Microvalves for Precise Dosing

Proportional Flow Control on a Chip

Maarten Groen

MICROVALVES FOR PRECISE
DOSING
PROPORTIONAL FLOW CONTROL ON A CHIP

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Abstract

Precise control of fluid flow becomes increasingly challenging as systems and instruments are scaled down. Smaller dimensions allow smaller flow ranges, but also leave smaller margins for error in performance. Reliable and effective fabrication and assembly procedures are therefore a primary requirement for any microfluidic system, if it is to be successful in real-world applications. This thesis describes the achievements made in ongoing efforts to create miniaturized, proportional control valves for minute gas flows. In this research we focus on MEMS-based fabrication processes, as they allow high precision manufacturing of miniature devices with high parallelism. This enables higher fabrication yields, improved reliability and increased integration.

We present four valve designs that can be divided into two main categories, being either based on silicon-on-insulator (SOI) technology or based on surface channel technology (SCT). The first group focuses on obtaining a proportional flow controller using simple, straight-forward fabrication processes with a small number of process steps, for use in an ambulant blood pressure waveform (BPW) measurement system. Two single-wafer valve designs are presented, both offering built-in capacitive sensing of the valve displacement which can be used to correct for actuator hysteresis and improve control precision. Nitrogen gas flows up to 13 g h^{-1} at a pressure of 500 mbar are demonstrated in good agreement with analytical and numerical models, with leakage below 0.1 mg h^{-1} at 1 bar. Maximum throughput is estimated at 25 g h^{-1} at 1 bar. A fully functional control valve assembly is demonstrated with integration of a miniaturized piezoelectric bimorph actuator. Time-dynamic characterization demonstrates that the control valve is suitable for high-speed flow control, with a mechanical bandwidth of 8 kHz and a frequency-independent response up to 3 kHz.

The second group demonstrates two proportional control valve designs in an already existing (SCT) fabrication process, to allow integration with existing components such as flow sensors. The first valve design allows flow control from a chip inlet or outlet to a fluidic channel embedded in the silicon surface, with a flow range of $> 1250 \text{ mg h}^{-1}$ at 600 mbar and a leak flow below 0.05 mg h^{-1} at

1 bar. The second valve design supports smaller flows ($> 70 \text{ mg h}^{-1}$ at 200 mbar), but allows fully on-chip flow control between any two surface channels. A good fit is obtained between the measured flow profiles and analytical flow models of both valves.

The valve designs aimed at the BPW monitoring system are shown to be very well suited for the aimed application, although a smaller bimorph piezo may be required to meet the demanded physical dimensions. The combination of capacitive displacement sensing with bimorph piezoelectric actuation allows for high-speed, high-precision flow control with low power usage. The SCT-based microvalves demonstrate proportional, on-chip flow control, suitable for integration with existing flow sensors. Their designs are tailored to match an existing high-resolution mass flow sensor which has a limited flow range, but the designs of both the valves and the sensor can be scaled up to increase the range.

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1

Introduction

1.1 Microscale fluidic systems

Control of small-scale fluid flows, be it of gases, liquids, plasmas or multiphase mixtures, forms the very essence of the field of microfluidics. The impressive number of microfluidic publications from the past two or three decades shows that ‘control’ is a broad term indeed. Reaction chemistry [1, 2], (bio)chemical analysis [3, 4], precision food processing [5, 6], environmental monitoring [7, 8] and point-of-care medicine [9, 10] are just a few examples of possible applications.

The driving force behind microfluidic research and development is miniaturization. There are several advantageous properties that only exist in miniaturized fluidic devices. At micrometer scales fluid flow is typically laminar, which can for example be used to create liquid-liquid interfaces that do not readily mix [11, 12]. The increased surface area to volume ratio can be used to improve chemical reactivity between a fluid and surface reactants or catalysts [13]. The relative increase in surface area also strongly increases the heat transfer coefficient at the fluid-solid interface which is beneficial for cooling applications [14].

There can however also be many advantages to simply reducing the dimensions of existing fluidic systems. Apart from the obvious increase in portability, decreasing device dimensions can lead to a higher level of integration. In particular the

field of micro total analysis (or “lab-on-a-chip”) research focuses on implementing multiple fluidic functions on a single, miniature device [15, 16]. Smaller devices may also allow smaller actuators, with an equivalent reduction in power usage and, for some actuation types, an increase in speed. Decreased internal and dead volumes of control or sensing devices leads to improvements in speed and accuracy. Furthermore, smaller fluid sample size reduces waste.

It should be noted however that, while showing a lot of promise, the field of microfluidics has so far produced only a limited number of commercial products. In designing microscale fluidic systems it is therefore key to consider not only the demands of the application, but also the complexity of the found solution and the feasibility of commercializing it [17].

1.2 Proportional flow control

In many fields relating to microfluidics there is a demand for accurate, proportional control of fluid flow. This control could for example relate to fluid sample size in analytical systems, constant flow of reactants in reaction chemistry, or automated drug administration in medicine. The key requirement here is the ability to control (total) flow to a specified value, rather than on-off control (binary valves) or passive one-way control (flow rectifiers).

Figure 1.1: Functional model of a typical mass flow controller (dashed box), consisting of a control valve, flow sensor and control circuit.

The basic operating principle of a proportional flow controller can be modeled as shown in figure 1.1. It consists of a (mass) flow sensor, a proportional control valve and control electronics connected in a closed control loop. The electronics control the valve unit to always keep the measured fluid flow equal to a configured setpoint. The performance of a (proportional) flow controller can be defined by many parameters, including flow range, control precision, leakage flow, power usage, bandwidth, physical dimensions and internal and dead volume. The control precision is mainly determined by the resolution of the flow sensor and the repeatability of the valve control. Effects like hysteresis or creep will have a negative impact on the control resolution. The flow range is mostly determined by the geometry of the control valve and the maximum actuator stroke, but is also

limited by the sensor accuracy at very small flow ranges. The speed or bandwidth with which the flow can be controlled is determined by the slowest component in the system, which in microfluidics is often the valve actuation system, but bandwidth can also be limited by a large internal volume of the fluidic system. Properties like power usage and physical dimensions vary strongly with the geometry, actuator type and assembly method.

There are many potential applications for a miniaturized proportional mass flow controller, and performance demands are not necessarily the same in all cases. Some examples include:

- Evaporation systems for the production of solar cells, requiring a very stable flow rate without overshoot;
- Microreactors for flow chemistry, demanding chemical compatibility and low leakage for safety reasons;
- Gas and liquid chromatographs, requiring a very stable flow rate and a large dynamic control range to facilitate both high-pressure and low-pressure flows;
- Dosing systems for the food production and pharmaceutical industries, demanding high control precision and easily cleanable or replaceable components;
- Flow control for medical purposes, e.g. in blood pressure measurement systems, requiring compatibility with the human body.

It is clear that the specific set of demands is determined by the application. In order to successfully design a microfluidic system that is suitable for commercialization, it is therefore also important to design for the application.

1.3 Aim of the research

Within the framework of the ‘Microvalve for precise dosing’ project funded by the Point-One Phase2 innovation program of the Dutch Ministry of Economic Affairs (PNE101004), it is the aim of this research to realize an actuated micro control valve for use in a miniaturized proportional flow dosing system. In order to make the micro valve suitable for industrial application and commercialization, it is key to combine a robust design with a low-complexity fabrication process. The aimed specifications of the microvalve are derived from two different applications, and are summarized in table 1.1.

Table 1.1: Typical microvalve specifications for two proportional flow controller applications.

	Blood Pressure Waveform Monitoring	Gas Chromatography
Fluid type	Air	GC gases
Flow range	200 sccm (15 g h^{-1})	500 sccm (37.5 g h^{-1})
Dynamic control range	1 : 100	$1 : 10^5$
Response time	< 1 ms	< 1 ms
Operating pressure	Up to 1 bar	Up to 10 bar
Differential pressure	Up to 300 mbar	Up to > 5 bar
Leak rate	–	$10^{-6} \text{ mbar l s}^{-1}$ ($\approx 5 \mu\text{g h}^{-1}$)
Operating power	< 100 mW	< 100 mW
Dimensions	< 5 mm $\times$ 5 mm $\times$ 5 mm	< 10 mm $\times$ 10 mm $\times$ 10 mm

The first application is a portable blood pressure waveform (BPW) monitoring system that requires high speed control (< 1 ms response time) of flows with a full range on the order of 200 sccm at moderate pressures (hundreds of millibars). Due to the portable application, the aimed physical dimensions are very small (< 5 mm $\times$ 5 mm $\times$ 5 mm). The application demands a constant air flow, so there is no strict leakage specification.

The second application is a high-resolution mass flow controller to be used in miniaturized gas chromatography systems. In this application, the requirement of controlling both high and low-pressure gas sources leads to a very high demanded dynamic control range. The signal-to-noise ratio in the detectors is strongly dependent on the stability of the flow rate and the sample purity, so a good stability of the valve in open state as well as low leakage of the valve in closed state is necessary. The leakage specification of $10^{-6} \text{ mbar l s}^{-1}$ helium is typical for most macro-scale applications. It will be particularly challenging to obtain this performance at the microscale, because closure forces are limited in small-volume, low-power actuators.

In recent research a hybrid calorimetric and Coriolis-based micromachined mass flow sensor has been developed, capable of measuring mass flows across more than five orders of magnitude [18, 19]. It currently supports flows up to 1.2 g h^{-1} (16 sccm for nitrogen gas), but this may be scaled up by increasing the physical dimensions of the design. To apply this mass flow sensor in a high-resolution mass flow controller, the microvalve should be able to control flows of the same order of magnitude. Ideally a microvalve for this application would be integrated on-chip with the flow sensor, to reduce internal volume of the fluidic system and therefore

decrease response time.

A typical value for the closed-loop settling time of an entire mass flow controller is 100 ms, which is generally dominated by the response time of the flow sensor and the stabilization of the fluid flow. To ensure that the valve does not become a limiting factor, an open-loop response time of 1 ms is demanded for both applications. Finally, the physical dimensions should be kept as low as possible, so that the flow control system as a whole remains compact and easy to integrate.

1.3.1 Mass flow conversion

The flow range specifications above are given in sccm, meaning cubic centimeter per minute at standard temperature and pressure, which is a unit of mass flow commonly used in industry. It expresses the mass flow as an equivalent volume flow at specific conditions. To arrive at a unit of grams per hour, we can apply the ideal gas law to convert the volumetric flow at standard conditions Q_{std} to a volumetric flow at actual conditions Q_{act} :

$$Q_{\text{act}} = Q_{\text{std}} \frac{P_{\text{std}}}{P_{\text{act}}} \frac{T_{\text{act}}}{T_{\text{std}}}. \quad (1.1)$$

Here P_{std} and P_{act} are the absolute pressures at standard and actual conditions, and T_{std} and T_{act} are the absolute temperatures at standard and actual conditions, respectively. In this thesis we follow the European definition of standard temperature and pressure, so that $T_{\text{std}} = 0^\circ\text{C}$ and $P_{\text{std}} = 1.013\text{ bar}$. For dry nitrogen gas flows, which are used in the characterization experiments in this research, the density at those conditions equals $\rho_{\text{N}_2} = 1.250\text{ kg m}^{-3}$ and the relation between both units becomes $1\text{ sccm} = 75.0\text{ mg h}^{-1}$.

1.4 Thesis outline

This thesis describes the solutions found for the challenges posed above. In chapter 2 the main considerations for the design of proportional micro control valves are described and analyzed. Existing microvalve designs reported in literature are broken down to their basic operating principles and evaluated for their suitability to the applications. Where possible, the applied actuation mechanisms are evaluated separately from the microvalve designs. A comparison is made between all described solutions, and the optimum solutions for our applications are selected.

Based on this analysis, an initial microvalve design is presented in chapter 3 that focuses on obtaining accurate proportional control in a very straightforward

fabrication process, aimed at the BPW monitoring application. The valve is made in a single silicon-on-insulator (SOI) wafer with a two-mask fabrication process, requiring only three etching steps and no wafer bonding. To enable high-resolution flow control the valve includes a capacitive displacement sensor, which is used to correct for observed actuator hysteresis. The valve's fluidic and mechanical characteristics are compared to analytical and numerical (flow) models derived for the design.

Although the straightforward fabrication process of this valve is a major advantage, the design does not allow easy integration of an actuator. The requirement of actuation is one of the main challenges in microvalve technology, since actuation forces and strokes are typically very limited at small scales. Chapter 4 therefore presents a revised design of the SOI-based microvalve, aimed specifically at integration of a high-speed miniature actuator. Focus lies particularly on achieving a sufficiently large actuator stroke, so that the valve can both close fully and be opened as far as possible. Chapter 5 studies further optimization of the assembly method to reduce the number of manual processing steps.

Having demonstrated high-speed proportional control in a straightforward, single-wafer design in chapters 3 and 4, chapter 6 proposes a second approach to the challenges posed in this research. Given the highly sensitive micromachined mass flow sensor reported in [19], two microvalve designs are presented that can be fabricated entirely in the existing process technology of this sensor. This allows full on-chip integration of a proportional control valve with the mass flow sensor, enabling creation of a mass flow controller with very high dynamic range and a minimized fluidic response time. One valve design is implemented with in- and outlet channels in the plane of the chip, which enables on-chip flow routing between several fluidic components. The other design controls flow between a fluidic chip inlet and a channel in the chip surface. It can be used to control flow between on-chip fluidic components and off-chip devices.

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2

Microvalve design principles

2.1 Introduction

Proportional mass flow control essentially requires three components, being a (mass) flow sensor, an electronic controller and an active, proportional control valve. Proceeding from the demands specified in chapter 1, a survey is made in this chapter of the primary design considerations of a micromachined, proportional control valve. Although they are sometimes related, we try to evaluate both the general operating principles of the valves and their actuation schemes separately. Proceeding from these analyses, we identify the design concepts and actuation schemes that we think are best suited for the realization of a mass flow controller that meets the demands.

2.2 Basic valve operation

Given a certain differential pressure, controlling fluid flow requires control over the flow resistance of at least one element in the flow circuit. In most cases, this resistance change is achieved by changing the mechanical geometry. Some non-mechanical alternatives have been reported, such as electro-capillary [1, 2] or

This chapter is based on “Design considerations for a micromachined proportional control valve” by M. S. Groen, D. M. Brouwer, R. J. Wiegerink and J. C. Lötters, published in *Micromachines*, 3:396–412, (2012),

Figure 2.1: Basic structure and operating principle of a microvalve. The moving plate covers an orifice in the valve seat, thus changing the flow resistance R_{flow} .

diffuser microvalves [3, 4], but since there is no actual closure these systems cannot guarantee low leakage. They are therefore not considered in this review.

A basic design of a mechanical control valve is shown in figure 2.1, consisting of an orifice in a fixed surface (the valve seat) which is covered by a vertically translating plunger (the valve plate). The maximum flow supported by the valve is defined by the structural dimensions - specifically, the diameter of the orifice and the maximum separation between the two plates (the valve stroke). The leakage performance of a valve is determined by the closing surface area, the relative surface roughness and flatness of the valve plate and seat, and the closing separation between them. In most cases the closing separation is determined by the force with which the plate is pressed against the seat. It is however also possible to close a valve by bringing the plate and seat very close to each other, without actual mechanical contact.

Mechanical microvalves are commonly divided into active and passive systems, meaning with and without a powered actuator. Passive valve designs are not evaluated in this study, because they are either one-way flow rectifiers [5–7], or designed to control a single, specific flow [8–10]. In order to achieve precise control over a range of flows, a powered actuator is required. The maximum actuator force often determines the maximum differential pressure across the valve, as the actuator needs to be able to counteract the fluid forces acting on the plates. The maximum actuator stroke limits the valve stroke, unless a mechanical amplification scheme is applied. The actuator also defines the power dissipation of a control valve, as well as its open-loop response time.

2.3 Valve concepts

At the macroscopic scale a wide range of valve designs exists for a large variety of applications. Gate valves, ball valves, poppet valves and needle valves are just a few common examples. In contrast, the diversity in microvalve designs is far smaller; in fact, the vast majority of valve designs at the micro scale are based

Figure 2.2: Schematic drawings of several microvalve design concepts. (a) Vertically translating plate; (b) Tilting plate; (c) Horizontally translating (sliding) plate; (d) Vertically translating membrane (cross-section); (e) Bending plate; (f) Needle (cross-section); (g) Scaling valve array.

on the same principle, namely using a vertically translating plate to cover a tube or orifice. This concept - illustrated in figure 2.2a - was used for the first valve micromachined in silicon [11], and has been applied with modifications in a large number of later designs. However, other valve concepts do exist, although they are applied less frequently. This section will give an overview of these concepts and will highlight their key advantages and drawbacks.

2.3.1 Vertically translating plate valves

Refinements of the vertically translating plate concept have led to many different microvalve designs. With the development of advanced silicon micromachining processes, the nickel diaphragm used in [11] was commonly replaced with silicon, or silicon-derivative materials. However, there are two important disadvantages of using hard materials such as nickel or silicon.

First, obtaining a leak-tight seal between two plates with high Young's modulus is challenging, as they will not readily deform to match each other's surface topography. It has been reported that the flatness of typical silicon wafers (~ 60 nm

Figure 2.3: Trapping of particles in grooves etched in a hard plate.

peak-to-peak across sub-millimeter scales) is already too large to obtain leak-tight closure on the order of $10^{-6} \text{ mbar l s}^{-1}$ [12]. A possible solution for this problem has been reported in [13]. It uses conformal layer deposition to fabricate a NiFe valve plate shaped (nearly) identical to a SiN seat surface, which are released from each other using sacrificial layer etching. Because of the nearly perfect fit between plate and seat, an exceptionally low leakage flow of $6 \cdot 10^{-9} \text{ mbar L s}^{-1}$ was achieved [14]. A similar approach using a combination of nickel and parylene materials led to a leak flow of $8 \cdot 10^{-6} \text{ mbar L s}^{-1}$ [15].

The second disadvantage to using hard materials is that any particle landing in between the plate and seat will prevent the valve from closing fully. Thus, in order to crush such particles and allow the valve to close, actuator pressures on the order of thousands of bars become required [16]. A possible solution to this particle sensitivity is to create a corrugated valve seat with grooves in the seat surface, trapping particles away from the closing surfaces as shown in figure 2.3 [17, 18]. The achieved leak flow reported for one such an approach was $2 \cdot 10^{-5} \text{ mbar l s}^{-1}$ at tens of bars of differential pressure [19].

2.3.2 Flexible membrane valves

A common modification of the translating plate concept is to replace the rigid plate by a flexible plate or membrane that wraps over or into the valve seat, as shown in figure 2.2d. The advantage of using membrane closure is that the material can elastically deform to match the precise shape and curvature of the valve seat, improving the seal and so reducing leakage flow. Dust particles can also be enveloped by the membrane, reducing the required actuator force at the cost of a slight increase in the required stroke. This reduction in force requirements may however be offset by an increase in stiction, which occurs when the seat and membrane remain in close contact for a prolonged period of time and the elastic material conforms more closely to the underlying morphology. A possible solution to this problem is to gradually peel the plunger off the seat surface [20].

When the flow channel is fabricated entirely out of flexible material, the design comes close to the pinch valve concept at the macroscopic scale. Such valves are highly tolerant of particles and other contaminants, which makes them well suited

Figure 2.4: Electrostatic tilting plate microvalve [29].

to the control of slurry-like fluids and use in contaminated environments.

The primary disadvantage of membrane closure is the increased fragility. Specifically thin membranes made from high Young's modulus materials may easily rupture, due to the large stress caused by the deformation strain. Excessive strain can for example occur if a sharp-edged particle is introduced into the system, or when the pressure difference between the channel and the surroundings becomes too great. It is therefore common to use materials with low Young's modulus such as silicone rubbers or other elastomers [17, 20–26].

Compared to rigid materials, elastomers typically have a reduced chemical resistance and higher permeability to moisture and gases. Some highly inert elastomers do exist, such as the commercially available Viton® and Kalrez® fluoroelastomers [27]. The latter is however only available as pre-fabricated sheets and seals, making it difficult to apply in a microfabricated design. Another solution for gas penetration is to use hybrid materials, for example coating PDMS with a thin layer of parylene [17, 28].

2.3.3 Tilting plate valves

A second modification of the translating plate concept is to fix one side of the plunger to the surrounding bulk. This turns a translating motion into tilting motion, as shown in figure 2.2b. With an appropriately chosen pivot point, this geometry can supply an amplification of actuator stroke (or force). An early tilting plate design was reported in [29], using a flexible beam fixed on one side to the bulk as shown in figure 2.4. An electrode embedded in the beam enabled direct electrostatic attraction to the substrate, but due to the electrostatic pull-in effect the valve suffered from very poor control precision (see section 2.4.3).

A design better suited to flow control applications is used in the commercially available NC-1500 Fluistor, shown schematically in figure 2.5. This valve uses thermal actuation, where the formation of evaporation bubbles creates a small expansion of a movable membrane. This small initial stroke is then transferred so that the actuator and plunger tilt away from the valve seat over a larger distance [30, 31].

Figure 2.5: Design and operating principle of the NC-1500 Fluistor, a commercially available tilting plate microvalve [30].

Figure 2.6: Annular disc actuation scheme presented in [32].

2.3.4 Bending plate valves

A more elaborate variation on the tilting plate concept utilizes specific materials, or combinations of materials, to fabricate a plunger that detaches itself from the valve seat. Material properties, such as thermal expansion, piezoelectric strain or phase-change shape memory, can be applied to obtain plates that bend or curl away from the valve orifice, as shown in figure 2.2e. The gradual ‘peeling’ of the plate can significantly reduce the force required to overcome plate stiction. It can also allow for precise flow control, provided the actuation has sufficient resolution. A drawback is the large bending moment created by the fluid pressure on the necessarily compliant plate, which leads to low leakage performance at large differential pressures.

A variation of the bending plate scheme that could circumvent this problem was presented in [32], as shown in figure 2.6. It uses an annular piezoelectric disc that contracts radially in the in-plane direction, eliminating any moving parts in the third dimension and so reducing the total volume of the device. Because the actuation direction is perpendicular to the fluid pressure, the stiffness in the direction of the fluid pressure can be increased without penalty. A disadvantage of the design is that fluid flows along the actuator system, meaning the fluid is exposed to the electric fields used for actuation of the piezo.

2.3.5 Horizontally translating (sliding) plate valves

A concept intuitively well suited to flow control applications is the use of a horizontally translating or sliding plate to vary the overlap between two orifices,

Figure 2.7: Horizontally translating plate microvalve using thermal actuation in a pivoting design [33]. The dashed line marks the perforated plate which overlaps with the inlet and outlet orifices.

shown in figure 2.2c. Apart from straight-forward control, a big advantage of such a design is that it can be made to require zero power in the steady state. The sliding motion can be executed linearly, in rotation or using a pivoting structure. The latter design is used in a valve reported in [33] and shown in figure 2.7. Thermal expansion of silicon rods pushes a perforated plate across the valve seat, changing the overlap of the orifices.

In contrast to its good control properties, the sliding or rotating plate concept suffers from very poor leakage performance. This is because the sliding movement takes place in the same plane as that in which the channel closure needs to be achieved. For low-force, high-precision actuation it is desirable to have low friction between the two plates, whereas good closure requires pretension between the plates, which leads to friction. Thus there is an inherent tradeoff between friction and closure. The valve presented in [33] illustrates this problem, as it is designed to always keep the plate floating.

2.3.6 Needle valves

The needle valve is a macroscopic scale design commonly used when precise flow control is required. It uses a tapering plunger moved into a tapered valve seat, commonly implemented as a sharp needle such as shown in figure 2.8. The good control properties stem from the large ratio between the needle's length and its diameter: A large axial translation leads to only a small change in radial closure, allowing for very precise flow control. These valves can also be made to have low leakage, by using the large closing area between the needle and the tapered seat.

The needle valve concept may be applied at the microscale as well. A high length-to-diameter ratio can be achieved by designing a needle valve in planar geometry, making it a special type of horizontally translating valve. It can also be

Figure 2.8: Macro-scale needle valve, using a tapered plunger (needle) to increase closure contact and controllability [34].

designed in out-of-plane geometry as shown in figure 2.2f, which may be better suited to low-leakage applications. Most MEMS fabrication processes however only offer limited design freedom in directions normal to the wafer surface, making it difficult to obtain a high length-to-diameter ratio with this approach.

2.3.7 Scaling valve array

The scaling array concept shown in figure 2.2g is not a valve design in itself, but can be used as an extension to all valve designs presented above. By fabricating an array of microvalves and opening and closing each valve separately, the total flow through the array can be controlled digitally. This allows the use of on/off bistable microvalve designs, or actuation schemes unsuited to proportional control, while maintaining the required flow control precision. The array can be constructed with all valves equal, by which the flow scales linearly with the number of open valves, or with exponentially scaling valve dimensions, which would allow binary control.

Applying the scaling array concept increases the total device footprint, but also increases the leakage flow considerably. For fully binary control with 1:10000 resolution, a total of 14 on/off valves would be required. Consequentially, the leakage demands would also be increased by a factor of 14.

2.4 Actuators

As stated in section 2.2, the type and specifications of the actuator have great influence over the performance of a microvalve. Because the choice of the optimal actuator and the design and operation of the microvalve are mutually dependent on each other, it is difficult to choose any one actuator type as the best. To allow a relatively objective comparison between microvalve actuators, this study focuses on the response time, for which the requirements have been defined in section 1.3, and the work density, which measures the maximum work an actuator of a certain volume can deliver.

Power consumption is also an important measure for actuator performance, but it can be greatly influenced by the operating mode of the microvalve. Therefore no explicit comparison will be made for this metric. So-called ‘stick and slip’ stepper actuators are not considered here, as they are extensions of more general actuator types. Typically, they offer increased stroke lengths at the expense of decreased actuation speed [35].

2.4.1 Thermal expansion actuators

Thermal expansion actuators are based on Joule heating of materials, usually applying bilayer strain mismatch or other forms of stroke amplification [36–38]. A more elaborate approach, known as thermopneumatic actuation, uses the expansion of a trapped working fluid [30, 39–41]. This gives more flexibility in the choice of membrane material, but in the case of a liquid working fluid the sealing of the fluid chamber often makes the fabrication process more complex. Typical settling times for thermopneumatically actuated microvalves in literature are on the order of hundreds of milliseconds to seconds [42], rendering them unusable for the flow control applications targeted here.

An actuation scheme very similar to thermopneumatics is using materials capable of shape-changing under thermal, chemical, optical or electrical stimuli. Recent examples are hydrogel [43–45], ionogel [46, 47] or electrochemical actuators [48]. Similar to thermopneumatic actuators, they suffer from very long response times on the order of seconds to minutes, which is too slow for mass flow control.

The work density of a thermal expansion actuator for an ideal elastic load has been derived in [49]. For a slim, prismatic beam of volume V at an average temperature difference ΔT_{avg} with the surroundings, the maximum work density ξ_{max} is:

$$\xi_{\text{max}} = \frac{F_{\text{bl}} \Delta l_{\text{max}}}{8V} = \frac{\alpha^2 E_y \Delta T_{\text{avg}}}{8}. \quad (2.1)$$

Where F_{block} is the blocking force at zero extension, Δl_{max} is the maximum stroke without load, α is the thermal expansion coefficient of the beam material and E_y is the Young’s modulus of the beam material.

Because thermal actuation is based on a temperature change, the speed of these actuators is limited to the heating and cooling of materials. The time constant for thermal expansion of a slim, uniform beam of length l_0 has been approximated in [38] to be:

$$\tau = \frac{l_0^2 C_p \rho}{\pi^2 \lambda}. \quad (2.2)$$

Where C_p is the specific heat capacity, ρ the density and λ the thermal conductivity

Table 2.1: Typical material properties of common materials in thermal expansion actuators, with their corresponding work density and response time [49].

	E_y [MPa]	α [K ⁻¹]	C_p [J kg ⁻¹ K ⁻¹]	ρ [kg m ⁻³]
Silicon	$1.6 \cdot 10^5$	$2.6 \cdot 10^{-6}$	700	2400
Polyimide	$2.5 \cdot 10^3$	$5.5 \cdot 10^{-5}$	2000	1420
	λ [W m ⁻¹ K ⁻¹]	ΔT_{avg} [K]	ξ_{max} [J m ⁻³]	τ [s]
Silicon	157	600	$4.9 \cdot 10^4$	$1.1 \cdot 10^{-3}$
Polyimide	0.16	250	$5.9 \cdot 10^4$	1.8

of the material.

Two common expander materials are (poly)silicon and polyimide. Table 2.1 shows the work densities and response times for 1 mm long beams of these two materials. Although polyimide requires a lower temperature to reach the same work density as polysilicon, and thus is more energy efficient, its low thermal conductivity makes it too slow for valve applications. The response time of polysilicon is within range of the demands listed in section 1.3.

2.4.2 Shape memory alloy actuators

Shape memory alloy (SMA) is a special type of thermal actuator that applies a specific material phase change which occurs in some metal alloys, such as NiTi, under influence of temperature. SMA actuators are known for offering higher work densities than any other MEMS actuator, on the order of 10^6 or 10^7 J m⁻³ [50, 51], and therefore have been applied in a wide range of micromechanical systems over the past decades [52, 53]. However, similar to many other thermal actuators, thermal phase change actuators suffer from long response times, ranging from tens of milliseconds to several seconds [42, 54, 55]. Another major disadvantage is that the performance of SMA actuators often significantly decreases after 10^4 or 10^5 cycles [56, 57], while proportional controllers require multiple orders more actuator cycles during their lifespans.

2.4.3 Electrostatic actuators

Electrostatic actuators are commonly used in MEMS because they are relatively easy to integrate in silicon fabrication processes. They offer very high speeds if

resistance and capacitance are kept low, and low power usage in steady state. However, the strongly nonlinear relation between electrode separation and electrostatic force leads to the so-called ‘pull-in’ effect, which makes direct electrostatic actuation unsuitable for proportional control. A (partial) solution to this problem can be achieved by making patterns of electrodes that are actuated in sequence to create step-by-step attraction between the two surfaces, or to make bending or ‘zipper-like’ electrodes that counter the pull-in force with an opposite bending force [58].

Another solution to the pull-in effect of electrostatic actuation is the laterally driven comb drive, which uses the fringing field between interdigitated electrodes to generate work [59, 60]. It supports larger strokes than gap-closing designs (on the order of 100 μm), but can deliver only very small forces ($< 1 \text{ mN}$).

The maximum energy that a lateral comb drive transfers to an elastic suspension has been derived to be [49]:

$$W = \frac{\epsilon n h U^2 s}{2 g_f}. \quad (2.3)$$

Where n is the number of finger pairs, ϵ is the dielectric constant of the gap between the fingers, g_f is the gap spacing, h is the height of the comb fingers, U is the voltage across the gap and s is the stroke of the actuator, which in the ideal case equals the finger length l_f (see figure 2.9). If the gap width g_f is limited by the spatial resolution of the fabrication process, the minimum thickness of the fingers t_f will be equal to g_f . If the minimum width of the comb drive equals two times the finger length l_f , the minimum volume of the comb drive then becomes:

$$V_{cd} = 2 l_f h \cdot n (t_f + g_f) = 4 n g_f l_f h. \quad (2.4)$$

The maximum work density then equals:

$$\xi_{\max} = \frac{W}{V_{cd}} = \frac{\epsilon U^2 s}{4 g_f^2 l_f} = \frac{\epsilon E_e^2}{4}. \quad (2.5)$$

Where E_e is the electrical field in the gap. Assuming an electrical field of 100 MV m^{-1} , the maximum work density for a lateral comb drive in air becomes $2.2 \cdot 10^4 \text{ J m}^{-3}$. Considering that an actual comb drive will have a larger volume than the minimum derived above, this value will be lower in practice. Comb drives can typically be driven at frequencies on the order of 10^4 Hz or higher [59], meaning that the response time is well below 1 ms.

Figure 2.9: Definitions of structural dimensions in a lateral comb drive, with motion along the x -axis.

2.4.4 Electromagnetic actuators

Electromagnetic actuators use the net Lorentz force on a current-carrying wire in a magnetic field. In the micro domain the current density can be high, because the heat generated due to resistivity in relation to the heat transport is low. The current density is not limited by the temperature rise of the coil, as in macro-scale Lorentz actuators, but by electromigration. Because of this limit, the current density J can remain constant when scaling down the device [49]:

$$J \sim r^0. \quad (2.6)$$

Where r represents the scaling factor, defined as the length scale of typical dimensions in a device. The deliverable work density of a Lorentz actuator can be described as:

$$\xi = \frac{F_L s}{V_{cm}} = J B_{avg} s \sim r^1. \quad (2.7)$$

Where F_L is the Lorentz force, s is the total stroke, V_{cm} is the coil volume opposing the magnet and B_{avg} is the average flux density in the coil. Therefore, the work density scales linearly with reducing dimensions, meaning the performance decreases significantly at higher levels of integration. The work density for a realizable Lorentz actuator design measuring $700 \times 500 \mu\text{m}^2$ has been calculated in [49] to be approximately 150 J m^{-3} .

An advantage of electromagnetic actuators is that they can be placed outside of the valve chamber, which allows easier interfacing and avoids electrical fields in the controlled fluid. This approach commonly applies large external coils, with a moving member attached to the valve mechanically [61–65], or with the magnetic field coupling to magnetic materials inside the valve [11, 66, 67]. A micromachined design applying this concept is demonstrated in [66], using NiFe flux guides to couple the magnetic field to a NiFe membrane in the valve chamber. However, the highly limited work density leads to very poor actuator performance

in small-scale designs. An integrated, planar electromagnetic coil design for use in a microvalve was reported in [68], which has a diameter of approximately 1 mm and was reported to generate an actuation force of 0.8 mN. For a total valve area of 1 mm², this actuator would be capable of withstanding only 8 mbar of differential pressure. A recent design could deliver up to 80 nN of normal force at a valve radius of 21 μm, corresponding to 0.6 mbar of pressure [69].

2.4.5 Piezoelectric actuators

Piezoelectric actuators use the strain of piezoelectric materials that is induced when they are submitted to an electric field. This strain is limited to approximately 0.1 % of the original size, but it can be delivered with very large forces on the order of kilonewtons. The limited stroke is commonly amplified using bimorph cantilevers or a leverage mechanism, or by creating stacks of actuators. Piezo actuators offer high speeds and very low power consumption in the steady state [70], but typically require high voltages on the order of 100 V and tend to show severe hysteresis in their electromechanical behavior. They also often contain hazardous substances, such as lead in Pb[Zr_xTi_{1-x}]O₃ (PZT), meaning the actuator must remain separated from the fluid in many applications.

For a beam of piezoelectric material with length l_0 and strain coefficient d_{31} , the maximum work density using an ideal elastic load can be derived to be [49]:

$$\xi_{\max} = \frac{F_{\text{bl}} \Delta l_{\max}}{8 A l_0} = \frac{d_{31}^2 E_{3,\max}^2 c_{11}}{8}. \quad (2.8)$$

Where F_{block} is the blocking force, $\Delta l_{\max}$ is the maximum stroke, $E_{3,\max}$ is the maximum electrical field between the electrodes, A is the surface area of the beam cross-section and c_{11} is the ratio of longitudinal stress to longitudinal strain. For PZT, currently the best performing piezoelectric material, typically $c_{11} = 150 \cdot 10^9 \text{ N m}^{-2}$, $d_{31} = 100 \cdot 10^{-12}$ and $E_{3,\max} = 40 \cdot 10^6 \text{ V m}^{-1}$. This results in a maximum work density of $3 \cdot 10^5 \text{ J m}^{-3}$. Commercially available stack piezos smaller than $10 \text{ mm}^3 \times 10 \text{ mm}^3 \times 10 \text{ mm}^3$ typically offer actuation forces on the order of hundreds of Newtons with strokes up to 10 μm, corresponding to work densities on the order of tens of kJ m^{-3} .

Micromachining of thin-film piezoelectrics can be accomplished in silicon technology [71–73]. This is of interest to large-scale integration applications, but with layer thicknesses on the order of micrometers such actuators will not have enough volume to offer large work. Furthermore, the integration of piezoelectric films in the microfabrication process can significantly reduce the thermal budget available for further processing steps [74]. For this reason most piezoelectric

Table 2.2: Summary of the valve concepts described in section 2.3.

Flow control			Leakage performance		Design freedom	
Translating plate		○		○	+	Many designs reported
Translating mem- brane		○	+	Matches seat topography, may be permeable to gases	–	Limited to thin or elastic ma- terials
Tilting plate	+	Large stroke: ΔR_{flow} ratio		○		○
Bending plate	+	(Assuming sufficient actua- tor resolution)	–	Pliable materials susceptible to buckling	–	Limited to specific (actuat- ing) materials
Sliding plate	++	Plate position determines R_{flow} directly	—	Tradeoff between friction and closure		○
Needle	++	Very large stroke: ΔR_{flow} ra- tio	○	Large contact surface, but needs high conformity		○
Scaling array	++	Resolution scales with num- ber of sub-valves	—	Demands scale with number of sub-valves		○

Table 2.3: Summary of the actuator types described in section 2.4.

	Work density [J m^{-3}]		Response time		Integration
Thermal expansion	$< 10^5$	○	Strongly material-dependent	+	Standard materials supported
Shape memory alloy	$< 10^7$	—	$> 10 \text{ ms}$ - 10 s		○
Electrostatic	$< 10^4$	+	$< 1 \text{ ms}$	+	Standard materials supported
Electromagnetic	$< 10^3$ at microscale	○	Limited by drive current electronics		○
Piezoelectric	$< 10^5$	+	$< 1 \text{ ms}$	–	Volume limitations, low thermal budget

actuators applied in fluid control are bulk stacked piezos [75–81].

2.5 Conclusions

In this chapter we have described design considerations for a micromachined proportional control valve for very small flows. We have surveyed a number of basic microvalve design concepts and analyzed their suitability for use in the applications described in chapter 1. A valve's capability to accurately control flow is deemed the most important specification, but the leakage performance is also given high weight as good closure is difficult to obtain at the micro scale. The results of this analysis are summarized in table 2.2.

Precise flow control can best be achieved by using a horizontally translating (sliding) plate design or a needle valve, although the limited design freedom may lead to complex fabrication processes. Furthermore, the first solution suffers from severe leakage, whereas the latter poses high demands on the relative roughness and flatness of the needle and the valve seat. An alternative is the tilting plate design, which can be made to have the same advantage as the needle valve.

The best design freedom is available for vertically translating plate designs. Although it does not offer inherent advantages for precise control or low leakage, it allows for a wide range of materials, shapes and fabrication methods. For this reason the translating plate design is the most commonly applied type in microvalves. Particularly when straight-forward fabrication processes are required, the wide design freedom makes the vertically translating plate concept the most interesting.

Good closure performance can be obtained from using soft, elastic materials, capable of shaping themselves to match the valve seat surface and of embedding contaminants inside the microvalve assembly. They do however suffer from increased valve stiction, reduced chemical resistance and increased permeability for gases, which often makes the use of harder, more resistant materials preferable. For hard materials, leak flow may be reduced by obtaining a high conformity between seat and plate.

Proportional control of fluids requires an actuator, and only a few types of actuator are capable of fulfilling the requirements outlined in section 1.3. The properties of the discussed actuators are summarized in table 2.3. Thermal expansion and shape memory alloy actuators are used in many designs, but cannot offer high enough speeds for high bandwidth control applications. Electrostatic actuators are very fast, but the pull-in effect in gap-closing designs makes them poor control actuators. The lateral comb drive circumvents this problem, but while

it can be designed for relatively large strokes, it can only deliver very small forces.

Micromachined electromagnetic and piezoelectric actuators suffer from severely decreased performance with respect to their macro-scale counterparts: The work density of electromagnetic actuators decreases with increasing level of integration, while micromachined piezoelectrics are very limited in layer thicknesses. This problem is commonly avoided by using macroscopic scale components, although this is disadvantageous for wafer-scale fabrication as assembly of fine machined devices can not easily be automated.

Given its high work density, high speed and low power usage, piezoelectric actuation is considered the best candidate for the aimed proportional control valves, but unless the integration compatibility is significantly improved it will remain challenging to obtain the required performance at a manageable level of complexity. The second main disadvantage in piezoelectric actuation is the inherent hysteresis effects, which can make it challenging to obtain a high control resolution.

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3

SOI-based microvalve with capacitive displacement sensor

3.1 Introduction

Cardiovascular disease and hypertension are partners in a vicious circle leading to death. With cardiovascular disease being killer number one in western society [1], it is key to have screening methods in place to identify its early onset and break the vicious circle. Predisposition to hypertension expresses itself in the arterial blood pressure waveform. This expression is not only limited to the maximum and minimum of the waveform, but also to its morphology. Non-invasive, real-time monitoring of the blood pressure waveform (BPW) can therefore be used for screening for cardiovascular disease.

A proven technology for non-invasive BPW monitoring applies a finger cuff which is pressurized to keep the finger in a continuous state of vascular unloading. A photo-plethysmograph is used to measure the total volume of blood in the finger by infrared absorbance. This measurement is used to control the gas pressure inside the cuff to keep the volume of the unloaded artery constant (see figure 3.1) [2]. By keeping the cuff pressure equal to the arterial pressure, the brachial

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Figure 3.1: Application of the microvalve in a portable blood pressure sensing device. Using a piezoelectric bimorph actuator, the microvalve controls air flow into the finger sleeve to keep the photocurrent equal to the reference [2]. The capacitive measurement is used as a low-latency feedback signal.

artery blood pressure can be calculated. This approach has been successfully commercialized by Finapres Medical Systems, but current solutions for ambulant applications remain bulky and further downscaling is limited by the flow control system.

As part of ongoing efforts to create an ambulant, non-invasive BPW monitoring system, work has been done on the realization of a micro control valve for a miniaturized gas flow control system. Apart from small dimensions ($< 5\text{ mm} \times 5\text{ mm} \times 5\text{ mm}$) and proportional control of gas flow at rates on the order of 15 g h^{-1} for moderate pressures, this microvalve should facilitate high-bandwidth control to closely follow the BPW.

A large number of micro control valves have been reported in literature in the past, but only a small portion of them are suitable for proportional control [3–5]. Relatively simple designs suitable for low-cost fabrication commonly apply easily moldable materials such as PDMS [6–10], which suffer from inherently short lifespans due to their pliability. More robust designs are typically realized in silicon or silicon-derivative materials, but often require complex fabrication processes [11–13]. A piezoelectrically actuated microvalve has recently been presented with a relatively straight-forward fabrication scheme consisting of two patterned wafers that are eutectically bonded together [14]. A design for a passive check valve has been reported that was realized in a single silicon wafer using only two mask steps in its fabrication process, but this design cannot be used to control flow to a specific value [15].

Other key limitations in many reported control valve assemblies include actuator hysteresis and high settling times, which can limit control precision and bandwidth [4, 6, 16–18]. The two most common actuator types that allow high-speed proportional control are electromagnetic and piezoelectric actuation. Electromagnetic actuation suffers from decreasing work densities at smaller

scales, which is why piezoelectric actuation is commonly applied in downscaling scenarios (see chapter 2). Piezoelectric actuation however typically suffers from significant hysteresis.

Capacitive displacement sensing is commonly applied in positioning systems to correct for hysteresis and improve control precision [19]. This can be applied to microvalve control as well: Provided the relation between displacement and gas flow is known, low-latency capacitive displacement sensing can be used as a feedback signal for position-based valve control. Capacitive sensing has also been applied for on-chip gas pressure sensors [4, 20, 21] and for measuring actuation fluid pressure in thermopneumatic valve designs [22]. Such capacitive microvalves however tend to require relatively complex fabrication processes.

This chapter describes a MEMS-based microvalve with capacitive displacement sensing for proportional control of gas flow, using only a single silicon wafer in its fabrication scheme. The design and fabrication process are described in detail, and the fluidic and mechanical properties of the design are derived analytically and verified experimentally. We also demonstrate the assembly of a miniaturized piezoelectric actuator with the microvalve chip. It is shown that the capacitive displacement sensor in combination with the assembled actuator makes the valve well suited for proportional control of gas flow.

3.2 Analysis

3.2.1 Design and operating principle

In the analysis of different design schemes in section 2.3 it was found that linear flow control can best be achieved by using a sliding plate design (see figure 2.2b), but problems like stiction and large leak flow make this approach less likely to result in robust, reliable solutions. In this work a translating plate design is chosen as it allows very large design freedom to simplify fabrication, and more convenient assembly of a miniaturized actuator.

The realized valve design is shown schematically in figure 3.2. It consists of a rigid, circular valve plate, suspended above a circular flow inlet by a number of flexing beams. The plate is free to move up and down with regards to the valve seat, with an initial separation defined by the separating layer. Movement of the valve is analyzed using finite element (FEM) simulations in COMSOL Multiphysics® v.4.3a using the structural mechanics toolbox, selecting poly-silicon as the material to approximate single-crystal silicon. Figure 3.3 shows that the inner ring and beams move closely together with the central plate, with the outer beams providing most of the displacement. The design is optimized to allow a maximum bending

Figure 3.2: (Top) Cross-sectional schematic view of the valve design. The blue dashed arrow indicates gas flow, valve movement is shown in red. (Bottom) Electrical model of the capacitive microvalve, consisting variable and constant capacitances connected in parallel.

stress of approximately 80 MPa, considerably lower than the breaking stress of single-crystalline silicon (> 1 GPa) [23]. Gas flow is indicated by the dashed arrow in figure 3.2, flowing from the inlet, through the radial flow channel between valve plate and valve seat, and out at the top of the structure. The flow can be controlled by varying the separation between plate and seat.

3.2.2 Laminar flow model of the radial channel

For laminar, incompressible and fully developed flow, the volume flow rate Q_{vol} through a radial channel between circular plates is proportional to the cube of the separation S [24]:

$$Q_{vol} = \frac{\pi \cdot S^3}{6\mu} \cdot \frac{P_1 - P_2}{\ln\left(\frac{R_2}{R_1}\right)}. \quad (3.1)$$

Where $R_1 = 175 \mu\text{m}$ and $R_2 = 275 \mu\text{m}$ are the inlet and plate radii (see figure 3.2), μ is the dynamic viscosity of the gas and P_1 and P_2 are the inlet and outlet pressure, respectively. We can approximate the compressible gas flow based on this formula when assuming an average density across the entire flow channel. A more elaborate

Figure 3.3: Mechanical FEM simulation of the microvalve design, showing that most of the displacement is provided by the outer beams, while the inner beams move together with the plate. Displacement is not to scale. The surface color maps the internal von Mises stress for a displacement of $10\ \mu\text{m}$.

model of compressible radial flow based on empirical observations can be found in [25]. Assuming that the density ρ scales linearly with pressure, the mass flow can be approximated by:

$$Q_{\text{mass}} = Q_{\text{vol}} \cdot \frac{P_1 + P_2}{2P_2} \rho(P_2). \quad (3.2)$$

Note that equation 3.2 is valid for viscous flow ($Kn < 0.01$), which is valid at 1 bar pressure and $4\ \mu\text{m}$ separation. At lower pressures and separations however, the Knudsen number increases (e.g. $Kn = 0.07$ at 200 mbar and $1\ \mu\text{m}$) and flow enters the transition regime. In this case the analytical model gives an overestimation of the actual flow.

Equations 3.1 and 3.2 are verified using FEM simulations using a weakly compressible, laminar flow model with a no-slip condition for the channel walls. Mass flow is calculated in COMSOL by integrating the product of the flow velocity field and the local gas density over the entire channel volume. Figure 3.4 shows the results of the analytical and numerical models for nitrogen gas at $20\ ^\circ\text{C}$ flowing from 800, 500, 300 and 200 mbar overpressure to atmosphere. Modeling only the radial flow channel with a laminar in- and outflow, the numerical results match equation 3.2 with less than 1.6 % deviation. Adding a flow inlet and outlet in series with the radial channel reveals a decrease in mass flow at higher flow velocities. The flow velocity distribution in figure 3.5 shows that this is caused by added flow resistance at the entrance and exit of the channel, as illustrated by the acceleration of the gas just outside of the channel.

The validity of the compressible laminar flow module in COMSOL is verified by the Mach number remaining low (< 0.3). Laminar flow conditions are also valid as the Reynolds number is below unity in all cases. Based on equation 3.2 at

Figure 3.4: Analysis of the expected nitrogen gas flow through the microvalve at various pressures. The solid line indicates the analytical solution, the single points indicate FEM simulations in COMSOL Multiphysics® v.4.3a of the channel (hollow points) and the channel with inlet and outlet (filled points).

Figure 3.5: Flow velocity distribution through the radial channel and (part of) the inlet and outlet. Pressure is 1 bar, separation is $4\mu\text{m}$. Radial symmetry has been used (rotational axis to the left of the shown area). Flow acceleration near the inlet and outlet indicate that the flow resistance is increasing near the channel.

1 bar of pressure, the requirement of 15 g h^{-1} gas flow is reached at a separation of approximately $7\text{--}7.5\mu\text{m}$. Depending on the initial separation between plate and seat, this may require an actuator capable of both pushing and pulling the plate. At a more moderate pressure of 200 mbar the required separation increases to $13\text{--}14\mu\text{m}$.

Note that across the applied pressure range ($< 1\text{ bar}$), changes in the viscosity of nitrogen gas can be neglected [26].

3.2.3 Electrical model of the capacitive sensor

Given electrically conducting material for both the valve plate and the valve seat, the electrical capacitance between the plates can be measured to sense the total valve separation. In a first-order approximation the capacitance will have an inverse linear relation to the separation, as can be seen from a simple parallel plate capacitor model:

$$C = \frac{\epsilon_0 \epsilon_r A}{S}. \quad (3.3)$$

Here A is the surface area of the parallel plates, S is the separation, ϵ_0 is the vacuum permittivity and ϵ_r the dielectric constant of the medium between the plates. This means that the capacitive sensor becomes increasingly sensitive as the valve is closing, which is advantageous for controlling small-scale flows.

A more precise model takes into account the variable capacitances existing between the suspension beams and the seat surface. Given that the inner beams move down together with the central plate (see figure 3.3), we can model their capacitance as an extra surface area at separation S . The capacitance of the outer beams, bending down from the initial separation $S_0 = 4\mu\text{m}$ to the actual separation S , can be calculated by integrating the position-dependent separation over the length of the beam:

$$C_b(S) = \epsilon_0 \epsilon_r W_b \int_{l=0}^{l=L_b} \frac{1}{s(l)} dl. \quad (3.4)$$

Where W_b and L_b are the width and length of the beam, respectively, and $s(l)$ is the separation which changes linearly from S to S_0 along the length of the beam. The model is completed by adding a constant parasitic capacitance C_{par} that exists in the unmoving silicon parts. Using $\epsilon_r = 1.0$ for the surfaces separated by air or nitrogen gas, this gives:

$$C(S) = \frac{\epsilon_0 (R_2^2 - R_1^2) + A_{\text{inner}}}{S} + 4C_b + C_{\text{par}}. \quad (3.5)$$

Here A_{inner} is the surface area of the inner beams.

Figure 3.6 plots the modeled capacitance as a function of separation S . It also plots the inverse of $C(S) - C_{\text{par}}$ which compensates for the last term in equation 3.5. It is visible that after removing the parasitic terms, the inverse of the capacitance scales nearly linearly with the separation. With this model, a total capacitance of between 31 pF and 24 pF is expected for valve separations between from $0.5\mu\text{m}$ to $4\mu\text{m}$, respectively. A value of 23 pF is obtained for the expected parasitic capacitance C_{par} .

Figure 3.6: Capacitance model (blue curve) and linearized model compensating for parasitic capacitances (inverse capacitance, red curve) as a function of valve separation.

3.3 Fabrication process

We choose silicon as the base material for our control valve, as it offers great long-term stability and it can be manipulated and shaped with high parallelism using well-matured fabrication technologies. As has been demonstrated for a passive check valve in the past, the use of a silicon-on-insulator (SOI) wafer allows for relatively straight-forward fabrication of three-dimensional structures [15]. Two layers of single-crystalline silicon are dry-etched to obtain the movable valve plate and gas inlet, and a buried silicon oxide layer in between them is selectively etched away to release the moving parts and open the flow channel. An alternative approach for making free-hanging structures is to use anisotropic etching in KOH to release structures in a $\langle 111 \rangle$ -oriented silicon wafer, but this requires extra steps to selectively protect the silicon structures from being etched as well [27]. Also the use of a single conducting wafer would make capacitive readout more difficult. Note that both fabrication methods result in a valve that is normally open.

To enable capacitive measurements, the SOI wafers used in this research are highly doped with a resistivity of $0.005\text{--}20\Omega\text{ cm}$ for both the handle layer ($400\mu\text{m}$ thick $\langle 100 \rangle$ -silicon) and the device layer ($50\mu\text{m}$ thick $\langle 100 \rangle$ -silicon). The buried oxide layer is chosen to be $4\mu\text{m}$ thick. Designing the suspending beams to allow a displacement of $\pm 4\mu\text{m}$, a total separation of at least $8\mu\text{m}$ becomes possible when using a bidirectional actuator. Figure 3.7 shows a schematic process flow of the fabrication steps. The handle layer is first etched with an Alcatel AMS100 deep

Figure 3.7: Fabrication is done in a SOI wafer (a) using deep reactive ion etching (b-c) and vapor-phase (d) and liquid-phase (e) HF etching.

reactive ion etcher (DRIE) using a Bosch process, to create the gas inlet and to etch breaking grooves between the valve chips. Then in a similar DRIE process the valve plate and suspension are etched in the device layer. Bond pads for electrical connections are also defined in this step. Figure 3.8 pictures a SEM micrograph of part of the flexing beam structure of the valves. The etching process results in straight, smooth sidewalls showing that the beams are shaped in a well-defined manner.

After DRIE the individual valve chips are broken free from the wafer along the etched breaking grooves and cleaned to remove particles and contamination. The microvalves are then released using HF etching of the buried oxide. To avoid stiction by capillary force during wafer drying, the oxide etch step is separated into firstly a liquid phase 50% HF etch, carefully timed to leave a small amount of oxide separating the valve plate and valve seat, and secondly a vapor-phase HF etch to remove the remaining oxide. Given a good control over the etching speeds during DRIE and HF etching, this fabrication process is very robust and near-perfect yields can be achieved. The realized microvalve chip, shown to the right in figure 3.8, is 7.5 mm in diameter and 0.5 mm thick, which is suitable for ambulant applications but can be further miniaturized if so required.

Note that the use of silicon means that after processing, all surfaces will be covered in a thin native oxide. This is advantageous, as it helps prevent the capacitive sensor from short-circuiting when the plate is pushed down.

Figure 3.8: SEM closeups (top left and right) and full photo (bottom) of the realized valve chip.

3.4 Characterization

3.4.1 Measurement setup

Figure 3.9 shows the integrated test setup used to characterize the microvalve. A PI P-603.3S2 piezoelectric actuator is controlled in feedback with an integrated strain gauge displacement sensor. A PC software program controls this external actuator to change the valve plate displacement. The force applied to the microvalve is measured with a Futek LSM250 loadcell at 10.00 V supply voltage, and is transferred to the valve plate through a stiff glass needle.

Figure 3.9 also shows the mechanical model of this setup. The feedback-controlled actuator enforces a displacement d_{piezo} . The loadcell is modeled as a simple spring with a stiffness K_{loadcell} , which is assumed constant in its measurement range of ± 1.1 N. K_{loadcell} is measured to be 4.52 kN. Note that since the metal frame and the stage of the setup have a finite stiffness, this result comprises both the compliance of the loadcell and that of the setup. With this, the displacement of the microvalve can be calculated as:

$$d_{\text{valve}} = d_{\text{piezo}} - F_{\text{loadcell}}/K_{\text{loadcell}}. \quad (3.6)$$

For fluidic measurements a Bronkhorst P-602CV pressure controller is used to control a dry nitrogen gas flow through the valve, which is subsequently measured

Figure 3.9: (Left) Photograph of the test setup used for mechanical characterization. A piezoelectric actuator enforces a displacement, which is transferred through a loadcell and a glass needle to the valve, mounted on an x, y, z stage. (Right) Mechanical model of the setup.

Figure 3.10: Measurement scheme used for flow characterization of the valve.

using a Bronkhorst F-111B gas mass flow meter with a maximum flow range of 1.97 g h^{-1} (see figure 3.10). Electrical capacitance is measured using an HP 4194a impedance analyzer at 100 kHz.

3.4.2 Flow properties

A detailed characterization of the fluidic behavior is required to reliably use the microvalve's capacitive displacement sensor in a control loop. Using the setup described above we have measured the gas flow as a function of valve separation at

Figure 3.11: Measured mass flow of dry nitrogen gas at various differential pressures (points) compared to their numerical flow models (solid lines).

various differential pressures. All measurement points are taken after stabilization of the separation and/or pressure (several seconds), and then averaged over five seconds of flow. The standard deviations of these averages are less than 0.25 % of full scale. Analysis of these measurements revealed that the maximum downward stroke equaled $3.5\ \mu\text{m}$, rather than the expected $4\ \mu\text{m}$ which is the thickness of the buried oxide. This means the valve did not close further than $0.5\ \mu\text{m}$ separation, as is visible in figure 3.11 showing the measured flow as a function of separation. At the maximum separation ($4.0\ \mu\text{m}$), the flow exceeds the measurement range of the flow sensor ($1.97\ \text{g h}^{-1}$) at a pressure of 900 mbar.

The measured flow is somewhat lower than the predicted values. This can be partially attributed to the pressure drop over the flow meter ($35\ \text{mbar}$ at $1.5\ \text{g h}^{-1}$) which lowers the effective valve inlet pressure. As mentioned in section 3.2.1 the analytical models also assumed viscous flow, which would overestimate the flow for our valve in the transition regime.

The most likely cause of the minimum separation of $0.5\ \mu\text{m}$ is there being contamination or particles in between the valve plate and the valve seat, preventing the valve from closing further. This means special care needs to be taken to prevent such contamination in applications where a low leakage flow is required. In the envisioned application a constant flow is required to keep the finger cuff pressurized, so small leakage flows ($< 0.01\ \text{g h}^{-1}$ at $\leq 1\ \text{bar}$ pressure) are not a concern.

Keeping the separation constant and sweeping the gas pressure results in the flow profiles shown in figure 3.12. Note that, as the pressure is increased,

Figure 3.12: Measured mass flow of dry nitrogen gas as a function of pressure, at constant separation. Pressure acting on the valve plate is countered using capacitive readout in feedback with the actuator.

Figure 3.13: Inverse of the measured capacitance (squares) and the analytical model (solid line) as a function of separation.

a change in valve plate position is expected due to the pressure acting on the valve plate. To avoid this influencing the characterization process, the built-in capacitive displacement sensing is used to keep the valve separation constant: For each separation value the capacitance is measured at zero pressure, and as the pressure is ramped up this capacitance is kept constant using the piezoelectric actuator to correct the valve position.

3.4.3 Capacitive sensor

We have investigated the performance of the capacitive displacement sensor in the realized microvalve chip. The total measured capacitance lies in the range of 28–44 pF for separations ranging from 0.5 to 4 μm . Figure 3.13 plots the inverse of the measured capacitance as a function of separation, with the constant parasitic capacitance C_{par} subtracted from the measurement values.

Fitting the model to the measurements results in a parasitic capacitance of $C_{\text{par}} = 27.5 \text{ pF}$. This agrees well with the 23 pF calculated from the model, as an increase in parasitic capacitance is expected from wiring and interconnects. At low separations the measurements show a larger capacitance than predicted by the analytical model, especially when approaching the minimum separation of 0.5 μm . Most likely the contamination or particles that limit the separation also cause this increase in capacitance due to the higher dielectric constant compared to nitrogen gas.

3.4.4 Actuation

To investigate the use of the capacitive sensor in a practical system with integrated actuator, we designed and 3D-printed a chipholder to mount a Noliac CMBR02 bimorph ring bender piezo together with the microvalve (see figure 3.14). Figure 3.15 shows the measured flow at various constant gas pressures, while first closing and then opening the valve. The flow is measured as a function of the voltage applied to the piezoelectric bimorph, and as a function of inverse capacitance. Due to limitations in the setup the bimorph is used in single side voltage control, meaning it can close and reopen the valve entirely, but the valve plate cannot be pulled. The plate-seat separation is therefore limited to 4 μm . Note that an increasing voltage means the valve is closing.

The results show a < 300% difference between opening and closing flow when controlling the valve through the piezo voltage. However, this large hysteresis is compensated almost entirely when using the capacitive displacement sensor, with a maximum measured hysteresis of < 5%. This can be an advantage in proportional control applications, as the system could use high-speed capacitive readout instead of having to wait for a flow sensor to be able to correct for actuator nonlinearities. Note that the integration of the bimorph piezo can somewhat change the shape of the flow path in the valve, for example due to alignment errors during glueing. For this reason the capacitance versus displacement characteristic of figure 3.13 may not apply to the microvalve with integrated piezo. It is however sufficient to characterize the capacitance versus flow behavior to allow closed-loop control of the active valve.

Figure 3.14: Schematic cross-sectional drawing (top) and photograph (bottom) of the assembled flow controller, consisting of a chipholder, the microvalve and a piezoelectric bimorph actuator. The electrical wires connect to the actuator and the capacitive sensor.

Figure 3.15: Dry nitrogen gas flow as a function of piezo voltage (left) and of inverse capacitance (right). Influence of hysteresis of the piezoelectric actuator is largely eliminated when using capacitive displacement control.

3.5 Conclusion

A micromachined control valve with straight-forward fabrication for use in ambulatory blood pressure waveform measurements has been designed and realized. The valve contains a built-in capacitive displacement sensor, which enables high-speed monitoring of the valve separation for increased control precision.

Flow behavior of the valve has been modeled analytically and numerically, with the numerical models including influence of the inlet and outlet of the valve. Measurements have revealed a minimum separation of $0.5\text{ }\mu\text{m}$, likely due to contamination between the valve plate and seat. This leads to a leak flow of $< 0.01\text{ gh}^{-1}$ at pressures up to 1 bar. A total mass flow of $> 1.97\text{ gh}^{-1}$ of dry nitrogen gas is measured at 900 mbar pressure and $4.0\text{ }\mu\text{m}$ separation. A good match is found between the theoretical models and the practical valve behavior across the entire measured range.

Capacitive behavior has been analyzed and characterized, and a near-linear relation between valve separation and inverse capacitance has been found. Upon integration of a piezoelectric bimorph actuator, we have also demonstrated that actuator hysteresis can be corrected from 300 % to less than 5 % using capacitance-based valve control. With these results, the potential of using the microvalve for proportional control of gas flow has been demonstrated.

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4

Integration of a piezoelectric actuator

4.1 Introduction

The microvalve design described in chapter 3 enables proportional control of gas flows up to at least 2 g h^{-1} , has integrated capacitive displacement sensing, and uses only a single-wafer, two-mask fabrication process. While the straight-forward fabrication is a major advantage, the design requires further improvements to be useful for the aimed applications. Although the flow range is suitable for the application in gas chromatography systems, the blood pressure waveform monitoring system requires significantly higher flow rates. For this, an actuator needs to be integrated on the valve that is capable of lifting the valve plate above its resting separation. The applications also demand response times on the order of milliseconds, which has not yet been proven for the described valve design. Additionally, the design lacks a fluidic outlet port, only controlling flow from a fluidic inlet, through the microfluidic chip to atmosphere.

The integration of an actuator is not always considered in the design of proportional control valves reported in literature. Unfortunately most of the devices that do are still relatively large, with particularly the actuator requiring larger volumes. Device dimensions are often on the order of centimeters, mostly

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either because of the downscaling limitations of electromagnetic actuators or because stacked piezos are used which have a very small stroke per unit length [1–9].

Although small-volume microvalves with response times on the order of milliseconds have been reported in literature [3, 10, 11], they often suffer from complex fabrication processes, poor control precision or hysteresis. A relatively simple design using a stacked piezo actuator was fabricated in a two-wafer fabrication process, but still required seven lithographic mask steps and wafer bonding [12]. The microvalve presented in chapter 3 is realized in a single-wafer fabrication process, and can compensate for hysteresis with its capacitive displacement sensor. However, the piezoelectric actuator is mounted on the microvalve using a 3D-printed support structure that requires high-precision manual assembly. This makes it unsuitable for large scale production and increases the device dimensions to centimeter scale. In this chapter we therefore present an improved design of this valve, controlling flow between an inlet and outlet. We achieve a high level of integration by replacing the 3D-printed support structure with a miniature bimorph piezoelectric actuator that can be mounted directly onto the fluidic chip. In our experiments it is found that direct adhesive bonding of the piezo bimorph to the silicon causes a severe reduction in the piezo displacement. This problem is analyzed using finite element modeling and a solution is found and implemented using a support ring of flexible material between the piezo and the silicon. The realized micro control valve is validated with fluidic and mechanical characterization measurements.

4.2 Analysis

4.2.1 Design and operating principle

Figure 4.1 shows the basic design of the microvalve, based on the design described in chapter 3. It consists of a circular valve plate suspended above the valve seat by a closed, flexible membrane, such that a radial flow channel exists between the valve plate and the valve seat. The height of the radial channel changes by vertical movement of the plate, changing the flow resistance of the valve. An electrical capacitance C_{valve} exists between the plate and the seat, bearing an inverse relationship with their separation S :

$$C_{\text{valve}} \approx \frac{\epsilon_0 \epsilon_r A}{S} + C_{\text{par}}. \quad (4.1)$$

Figure 4.1: Artist impression and operating principle of the SOI-based microvalve. Flow is regulated by vertical translation of the valve plate with regards to the valve seat. The electrical contact points allow capacitive displacement sensing of the valve membrane. Gas flow is indicated by the red arrow.

Where ϵ_0 is the vacuum permittivity, ϵ_r is the dielectric constant of the medium between the plate and the seat, A is the surface area of the valve membrane and C_{par} is the constant, parasitic capacitance arising from the immovable parts of the valve membrane. Since capacitance can be measured without waiting for flow stabilization, this makes it possible to use the capacitance as a high-speed control signal for position based valve control. At the same time the capacitive sensor can be used to compensate for hysteresis in the actuator.

Like the previously reported valve design, the valve is realized in a silicon-on-insulator (SOI) wafer. Apart from simplifying the fabrication process, this has the advantage that the valve plate and seat both have a low surface roughness, which is beneficial for the leakage performance when the valve is closed. The valve plate is formed in the SOI device layer and the inlet and outlets are shaped in the handle layer. The buried oxide (BOX) between the valve plate and valve seat is removed as a sacrificial layer, leaving the valve plate free to move up and down.

In contrast to the previous design, both the valve inlet and outlets are on the

bottom side of the chip. This means that extra care must be taken to prevent leakage paths from inlet to outlet during interfacing of the fluidic chip. However, the distance between inlet and outlet channels can not be made arbitrarily large, as this would increase the etching time of the BOX layer and increase the valve's dead volume (see figure 4.1b). To allow easier assembly of the microvalve chip to a fluidic chipholder, a glass support wafer is added that increases the inlet to outlet spacing. Note that this wafer is not required for correct operation of the valve and may be replaced with other fluidic components, depending on the application.

4.2.2 Valve dimensions

The dimensions noted in figure 4.1b are summarized in table 4.1. The valve membrane diameter is $w_{\text{membrane}} = 4.6 \text{ mm}$, with the valve plate in the center defined as $w_{\text{plate}} = 800 \mu\text{m}$. The circular flow inlet is situated beneath the valve plate, with a diameter of $w_{\text{in}} = 600 \mu\text{m}$. This width is chosen far greater than the height of the radial channel, to ensure that the flow resistance of the inlet channel is negligible compared to the valve resistance. Similarly the flow outlets are made to be $w_{\text{out}} > 500 \mu\text{m}$ wide. The inlet to outlet spacing is chosen to be $s_{\text{io}} = 600 \mu\text{m}$, which is expected to provide enough bonding surface to enable a leak-tight bond between the microvalve chip and the glass support wafer. The inlet-to-outlet spacing of the glass support wafer is $s_{\text{glass}} = 8.7 \text{ mm}$.

The valve membrane thickness is chosen to be $t_{\text{membrane}} = 25 \mu\text{m}$ thick, which is half the starting thickness of the device layer. This ensures that mechanical contact between the valve membrane and the piezo actuator occurs only at the central plate, so that the valve membrane remains free to deform. FEM analysis shows that at $25 \mu\text{m}$ thickness the membrane can be displaced tens of micrometers at very low forces ($< 2 \text{ mN}$). Maximum bending stress remains very low, on the order of tens of megapascals. Note that the valve membrane is physically isolated from the surrounding device layer silicon, to reduce the constant parasitic capacitance in the valve.

The size of the valve cavity underneath the membrane depends on the etching time of the BOX layer, and is approximately $w_{\text{cavity}} \approx 3.5 \text{ mm}$ for the chosen inlet to outlet pitch. At these dimensions, the dead volume of the valve at its resting position remains small at approximately 12 nL .

The thickness of the BOX layer determines the required actuator stroke to fully close the valve, but also determines the maximum valve separation and therefore the maximum flow supported by the valve. Since the valve design in chapter 3 applied a monodirectional actuator, the valve plate could not be pulled above the $4 \mu\text{m}$ BOX layer thickness and flow was limited to approximately 1.1 g h^{-1}

Table 4.1: Summary of the valve dimensions.

Parameter	Variable	Value
<i>Wafers:</i>		
Device layer thickness	t_{DL}	50 μm
Handle layer thickness	t_{HL}	400 μm
Buried oxide thickness	t_{BOX}	4 μm
Glass wafer thickness	t_{glass}	525 μm
<i>Valve:</i>		
Membrane width	w_{membrane}	4.6 mm
Membrane thickness	t_{membrane}	25 μm
Plate width	w_{plate}	800 μm
Plate thickness	t_{plate}	50 μm
Cavity width	w_{cavity}	3.5 mm
<i>Fluidic connections:</i>		
Inlet channel width	w_{in}	600 μm
Outlet channel width	w_{out}	> 500 μm
Inlet-outlet spacing	s_{io}	500 μm
Glass inlet-outlet spacing	s_{glass}	8.7 mm

at 500 mbar of pressure. With the integration of a bidirectional piezo actuator, our new design will open to at least twice the BOX thickness. Since radial flow scales with the third power of the separation (in the viscous, laminar regime) [13], we estimate that in order to reach a total flow of 15 g h^{-1} at 500 mbar a channel height of approximately $10 \mu\text{m}$ is required. To make sure the actuator can always close the valve entirely, the buried oxide thickness is chosen to be $4 \mu\text{m}$ thick, and the required actuator stroke becomes $\pm 6 \mu\text{m}$.

4.2.3 Piezo integration

We use T216-A4NO-273X bimorph disks by Piezo Systems Inc. as piezoelectric actuators for our microvalve. The specifications of these actuators are summarized in table 4.2. The free stroke S_{free} is well above the minimum required displacement of $\pm 6 \mu\text{m}$. However, initial experiments revealed that the performance of the actuator diminished severely when it was adhesively bonded to the silicon surface. The maximum stroke was reduced to approximately $1 \mu\text{m}$, making it impossible for the valve to close fully.

Table 4.2: Specifications of the T216-A4NO-273X bimorph actuator.

Parameter	Variable	Value
<i>Mechanical:</i>		
Monomorph thickness	t_{mono}	190 μm
Total thickness	t_{piezo}	410 μm
Diameter	w_{piezo}	12.7 mm
Stiffness	K_{piezo}	125 kN m^{-1}
Resonance frequency	f_{piezo}	7.3 kHz
<i>Piezoelectric:</i>		
Rated voltage (AC)	V_{max}	$\pm 180 \text{ V}$
Free stroke	S_{free}	$\pm 19.1 \mu\text{m}$
Blocking force	F_{block}	$\pm 2.4 \text{ N}$

This efficiency loss occurs because the adhesive bonding forms a fixed constraint for the piezo across the bonding area, which keeps both monomorph layers from contracting and expanding fully. In order to reach a large stroke, the piezo bimorph requires a support that is flexible in radial direction and allows rotation of the piezo at or near the outer edge. We have therefore designed a flexible piezo support structure, consisting of a ring of flexible material that is placed between the piezo disk and the silicon surface. The flexible support can expand and contract to accommodate expansion and contraction of the piezo bimorph. The ring support can also bend to allow the piezo to rotate, effectively transforming the fixed constraint into a pinned constraint.

We have used finite element (FEM) analysis in COMSOL Multiphysics® v.4.3a to study the critical parameters of this flexible ring support and determine their optimum values to maximize the stroke of the piezo actuator.

4.3 FEM Analysis

4.3.1 Piezo model

To create an approximate FEM model of the piezo actuator, we focused on its specified mechanical stiffness, blocking force and free stroke (see table 4.2), as together they describe both the mechanical and piezoelectric behavior. Fig. 4.2 shows the radially symmetric model used for the analysis. The symmetry axis allows vertical (z -axis) displacement at the center of the piezo, while the piezo's outer bottom edge is constrained with an enforced displacement of $z = 0$. The x, y

Figure 4.2: Axisymmetric model used for FEM analysis of the piezo actuator, consisting of two oppositely poled monomorph layers with a prescribed displacement $z = 0$ at the outer edge. Rotational symmetry axis is marked by the dashed line to the left.

and rotational axes at the outer edge are unconstrained, leaving the entire piezo free to bend.

We selected COMSOL's built-in PZT-5A material for both piezo layers, and inverted the z -axis of the bottom layer to account for the opposite piezoelectric poling. To match the material properties in our model to the actual piezo actuator (also a PZT-5A ceramic), we varied the bimorph layer thickness until the mechanical stiffness equaled K_{piezo} . This is the case at a monomorph thickness of $168 \mu\text{m}$, which is close to the $190 \mu\text{m}$ thickness of the actual piezo monomorphs.

To obtain the same piezo performance in our model as in the actuator, we varied the electric field applied in the model until the free stroke and blocking force matched the specifications. The free stroke is simply the simulated piezo displacement when no external force is applied. The blocking force equals the boundary load (applied near the center of the disk, left in the image) necessary to push the bimorph back to a fixed surface at its starting position. At an applied electric voltage of 250 V the modeled bimorph reached a free stroke of $20.0 \mu\text{m}$ at a blocking force of 2.5 N . Since these values match reasonably well with the specifications, the model can be used as a qualitative approximation of the real actuator.

4.3.2 Flexible support model

We have studied the effects of a flexible ring support mounted between the piezo actuator and the silicon microvalve, using the axisymmetric COMSOL model shown in figure 4.3a. The piezo model described in the previous section is used, with a ring of flexible material fixed at the outer edge of the piezo (right side of the image). A cylindrical piece of glass is added in the center, as this will be used to transfer the piezo motion to the microvalve. The bottom of the flexible support is given a fixed constraint, and a boundary load F_{gas} is applied at the glass cylinder:

$$F_{\text{gas}} = P_{\text{gas}} \cdot A_{\text{valve}}. \quad (4.2)$$

Where A_{valve} is the surface area of the valve membrane.

Figure 4.3: (a) Axisymmetric FEM model of the piezo actuator with a flexible ring support (b) There is a tradeoff between increased piezo displacement and increased stretching of the flexible support due to the gas pressure.

The flexible support is expected to increase the maximum displacement of the piezo, as it allows the piezo layers to expand and contract without much constraint. At the same time however, the gas pressure P_{gas} pushing up against the valve would cause the flexible support to be stretched upward, raising the piezo and the valve plate with it (see figure 4.3b). This means the net displacement of the valve d_{valve} is smaller than the displacement of the piezo d_{piezo} . It is expected that an optimum exists for the flexibility of the support layer, where the net valve displacement is maximized.

Note that in reality an adhesive layer exists between the piezo and the support, which can be modeled using the thin elastic layer boundary condition in COMSOL. It is however found that inclusion of this thin elastic layer has no significant effect on the simulation results, which is in line with the adhesive layer's flexibility being negligible compared to the flexibility of the support material.

The materials used in our analysis are silicone rubbers, as they are readily available both premade and synthesizable with a range of mechanical properties. The first material is a silicone rubber named RM10BN available as premade sheets with a hardness of 60° Shore A, which corresponds to a Young's modulus of approximately 5 – 10 MPa [14]. The second material is polydimethylsiloxane (PDMS), which is a common elastomer used in microtechnology and can be shaped with lithographic techniques. It has a Young's modulus on the order of hundreds of kilopascals [15].

4.3.3 Simulation results

We simulated the maximum downward displacement of the piezo as a function of the width of the flexible support ring w_{ring} , the thickness of the material T_{ring} , and the gas pressure P_{gas} . Figure 4.4 shows the results for PDMS ($E = 750 \text{ kPa}$) and RM10BN ($E = 10 \text{ MPa}$) respectively. A Poisson ratio of $\nu = 0.49$ was used for both materials.

Figure 4.4: Results of FEM analysis of the maximum downward stroke achievable with a piezo actuator with a flexible ring support made of PDMS (left) or RM10BN silicone rubber (right), as a function of ring width and ring thickness. Note that at higher pressures the flexible support is stretched further upwards, decreasing the net stroke of the actuator.

Figure 4.5: Piezo and ring support deformation results from the FEM model shown in figure 4.3 at a pressure of 1 bar, showing that the wider ring support (b) is less sensitive to gas pressure. Rotational symmetry axis is marked by the dashed lines to the left.

It is found that at zero pressure both materials enable a stroke of approximately $17\text{ }\mu\text{m}$ at a support width of 1 mm. As was expected the net stroke decreases as the gas pressure increases, due to the upward stretching of the support material. This stretching effect is stronger in the more flexible PDMS material, which makes the PDMS system most sensitive to gas pressure. Both materials facilitate a maximum stroke of $10\text{ }\mu\text{m}$ at 1 bar of pressure. For the PDMS material this maximum is reached at a width of $w_{\text{ring}} = 1\text{ mm}$ and a thickness of $T_{\text{ring}} = 100\text{ }\mu\text{m}$. For the RM10BN silicone rubber the maximum lies at $w_{\text{ring}} = 1\text{ mm}$ and $T_{\text{ring}} = 400\text{ }\mu\text{m}$.

It is visible in figure 4.4 that the difference between high pressure stroke and low pressure stroke is larger for slimmer supports. This means that slimmer supports are more susceptible to the gas pressure pushing them up. This can be explained when looking at the material displacement, as shown in figure 4.5. This result plots the deformation of the piezo and ring support using a voltage of 250 V, a pressure of 1 bar and a ring thickness of $700\text{ }\mu\text{m}$, using $w_{\text{ring}} = 1\text{ mm}$ and $w_{\text{ring}} = 2\text{ mm}$. For the slim support (a), the material deformation at the inner and outer support edges are approximately equal. For the wider ring support (b), the deformation at the inner edge decreases significantly. This is because the inner edge is now close to the 'neutral' pivot point of the piezo, where displacement is zero. Around this point the work done by the piezo is (near) zero, so a wider support does not decrease the piezo's efficacy. It does however increase the total stiffness in the normal direction, meaning this wider support will be stretched less easily by the gas pressure.

Apart from decreasing the sensitivity to gas pressure, a wider ring support simplifies handling and bonding during assembly of the valve. In this work we have therefore chosen to apply premade RM10BN silicone rubber sheets with the ring support as wide as possible. The thickness is chosen at 1 mm, primarily to leave room for electrical wiring to the bottom of the piezo (see figure 4.8). At this

thickness, the maximum stroke at 1 bar pressure in our FEM model is relatively insensitive to support ring width. This allows us to choose a support width of 2 mm.

4.4 Fabrication

4.4.1 Microfluidic chip

The fabrication process of the microvalve chips is based on the process described in chapter 3 and illustrated in figure 4.6 (see also appendix A). We start with a <100>-oriented SOI wafer with a 50 μm device layer, 4 μm BOX layer and 400 μm handle layer (figure 4.6a). Both device layer and handle layer are highly doped with a resistivity of 0.005 - 0.020 Ωcm , to enable electrical sensing of the valve capacitance. A 1 μm thick oxide layer is grown on the wafer using wet thermal oxidation, which is then patterned and etched in a buffered HF solution (7:1 HF:NH₄F solution, figure 4.6b). This patterned oxide will act as an etching barrier in subsequent dry etching steps.

A second lithographic pattern is created on the device layer using photoresist, and the silicon is etched with an Alcatel AMS100 deep reactive ion etcher (DRIE) using a Bosch process, to a depth of approximately 30 μm (c). The photoresist is then stripped and a second DRIE step is performed until the shallow structures are etched approximately 25 μm . This also etches the deep structures down to the BOX layer (d). The oxide mask is then removed using a 50% HF solution (e). To protect the exposed BOX layer from subsequent HF etch steps, a 1 μm silicon-rich nitride layer is grown using LPCVD (f). The handle layer is then patterned and etched with a Bosch DRIE process to create the flow inlets and outlets (g).

The valve membrane is released using a combination of liquid and vapor phase HF etching. First the majority of the buried oxide underneath the valve membrane is etched using a 50 % HF solution, carefully timed to leave a small amount of oxide separating the valve plate and valve seat (h). This oxide prevents stiction by capillary force during wafer drying. Note that the protective silicon nitride layer is also etched in this step. The wafer is then diced and the individual microfluidic chips are cleaned and dried. After drying the remaining oxide is removed using a vapor phase HF step (i).

To create the glass support chip, a 500 μm thick borosilicate glass wafer is patterned on both side with photoresist foil and powderblasted from both sides, leaving approximately 300 μm deep channels. After cleaning the glass wafer is diced into separate chips. Figure 4.7 shows a SEM close-up of a realized valve, showing the valve plate, the valve membrane and the deeply etched ring separating

Figure 4.6: Process flow of the microvalve: (a) SOI wafer; (b) Oxidation and patterning; (c) DRIE of silicon; (d) Resist strip and continued DRIE of silicon; (e) Oxide strip in 50% HF; (f) LPCVD of nitride capping layer; (g) Bottom side DRIE; (h) Etching of BOX and nitride using 50% HF; (i) Etching of BOX using vapor HF; (l) Adhesive bonding to powderblasted glass chips.

the membrane and the surrounding device layer silicon.

4.4.2 Assembly

The glass and silicon chips are bonded together individually, using Loctite 358 high-viscosity, UV curable adhesive. The bonding process is similar to the method presented in [16]. A thin layer of adhesive is distributed on the glass chip surface using a rubber roller, after which the silicon chip is manually aligned and brought into contact with the glass chip. The transparent glass allows easy verification of both the alignment and the mechanical contact between the two chips. The chips are then placed in a UV exposure system to cure the adhesive, creating a solid, leak-tight seal between glass and silicon (figure 4.6j).

The flexible ring support is cut out of 1 mm thick RM10BN silicone rubber sheets and gaps are cut into the ring to create room for the piezo wiring (see figure 4.8). The flexible support is mounted on the silicon using Loctite 4850 flexible cyanoacrylate adhesive. Loctite 480 toughened cyanoacrylate is used to adhesively bond a piece of glass to the valve plate, which will connect the valve plate to the piezo disk. We use diced pieces of a 1 mm glass wafer for this, as they have a well defined thickness.

The piezoelectric actuator is connected on both sides with thin flat cable wires, using silver-based conductive paint for electrical contact and cyanoacrylate adhesive for mechanical fixation. The actuator is then bonded to the silicone ring

Figure 4.7: SEM image of the valve membrane, showing both shallow and deep etched structures. The beam visible at the bottom left is for connecting the capacitive readout.

Figure 4.8: Artist impression of the final assembly of the microfluidic chip with the piezoelectric actuator, using the silicone rubber support.

using Loctite 4850 adhesive, while Loctite 480 is used for the bond between the actuator and the glass connector piece. To connect the microvalve chip to the fluidic characterization system, it is mounted in a fluidic chipholder with Loctite Precision epoxy adhesive. Finally, electrical connections for capacitive sensing are made using platinum wirebonds. A realized microvalve with electrical connections and PMMA chipholder is shown in figure 4.9.

4.5 Characterization

4.5.1 Measurement setup

We have characterized the frequency-dependent electrical and mechanical properties of the microvalve using the measurement scheme illustrated in figure 4.10. The piezo actuator is driven by a sine voltage at frequencies $1 \text{ Hz} \leq f_{\text{act}} \leq 10 \text{ kHz}$, which causes the valve capacitance to change at the same frequency. This capacitive signal is modulated on a sine wave with a frequency of $f_{\text{lock}} = 100 \text{ kHz}$ and $500 \text{ mV}_{\text{pp}}$ peak to peak amplitude, connected to the valve plate (device layer). The valve seat (handle layer) is connected to a charge amplifier, the gain of which can be expressed as:

$$V_{\text{out}} = \frac{C_{\text{valve}}}{C_f} \cdot V_{\text{in}}. \quad (4.3)$$

Figure 4.9: Photograph of the realized microvalve with integrated piezoelectric actuator, mounted on a PMMA fluidic chipholder.

Where C_{valve} is the valve capacitance and $C_f = 47 \text{ pF}$ is the feedback capacitance of the amplifier. The output of the charge amplifier is sampled and demodulated by a Stanford Research 830m lock-in amplifier. To further reduce noise the demodulated signal is measured by a second lock-in amplifier, locked at the actuation frequency. The mechanical displacement is measured at the center of the piezo, using a Polytec MSA-400 vibrometer.

The time-dynamic response provides information about the mechanical switching speed of the valve. Although in an ideal case a time-dynamic fluidic characterization could be performed together with the mechanical experiments, such measurements would show the convolution of the fluidic response of the microvalve with that of the measurement setup. Since the internal volume of our microvalve is very small, it would be very difficult to obtain values that are not heavily dependent on the circumstances in which they are measured. The fluidic response of the valve is therefore characterized in the steady state, to eliminate the influence of the measurement setup.

Fluidic characterization of the realized microvalves is done using a gas pressure controller (Bronkhorst P-602CV) backed by a dry nitrogen gas source, connected to a mass flow meter with a maximum range of 13 g h^{-1} (Bronkhorst F-111B). The mechanical response during flow measurements is measured using a loadcell, connected above the center of the piezo actuator (Futek LSM250 at 10.00 V supply voltage).

A spark discharge from the valve's electrical wires was observed at a voltage of 120 V, so for safety reasons the actuation voltage was limited to $\pm 100 \text{ V}$.

Figure 4.10: Measurement scheme for the frequency-dependent characterization of the valve's electrical and mechanical properties. The light gray area denotes the microvalve assembly.

4.5.2 Dynamic response

We have characterized the dynamic response of the microvalve in a range of 1 Hz - 10 kHz, at actuation voltages of 1.0 V_{pp}, 3.0 V_{pp} and 10.0 V_{pp} and at pressures between 0 mbar and 1000 mbar. Figure 4.11a shows the displacement amplitude measured at the center of the piezo, as well as the total phase shift (at V_{act} = 3.0 V_{pp}) between the actuation signal (output of the function generator) and its mechanical response. The dynamic response was found to be independent on pressure for the measured voltage and pressure ranges, up to at least 5 kHz (plots show results at zero pressure). Figure 4.11b plots the peak to peak capacitance change of the displacement sensor, calculated from the output of the charge amplifier using equation 4.3. The figure also plots the phase shift between the lock-in output and the 3.0 V_{pp} actuation signal. Note that at very low frequencies ($f < 10$ Hz) the measurement time for each data point is on the order of tens of minutes due to the long integration time of the lock-in amplifier, which leads to increased measurement errors caused by drift in the piezoelectric actuator. This effect is most notable at the lowest actuation voltages.

In the measured frequency range the microvalve behaves as a second order mass-spring system with a flat amplitude response for frequencies up to 3 kHz, above which the first mechanical resonance (-90° phase shift) is visible at 6.8 kHz. This is close to the specified resonance frequency of the piezo actuator (7.3 kHz). The capacitive displacement sensor (figure 4.11b) shows a flat response up to a bandwidth of approximately 700 Hz. This bandwidth is limited by the sampling rate of the lock-in amplifier, which means that with a dedicated high-speed filter a higher performance should become possible.

At the measured actuation voltages the valve response scales linearly with piezo

Figure 4.11: Dynamic response of the microvalve, showing the displacement amplitude at the center of the piezo (a) and the peak-to-peak capacitance variation of the capacitive sensor (b) at various actuation voltages.

voltage amplitude. Figure 4.12 shows the ratio between actuator displacement and actuator voltage (measured in nm/V_{act}) as well as the sensitivity of the capacitive sensor (measured in $V_{cap}/\mu\text{m}$). Both plot groups show identical results for all three actuator voltages, which validates the reliability of the capacitive sensor. The above results show that our piezoelectric microvalve is suitable for high-speed flow control with response times on the order of milliseconds. During the course of the measurements the piezo was exposed to (low voltage) AC signals for at least 10^7 cycles. No degradation of piezo performance was observed during this time.

Figure 4.12: The actuation ratio (displacement per volt) and capacitive sensitivity are nearly independent of actuation voltage across the entire measurement range. Note that the outliers at 1 and 2 Hz are probably caused by piezo drift due to long measurement time.

4.5.3 Steady-state response

Figure 4.13 shows the steady-state mechanical response of the piezoelectric actuator. The displacement is measured at the center of the piezo as it moves up and down from its neutral position. Note that the constant displacement offset caused by stretching of the ring support is not measured using this method. Apart from this displacement offset the piezo displacement curves are found to be independent of gas pressure, which is in line with the piezo being much stiffer than the support. The maximum piezo displacement drift across all measurements is found to be 4% of full scale. The maximum displacement is $\pm 7.2 \mu\text{m}$ at $\pm 100 \text{ V}$, which translates to a full stroke of $\pm 13 \mu\text{m}$ at $\pm 180 \text{ V}$ when assuming a continued response as the one shown in figure 4.13.

The fluidic response of the valve is shown in figure 4.14, plotting the measured mass flow Q_{mass} as a function of the measured capacitance variation, compensated for the parasitic capacitance ($C_{\text{var}} = C_{\text{valve}} - C_{\text{par}}$). The flow through the fully opened valve exceeds the measurement range of the flow meter (13 g h^{-1}) at a pressure of 500 mbar. The flow curves in figure 4.14 show a significant hysteresis, which suggests that the capacitance does not always correspond to a unique value for the valve separation. This is probably caused by a changing tilt angle between the valve plate and valve seat: Mass flow in the radial channel increases with the

Figure 4.13: Static mechanical response, average of measurements at pressures of 100, 200, ..., 1000 mbar. Error bars show the minimum and maximum values across all pressures. The total piezo stroke equals $\pm 7.2 \mu\text{m}$ at $\pm 100 \text{ V}$.

third power of the separation [13], and capacitance increases with the inverse of the separation. If the tilt angle changes between opening and closing the valve, this highly non-linear behavior will lead to different mass flows at equal capacitance values, or vice-versa.

Given that the hysteresis loops are closed and repeatable, the valve plate tilt angle must depend on the direction of the piezo movement. This means that the actuator movement is not following the same path in both directions. Because of the top-mounted piezo actuator it is not possible to observe the shape and movement of the valve membrane, for example by white light interferometry. A possible cause could be a local imperfection in the bond between actuator and support, leading to static and dynamic friction and consequently to mechanical hysteresis. In order to prevent this from happening, care must be taken to provide a reproducible, well-defined connection between valve and actuator, for example by applying surface treatments or by using different materials or adhesives.

Figure 4.15 shows the measured flow as a function of pressure, at various actuation voltages. The flow scales nearly linearly with pressure with a slight nonlinear increase at higher pressures, probably caused by stretching of the flexible ring support. It is therefore expected that the maximum flow at 1 bar pressure is at least 25 g h^{-1} . If care is taken to prevent electrical discharges at higher voltages, it is possible to increase this flow range further by driving the actuator to its maximum stroke. When the valve is closed the leak flow is below the detection threshold of 0.5 mg h^{-1} up to a pressure of 500 mbar. At 1 bar the measured leak flow is 28 mg h^{-1} .

Figure 4.14: Static fluidic response, showing mass flow as function of valve capacitance, compensated for the parasitic capacitance C_{par} .

Figure 4.15: Mass flow as function of pressure, at various actuation voltages. A slight nonlinear increase can be seen at higher pressures, due to stretching of the flexible ring support.

4.6 Conclusions

We have demonstrated the realization of a proportional micro control valve with a top-mounted piezoelectric bimorph actuator. The microvalve is realized in a straight-forward fabrication process based on a single SOI wafer. A glass support wafer was applied to facilitate easier mounting of the microfluidic chip in the experimental setup, but is not required for valve operation. The bimorph actuator is mounted to the microfluidic chip using adhesive bonding. We have achieved a

high level of integration of the actuator using a flexible silicone rubber support to maintain maximum actuator stroke. The dimensions of the control valve with actuator are $17\text{ mm} \times 17\text{ mm} \times 1.9\text{ mm}$. With the addition of the glass support wafer used for fluidic interfacing the thickness increases to 2.4 mm , bringing the total volume of the prototype to 0.69 cm^3 .

The valve has been characterized mechanically and fluidically. The system behaves as a second order mass-spring system with a resonance frequency of 6.8 kHz , with a flat mechanical response up to 3 kHz . The capacitive readout was limited by the sampling rate of the lock-in amplifier, with a bandwidth of 700 Hz . These results show that the microvalve is suitable for high-speed flow control with response times on the order of milliseconds.

The valve supports flows up to at least 13 g h^{-1} at a pressure of 500 mbar with an expected maximum of at least 25 g h^{-1} at 1 bar , using a maximum actuation voltage of 100 V . This range can be increased by driving the actuator to its full stroke at 180 V . Flow through the valve scales nearly linearly with pressure. The relation between capacitance and flow was found to suffer from hysteresis, which can be attributed to tilting of the valve plate that changes with the direction of the piezo movement. This may be solved by ensuring a strong and well-defined adhesion bond between valve and actuator.

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5

On-chip flexible actuator supports

5.1 Introduction

The piezoelectric bimorph actuator used in chapter 4 has the advantage of offering relatively large stroke and force while requiring only a small volume. In particular the thickness is greatly reduced compared to a (stacked) linear piezo, since the bimorph bending transforms part of the actuation force to increased stroke. However, a bimorph also places increased demands on the assembly and mounting method, as the bending actuator requires freedom of movement to contract and expand. If the bimorph is fixed to the chip surface, for example by an inflexible adhesive, this mechanical constraint impedes bending and the actuator stroke is reduced.

It was shown in chapter 4 that the application of a flexible support layer between the microfluidic chip and the piezo bimorph is sufficient to provide the required freedom of movement. The design however required several manual assembly steps, which has a negative impact on fabrication yields. This chapter investigates design options that would allow better integration of the actuator with the silicon chip.

Figure 5.1: Mechanical stiffness of a flexible piezo support in the normal, radial and rotational directions.

5.2 Analysis

5.2.1 Requirements

For this investigation we start from the microvalve design presented in figure 4.1, using the same T216-A4NO-273X bimorph disks as used in chapter 4. This means the actuator is to be top-mounted on a SOI-based silicon chip shaped with deep reactive ion etching (DRIE). The thickness of the handle layer and device layer of the SOI wafer may be changed from the values used in the existing valve design, as the valve operation does not depend on these dimensions. The thickness of the buried oxide is however kept at $4\mu\text{m}$, as it has a strong impact on the required actuator stroke and maximum flow through the valve.

The primary demand of the actuator assembly is that it provides sufficient stroke to fully close and open the microvalve. The existing microvalve requires a minimum net stroke of $S_{\min} = 4\mu\text{m}$ to fully close the valve. When the gas pressure is increased, the force acting on an open valve causes an increase in valve separation that depends on the flexibility of the bimorph and its support. This means that there is a tradeoff between the support's stiffness in the normal direction k_z , which should be high to withstand the gas forces, and stiffness in the radial and rotational directions k_r and k_θ , which should be low to allow actuator movement (see figure 5.1).

Table 5.1 summarizes the demands placed on the actuator support structure and the physical dimensions that act as boundary conditions for its design. The merit of each design is evaluated by the resulting net valve stroke and the maximum stresses in the support at $P_{\text{gas}} = 1\text{ bar}$. Stresses should not exceed the fracture strength of the materials, and should not exceed the rated lap shear strength at adhesive interfaces. Since silicon is a brittle material, local imperfections such as jagged or sharp edges can lead to fracture even for relatively low global stresses. The maximum stress in silicon should therefore be well below its tensile strength of several gigapascals [1]. A limit of 500 MPa is selected for this study.

Table 5.1: Demands and boundary conditions of the actuator and its support structure.

Parameter	Variable	Value
<i>Physical dimensions:</i>		
Bimorph disk radius	r_{piezo}	6.35 mm
Valve membrane radius	r_{valve}	1.7 mm
Valve plate radius	r_{plate}	400 μm
Buried oxide thickness	t_{BOX}	4 μm
<i>Performance demands:</i>		
Minimum stroke	S_{min}	$\pm 4 \mu\text{m}$
Maximum pressure	P_{gas}	1 bar
Maximum silicon stress	σ_{Si}	< 500 MPa
Maximum adhesive stress:		
In bulk		< 1.5 MPa
At adhesive interfaces	σ_{adh}	< 1 MPa

We select the commercially available Loctite 5590 two-component epoxy adhesive as a model of flexible adhesive material, as it cures to an elastomeric material with a Young's modulus as low as $E_{5590} = 3.6 \text{ MPa}$. The lap shear strength of Loctite 5590 is rated at 2 MPa for an aluminum to aluminum bond. Since ceramics typically bond less well than most other materials, we set the maximum stress at adhesive surfaces to 1 MPa. The adhesive has a rated tensile strength of 3 MPa, the maximum adhesive bulk stress is set to half of that. The fracture strength of the bimorph actuator is not specified, but the maximum gas force (< 1 N) is well below the blocking force of the actuator (2.4 N) and it is assumed that the actuator is not strong enough to break itself.

5.2.2 Possible solutions

The fabrication process of the microvalve shown in section 4.4 allows several different approaches to the assembly problem. The most straight-forward solution would be to apply direct adhesive bonding, as illustrated in figure 5.2a. A layer of flexible adhesive is applied to the bimorph, for example by spin-coating, after which the bimorph is brought into contact with the silicon chip. The unetched silicon surfaces make contact with the adhesive so that the bimorph is bonded to the valve plate and the bimorph supports.

The main advantage of this approach is its simplicity. Dispensing the adhesive on the entire bimorph surface avoids the need for precision dispensing or

Figure 5.2: Geometries that can provide a flexible support for the bimorph actuator. (a) Thick, flexible adhesive layer between bimorph and silicon, causing adhesive overflow (hashed). (b) Same as (a), but using spacers to define adhesive thickness and reduce overflow. (c) Flexibility provided by bendable silicon structures instead of flexible adhesive.

patterning, and the use of spin-coating would allow precise control over adhesive thickness. The thickness of the adhesive layer after bonding is however very difficult to control, as it depends strongly on the contact force during bonding. This means that it is difficult to obtain a proper stiffness in the normal direction. It also means that the overflow of adhesive material away from the contact surfaces is difficult to control, which might lead to undesired contacting of adhesive material to the etched silicon surfaces (illustrated as the hashed regions in figure 5.2a). This method of direct adhesive bonding is therefore best suited for thin adhesion layers, and relatively high device layer thicknesses.

A design that could solve the disadvantages of direct adhesive bonding is shown in figure 5.2b. By creating physical spacers at the bonding contact points,

the thickness of the adhesive layer can be accurately defined. The adhesive outside of the spacers is used to hold the bimorph in place, so the spacers can be made much smaller than the required total bonding area. This allows the use of a narrow silicon ridge at the edge of the chip, if it can be made flexible enough to enable the required piezo bimorph motion. With this approach the final thickness of the adhesive can be defined very precisely. Mechanical stress is a primary concern in this design, as the support must be strong enough to withstand the bimorph deformation. Note that some overflow of adhesive can still occur in this design, in particular because the adhesive thickness should be slightly thicker than the silicon gap to ensure contact with the silicon surface. The overflow is however reduced significantly as the spacer-bimorph contact area is much smaller than the total bonding area.

A more elaborate approach would be to create a tilting silicon support as shown in figure 5.2c. By releasing part of the device layer from the buried oxide, a hinging structure can be defined that provides both rotational and translational freedom. With this approach the bimorph movement is supported entirely by the silicon structures, so a stronger, non-flexible adhesive may be used to bond the bimorph to the silicon. The challenge of this approach is to create a silicon structure that has low k_r and k_θ , while having a high enough normal stiffness k_z . It is also likely to involve a more complex fabrication process. Our study will therefore focus mainly on the second solution, to obtain a bond with flexible adhesive using silicon spacers to define the adhesive thickness.

5.3 Finite element modeling

The mechanical properties of the support influence the boundary conditions and constraints of the actuator, and therefore its performance. An iterative approach is therefore required to determine the optimum support dimensions. We use FEM analysis to investigate the effects of different support assemblies on the net stroke of the actuator, based on the model described in section 4.3.1. The material, geometrical and electrical parameters of the piezo bimorph model are as described in chapter 4, while the silicone rubber support is replaced by the silicon spacer support. The use of axial symmetry means that we only model continuous, ring-like shapes. This can be viewed as a 'worst case' approach, as a radially symmetric structure is stiffer in the radial direction than a discontinuous structure such as a ring of individual pillars.

Since primary interest of the analysis lies in the support or bonding layers, we explicitly model the adhesive materials with high-resolution meshes. Mesh

Figure 5.3: Model used for FEM analysis of the silicon spacer, assuming the worst case of no adhesive material present (not to scale). The silicon shape is filleted to avoid singularities.

Figure 5.4: Net stroke from and maximum stress in a silicon spacer fixed to the bimorph actuator, using the FEM model of figure 5.3. Stroke increases and stress decreases with increasing r_{spc} .

points are manually distributed at the boundaries of all small-scale geometry components, after which a fine free triangular mesh is applied to fill the 2D surfaces. By scaling the manual mesh distribution with the geometry, this allows us to maintain a fine mesh even in extremely thin adhesion layers ($< 100\text{nm}$). Care is also taken to avoid geometrical singularities in the model. Since stress is the spatial derivative of displacement, sharp inside corners can not converge with increasing mesh refinement. Sharp corners are therefore rounded with fillets where necessary.

5.3.1 Directly bonded spacer

The worst case scenario for spacer adhesive bonding is when the bimorph is fixed directly to the spacer with no flexible adhesive material inbetween, as stresses will be maximized and stroke will be minimized in this case. We therefore use the FEM model shown in figure 5.3 to determine the minimum requirements for

Figure 5.5: Net stroke and maximum stress as a function of h_{spc} and w_{spc} .

the support spacer position and dimensions. The piezo bimorph is actuated with 250 V and loaded with gas pressure P_{gas} at the center (see also section 4.3.1). Near the outer edge it is fixed to a silicon spacer ring with radius r_{spc} , width w_{spc} and height h_{spc} . The spacer is modeled as a polysilicon material with a fixed constraint at the bottom. The net stroke is determined by measuring the z-displacement at the center of the bimorph. The 2D surface maximum of the Von Mises stress is taken as a measure for the maximum material stress in the silicon.

Figure 5.4 shows the resulting stroke and maximum stress using this model, as a function of the radial position of the spacer r_{spc} . The spacer height is set to $h_{\text{spc}} = 100 \mu\text{m}$ with $w_{\text{spc}} = \{20 \mu\text{m}, 40 \mu\text{m}\}$ to model both a relatively flexible and stiff support spacer. It is visible that for both a thin and a thick spacer the stroke increases with increasing r_{spc} , while the stress remains relatively constant. We therefore set the position of the spacer to the maximum, which is the radius of the bimorph $r_{\text{piezo}} = 6.35 \text{ mm}$.

The results also demonstrate that the maximum stroke is strongly dependent on the physical dimensions of the spacer. We have therefore analyzed the stroke and stress as a function of w_{spc} and h_{spc} . We allow a maximum aspect ratio of 1:10, as this can still be achieved with (deep) reactive ion etching without too much risk of underetching or overetching [2]. The results in figure 5.5 show that stroke decreases approximately linearly with increasing spacer width. This is as expected, as a wider silicon ring is stiffer and therefore more constraining for the bimorph

actuator. Stroke also appears to decrease with increasing spacer height, but this is because the width minimum is increasing due to the limited aspect ratio. Note that the spacer flexibility depends more strongly on the width than on the height, analogous to for example the stiffness of a clamped-clamped beam.

Overall the stress decreases strongly with increasing height. For all heights it is found that stress first increases with increasing w_{support} , until it reaches a maximum and begins to decrease. This may be because at larger widths, the stroke is reduced so much that the spacer bending becomes negligible. Starting from a height of $h_{\text{spc}} = 100 \mu\text{m}$ the stress drops below the demanded 500 MPa. For this height a width of $\leq 30 \mu\text{m}$ is required to achieve the required stroke. Increasing h_{spc} to $200 \mu\text{m}$ decreases stress to 300 MPa and nearly doubles the stroke.

5.3.2 Flexibly bonded spacer

A rigid bond between spacers and bimorph can be achieved using a strong, inflexible adhesive, but the relatively small surface area of the support spacers may not be enough to hold the bimorph in place. To increase the bonding area a larger amount of adhesive is added inside of the spacer ring, as illustrated in figure 5.6. This adhesive needs to be very flexible to maintain the flexibility of the support as a whole.

The model shown in figure 5.6 adds two adhesive parts to the geometry, one at the support and one at the valve plate. In both parts the adhesive consists of a thin layer where there is contact between the spacer and the bimorph, and a thick layer where the silicon is etched to a depth of h_{spc} . The spacer height therefore defines the thickness of the thick adhesive layer. To avoid singularities the top of the silicon spacer is filleted inward and the bottom outward. Based on the previous analysis the spacer dimensions are chosen to be $h_{\text{spc}} = \{100 \mu\text{m}, 200 \mu\text{m}\}$ and $w_{\text{spc}} = \{10 \mu\text{m}, 20 \mu\text{m}\}$.

The thin adhesive layer between spacer and bimorph has a thickness t_{adh} that is determined by the contact force during bonding. The width of the thick adhesive part $w_{\text{adh}} = w_{\text{sup}} - w_{\text{spc}}$ is initially set to $25 \mu\text{m}$, the height equals $h_{\text{adh}} = h_{\text{spc}} + t_{\text{adh}}$. In practice the adhesive at the support spacer will overflow both inward and outward during the bonding process, leading to slightly increased adhesive dimensions.

The thin adhesive layer at the valve plate has a thickness t_{ap} , which is not equal to t_{adh} . Since the valve plate is movable, it is likely that the plate is pushed down during the bonding procedure. Our model assumes the worst case, which has the plate pushed down to the valve seat ($t_{\text{ap}} = t_{\text{adh}} + 4 \mu\text{m}$). Note that in this case the valve would be normally closed. The required actuator stroke to close the valve

Figure 5.6: FEM model including a flexible adhesive at the valve plate (left) and the silicon support (right). Both parts consist of a thin adhesive layer at the contact point, with the main (thick) adhesive layer surrounding it (not to scale).

Figure 5.7: Net stroke from and maximum stress in an adhesively bonded spacer, using the FEM model of figure 5.6 with $w_{adh} = 25 \mu\text{m}$. Both stress and stroke decrease with increasing support flexibility.

would then be zero, but the required stroke to reach maximum flow is increased by $4 \mu\text{m}$ ($S_{min} = 8 \mu\text{m}$). Ideally the spacer at the valve plate should be chosen as narrow as possible to prevent adhesive overflow, but a certain minimum width w_{spp} is required to maintain adhesive stiffness in the normal direction. Given the valve plate radius $r_{plt} = 400 \mu\text{m}$ we choose $r_{spp} = 200 \mu\text{m}$, leading to a radial adhesive overflow of approximately $100 \mu\text{m}$.

We model the adhesive material based on Loctite 5590, using the specified Young's modulus of $E_{5590} = 3.6 \text{ MPa}$. A Poisson ratio of $\nu_{5590} = 0.49$ is used and the material density is set to equal the uncured mixed product at 1400 kg m^{-3} .

Figure 5.8: Stress distribution in the thin adhesive layer is concentrated mostly at the interfaces with bimorph (above) and spacer (below right).

Figure 5.9: Stress distribution in a partially delaminated spacer adhesive is concentrated at the edge of the air gap.

5.3.3 Adhesive stress

The simulation results of this model are shown in figure 5.7. Stress decreases for increasing t_{adh} and is also lower for a greater support height, which is in line with the support structure being more flexible in these cases. The net stroke is relatively constant for small adhesive thicknesses. As t_{adh} increases above $5\mu\text{m}$ however, the stroke starts decreasing more rapidly. This is because the stiffness in the normal direction is decreasing and the gas force becomes sufficient to open the valve. An optimum is found around $t_{adh} = 1\mu\text{m}$, but note that is difficult to obtain a specific adhesive thickness when pushing the bimorph and silicon together.

The resulting stresses are the lowest when using $h_{spc} = 200\mu\text{m}$ and $w_{spc} = 20\mu\text{m}$, but the stress in the adhesive layer still exceeds 1 MPa in all simulated cases. Since most of the stress occurs at the adhesive interfaces (see figure 5.8), this means that the adhesive may not remain bonded to the silicon spacer at these high-stress areas.

The effects of local delamination are studied by introducing a gap between the adhesive material and the silicon spacer. Figure 5.9 shows the stress distribution in delaminated state, revealing that stress concentrations at the edge will probably lead to tearing along the adhesive interface. In the worst case this tearing will continue until the adhesive is released entirely from the silicon spacer. This

Figure 5.10: Net stroke and maximum stress as a function of w_{adh} , modeled with silicon spacer (fixed) and without (free). Dimensions of the silicon spacer are $w_{spc} = 10\mu\text{m}$ and $t_{adh} = 1\mu\text{m}$.

situation can be modeled by removing the support spacer entirely, so that the bimorph is supported by adhesive only. Figure 5.10 shows the resulting stroke and stresses as a function of w_{adh} , both with spacer ring (adhesive still fixed) and without (adhesive fully released). The study is performed using $h_{spc} = 200\mu\text{m}$, $w_{spc} = 20\mu\text{m}$ and $t_{adh} = 1\mu\text{m}$, which are the optimum values determined earlier.

In the fixed state (no adhesive delamination) the stroke remains relatively constant for narrow adhesives, meaning that the total support flexibility is dominated by the silicon spacer. As w_{adh} increases beyond $300\mu\text{m}$ the support becomes less flexible and the stroke begins to decrease. Stress in the silicon is well below fracture, but the adhesive layer stress is on the order of the rated lap shear strength.

If the stress leads to full delamination of the adhesive from the support spacer, the adhesive stress reduces to less than 1MPa with a minimum of 0.3MPa at $w_{adh} = 500\mu\text{m}$. This means that while the thin adhesive layer may fully delaminate from the support spacer, the main adhesive layer should be strong enough to hold the bimorph in place. In delaminated state the bimorph is no longer supported by the support spacer but only by the adhesive material, which means that the flexibility in the normal direction increases significantly. This leads to a strongly reduced net stroke at small w_{adh} . An optimum for both fixed and free states is found at $w_{adh} = 500\mu\text{m}$.

The optimum dimensions derived from the FEM analyses are summarized in table 5.2, with the support performance at those dimensions detailed in table 5.3.

Table 5.2: Optimum dimensions found in FEM analysis of the silicon spacer support (see figures 5.3 and 5.6).

Parameter	Variable	Value
<i>Optimized dimensions:</i>		
Spacer position	r_{spc}	6.35 mm
Spacer height	h_{spc}	200 μm
Spacer width	w_{spc}	20 μm
Support width	$w_{\text{sup}} = w_{\text{adh}} + w_{\text{spc}}$	520 μm
<i>Assumed dimensions:</i>		
Thin adhesive thickness:		
At support	t_{adh}	$> 0.1 \mu\text{m}, < 10 \mu\text{m}$
At valve plate	t_{ap}	$t_{\text{adh}} + 4 \mu\text{m}$
Spacer width at valve plate	$w_{\text{spp}} = 2r_{\text{spp}}$	400 μm

Table 5.3: Support performance at optimum dimensions.

Parameter	Variable	Value, $t_{\text{adh}} =$	
		0.1 μm	10 μm
<i>Fixed to support spacer:</i>			
Stroke	S	10.8 μm	9.4 μm
Maximum silicon stress	σ_{Si}	113 MPa	7.7 MPa
Maximum adhesive stress	σ_{adh}	4.5 MPa	1.4 MPa
<i>Delaminated from spacer:</i>			
Stroke	S	10.2 μm	8.7 μm
Maximum adhesive stress	σ_{adh}	0.3 MPa	0.3 MPa

Figure 5.11: Cross-sectional etch profiles of deep reactive ion etching, with and without tapering.

Figure 5.12: Variation of the valve stroke $v(S)$ as a function of variations in h_{spc} and w_{adh} , at selected values of $t_{\text{adh}} = \{0.1 \mu\text{m}, 1 \mu\text{m}, 10 \mu\text{m}\}$. Note that stroke variations are calculated with respect to the stroke at 0 % variation.

5.4 Parameter variation sensitivity

The practical usability of the above results are verified by studying their sensitivity to variations in the fabrication process. We use the model described in section 5.3.2 to derive the stroke and stresses while scaling the physical dimensions h_{spc} , w_{spc} and w_{adh} between -25% and 25% , with 5% steps. The thin adhesive thickness t_{adh} is varied between 0.1 , 1 and $10 \mu\text{m}$.

To reduce the number of variables to solve for, we make use of the fact that variations in the spacer height $v(h_{\text{spc}})$ and the spacer width $v(w_{\text{spc}})$ are correlated through the dry etching processes. Figure 5.11 schematically shows the cross-sectional profiles of a trench etched using deep reactive ion etching, using a positive, neutral and negative taper. For a positively tapering profile, a variation in etch depth also means a variation in average trench width by the same factor. Similarly for a negative taper the variation is equal but opposite. We therefore define $v(w_{\text{spc}}) = \pm v(h_{\text{spc}})$, to solve for both positive and negative tapers. We also solve for neutral tapers (no tapering) by keeping w_{spc} constant.

The surface plots in figure 5.12 show the resulting stroke variation as a

percentage of the stroke at 0 % variation. Negative values mean a loss in net stroke, while positive values mean an increase in stroke performance. It is visible that the stroke increases as w_{adh} decreases, which is in line with the support becoming more flexible. This advantage would however be lost if the adhesive delaminates from the support spacer, at which point a larger w_{ad} is advantageous (see figure 5.10). Positively tapered spacers are relatively insensitive to variations in spacer height, since they become wider as h_{spc} increases. Negatively tapered spacers become thinner and significantly more flexible with increasing h_{spc} , which translates to a stroke variation between -20 % and 8 % across the tested range.

Figure 5.13 shows the variations in the Von Mises stress in both the silicon and the adhesive. Better performance is indicated by negative stress variations. As before, the performance generally improves with increasing h_{spc} (more flexible spacer). The spacer dimensions can however not be scaled up indefinitely, as with increasing etch depth it becomes increasingly difficult to maintain a high aspect ratio.

The silicon stress never increases by more than 30 % in all cases, except for the silicon stress at $t_{adh} = 10 \mu\text{m}$ which is already very low (see table 5.3). The adhesive stresses however do increase significantly in some cases. Particularly for $t_{adh} = 0.1 \mu\text{m}$ and negative tapers the stress is strongly dependent on h_{spc} , with stress variations between -60 % and 90 %. Note however that the stress at $t_{adh} = 0.1 \mu\text{m}$ is already above the lap shear strength of the adhesive, so the adhesive will probably delaminate from the silicon spacer and the piezo will be held by the adhesive alone.

The above results show that the stroke is relatively insensitive to variations in the fabrication process, while the adhesive stress has a higher sensitivity. For some parameter combinations the adhesive stress can decrease below the limit of 1 MPa, but in most cases a delamination of the support spacer can be expected. The silicon stress remains well below the limit of 500 MPa in all cases. Overall it is found that positively tapered etch profiles are considerably less sensitive to variations in h_{spc} , because they are compensated by equal variations in w_{spc} . Across the calculated ranges it was found that for untapered geometries (w_{spc} constant) the results lie approximately halfway between the positive and negative taper results.

5.5 Conclusions

We have used FEM analyses to study the optimum dimensions of a flexible support for adhesive bonding between a valve chip and a piezoelectric actuator.

Figure 5.13: Variation of adhesive stress $v(\sigma_{adh})$ (above) and silicon stress $v(\sigma_{Si})$ (below), as a function of variations in h_{spc} and w_{adh} at selected values of $t_{adh} = \{0.1 \mu m, 1 \mu m, 10 \mu m\}$. Note that stress variations are calculated with respect to the stress at 0 % variation.

The studied design uses a narrow spacer to define the bonding thickness of a wide layer of highly flexible adhesive. We vary the physical dimensions of the support to maximize the net actuator stroke and minimize material stresses. Good performance is found for spacer height $h_{spc} = 200 \mu m$, spacer width $w_{spc} = 20 \mu m$

and total support width $w_{\text{sup}} = 520\mu\text{m}$. At these dimensions the net stroke is calculated to be $> 9.4\mu\text{m}$, which is sufficient even for a normally closed valve.

The maximum silicon stress remains low at 113 MPa, but the thin adhesive layer is stressed at several megapascals which is on the order of its rated lap shear strength. This can be expected to lead to delamination at the support spacers, after which the actuator will be held in place by the thick adhesive only. In this case adhesive stress is reduced to 0.3 MPa and net stroke is reduced to $> 8.7\mu\text{m}$, which are both within the demanded range. This shows that the flexible spacer support design is a potentially viable solution for top-mounting a piezoelectric bimorph on a silicon chip.

A parameter variation study is done to determine the sensitivity of the performance to inaccuracies in the fabrication process. The stroke is relatively insensitive to variations, although a decrease up to -20% is found in some extreme cases. Better performance is possible for higher and narrower spacers, but this makes the etching processes more challenging. Mechanical stresses vary across a larger range, but do not vary enough to alter the conclusions above. This means the design is relatively robust with regard to fabrication tolerances.

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6

Proportional control valves integrated in SiN surface channel technology

6.1 Introduction

Proportional fluid control principally requires two things: A device capable of monitoring the exact flow through a system, and a means of influencing the flow if it differs from the desired value. In the ideal case, these two elements would be integrated into a single device or a single chip. This would simplify fabrication and assembly processes, improve yields, allow the realization of more complex fluidic networks, and reduce internal volumes and therefore shorten the response time.

Only few publications in literature explore the possibility of integrating a small-scale proportional control valve with a flow sensor. A limited level of integration is obtained when separate valve and sensor chips are joined into a combined fluidic system, such as presented in [1–3]. The downside of this approach is the unavoidably large internal volumes in the fluidic interconnects between the separate devices, and the increased complexity of the assembly. True integration requires the realization of valve and sensor in a single chip. Some solutions have

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been reported, applying either thermal mass flow sensors [4–6] or (differential) pressure sensors [7, 8]. However, despite their advantage of on-die integration, most realized devices have very limited sensing resolution on the order of a few percent of full range.

In recent work a combined thermal and Coriolis-based microsensor has been demonstrated for mass flows of both liquids and gases, capable of accurately measuring flow across five orders of magnitude [9]. The ability to directly measure mass flow independent of fluid composition makes this sensor very suitable for precise dosing systems, if it is combined with a proportional control valve. The specifications of such a control valve would include a flow range up to 1.2 g h^{-1} to match the current sensor range, and operation at pressures on the order of 1 bar.

In this chapter we present the design, realization and characterization of two types of proportional control valves, which are compatible with the fabrication of the micro-Coriolis flow sensor. This allows on-die integration of both components, eliminating the need for wafer bonding steps or fluidic interconnects and enabling realization of a proportional mass flow controller on a single chip.

6.2 Analysis

6.2.1 In-plane and out-of-plane valve designs

The fabrication process of the micro-Coriolis mass flow sensor, presented in [10], is based on the silicon nitride surface channel technology first described in [11] but applies a silicon on insulator (SOI) wafer instead of a regular silicon wafer. The valves reported in this paper are realized in the same SOI-based surface channel technology (SCT). Figure 6.1 shows an artist impression of the two SCT microvalve designs. Both consist of a translating silicon plate with a radial flow channel between valve plate and valve seat. The out-of-plane design (figure 6.1a) places the valve plate directly above a through-wafer fluidic inlet or outlet. Moving the valve plate up and down then controls flow through the radial channel, between the inlet/outlet and a surface channel.

The in-plane design (figure 6.1b) allows flow control from one surface channel to another surface channel. Similar to the out-of-plane design it consists of a large outer cavity with a valve plate in the center. Fluid flows from a surface channel into the outer cavity, and then proceeds through the radial channel between the valve plate and seat. Contrary to the out-of-plane design the flow does not exit the chip, but instead flows into an inner cavity inside the valve plate. This inner cavity is connected to three 'outflow' surface channels that lead through the valve plate, through the outer cavity, back to surface channels outside of the valve.

Figure 6.1: Artist impression and operating principle of the two surface channel technology (SCT) microvalves: (a) Out-of-plane design, (b) in-plane design. Both valves regulate flow using a vertically translating valve plate, creating a radial flow channel between the plate and the valve seat. Gas flow is indicated in blue (in) and yellow (out).

To obtain a single, symmetric point of actuation, a central plate is added at the center of the inner cavity (not shown in figure 6.1b). It is connected to the ring of the valve plate by three 'spokes'. Figure 6.2a shows a top view of the full valve plate shape.

Since flow does not leave the surface channel network, the in-plane design allows on-chip routing of flow between several fluidic components. In contrast, the out-of-plane design must be placed above a chip inlet/outlet and can regulate flow between that inlet/outlet and other on-chip fluidic components. The out-of-plane design also offers a larger flow range, low leakage and smaller device footprint.

6.2.2 Laminar flow model

To obtain an estimation of the resulting flow through both valves as a function of separation and pressure, we begin with the laminar flow model for purely radial gas flow as described in [12]. Defining a flow resistance as the ratio between volume flow and pressure drop, we can derive an equation for the radial channel resistance:

Figure 6.2: (a) Top view of the valve plate structure of the in-plane valve design, showing the central plate connected by three spokes to the outer ring. (b) Mechanical model of a gas pressure P_{gas} pushing against a compliant membrane and valve plate, both modeled as linear springs.

$$R_{radial} = \frac{3\mu \ln\left(\frac{r_2}{r_1}\right)}{4\pi\left(\frac{S}{2}\right)^3}. \quad (6.1)$$

Where r_1 and r_2 are the inner and outer radii of the radial channel, S is the separation between the valve plate and seat, and μ is the dynamic viscosity of the gas (see figure 6.4 for an illustration of the critical physical parameters).

The radial flow model of equation 6.1 is best suited to the out-of-plane design, since the valve plate and the fluidic inlet form a purely radial geometry. The in-plane valve has a more complex geometry and therefore demands a modification of the model. First of all the model assumes a rigid valve plate, so that the separation between plate and seat is considered equal everywhere underneath the plate. In the case of the in-plane design the valve plate is etched to contain an inner cavity, which lowers the stiffness significantly. In particular, a finite stiffness exists between the center and the outer ring of the valve plate (see figure 6.2a). This means the displacement at the actuation point (central plate) is not necessarily equal to the change in separation of the radial channel (outer ring).

We can determine the actual separation of the radial channel using the mechanical model shown in figure 6.2b. The fluid pressure underneath the outer cavity membrane pushes the silicon ring upward, while an actuator enforces a position at the central silicon plate. Modeling the combined compliance of the valve plate structure and the cavity membrane as a linear spring with stiffness $K_{valve} = K_{plate} + K_{membrane}$, the actual valve separation S_{actual} can be calculated as a function of gas pressure P_{gas} :

$$S_{actual} = d_{act} - \frac{P_{gas} \cdot A_{membrane}}{K_{valve}}. \quad (6.2)$$

Here A_{membrane} is the total surface area of the outer cavity membrane and d_{act} is the displacement of an actuator at the center of the valve plate. Using equation 6.2 we can fit the practical behavior of a realized microvalve to the theoretical model of equation 6.1, using K_{valve} as fitting parameter.

Flow between the inner and outer cavities of the in-plane valve ideally follows a radial pattern. However, as is visible in figure 6.4, the outer cavity contains three outflow tubes that divide the outer cavity into three compartments. These three tubes are necessary for defining the degrees of freedom of the valve plate. Outside of the valve structure, these three tubes are combined again into a single channel.

Due to constraints in the fabrication process, the separation between the outflow tubes and the cavity floor is on the order of a few micrometers. This leads to a relatively large flow resistance between each of the three outer cavity compartments. This means that the radial flow will proceed almost entirely from a single compartment, where the inlet surface channel is connected (area 1 in figure 6.4). Due to the complex geometry it is very difficult to create an exact analytical model for the flow in this system, but an estimation can be obtained if we neglect all flow coming through areas 2 and 3, and assume purely radial flow between area 1 and the inner cavity. This means that only one third of the perimeter of the radial channel is used for flow, increasing the effective flow resistance by a factor of three:

$$R_{\text{effect,ip}} \approx 3R_{\text{radial}}. \quad (6.3)$$

The valves are connected to surface channels with a semi-circular cross-section, which lead away from the valve to the inlets and outlets of the microfluidic chip. Using the Hagen-Poiseuille equation we can estimate the flow resistance of a surface channel R_c to be:

$$R_c = \frac{128\mu L_c}{\pi d_h^4} \quad \text{with} \quad d_h = \frac{4A}{P}. \quad (6.4)$$

Where L_c is the channel length, d_h is the channel's hydraulic diameter, A is the cross-sectional surface area and P is the cross-sectional perimeter. The expected values of the cross-sectional parameters are estimated based on [10] and listed in table 6.1. The surface channels have an estimated hydraulic diameter of $d_h = 52\mu\text{m}$. The outflow channels of the in-plane valves are narrower, with a hydraulic diameter of $d_h = 26\mu\text{m}$.

The validity of the Hagen-Poiseuille equation is verified using finite element analyses in COMSOL Multiphysics® v.4.3a, using a weakly compressible, laminar flow model with a no-slip condition at the channel walls. The highest flow

Table 6.1: Estimated, actual, and best fit values for the critical valve parameters.

Parameter	Variable	Estimated / Designed value	Actual / Fit value
<i>General</i>			
Membrane width	w	200 μm	200 μm^{a}
Membrane thickness	t	3.5 μm	–
Surface channels:			
Hydraulic diameter	d_{h}	52 μm	59 μm^{b}
<i>Out-of-plane valve</i>			
Radial channel:			
Inner radius	r_1	175 μm	180 μm^{a}
Outer radius	r_2	360 μm	350 μm^{a}
Separation at rest	S_{rest}	2 μm	2.8 μm^{a}
Minimum separation	S_0	–	1.1 μm^{b}
<i>In-plane valve</i>			
Radial channel:			
Inner radius	r_1	205 μm	200 μm^{b}
Outer radius	r_2	250 μm	265 μm^{b}
Separation at rest	S_{rest}	2 μm	4.3 μm^{a}
Minimum separation	S_0	–	0.9 μm^{b}
Outflow channels:			
Hydraulic diameter	d_{h}	26 μm	25 μm^{b}
Length	l_{outflow}	1 mm	1 mm ^a
Valve plate stiffness	K_{valve}	–	120 kN ^b

^a Measured value.^b Best fit value.

velocities occur in the out-of-plane valve, since it supports the highest pressures. The simulation results show that laminar, viscous flow conditions apply, with the Reynolds number remaining below 5 in all cases and the Knudsen number on the order of $Kn \approx 2 \cdot 10^{-3}$. The flow can be approximated as incompressible (Mach number < 0.3) up to a pressure of at least 400 mbar in the worst case (all pressure drop across the surface channels, no pressure drop across the valve). At higher pressures the accuracy of the model will decrease since the flow velocities start

approaching the speed of sound.

We can add the resistances from the radial and Poiseuille flow models to arrive at the total flow resistance of the valves. Assuming an average density ρ_{avg} across the flow systems then leads to an approximate expression for the mass flow Q_{oop} (out-of-plane) and Q_{ip} (in-plane) [13]:

$$\begin{aligned} Q_{\text{oop}} &= \frac{P_{\text{in}} - P_{\text{out}}}{R_{\text{radial}} + R_{\text{c}}} \cdot \rho_{\text{avg}} \\ Q_{\text{ip}} &= \frac{P_{\text{in}} - P_{\text{out}}}{R_{\text{effect,ip}} + R_{\text{c}}} \cdot \rho_{\text{avg}}, \end{aligned} \quad (6.5)$$

with

$$\rho_{\text{avg}} \approx \frac{P_{\text{in}} + P_{\text{out}}}{2P_{\text{out}}} \cdot \rho(P_{\text{out}}). \quad (6.6)$$

Where P_{in} and P_{out} are the inlet and outlet pressure and $\rho(P_{\text{out}})$ is the density at the outlet pressure. Note that the pressure drop across the chip's fluidic inlets and outlets is neglected, as their diameters are much larger than the width of the surface channels.

6.2.3 Valve dimensions

For the out-of-plane design, the optimal inner and outer radii of the radial channel (r_1 and r_2 in equation 6.1) are determined based on the required flow range of 1 g h^{-1} . Since the valve plate can move both up and down from its rest position ($S_{\text{rest}} = 2 \mu\text{m}$, defined by the thickness of the BOX layer and nitride layer deposition), it is assumed that the maximum valve separation equals at least $4 \mu\text{m}$. Using this separation, a mass flow of 1 g h^{-1} can be achieved when $r_1 = 175 \mu\text{m}$ and $r_2 = 360 \mu\text{m}$ (see table 6.1). Note that the inner radius r_1 determines the radius of the chip's fluidic in/outlet.

The in-plane valve design contains narrow outflow channels, which have a higher flow resistance than the other surface channels. This limits the maximum flow through the valve, so to counteract this the radial channel is shortened to $r_1 = 205 \mu\text{m}$ and $r_2 = 250 \mu\text{m}$. At these dimensions, the flow at $4 \mu\text{m}$ separation and 1 bar is expected to be 0.5 g h^{-1} .

We used finite element analyses in COMSOL Multiphysics® v.4.3a to derive optimal dimensions for both valve designs. First the width of the (outer) cavity membrane was determined using the axisymmetric model shown in figure 6.3a. The membrane thickness $t = 3.5 \mu\text{m}$ is a constraint set by the fabrication process. Silicon nitride is used as material for the membrane, and polysilicon is used to approximate the single-crystalline silicon bulk material. The valve plate is

Figure 6.3: (a) Axisymmetric model used for FEM analysis of membrane stress (rotational symmetry axis on the right). (b) Resulting maximum von Mises stress as a function of membrane width w and gas pressure P_{gas} when the valve is closed ($d_{\text{plate}} = 2\mu\text{m}$).

pushed down with a $2\mu\text{m}$ prescribed displacement, while a distributed load acting on the membrane is used to model the gas pressure. Figure 6.3b plots the maximum von Mises stress in the structure when closing the valve, as a function of membrane width w . A minimum is found at $w = 200\mu\text{m}$. At smaller widths the increasing membrane stiffness is the dominant cause of stress. At larger widths the influence of the gas pressure under the membrane becomes dominant, deforming the membrane beyond the prescribed displacement of the valve plate. At $w = 200\mu\text{m}$ and 1 bar pressure the calculated stress is approximately 300 MPa, which is well below the fracture stress of silicon nitride [14].

For the in-plane microvalve we also analyzed the required length of the flexible outflow tubes. It is found that a minimum length of 1 mm is required for a maximum stress of 300 MPa. Since the outflow tubes need to be suspended in the outer cavity membrane, this leads to the design shown in figure 6.4. Note that most of the outer cavity is formed by the membranes supporting the outflow tubes. This leads to a large total surface area of the outer cavity in the in-plane valve, compared to the out-of-plane valve.

At low separations the flow resistance is determined mostly by the radial channel, whereas at high separations the flow is limited by the static flow resistance of the surface channels (see equation 6.5). The small hydraulic radius of the outflow

Figure 6.4: Design of the out-of-plane (left) and in-plane (right) valves, illustrating the dimensions listed in table 6.1. Note that the outflow tubes in the in-plane design separate the outer cavity in three compartments.

channels increases the static flow resistance of the in-plane valve design, which means the total flow supported will be lower than in the out-of-plane design.

6.3 Fabrication

Figure 6.5 shows a simplified process flow of the fabrication. A more detailed description can be found in [10]. We start with a $\langle 1\ 0\ 0 \rangle$ -oriented silicon on insulator (SOI) wafer with a 50 μm thick device layer, a 400 μm handle layer and a 5 μm buried oxide (BOX) layer. First the fluidic inlets and outlets are created in the handle layer using deep reactive ion etching (DRIE) of silicon (figure 6.5a). The surface channels and cavities are then created in the device layer using isotropic etching of silicon through a silicon nitride slit-pattern mask (b). By varying the density of this slit pattern, it is possible to create both deep and shallow structures in a single etch step (see figure 6.6). To release the valve structures we selectively etch the BOX layer using first a liquid phase HF etch, followed by a vapor phase HF etch to prevent stiction by capillary forces (c). Using LPCD of silicon nitride, the slit patterns are then closed to seal the surface channels, form the channel walls and create a closed fluidic system (d).

When the valve is closing, the outflow channels in the in-plane valve need to bend down. To reduce their bending stiffness, the silicon around them is removed in a final isotropic etch (figure 6.5e). Note that the nitride shell surrounding

Figure 6.5: Process flow of the SCT microvalves: (a) DRIE of inlets/outlets; (b) Isotropic etch through nitride slit pattern; (c) HF etching of the buried oxide; (d) Sealing of nitride membranes with LPCVD nitride; (e) Isotropic etch to release outflow channels.

the outflow tubes is grown inside the $5\mu\text{m}$ BOX layer, so there is only a few micrometers clearance beneath them. This is the cause of the three separated compartments in the outer cavity.

The realized valve structures are shown in figures 6.7, 6.8 and 6.9. The in-plane valve (figure 6.8) consists of a hollow valve plate, suspended by three outflow channels and the outer cavity membrane. The three outer cavity compartments are clearly visible. Three cross-sectional drawings are added to illustrate the three-dimensional shape of the valve. Cross-section A-A' is cut along an outflow channel (to the left) and the inflow channel (to the right), also cutting through both cavities and the valve plate. Cross-section B-B' is cut perpendicular to the outer cavities and the outflow channel. Cross-section C-C' is cut perpendicular to the inflow

Figure 6.6: SEM picture showing the effect of nitride slit patterns on etch depth. Note that this was an etching test structure for the in-plane valve, so no radial channel or outflow channels were created.

Figure 6.7: Photograph of the realized microfluidic chips, showing the out-of-plane design on the left and the in-plane design on the right. Both chips are 7.5 mm x 7.5 mm in size.

surface channel.

In the close-up image of figure 6.8b it is visible that the final silicon etch in the fabrication process (figure 6.5e) has also etched away part of the valve plate. Specifically, the outer ring has been partially removed (visible as light grey cavities). This decreases the stiffness of the valve plate, increasing the influence of K_{valve} in equation 6.2).

Figure 6.8: (a) Microscope image of the realized in-plane microvalve, showing the silicon valve plate (dark grey) in the center of a sealed silicon nitride membrane. The inflow surface channel is visible at the right (light grey). The thin-film metal markings (white) are used for actuator alignment.. (b) Close-up of the valve plate structure. (c) Cross-sectional drawings, cut along the dashed lines in (a).

The out-of-plane design (figure 6.9) shows a solid valve plate in the center of a circular silicon nitride membrane. Fluid enters the cavity through a surface channel (light grey at the right side of the image) and flows beneath the valve plate (dark grey) to the outlet at the bottom of the chip (not visible).

Figure 6.9: Microscope image of the realized out-of-plane microvalve.

Figure 6.10: Measurement scheme used for mechanical and fluidic characterization of the SCT microvalves [13].

6.4 Characterization

Fluidic and mechanical characterization of the realized microvalves is done using the scheme illustrated in figure 6.10, based on the setup described in chapter 3. An external piezoelectric actuator applies a controlled displacement up and down, with a loadcell connected to measure the force being exerted. The valve chip itself is placed in a PMMA fluidic chipholder with Viton micro o-rings (1 mm inner diameter and 0.74 mm cord thickness) sealing the fluidic connections.

Backed by a dry nitrogen gas source, a gas pressure controller (Bronkhorst P-602CV) is connected to the inlet of the fluidic chipholder, and a gas mass flow meter (Bronkhorst F-111B) is connected to both the inlet and the outlet. When fully closing a microvalve in the chipholder, the measured outlet flow reduced to zero while the inlet flow remained on the order of 2.5 mg h^{-1} at 200 mbar

pressure. This means there is a leakage inside the chipholder, probably caused by non-optimal compression of the o-rings. This problem is probably caused by the increased influence of fabrication tolerances of the chipholder when smaller o-rings are used, and might be solved by using adhesive bonding instead of o-rings between the chip and chipholder. Since the outlet flow goes to zero with full valve closure, it is clear that the pressure drop across the fluidic system occurs mostly inside the microfluidic chip. Most of the system leakage therefore occurs at the inlet side of the chipholder, which is why we chose only to use the outlet flow measurements.

6.4.1 Flow properties

Figure 6.11 shows the measured mass flows as a function of pressure and separation of the realized out-of-plane microvalve. All measurement points are taken after mechanical and fluidic stabilization (two seconds), and then averaged over three seconds of flow. The standard deviations of these averages were less than 0.15% of full scale. Fitting the analytical model of equation 6.5 to the measurements a good fit is found using the values shown in table 6.1. Measured values were used instead of fit values where available. The fitted hydraulic radii correspond well with the values estimated in section 6.2.2. The values for the radial channel dimensions were estimated based on the lithographic mask and are measured on actual, realized devices. Note that the length of all surface channels is measured directly from the mask design, as they do not change significantly during fabrication.

As is visible in figure 6.11, it is found that the valve appears to have a minimum separation of $1.1\text{ }\mu\text{m}$. It is likely that some contamination or particles exist between the valve plate and seat, preventing the plate from closing fully. Since the angle between the actuator movement and the valve plate could not be precisely controlled, it is also possible that a tilted plate is causing an apparent offset displacement in the flow profiles.

The maximum flow supported by the out-of-plane microvalve at 600 mbar pressure is approximately 1250 mg h^{-1} . Leak flow of the out-of-plane microvalve is below the detection threshold of 0.05 mg h^{-1} at pressures up to 1 bar.

The characterization results of the in-plane valve are shown in figure 6.12, again fitted to the flow model of equation 6.5 using the fitting parameters listed in table 6.1. A minimum separation of $0.9\text{ }\mu\text{m}$ is found and the maximum flow is $> 70\text{ mg h}^{-1}$ at 200 mbar. The best fit for the combined plate stiffness is found to be $K_{\text{plate}} = 120\text{ kNm}^{-1}$. At this stiffness a gas pressure of 200 mbar would bend the valve plate approximately $0.5\text{ }\mu\text{m}$ upwards. This leads to an observed leak

Figure 6.11: Dry nitrogen gas flow through the out-of-plane valve, as a function of valve separation and gas pressure. Measured values (points) are plotted with the fitted analytical model (lines). Note that the gas pressure pushes the valve open further than the minimum expected $4\mu\text{m}$.

Figure 6.12: Measured dry nitrogen gas flow through the in-plane valve (points), plotted with the fitted analytical model (lines), as a function of valve separation and gas pressure.

flow of 9.0 mg h^{-1} at a pressure of 200 mbar, which is 13 % of the measured flow range. It is therefore important to increase the stiffness of the valve plate in future designs. This can be done by increasing the width of the three spokes and with better control over the isotropic silicon etch of the outflow channels to prevent etching of the outer ring (see figure 6.8b).

6.5 Conclusion

We have presented two microvalve designs realized entirely in a silicon nitride surface channel technology. This technology has been used to realize high precision and high dynamic range micro-Coriolis mass flow sensors [9], which makes our valves well suited to on-die integration of both components to create a single-chip proportional mass flow controller. We have predicted the fluidic behavior of both valve designs using analytical laminar flow models and have characterized the mechanical and fluidic properties of the realized devices. The analytical model was in good agreement with the measurements.

The in-plane microvalve offers fluidic control between two surface channels, allowing on-chip flow routing between several fluidic components. It allows flows of approximately 70 mg h^{-1} at 200 mbar, which makes it suitable for applications demanding very low dosing ranges. The in-plane design revealed a considerable leak flow, up to 13 % of the measured flow range. This leakage is caused by the mechanical compliance of the valve plate, which makes it possible for the valve to be pushed open by the inlet gas pressure. To solve this problem, future designs will need to focus on increasing the valve plate stiffness.

The out-of-plane design can control flow between a surface channel and a fluidic chip inlet or outlet, which allows a geometry with a smaller chip footprint than the in-plane design. It allows up to 1250 mg h^{-1} of gas flow at 600 mbar pressure, which is the full flow range of the micro-Coriolis flow sensor. Leak flow through the out-of-plane valves is below the detection threshold of 0.15 mg h^{-1} , making it well suited to applications that require complete closure of fluidic channels.

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7

Conclusions and outlook

7.1 Conclusions

This thesis describes the design and realization of four separate MEMS-based proportional control valves for minute gas flows. The four valve designs can be divided into two main categories, being either based on silicon-on-insulator (SOI) technology or based on surface channel technology (SCT). The first group focuses on obtaining a proportional flow controller using simple, straight-forward fabrication processes with a small number of process steps. The second group demonstrates two proportional valve designs in an already existing fabrication process, to allow integration with existing components such as flow sensors.

7.1.1 SOI-based microvalves

Two novel SOI-based valve designs have been presented and realized that allow proportional control of gas flows in and through a single-wafer device. The use of silicon-on-insulator wafers as a basis for fabrication allows the valves to be created without the need for costly wafer bonding steps, and the two and three-mask fabrication processes are of great advantage with regard to fabrication yields. The first valve design supports gas flow with inlet and outlet at opposite sides of the

chip. While this may be beneficial in some applications, it makes it more difficult to mount an actuator on the device without blocking the flow paths. The second design therefore defines both inlets and outlets at the bottom side of the chip, making the fluidic system isolated from the actuation points at the top side.

Both valve designs have been characterized mechanically and fluidically. Dry nitrogen gas flows up to 13 g h^{-1} at a pressure of 500 mbar have been demonstrated, with an expected maximum of 25 g h^{-1} at 1 bar, using a maximum actuation voltage of 100 V. The actuator supports voltages up to 180 V, so higher flows should be obtainable in this design. Leak flows as low as 0.1 mg h^{-1} at 1 bar ($\approx 5 \cdot 10^{-4} \text{ mbar l s}^{-1}$) have been obtained. This may be further improved if particle contamination of the microvalve assembly can be prevented, for example using microfilters at the inlet ports.

Piezoelectric actuation is applied in this research because of its relatively high work density, high speed and low power consumption. In contrast to many existing solutions, we choose to use bimorph benders instead of stacked piezos, as the latter require much larger thicknesses to achieve the required stroke. The two main challenges in applying piezoelectric actuators in a micromechanical system are solving the inherent hysteresis effects and creating a reliable and reproducible method of interfacing.

The first problem can be solved by controlling the piezo in feedback with a displacement sensor. We have demonstrated the application of such a sensor in a micro control valve by using highly doped SOI wafers and sensing the electrical capacitance between the device and handle layers. It is shown that the capacitive displacement sensor can correct the flow hysteresis from 300 % to less than 5 %. The sensor bandwidth is currently limited by the readout electronics at 700 Hz.

The challenge of interfacing a piezoelectric bimorph actuator with the microfluidic chip is solved by adding a flexible silicone rubber support layer between the piezo and the silicon chip. The flexible support allows the piezo to bend and contract so that sufficient stroke is maintained at the piezo center, while still providing enough vertical stiffness to keep the actuator in place and prevent the gas pressure from lifting the piezo up and opening the valve. Using this technique an actuated micro control valve is realized with a high mechanical bandwidth of 8 kHz and a frequency-independent response up to 3 kHz. The total physical dimensions of this device are $17 \text{ mm} \times 17 \text{ mm} \times 2.4 \text{ mm}$.

An alternative to the silicone rubber support is also presented consisting of a thick layer of flexible adhesive between the bimorph and silicon surfaces, with silicon spacers created on the microfluidic chip defining the adhesive thickness after bonding. FEM analyses are used to derive the optimum dimensions of the support, maximizing the net stroke and minimizing the stress in the silicon and

the adhesive. A set of physical dimensions is found that provide good performance with $> 8.7 \mu\text{m}$ stroke and stress below the determined maximum. The design is found to be relatively robust with regard to fabrication tolerances.

7.1.2 SCT-based microvalves

We have also presented two control valves realized in a silicon nitride surface channel technology (SCT) fabrication process. The addition of proportional flow control devices to the SCT-based fluidic toolbox allows creation of complex fluidic systems fully integrated on a single chip. The valve designs apply a combination of dense and sparse nitride slit patterns to obtain deep and shallow cavities in the silicon, respectively. Finite element analysis is used to derive the optimum dimensions for both designs, and analytical and numerical models are used to predict the fluidic behavior.

The out-of-plane design allows active flow control from a chip inlet or outlet to a fluidic channel embedded in the silicon surface. Placing the valve plate directly above a chip inlet or outlet simplifies the fabrication process, by requiring only a single, deep cavity to be created in the silicon surface. The design therefore has a small device footprint of $< 1.2 \text{ mm}^2$. The in-plane design allows fully on-chip flow control between any two surface channels, without the need to be placed at a chip inlet or outlet. This does however increase the device complexity and increases the footprint to approximately 5 mm^2 .

Both designs are fabricated and characterized, and a good fit is obtained between the measured nitrogen gas flow profiles and the predicted flow models. The out-of-plane microvalve has a very low leakage flow ($< 0.05 \text{ mg h}^{-1}$ at 1 bar) and the flow range exceeds 1250 mg h^{-1} at 600 mbar. The in-plane design supports flows up to $> 70 \text{ mg h}^{-1}$ at 200 mbar. It is found that the outer ring of the in-plane valve plate is partially removed during one of the silicon etching steps, preventing full closure of the valve. This leads to a leakage flow of 9.0 mg h^{-1} at a pressure of 200 mbar. This problem can be solved by adjusting the etch timing of the device layer. The isotropic etch should be long enough to fully release the inflow tubes, but stop before the valve plate is reached.

7.2 Outlook

The obtained performance of the SOI-based microvalve designs makes them very well suited for the aimed application of ambulant blood pressure waveform monitoring. The combination of capacitive displacement sensing with bimorph piezoelectric actuation allows for high-speed, high-precision flow control with

low power usage. The flow rate of 7.5 g h^{-1} at 300 mbar is half of the flow range requirement of the application, but if the actuation voltage can be increased to the rated maximum of the piezo, the requirement should be met even at lower pressures. Encapsulating the high voltage contacts in an insulating layer with higher dielectric strength than air will probably be sufficient to prevent electrical discharges as observed in the prototype.

An important shortcoming in the design presented in chapter 4 is the individual placement and manipulation of components, which is costly and error-prone. It is therefore essential to keep the number of manual processing steps to a minimum. Elimination of the silicone rubber support layer between the bimorph and the silicon would reduce the manual assembly steps significantly, especially if electrical contact to the piezo can be made through the silicon device layer. The use of flexible adhesive bonding has been demonstrated to be a potentially viable solution, but it has not yet been tested in practice. Particularly the problem of local adhesive delamination at the support spacers may lead to non-linear effects, since the stiffness of the support as a whole becomes unequal for actuation in positive and negative directions. Such effects may also be frequency-dependent. The design also complicates the fabrication process somewhat, as the three-tiered etching of the device layer requires the use of an extra mask.

Another remaining challenge is reducing the relatively large chip dimensions. Although the valve itself is less than 4 mm in diameter, the bimorph actuator requires the valve chip to be much larger. This can only be remedied by replacing the bimorph by a smaller actuator with identical or better performance. Since the piezo bimorph used in this work does not have a surplus in either stroke or force, it is currently unclear how this may be accomplished.

The SCT-based microvalves have been demonstrated to be suitable for proportional, on-chip flow control. Their designs are tailored to match the existing calorimetric and Coriolis-based mass flow sensor, with a dynamic resolution of 1:10000 and a flow range of 1.2 g h^{-1} . The out-of-plane valve design supports flows of this magnitude. However, to be suitable for the aimed application of gas chromatography, both flow sensor and valve will need to be scaled up. Flow through the valves becomes dominated by the static flow resistance of the surface channels at valve separations of only a few micrometers. The greatest gain can therefore be achieved by increasing the surface channel diameters. It is however also possible to reduce the dynamic flow resistance of the valve. The simplest way to do this would be to increase the thickness of the buried oxide, but this would also increase the required actuation stroke. A second option is to decrease the radial width of the valving channel underneath the valve plate, but this would make the flow more sensitive to changes in valve separation and may therefore

reduce dynamic control precision.

Actuator integration has not yet been achieved on the SCT-based microvalves. Given the fragility of the flexible silicon nitride membranes, manual assembly of components on these devices would be difficult to achieve. In the case of an external (piezo) actuator, it would be preferable to devise a mounting solution where only a droplet of adhesive material would come into contact with the valve plate. Care would need to be taken to avoid the valve plate from being significantly displaced during the bonding process, for example by shrinkage of the adhesive. It would also be challenging to accurately define the thickness of the adhesive layer, since the SCT process currently does not allow etching of structures in the device layer silicon.

It is possible to apply alternative forms of actuation, such as electrostatic attraction between the valve plate and seat or deposition of a thin-film piezoelectric layer on top of the device layer. Electrostatic actuation would only enable valve closure, so that reopening of the valve would depend on elastic strain energy and the fluid pressure. For thin-film piezoelectrics the challenge would be to devise a fabrication method that is compatible with the SCT process, and to deposit a thick enough piezoelectric layer to obtain sufficient stroke and force. This approach may be limited by the constraints of the existing surface channel technology.

Detailed fabrication process

A.1 Introduction

This chapter will describe in greater detail the fabrication process of the SOI-based microvalve presented in chapter 4. Practical aspects of the critical process steps are noted where necessary, and some details are briefly explained in context. The color scheme of the materials in the illustrations is as shown below.

Note that the fabrication process used for the SCT-based microvalves described in chapter 6 will be detailed in [1].

A.2 Silicon-on-insulator (SOI) wafer

1. Wafer selection

- Device layer (DL): $50 \pm 1 \mu\text{m}$ $\langle 100 \rangle$ -silicon
- Handle layer (HL): $400 \pm 10 \mu\text{m}$ $\langle 100 \rangle$ -silicon

- Buried oxide (BOX): $4 \pm 0.2 \mu\text{m}$ DL-grown thermal oxide
- Doping: P++ 0.005 – 20 $\Omega \text{ cm}$ (DL and HL)
 - *Buried oxide is grown on thinnest layer (DL) to reduce wafer warping*
 - *Very high doping is used to allow low-resistive contacting of capacitive displacement sensor*

2. Thermal oxidation

- Wafer cleaning, pre-furnace clean
- Wet thermal oxidation, 1 μm (1150 °C)
 - *Oxide layer is used as a buried mask for silicon etching*

3. Oxide patterning (buried DRIE mask)

- Coating, 3.5 μm positive photoresist
- Lithography, "DL Deep & Shallow" mask
- Oxide etching in buffered HF, 1 $\mu\text{m} \approx 17 \text{ min}$
 - *Use HDMS priming and 30 min hardbake of photoresist for BHF etching*
 - *Use RIE of oxide if high resolution is required*

4. Photoresist patterning (top DRIE mask)

- Stripping of photoresist using HNO_3
- Coating, 3.5 μm positive photoresist
- Lithography, "DL Deep only" mask

5. Etching of top DRIE mask

- RIE of device layer silicon
- BHARS STD recipe, $> 20\mu\text{m} \approx 8\text{ min}$
 - *The top DRIE mask defines deep trenches*

6. Etching of buried DRIE mask

- Stripping of photoresist using O_2 plasma
- Wafer cleaning, post-RIE clean
- RIE of device layer silicon
- BHARS STD recipe, $40\mu\text{m} \approx 12\text{ min}$
 - *The buried DRIE mask defines shallow areas and also continues etching of deep trenches down to the BOX layer*

7. Removal of buried mask

- Wafer cleaning, post-RIE clean
- Stripping of thermal oxide in 50 % HF, $1\mu\text{m} \approx 1\text{ min}$
 - *This will also partially etch the exposed buried oxide*

8. LPCVD of silicon-rich nitride

- Wafer cleaning, pre-furnace clean
- LPCVD of silicon-rich nitride (G4 SiRN), $1\mu\text{m} \approx 2\text{ h}$
 - *The nitride layer will protect the buried oxide in the final HF etch*

9. Etching of handle layer

- Bottom side coating, 3.5 μm positive photoresist
- Lithography, "Handle Layer" mask
- RIE of silicon nitride, Multilayer recipe, 1 $\mu\text{m} \approx 4 \text{ min}$
- DRIE of silicon, Pulsed C_4F_8 STD recipe, 400 $\mu\text{m} \approx 27 \text{ min}$
 - *DRIE timing is critical as notching effects will influence radial channel length and therefore subsequent HF release etch timing*

10. Course release etch

- Stripping of photoresist using O_2 plasma
- Wafer cleaning, post-RIE clean
- Wafer dicing and cleaning
- Etching of buried oxide in 50 % HF, 300 $\mu\text{m} \approx 4 \text{ h}$
- Gentle rinsing in DI water
 - *Wafers are diced before release etch to avoid valve contamination*
 - *HF etching time is critical, leave approximately 10 μm of oxide*
 - *Use stirrer to prevent HF etch product bubbles forming on inlets & outlets*
 - *Overetching can lead to sticking of the valve membrane (stuck membranes sometimes release after a number of hours)*

11. Fine release etch

- Etching of remaining buried oxide using vapour-phase HF
 - *VHF timing depends on amount of remaining oxide*

A.3 Glass support wafer

12. Wafer selection and preparation

- 500 μm borosilicate glass
- Coating, powderblasting foil, both sides
- Lithography, "Glass Top" and "Glass Bottom" masks

13. Powderblasting top side

- Powderblasting top side, > 300 μm
 - *Determine powderblasting speed and cross-sectional tapering using dummy wafers*

14. Powderblasting bottom side

- Powderblasting bottom side, > 200 μm
- Stripping of photoresist using Acetone
- Wafer dicing and cleaning
 - *Adjust required powderblast time to ensure full opening of flow channels*

A.4 Adhesive bonding

15. Applying adhesive

- Sample cleaning, Acetone and IPA
- Applying UV-curable adhesive on top glass surface
 - *Adhesive bonding is done on individual chips since wafers are already diced*
 - *First roll out a small amount of adhesive on a clean wafer to obtain a thin layer of adhesive on the roller*

16. Bonding of silicon and glass chips

- Manual alignment and contacting of silicon and glass chips
- UV flood exposure through the glass chip in Spectrolinker, optimal crosslink program
 - *Chip alignment can be done manually by holding the glass chip above the silicon chip with tweasers*
 - *Place the (top-side down) silicon chip on a clean wafer surface during alignment to prevent damage to valve membrane*
 - *Release the glass chip just above the silicon surface and gently push down near the four corners to ensure good adhesive contact*

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Samenvatting

Nauwkeurige regeling van gas- en vloeistofstromen wordt steeds moeilijker naar mate systemen en instrumenten kleiner worden gemaakt. Kleinere fysieke dimensies maken het mogelijk kleinere stroombereiken aan te sturen, maar staan ook kleinere marges voor fouten toe. Betrouwbare en effectieve fabricage- en assemblageprocessen zijn daarom een eerste vereiste voor het succesvol aanwenden van microfluidische systemen in praktische toepassingen. Dit proefschrift beschrijft de resultaten van voortdurend onderzoek naar het realiseren van geminiaturiseerde, proportionele regelventielen voor hele kleine gasstromen. In dit onderzoek concentreren wij ons op MEMS-gebaseerde fabricageprocessen, omdat die het mogelijk maken om geminiaturiseerde systemen te produceren met hoge precisie en hoog parallelisme. Dit leidt tot hoger fabricagerendement, verbeterde betrouwbaarheid en een hogere mate van integratie.

We tonen vier ventielontwerpen welke onderverdeeld kunnen worden in twee hoofdcategoriën, zijnde gebaseerd op silicium-op-isolator (SOI) technologie ofwel gebaseerd op oppervlaktekanaals-technologie (SCT). De eerste groep focust op het creëren van een proportionele stroomregelaar met een eenvoudig en degelijk fabricageproces met een klein aantal processtappen, voor gebruik in een draagbaar bloeddruk golfvorm meetsysteem (BGM). Twee ventielontwerpen op basis van een enkele wafer worden gepresenteerd, met beide een ingebouwde capacatieve sensor voor de ventielverplaatsing welke gebruikt kan worden om actuatorhysterese te corrigeren en regelprecisie te verbeteren. Stikstof gasstromen tot 13 g h^{-1} bij een gasdruk van 500 mbar worden bereikt in goede overeenkomst met analytische en numerieke modellen, met een lekstroom van minder dan 0.1 mg h^{-1} bij 1 bar. De maximale doorvoercapaciteit wordt geschat op 25 g h^{-1} bij 1 bar. Een volledig functioneel regelventiel wordt gedemonstreerd met de integratie van een geminiaturiseerde piezoelektrische bimorph actuator. Tijd-dynamische karakterisering toont aan dat het ventiel geschikt is voor hogesnelheidsregeling van gasstromen, met een mechanische bandbreedte van 8 kHz en een frequentie-onafhankelijke respons tot 3 kHz.

De tweede groep toont twee proportionele regelventielontwerpen in een al

bestaand (SCT) fabricageproces, om integratie met bestaande componenten zoals stromingssensoren mogelijk te maken. Het eerste ventielontwerp maakt stromingsregeling mogelijk vanuit een chipinlaat of -uitlaat naar een fluidisch kanaal ingebed in het siliciumoppervlak, met een regelbereik van $> 1250 \text{ mg h}^{-1}$ bij 600 mbar en een lekstroom van minder dan 0.05 mg h^{-1} bij 1 bar. Het tweede ventielontwerp ondersteunt kleinere stromen ($> 70 \text{ mg h}^{-1}$ bij 200 mbar), maar maakt ook stromingsregeling tussen twee of meer oppervlaktekanalen mogelijk. Een goede overeenstemming is gevonden tussen de gemeten stromingsprofielen en analytische modellen van beide ventielen.

Het wordt aangetoond dat de ventielen gericht op het BGM systeem erg goed geschikt zijn voor de beoogde toepassing, hoewel een kleinere bimorph piezo nodig zou kunnen zijn om de vereiste fysische afmetingen te behalen. De combinatie van capacitieve verplaatsingssensoren met bimorph piezoelektrische actuatie maakt snelle, nauwkeurige stromingsbesturing mogelijk met een laag energieverbruik. De SCT-gebaseerde microventielen demonstreren proportionele stromingsregeling op een chip, geschikt voor integratie met bestaande stromingssensoren. De ontwerpen zijn toegesneden op een bestaande hoge-resolutie massa-stromingssensor welke een beperkte doorvoercapaciteit heeft, maar de ontwerpen van zowel de ventielen als de sensor kunnen worden opgeschaald om grotere capaciteiten te bereiken.

Dankwoord

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Maarten Groen

Zwolle, november 2015

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Biografie

Maarten Groen werd op 18 september 1982 geboren in Amersfoort. Na de middelbare school is hij een jaar in het bedrijfsleven actief geweest, waarna hij in 2002 begon aan zijn bachelor- en masterstudie elektrotechniek aan de Universiteit Twente. Na een tussentijdse pauze in 2006 en 2007 heeft hij zijn studie afgerond in 2011 met een specialisatie in microsystemen en microelektronica. Bij de vakgroep NanoElectronics heeft hij zijn masteronderzoek uitgevoerd naar de elektron transporteigenschappen van geïsoleerde FePt nanodeeltjes.

Hij is hierna begonnen als promovendus bij de vakgroep Transducers Science and Technology, waar hij onderzoek deed naar proportionele regelventielen op microschaal.

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