a thin layer of oxide to prevent catastrophic failure by dielectric breakdown of the electrostatic actuator. The oxidation in the furnace is preceded by an RCA wet cleaning step, which is standard procedure to prevent contamination of the furnace equipment. This wet cleaning step, however, imposes fluidic forces on the fragile valve plates and also causes stiction after the rinse water is dried. To improve the yield dramatically, alternative dry cleaning processes should be investigated.

A further interesting testing procedure would be to use pulse width modulated actuation voltage (PWM) in order to see whether the valve could be used as a proportional control valve as opposed to simply on/off. In some references in the literature, for example in [105], researchers have successfully used PWM voltage to turn an electrostatically actuated valve into a proportional valve, even though the electrostatic actuator is fundamentally governed by pull-in behavior.

Another aspect that would require further thought is particle rejection. Since the valve’s high sealing capability relies on ultra flat surfaces that are inherently non-compliant, the valve is especially susceptible to particles that are being washed in with the gas flow and may get stuck between the valve seat and the valve plate. The leakage rate is especially sensitive to the gap height and therefore, even the smallest particles could increase the leak rate. One way of dealing with this problem is to provide adequate filtering before admitting a gas into the valve system. Another way would be to provide a deep well around the valve seat as indicated in Figure 6.1. The well would provide a space where particles could be statistically trapped before they reach the valve seats. This technique is also used in macro scale machinery valves to prevent particle contamination.
A method has been envisioned where a zipper actuator could be used to increase the pressure that a microvalve can handle. Figure 6.2 shows how the concept works. Initially, the valve is closed and the valve plate is pressed onto the valve seat by the pressure.
differential between $p_1$ and $p_0$ (1). If an electric potential is applied, the free end of the valve plate is pulled up towards the top electrode (2) and continues to travel along (3). At a particular point, the force on the valve seat reaches a critical point where a crack opens and the pressure differential decreases or disappears (4). The valve plate is then being pulled further towards the top electrode (5, 6) and the valve is open. Such zipper actuators can exert much greater forces than electrostatic actuators where the plates remain parallel [116]. The difference to the existing "traveling wave actuators" described in Chapter 2 is that the valve plate in the valves described here does not have to be fabricated in a pre-bent shape, which is generally hard in microfabrication. It is possible to use the same fabrication process found in this work and only a change in the masks is required. We have fabricated prototypes of these kinds of valves as an experiment on our last wafer. About half of the wafer space was used. It is expected that these valves can open against a pressure of up to 70 atmospheres. Experimental verification is currently being performed.
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