

Microfabricated Active Valves for Liquid and Gas Handling

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**Microfabricated active valves for liquid and gas
handling**

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Le doyen:

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F. Zwahlen

Abstract

This study focuses on the design, fabrication and testing of microvalves developed to have good sealing properties and long lifetime. The most important characteristic of a valve is its tightness, which is related to the roughness of the surfaces which are in contact with each other, and the pressure applied on them. Because of the planarity of the materials used in microsystems technology, this fabrication approach should be suitable for valves. Nevertheless, very few micromachined valves have characteristics adapted to industrial products. The difficulty is that producing sufficient pressure on the moving part of the valve to guarantee the quality of the seal while maintaining a constant performance for a large number of cycles is not simple. These developments show that combining bulk deep reactive-ion etching (DRIE) and surface micromachining allows the fabrication of valves with characteristics adapted to specific applications.

Since gases have different properties than liquids, the valve channels and orifices have to be much wider for liquids than for gases to obtain comparable flow resistances. Therefore, two valves with completely different designs were developed, one for gases, the other for liquids. The gas valve was optimised to switch at high-speed flows under large pressures. The very high switching rate enables this valve to be actuated in pulsed width modulation in order to have a linear flow response. Applications for such a valve include: pilot valve to activate a pneumatic valve, gas chromatography, or micro satellite propulsion. Electrostatic force acting on a tantalum-silicon-nitride

membrane that closes a high-aspect-ratio hole actuates the valve. Tantalum-silicon-nitride, which presents an exceptional combination of mechanical and electrical properties, is used for the first time in a microsystems application.

The valve for liquid applications was designed to work with fluids, such human serum, that have an agglutination tendency. Previous studies showed that sliding valves, where the two parts moving relative to each other are constantly in contact, prevent agglutination better than other types of valves. Since frictional behavior is an important factor in a sliding valve, tribological studies have to be conducted for minimizing friction and wear. Therefore a test valve, which served simultaneously as a tribometer, was built for observing wear of coating layers under flow conditions. To have accurate measures, a design based on high-aspect-ratio bending beams suppressed friction in the guiding area, while producing a high normal force. Actuation in this case was provided by an external motor.

Recent progress in deep reactive-ion etching (DRIE) of silicon has enabled fabrication of the high-aspect-ratio structures essential to the achievement of the exceptional characteristics of these valves. For instance, both devices have structures smaller than 40 μm etched through a silicon wafer measuring more than 300 μm in thickness. Even if further developments are required to fabricate industrial valves based on these concepts, this study demonstrates in a concrete way that microsystems technology can be suited to fluidic applications previously associated only with the macroscopic world.

Résumé

Cette étude est focalisée sur la conception, la fabrication et le test de valves développées dans le but d'obtenir une bonne étanchéité et une longue durée de vie. La propriété recherchée la plus importante d'une valve est son étanchéité, qui est liée à la rugosité des surfaces en contact et la pression mécanique appliquée sur celles-ci. En raison de la planéité des surfaces utilisées dans la technique des microsystèmes, cette méthode de fabrication apparaît adaptée à la réalisation de valves. Néanmoins, actuellement, très peu de valves micro-usinées présentent des caractéristiques adaptées à des produits industriels. La difficulté technique principale réside dans la production d'une force normale suffisante pour obtenir une bonne étanchéité de la valve tout en ayant un comportement stable sur le long terme. Cette étude montre que la combinaison de micro-usinage de surface avec l'attaque profonde par plasma du silicium permet d'obtenir des performances adaptées à certaines applications.

Les gaz ayant des propriétés différentes des liquides, pour l'obtention de débits analogues, les canaux à l'intérieur de la valve doivent être beaucoup plus larges pour des liquides que pour des gaz. Ainsi, deux valves de conception complètement différentes ont été développées, l'une pour des liquides, et l'autre pour des gaz. La valve pour les gaz a été optimisée pour travailler à grandes vitesses et pressions de gaz élevées. Les très faibles temps de réponse de cette valve lui permettent d'être actionnée en modulation de largeur d'impulsion afin de varier linéairement le flux. Les applications d'une telle valve

comprennent notamment son utilisation en valve de commande dans des injecteurs pneumatiques, la chromatographie gazeuse, et la propulsion de micro satellites. Le principe de fonctionnement de cette valve est basé sur l'utilisation de la force électrostatique agissant sur une membrane en nitrure de tantale silicium afin d'obstruer un orifice de très faible diamètre. Le nitrure de tantale silicium, qui possède une exceptionnelle combinaison de propriété mécanique et électrique, est utilisé pour la première fois dans un microsystème fonctionnelle.

La valve pour liquide est destinée à des fluides présentant une tendance à l'agglutination, tels que le sérum humain. Des études ont montré, que les valves fonctionnant par glissement de deux surfaces en contact permanent, sont les mieux adaptées à ce genre d'utilisation. Pour réduire l'usure, les parties de la valve en contact sont recouvertes de couches apparentées au diamant. Ces couches étant mal connues, afin d'étudier leur friction, un tribomètre fonctionnant également en tant que valve a été micro-fabriquée. Sa conception est basée sur des barreaux flexibles en silicium ayant des proportions très élancées afin de permettre une grande souplesse dans une direction, et une grande rigidité dans l'autre. La structure particulière du dispositif permet de supprimer la friction dans les zones indésirables tout en ayant un guidage très précis. L'activation du système est réalisée par moteur électrique externe.

Des progrès récents de la technique de micro-usinage par plasma ont permis de réaliser des structures, ayant des proportions exceptionnelles nécessaires à l'obtention des caractéristiques de ces microvalves. Par exemple, ces deux valves ont des structures plus petites que $40\text{ }\mu\text{m}$ attaquées au travers d'un substrat en silicium de plus de $300\text{ }\mu\text{m}$ d'épaisseur. Même si d'autres études seraient nécessaires à la fabrication de valves industrielles basées sur ces concepts, ce travail montre que la technique de microfabrication peut être adaptée à la réalisation de composants fluidiques utilisés dans des applications macroscopiques.

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Nomenclature: TaSiN gas valve

Variables:

Symbol	Description	Unit
d	Distance or diameter	m
f	Displacement	m
y	Vertical position	m
h	Membrane height	m
w	Membrane width	m
l	Membrane arm length	m
S	Surface	m ²
k	Spring constant	N/m
p	Pressure	Pa
I	Geometrical moment of inertia	m ⁴
W	Energy	J
C	Electrical capacitance	F
Q	Volume flow rate	m ³ /s
ρ	Density	kg/m ³
T	Temperature	°K
m	Mass	kg
b	Damping constant	Ns/kg
t	Time	s
P	Power	W
freq	Frequency	Hz

Indices:

Symbol	Description
0	Initial
g ₀	Initial gap
g	Gap
me	Mechanical
el	Electrical
ga	Gas
i	Insulation layer
h	Hole diameter (orifice)

m	Membrane
y	Vertical direction
pot	Potential
eq	Equivalent
in	Inlet
ou	Outlet
w	Wire
c	Contact
t	Electrical track
pa	Parasitic
se	Security
p	Pull-in

Constants:

Symbol	Description	Value	Unit
ϵ_0	Permittivity in vacuum	$8.85 \cdot 10^{-12}$	s/(Ω m)
ϵ_{rSiO_2}	Silicon oxide relative permittivity	3.9	
$\epsilon_{rSi_3N_4}$	Silicon nitride relative permittivity	6	
E	Young modulus of Ta-Si-N	200	GPa
σ	TaSiN tensile stress	50-100	Pa
ν	Poisson's coefficient, value adopted for TaSiN	0.3	
M_{N_2}	Nitrogen specific mass	28	g/mol
ρ_{N_2}	Density of nitrogen at 293 °K	1.25	kg/m ³
γ	C_p/C_v , value for diatomic gases	1.4	
R	Perfect gas constant	8.31	J/(mol°K)
α	Head loss coefficient for N ₂	0.83	
η_{N_2}	Nitrogen viscosity constant	$1.66 \cdot 10^{-5}$	N/(m ² s)
ρ_{N_2}	Nitrogen density	1.25	kg/m ³
ρ_{water}	Water density	1000	kg/m ³

Nomenclature: Sliding silicon valve

Variables:

Symbol	Description	Unit
F	Force	N
m	Silicon mass being moved	kg
v	Speed	m/s
t	Time	s
k	Vertical spring constant	N/m
I_G	Geometrical inertia moment	m ⁴
f	Displacement	m
E	Young's modulus	Pa
G	Torsion modulus	Pa
σ	Stress	Pa
μ	Friction coefficient	
w	Beam width	m
h	Beam height	m
l	Beam length	m
n	Beam number	
N	Inter-beam number	
φ	Angle deformation function	°
p	Gas pressure	Pa
Q	Volume flow rate	l/min
ρ	Fluid density	kg/m ³
A	Surface	m ²
S	Section	m ²
d	Diameter	m ²

Indices:

Symbol	Description
x,y,z	Orthogonal directions
max	Maximum for dimensioning (nominal)
dim	For dimensioning
adm	Admissible
tot	Total for all beams
0	Values for 20 μm beam width, or initial values
c	Liquid channel
mea	Measured
ins	Instability
sim	Simulated
crit	Theoretical critical value for guaranteeing tightness
opt	Optimal
f	Friction
t	Traction (force)
N	Normal
k	Critical buckling
B	Bernoulli
P	Poiseuille

Constants:

Symbol	Description	Value	Unit
E_{Si}	Young's modulus of silicon	130	GPa
ν	Poisson's coefficient adopted	0.3	
α_k	Coefficient for stress increase due to geometrical basis beam shape	1.41	
b	Coefficient for leakage calculation	2-3	
C	General constant		
η_{water}	Water viscosity constant	$1 \cdot 10^{-3}$	N/(m ² s)

Chapter 1

Introduction

The research presented in this Ph.D. thesis addresses the design, fabrication and testing of two different active valves. The first one is a valve for gases actuated by an electrostatic force, the second a sliding valve for testing low friction coatings. Both valves involved the use of materials new to the field of Microsystems and were based on high aspect ratio structures.

1.1. Motivation

Actuated valves are devices used to control fluid flows in pipes or channels. The cross-section through which the fluid passes dictates the flow rate. Valves based on standard mechanics exist in all size ranges, from the millimeter size for medical applications to the meter size for gas pipelines. The actuation is generally electric or pneumatic.

Applications exist where micromachining technologies could be an advantage for the fabrication of small valves. This way of processing microdevices or microsystems is an extension of the surface patterning techniques of silicon used in the integrated circuit (IC) industry over the last few decades. The nice feature of this technology is the capability it offers to produce structures ranging from the micrometer to a few millimeters.

Reducing the size and mass of valves could be an advantage for some macroscopic applications. For instance, gas microvalves have been proposed for the propulsion of the next generation satellites, meant to be the size of a soccer ball [1.1,1.2]. One option to maintain the satellite in its orbit is to eject very small amounts of gas. For this application, a valve of reduced size and mass is essential. Another advantage of some micromachined valves is their capability for high switching speeds. For pilot valves that actuate larger-size pneumatic valves, fast switching is essential to achieve high speeds in the main valve. Such characteristics could also be used for automobile engines based on direct fuel injection, where in very short times fuel is introduced to the combustion cylinder.

Micromachined valves can achieve high reliability and lifetime properties due to the special materials used. The most common material in microsystems is monocrystal silicon, which does not vary its properties over time [1.3]. To minimize friction of surfaces in contact, thin film coatings can also be used [1.4]. The exceptional flatness and smoothness that can be achieved by surface micromachining of deposited thin film layers favors good sealing of valves. Applications using these features could involve fluid handling, where conventional valves have a limited lifetime that needs to be improved [1.5].

Since a reduction of the size of valve implies a reduction of the cross-section that the fluid passes through, small valves are not suited for all macroscopic applications. At the present time, few applications for micro valves exist in the micro world. Reduced size is a real advantage for chemical analyzing systems, consisting of several functional elements placed in a limited volume. Drug delivery systems destined for implantation into the human body also stand to benefit from miniaturized fluid handling components [1.5].

1.2. Types of actuation

Existing microvalves exploit numerous types of actuation, some more adapted to specific applications than others. The more common types of actuation encountered in reported valves are piezoresistive, electrostatic, and thermal [1.6]. Few

magnetically microsystems valves exist, probably due to the technological difficulty of making coils [1.6,1.7].

Piezoelectricity is the property which relates to the ability of some materials to modify their dimensions when submitted to an electrical field [1.8]. This feature can be used to deform valve membranes to close orifices. Such valves can have good characteristics usable for liquid and gases [1.6]. However, it is difficult to grow reproducible piezoelectric layers.

Electrostatic force created by the electrical charging of conductors can be used to deflect valve membranes. This force is very efficient in the micrometer range, since it depends on the reciprocal of the square of the distance separating the charged electrodes [1.9]. This force also has the advantage that virtually no time delay exists between electrode charging and creation of the force. However, due to the difficulty of implementing such actuation, few reliable valves using electrostatic actuation exist. They are mostly suited to gas applications.

Thermal actuation uses the force created by the dilatation of materials. Such an actuation has the advantage of good reliability and large force [1.10,1.11]. However, the electrical consumption of these valves is much larger than for the other types of actuation. Another disadvantage is the slowness of switching.

1.3. Liquid and gases

Liquids and gases exhibit different fluidic properties that necessitate different orifice or channel dimensions to obtain identical volume flow rates. For instance, water has a density about 770 times higher than air, and the viscosity of water is 60 times higher than for air [1.12].

The first situation considered is the flow through a circular restriction. The volume flow rate through an orifice of diameter d can be calculated for water and air (Figure 1.1). When a differential pressure $\Delta p = p_{in} - p_{ou}$ is applied to an orifice placed in a fluid of density ρ , a volume flow rate, Q_B , can be derived from the Bernoulli's equation (1.1) [1.13].

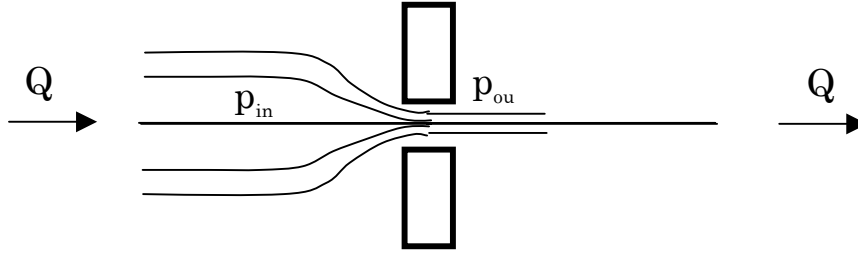


Figure 1.1: Orifice of diameter d immersed in a fluid of density ρ having a differential gas pressure $\Delta p = p_{in} - p_{ou}$ applied that creates a volume flow rate Q_B .

$$Q_B = \frac{\pi d^2}{4} \sqrt{\frac{2\Delta p}{\rho}} \quad (1.1)$$

Therefore, if the same differential pressure is to be maintained for both air and water, the diameter of the orifice used for water has to be 5 times larger than for air, to obtain the same flow rate for water and air.

The second case to be considered is the flow in a channel of constant cross section. The volume flow rate through a channel of diameter d can be calculated for water and air (Figure 1.2). When a differential pressure $\Delta p = p_{in} - p_{ou}$ is applied to a channel placed in a fluid of density ρ , a volume flow rate Q_P can be derived from Poiseuille's equation (1.2) [1.14].

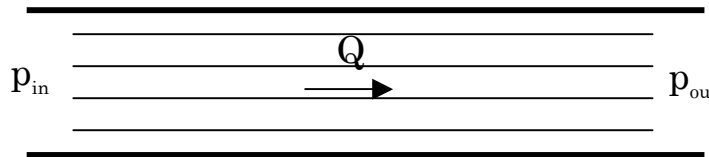


Figure 1.2: Channel of length l filled with fluid of density ρ with an applied differential gas pressure $\Delta p = p_{in} - p_{ou}$, resulting in a volume flow rate Q_P .

$$Q_P = \Delta p \frac{\pi d^4}{128\eta l} \quad (1.2)$$

In this case, the diameter of the orifice used for water has to be 3 times larger than for air to obtain the same flow rate, under the condition of constant differential pressure.

Depending on the shape of the valve, one of these two cases will dominate (Appendix A). In any case, fluid channels or orifices in valves have to be larger for liquids than for gases in order to have identical flow resistance.

1.4. Scope of the research

The research presented in this Ph.D. thesis addresses the development, fabrication and testing of two distinct devices. The first is a micromachined, active electrostatic Ta-Si-N valve for gas handling, while the second is a micromachined, active silicon microtribometer/valve designed for friction studies of diamond-like-carbon (DLC)-coated chips and liquid handling.

The micromachined, active Ta-Si-N electrostatic gas valve fabricated for gas handling was designed to test this material in a functional MEMS application. The design and mode of actuation proposed were selected to take advantage of electrostatic forces for rapid switching of gas flows under large pressures [1.15]. Several versions of the valve were fabricated to improve its reliability. Different types of Ta-Si-N electrostatic gas microvalves were characterized in flow conditions under diverse modes of actuation. The switching times were measured with and without flow [1.16].

The microtribometer/valve fabricated was designed to observe wear of removable flat silicon test inserts, coated with thin-film layers such as DLC and moving in an oscillating manner relative to each other [1.17]. Actuation in this case was achieved by an external linear motor. Silicon mechanical deformation was used to produce a force normal to the test chips. Many versions of the microtribometer/valve were fabricated to achieve the performance desired. The microtribometer/valve was mostly characterized as a tribometer. However, the good sealing properties of the valve principle were also demonstrated.

1.5. Organization of the thesis

The thesis comprises two distinct parts. The first part concerns the Ta-Si-N electrostatic gas valve (Chapter 2 to Chapter 5), while the second concerns the microtribometer/valve (Chapter 6 to Chapter 9). Each part is divided into three different sections, considering the design, fabrication, and characterization/testing of the valve in question.

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Chapter 2

TaSiN Electrostatic Gas Valve

Introduction

The research presented in Chapters 2 to 5 of this Ph.D. thesis addresses the development, fabrication and characterization of a micromachined, active electrostatic Ta-Si-N valve for gas handling.

2.1. Motivation

Tantalum silicon nitride (Ta-Si-N) presents an exceptional combination of chemical, mechanical and electrical properties that could be suitable for microsystems [2.1,2.2]. Many studies have demonstrated its qualities as a diffusion barrier to aluminum and copper [2.3-2.12]. However, its mechanical and electrical properties have never been tested in a functional micromachined application.

Micromachined electrostatic gas valves are composed generally of two electrodes that are attracted by an electrostatic force when actuated. The recovery force is ensured by the stiffness of the spring and therefore depends on the Young's modulus of the spring material used. Fabricating the electrodes as well as the spring in the same material simplifies greatly the processing. Published microelectromechanical systems (MEMS)

valves are usually made in doped silicon or polysilicon, which presents a sufficient electrical resistivity above 10 mΩcm [2.13]. However, the lower resistivity of Ta-Si-N, below 800 μΩcm [2.14], and a Young's modulus in the same order of magnitude as polysilicon, makes it the ideal candidate for such an application. Furthermore, Ta-Si-N presents the possibility of controlling its internal stress by annealing [2.2], which could be used to enhance the mechanical recovering force to avoid stiction.

Electrostatic actuation has many advantages over other principles of actuation. Compared to thermopneumatic actuation or bimetallic thermal actuation, electrostatic force can be modulated extremely rapidly. For flat parallel electrodes, the electrostatic force depends on the reciprocal of the square of the distance separating the electrodes and therefore when two electrodes are separated by thin film insulation [2.15], the very large attractive force can be used to improve quality of the seal. Furthermore, the energy consumption of an electrostatic actuator having good quality insulators tends to zero when maintained in one position.

The main drawback of electrostatic actuation is the necessity of relatively high voltages to have sufficient electrostatic pressure to handle flows [2.16-2.22]. Nevertheless, now commercial electronic products exist to enhance voltage from 5 to 200 V in a volume of about a cubic centimeter.

However, very few published electrostatic MEMS valves demonstrate good sealing, high-pressure handling capability and ultrafast switching [2.16-2.22]. The reported silicon bulk micromachined valves seem to be capable of handling higher pressure than surface micromachined valves, though their switching times are slower [2.21]. The reason why surface micromachined valves can only handle lower pressure is believed to be related to their large orifices, which are made by wet anisotropic etching. Combining surface membrane micromachining with deep reactive ion-etching (DRIE) enables the fabrication of orifices more than five times smaller than in reported surface micromachined valves [2.16-2.19,2.22]. This allows flows to be switched under much higher differential pressure of up to a few bars.

The applications for reduced size, low-consumption and fast-switching gas valves that can operate under large pressures are numerous. For instance, researches have been conducted to develop microsatellites the size of a soccer ball, which could be maintained in their orbit by ejection of gas jets [2.23,2.24]. Fast switching gas valve can also be used as pilot valves to thermopneumatically actuate large-size mechanical valves. Other applications could involve refreshing Braille screens or be in gas chromatography [2.19].

2.2. Scope of the research

The micromachined, active Ta-Si-N electrostatic gas valve fabricated for gas handling was designed to test this material in a functional MEMS application. The design and mode of actuation proposed were selected to take advantage of electrostatic forces for rapid switching of gas flows under large pressures.

Since Ta-Si-N is a deposited thin film layer, the valve has a surface-micromachined membrane that can block flow going through a small vertical channel made in bulk silicon by deep-reactive ion etching (DRIE). Design optimization was undertaken to reduce the drawbacks of electrostatic actuation by minimizing actuation voltage, and to allow for large flow rates even with small-size orifices. Development of particular membrane shapes based on finite element simulations (FEM) made it possible to lower the actuation voltage under flow conditions. The small valve size meant that many membranes could be placed on the same chip. In this way, a large gas flow could be distributed over a number of orifices, while retaining the high-pressure handling capability. The non-linear and unstable behavior of an electrostatic membrane actuator led to the valve being designed as an on/off switch or alternatively, to be actuated in pulse width modulation (PWM) to vary flow rate [2.25].

The combination of bulk and surface micromachining necessitate relatively complex fabrication processes. The development approach chosen was to first use a process based on numerous simple fabrication steps, and then to develop other versions based on fewer, more difficult steps. Therefore, three

different processes were developed and tested, the last one being the most reliable.

Due to the difficulty of establishing electrical contact with the membranes while guaranteeing valve tightness, different packaging approaches were investigated. Anodically bonding a glass cover containing channels to the silicon valve chips, and then gluing the valve chip onto a PCB was tried. However, the numerous difficult manipulations and lack of reliability led to the abandonment of gluing, PCB, wire bonding and anodic bonding of the glass cover as well. In the final package version, the silicon chip was sandwiched in between two seals, with the electrical contacts being made by spring needles.

2.3. Electrostatic actuation

When electrostatic pressure is used to deform a mechanical spring (Figure 2.1), the system can present instabilities and hysteresis, depending on the magnitude of the actuation voltage U and displacement y [2.25]. This particular behavior is due to the different forces exhibiting different physical properties within the system. Electrostatic force F_{el} varies as the reciprocal of the square of the distance separating the two electrodes d_g , while spring force F_{me} is a linear function of displaced distance y (2.1)(2.2)[2.15].

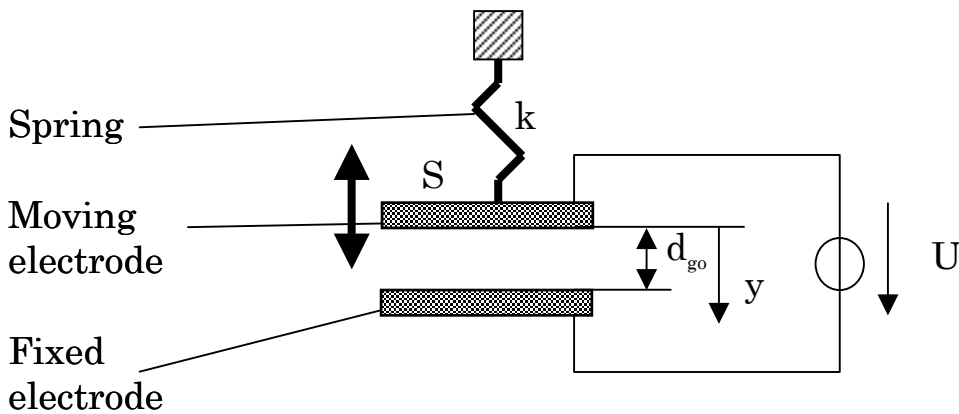


Figure 2.1: Electrostatic actuation principle of a deformable system having membrane of surface area S mechanically maintained by a spring of constant k .

$$F_{me} = -ky \quad (2.1)$$

$$F_{el} = \frac{\varepsilon_0 S U^2}{2d_g^2} = \frac{\varepsilon_0 S U^2}{2(d_{g0} - y)^2} \quad (2.2)$$

To reveal the stable and unstable domains, the two forces can be plotted as a function of y , the vertical displacement. For voltages below a limit value called the pull-in voltage U_p , there exist two points of equilibrium: one stable and one unstable (Figure 2.2). When the voltage is increased up to the pull-in voltage, the curve $F_{el}(y)$ moves upward, and the two points of equilibrium rejoin to become one.

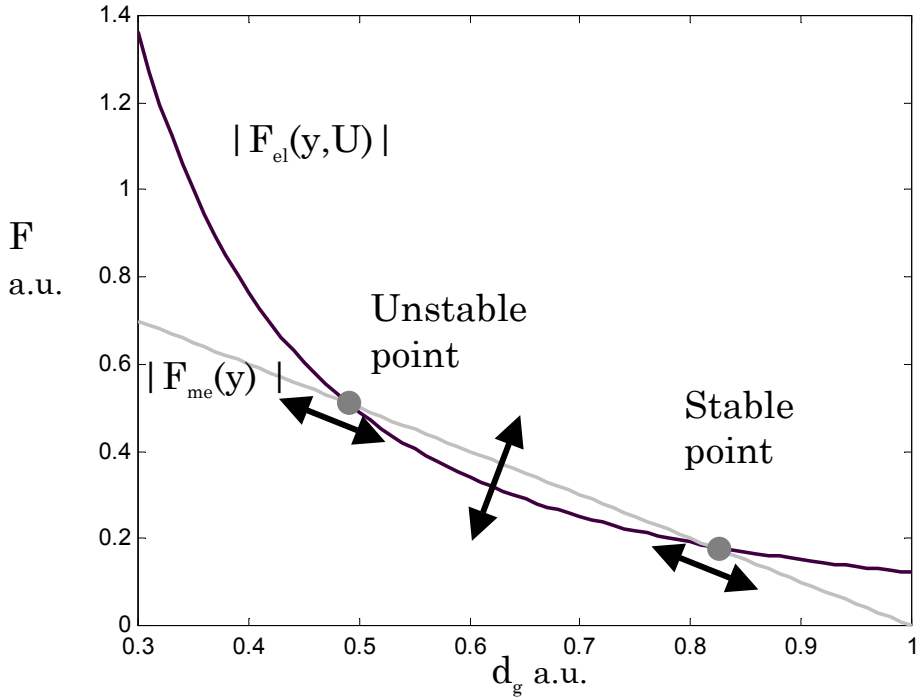


Figure 2.2: Mechanical and electrostatic forces as a function of vertical displacement for a fixed actuation voltage. One stable and one unstable point of equilibrium exist for actuation voltages below pull-in.

The stability around the point of equilibrium expressed mathematically with fulfillment of the following condition requirement (2.3):

$$\text{Stability condition: } \left| \frac{\partial F_{me}}{\partial y} \right| > \left| \frac{\partial F_{el}}{\partial y} \right| \quad (2.3)$$

The membrane displacement can be plotted for voltages from zero to values above pull-in. The membrane position is stable up to a certain point and then suddenly claps down to the counter electrode (Figure 2.3). The pull-in point can be calculated (2.4).

$$\text{Pull-in condition: } \left| \frac{\partial F_{me}}{\partial y} \right| = \left| \frac{\partial F_{el}}{\partial y} \right| \quad (2.4)$$

Solving this equation gives a pull-in gap at two thirds of the initial gap, independent of the spring constant.

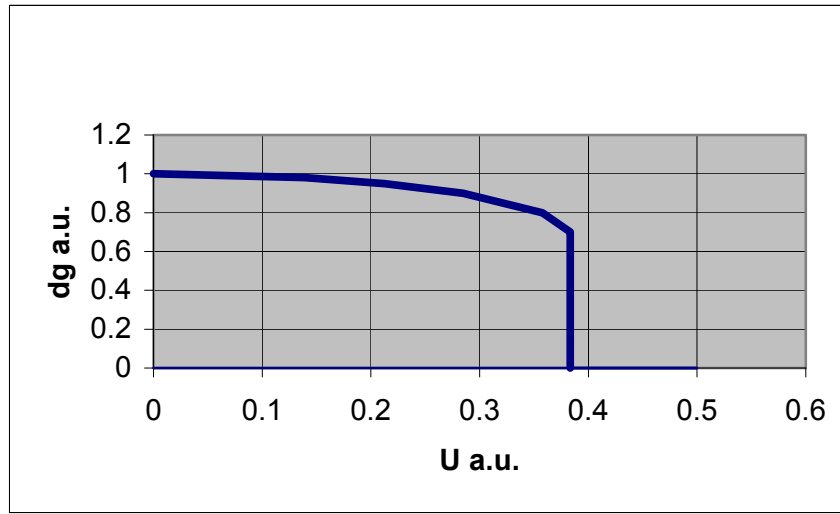


Figure 2.3: Membrane position when the actuation voltage is raised from 0 to values above pull-in. When the membrane has been displaced one third of the initial gap, the membrane moves suddenly down to the counter electrode.

Therefore, when the membrane is controlled by an applied voltage, its position is only stable for one third of the initial gap. This behavior shows that an electrostatic valve membrane is most suited to work as an on/off switch instead of controlling flow by intermediate membrane positions.

2.4. Pulse width modulation (PWM) actuation

Since controlling flow in a linear manner can be essential for some applications, a way of actuating the membrane without trying to place it in intermediate stationary positions was

investigated. The idea was to make the membrane work as an on/off flow switch but with varied open and closed times, with the flow rate being proportional to the duty cycle. This concept of pulse width modulation (PWM) is widely used in power electronics to control electrical motors or for DC/AC converters.

Only two reports were found that mention MEMS valve actuation in PWM [2.18,2.21]. The reason is probably that most existing valves are too slow and have cycles lifetimes which are too short to be actuated in this way. For PWM actuation, the switching frequency should be high enough to avoid flow fluctuations, which are filtered by the fluidic tubes and channels placed behind the valve that act as low pass filters [2.26]. Such actuation would be ideal for flow control flow unit, in which the valve is placed in a feedback loop where a flow meter measures flow rate.

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Chapter 3

TaSiN Electrostatic Gas Valve Design

This chapter focuses on the design of a reliable, normally open gas valve that can rapidly switch flows under important differential pressures. Due to the non-linear behavior of electrostatic actuators (section 2.3), the microvalve was developed either to be actuated as an on/off switch or to work in pulse width modulation (PWM). Based on the concept of a membrane moving perpendicularly with respect to an orifice (Figure 3.1), different designs of the electrostatic valve were investigated to reduce the actuation voltage or allow for increased flow rates. For minimizing the risk of failure and optimizing performance, simplified analytical calculations were conducted. Finite element simulations allowed the tensile stress of the Ta-Si-N membrane layer to be taken into account and validated the dimensions of the valve calculated analytically. The flow rate is estimated based on gas isentropic transformations in a nozzle.

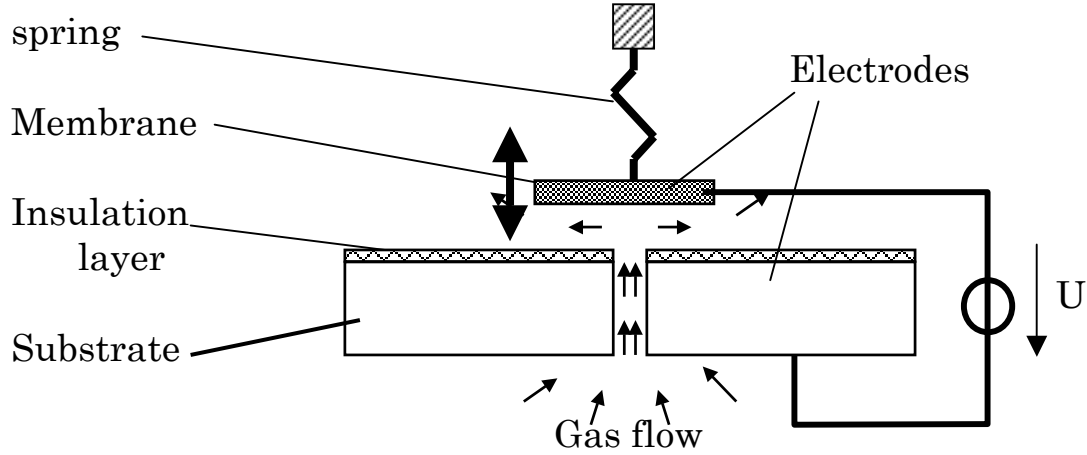


Figure 3.1: Schematic of the normally open Ta-Si-N electrostatic valve.

3.1. Introduction

The literature presents two types of electrostatic MEMS valves, those based on a combination of surface and silicon bulk micromachining and those using a stack of bulk micromachined silicon wafers [3.1]. The first category of valves has the advantage of a reduced mass to be moved and therefore higher switching speeds. However, bulk micromachined valves seem to be more robust [3.2-3.4].

The valve developed consists of a Ta-Si-N 1 μm thick membrane that can close a 10 μm orifice when actuated by an electrostatic force to block the gas flow. The actuation voltages range from 20 V to 150 V depending on the valve version, the membrane design and the fluidic conditions. The valves are diced into square chips measuring 4 by 4 mm^2 . The valve can be operated at differential gas pressure up to 2 bar and deliver flow rates of 50 ml/min.

The concept proposed differs in many aspects from existing surface micromachined membrane valves. There are few papers reporting on electrostatic valves having a thin film membrane closing an orifice [3.5-3.7], and none of these uses DRIE to fabricate small-diameter orifices, essential for large pressure handling capability. Furthermore, optimization of the shape of the thin film membrane in order to reduce the actuation voltage has to date not been reported, to the best of our knowledge.

The proposed design has been determined in large part by the fabrication process, which is based on a combination of surface and bulk micromachining (Chapter 4). Surface micromachining enables the structuring of sputtered freestanding electrically conducting membranes in Ta-Si-N. Deep reactive-ion etching (DRIE) of silicon is used to etch 50 μm holes through the wafer.

3.2. Valve concept

The concept chosen, which is a membrane moving perpendicularly to an orifice, is in accordance with the characteristics desired of fast switching, good sealing, and high pressure handling capability (Figure 3.2).

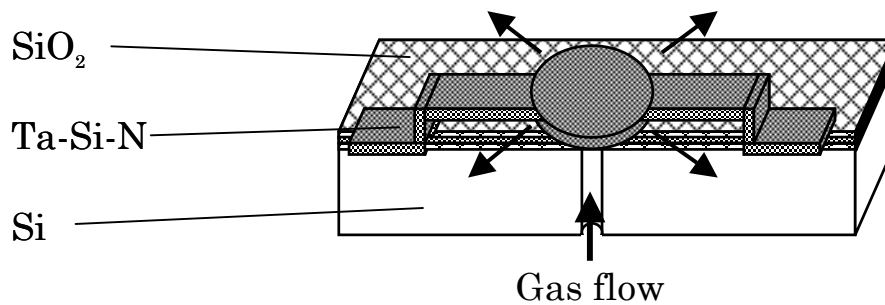


Figure 3.2: A freestanding membrane can be actuated by an electrostatic force to close a small orifice and block the gas flow. The repelling force is the spring force of the membrane.

The switching speed is related both to the mass in motion and the forces involved. Since the thickness of the Ta-Si-N membrane is limited to a few micrometers for technological reasons, the mass of the membrane is extremely small compared to bulk silicon microvalves. The mechanical repelling spring force, which is related to the shape, size and material of the membrane, should be large in order to prevent sticking. The main advantage of a double-clamped beam compared to a cantilever is the relative flatness of the central part when deformed, which should prevent sticking by producing a well-defined repelling force. The electrostatic force used to close the membrane can be relatively large and can be changed almost

instantly, depending on the actuation voltage. During the fast downward motion of the membrane, the gas around the membrane produces a pressure opposed to its displacement, due to the squeeze film effect. However, since the effect is symmetrical and only slows down the displacement, it will not be taken into account in the design of the membrane.

Comparison of different published MEMS valves indicates that the leakage is very dependent on the pressure holding the surfaces together [3.3,3.8-3.11]. Therefore, since the electrostatic force is proportional to the inverse of the square distance between electrodes (section 2.3), the thickness of the insulation layer has to be minimized while at the same time avoiding electrical breakdown.

Pressure handling capability depends on the gas force applied on the membrane. It is assumed that the gas produces the highest pressure on the membrane when the membrane blocks its flow. Therefore, to maximize the pressure handling capacity, the diameter of the orifice has to be kept small. As it is explained in section 4.1.1, the limitation of orifice diameter to about 10 μm comes from the fabrication process. The side effect of a small orifice is the high flow resistance, which limits the operating flow rates. To overcome this problem, it was decided to design one type of chip having many valves working in parallel. This approach reduces the global flow resistance without modifying the other characteristics of the valve.

3.3. Static physical model

The purpose of this model is to dimension the overall valve, its membrane and orifice based on analytical calculations. Under the no-flow condition, the pull-in voltage of the membrane corresponds to the actuation voltage. First, a physical model will be used to dimension the membrane based on the pull-in voltage. Then, the influence of the gas pressure on the closed membrane will be considered. Since the effect of the gas flow on the membrane during its actuation is extremely complex, it will not be studied in this design, but some aspects are considered in Chapter 5, as well as the switching dynamic.

By reducing the complexity of the valve shape and structure to keep the quantities of major influence (Figure 3.3), it is

possible to analytically describe the system. In order to understand the influence of the main parameters, some approximations are made.

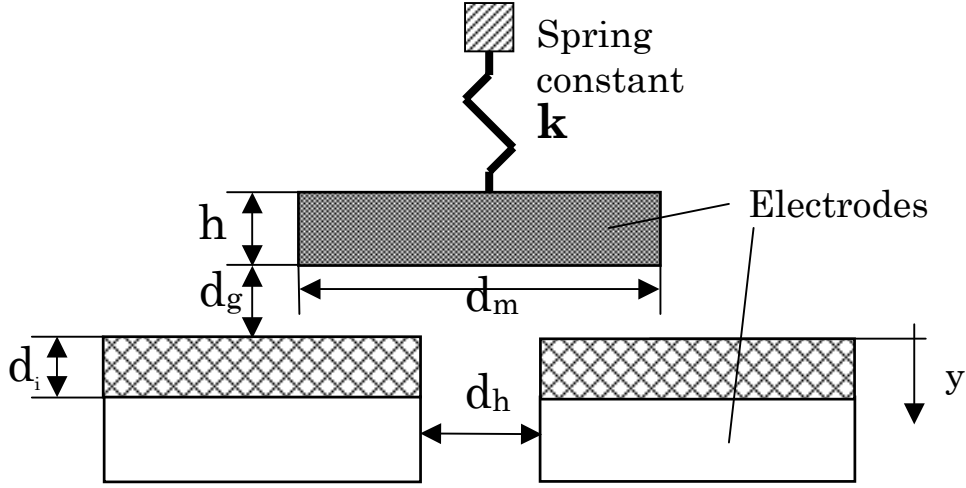


Figure 3.3: Schematic of the valve simplified physical model. The spring constant k is related to membrane elasticity. The central membrane of diameter d_m is separated by a gap d_g from the insulation layer of thickness d_i that covers the bottom electrode.

The spring constant k is related to membrane elasticity, which is determined by its shape, dimensions and material properties. The membrane spring, based on a double-clamped beam, is stiff and compact (Figure 3.4).

The membrane geometry is simplified in order to calculate a spring that is stiffer than in reality, which will increase the calculated pull-in voltage. The central circular membrane is considered to be rigid in the area wider than the lateral beams.

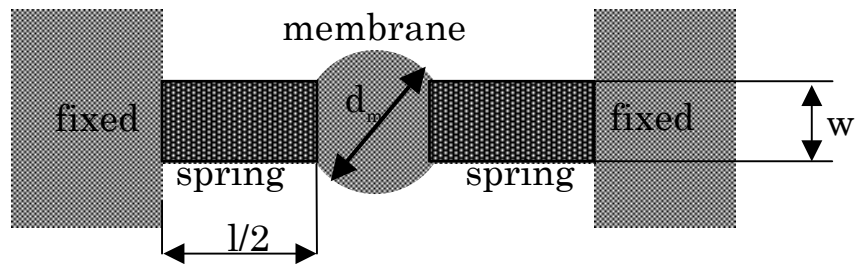


Figure 3.4: Top view of the Ta-Si-N membrane. The dark area is the part of the membrane acting as a spring.

3.3.1. Spring constant

Assuming the central membrane to be rigid enables the spring to be modelled as a double-clamped beam (Figure 3.5) of constant cross section.

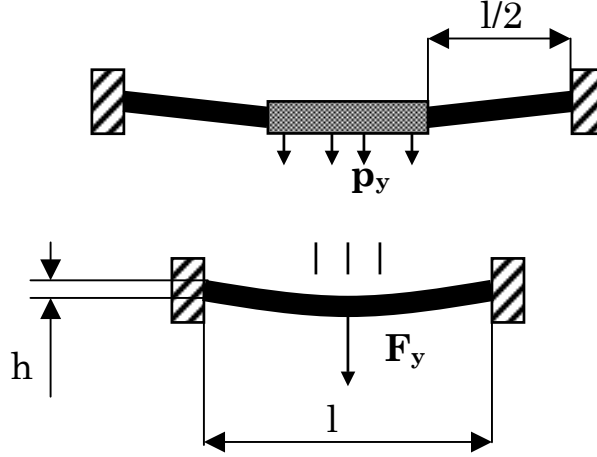


Figure 3.5: Schematic showing the simplification that results when the central membrane is assumed to be rigid.

Hook's law, which describes material elasticity, is the basis for calculating the bending of a double-clamped beam under the action of a central force F_y , given in equation (3.1) [3.12]. This equation takes into account the Young's modulus E of the material and the geometric moment of inertia I of the cross section of the beam.

$$f_y = \frac{F_y l^3}{192EI} \quad (3.1)$$

Therefore, as described in equations (3.3), (3.4), the width w and the height h of the beams can be related to the spring constant k .

$$I = \frac{wh^3}{12} \quad (3.2)$$

$$F_m = \frac{16Ewh^3}{l^3} f_y \quad (3.3)$$

$$\text{Where: } k = \frac{16Ewh^3}{l^3} \quad (3.4)$$

3.3.2. Electrostatic force

Based on Coulomb's theory, the electrostatic force F_{el} attracting two flat parallel plates set at different electrical potential can be calculated (3.5),(3.6)[3.13]. Unfortunately, it is not possible to derive a general analytical solution for curved surfaces. Therefore, as a rough approximation, the electrostatic pressure is considered only in the central circular part of the membrane.

$$F_{el} = \frac{\partial W_{pot}}{\partial y} \quad (3.5)$$

$$\text{With: } W_{pot} = \frac{1}{2}CU^2 \quad (3.6)$$

The electrical field crosses two dielectrics placed in series (3.7). The equivalent capacitance C_{eq} is calculated (3.7) and derived in the y direction (3.8).

$$\text{and: } C_{eq} = \frac{1}{\frac{1}{C_1} + \frac{1}{C_2}} = \epsilon_0 S_m \frac{1}{\frac{d_1}{\epsilon_{r1}} + \frac{d_2}{\epsilon_{r2}}} \quad (3.7)$$

$$\frac{\partial C_{eq}}{\partial y} = \epsilon_0 S_m \frac{\epsilon_1 \epsilon_2 (\epsilon_1 + \epsilon_2)}{(d_1 \epsilon_1 + d_2 \epsilon_1)^2} \quad (3.8)$$

Finally, equation (3.8) is replaced in the electrostatic force equation (3.9).

$$F_{el} = \frac{\epsilon_0 S_m U^2}{2} \frac{\epsilon_1 \epsilon_2 (\epsilon_1 + \epsilon_2)}{(d_1 \epsilon_2 + d_2 \epsilon_1)^2} \quad (3.9)$$

This equation can be simplified since the relative permittivity of the air gap is equal to 1 (3.10).

$$F_{el} = \epsilon_0 S_m U^2 \frac{\epsilon_i (1 + \epsilon_i)}{2(d_g \epsilon_i + d_i)^2} \quad (3.10)$$

For further calculations, the thickness of the insulation layer is neglected (3.11).

$$F_{el} = \frac{\epsilon_0 S_m U^2}{2d_g^2} \quad (3.11)$$

The introduction of numerical values allows checking the validity of this approximation. The SiO_2 insulation layer has a

relative permittivity ε_i of 3.9 and a thickness d_i of 0.25 μm . The air gap d_g at pull-in is 1 μm . In these particular conditions, the simplified equation (3.9) gives an electrostatic force F_{el} within 11 % compare to the exact equation (3.10), which is considered acceptable.

Finally, the membrane surface S_m is replaced in equation (3.11) to calculate the electrostatic force equation that will be used in further calculations (3.12).

$$F_{el} = \frac{\varepsilon_0 \pi d_m^2 U^2}{8 d_g^2} \quad (3.12)$$

3.3.3. Gas effect on the closed membrane

When there is no flow, the gas pressure on each side of the membrane is known. On the inlet side, it is the inlet pressure, and on the other side, the outlet pressure. The gas force that tends to reopen the valve is estimated in equation (3.13).

$$F_{ga} = \frac{\pi d_h^2}{4} (p_{in} - p_{ou}) \quad (3.13)$$

Therefore, small orifice favours high gas pressure handling capability. However, this calculation neglects leakage flow or transient behavior that could produce additional pressure on the membrane.

3.3.4. Gas flow considerations

Membrane effect

When the valve is open, having the membrane participate in an significant manner to limit gas flow will not help to achieve high-pressure handling capability and large flow.

Therefore, the gap at which the membrane restricts the flow as much as the orifice will be estimated. In nozzles, the narrowest section restricts the flow the most (Appendix A). Equating the surface of the orifice to the smallest section where

the gas circulates under the membrane gives the limit at which the membrane restricts the flow significantly (3.15).

$$\pi \frac{d_h^2}{4} = \pi d_h d_g \quad (3.14)$$

$$d_g = \frac{d_h}{4} \quad (3.15)$$

If the gap is smaller than this limit, the membrane restricts the flow in a considerable manner compared to the orifice. For instance, if the orifice diameter measures 10 μm , the gap height threshold is 2.5 μm . For smaller gaps, the membrane participates to limit flow.

Flow through a nozzle

For defined pressure conditions, the flow rate delivered by the valve can be estimated. In appendix A, it is shown that the fluid viscosity within the channel can be neglected and the valve considered as a nozzle with good accuracy at high pressures. The viscosity of the fluid will be considered by a head loss factor that accounts only for effects due to change of cross section.

For nozzles, conservation of energy defines flow rate. If fluid density is considered constant, Bernouilli's equation is valid, but for pressure variation above 0.25 bar, the change of gas density has to be taken into account [3.14]. When flowing through an orifice, since gas does not stay for long in contact with sidewalls, no thermal energy is exchanged, and an adiabatic process can be considered. Because gas is heated up when flowing through a narrow orifice, and cooled down when expanding in the outlet reservoir, the process is double adiabatic, which is called isentropic [3.15]. For isentropic processes, flow rate follows different laws below the speed of sound and at the speed of sound, or choked flow.

James et al. have developed applicable flow formulas for isentropic flow [3.16]. However, these formulas do not take into account viscosity effects due to change of cross section. To determine the head loss factor, data provided by fluid components manufacturers will be used. The Lee Company provides such information [3.17]. Unfortunately, the flow

formulas they use for calculation are slightly different from the one developed by James et al. In section 5.1.1, the simplified formula of Lee Company are derived from isentropic formulas from James et al. This approach allows calculating the head loss factor α due to nitrogen viscosity at a change of cross section, which can be replaced in theoretical formulas from James et al.

The theoretical formulas derived by James et al. are more accurate than the one used by the valve manufacturer, which contains additional approximations (section 5.1.1) [3.16,3.17]. Numerical comparison between the theoretical isentropic formulas and the one of Lee Company showed a matching below 5 % for a 10 μm diameter nozzle with inlet pressure ranging from 1 to 3 bar and ambient outlet pressure.

Because the microvalve is expected to work at inlet pressure larger than 1.9 times the outlet pressure, the flow can fall in the choked flow or sonic domain, which defines the way that the high flow rates are calculated (3.16)[3.15-3.17].

$$\text{Sonic domain: } Q = \alpha \frac{p_{in} \pi d_h^2 \sqrt{3}}{4 \rho \sqrt{\frac{\gamma R T}{M}}} \left(1 - \left(\frac{2}{\gamma + 1} \right)^{\gamma/(\gamma-1)} \right) \quad (3.16)$$

For N_2 , at ambient conditions:

$\rho_{\text{N}_2}=1.25\text{kg/m}^3$, $T=293^\circ\text{K}$, $\gamma=1.4$, $M_{\text{N}_2}=28\text{g/mol}$, $\alpha_{\text{N}_2}=0.83$,
 $R=8.31\text{ J/(mol K)}$

If a nominal inlet pressure p_{in} of 3 bar is considered, while the outlet pressure p_{ou} is 1 bar, the flow rate flowing through a 10 μm diameter orifice d_h can be calculated for nitrogen, which is a diatomic gas ($\gamma=1.4$) defined by its molar mass M , and density ρ at ambient temperature T . In these conditions, the flow is 2.4 ml/min.

3.4. Calculation of the valve dimensions

In this section, the basic valve is dimensioned according to the simplified model proposed in section 3.3. First, the fixed parameters, dictated by the fabrication process and the material properties, are established. Then the beam length is

chosen based on published simulations of a similar device, and the beam width is calculated. Finally, the working characteristics are checked based on the dimensions proposed.

3.4.1. Fixed parameters

Some parameters are limited or fixed for technological reasons (Table 3.1). These data will be the starting point for the calculation of valve dimensions.

Table 3.1: This table shows the parameter limitation for technological or physical reasons and the values adopted for the first valve version. One particularity of the first valve version is the orifice diameter where the bottom electrode has an orifice of 20 μm , while the flow orifice is 10 μm .

Designation	Technological/ physical limit	Value adopted for the first valve version
Insulation layer thickness [d_i]	2000-5000 Å	2500 Å
Insulation layer permittivity [ϵ_r]	Si_3N_4 :6 SiO_2 :3.9	6
Ta-Si-N membrane thickness [h]	Max. 2 μm	1 μm
Ta-Si-N Young modulus [E]	190-220 GPa	200 GPa
Ta-Si-N Poisson coefficient [ν]	0.3	0.3
Gap height [d_g]	Max. 2 μm	1.5 μm
Orifice diameter [d_o]	Min. $\sim 10 \mu\text{m}$	10/20 μm

Explanations and choices concerning:

- Insulation layer: for the first version of the valve, the insulation was made in PECVD silicon nitride, with a thickness of 2500 Å. However, it appeared that this layer had a tendency to peel off. Therefore, in subsequent versions a silicon oxide insulation layer was adopted (Chapter 4).

- Ta-Si-N membrane: since tantalum silicon nitride is deposited by sputtering a Ta_5Si_3 target in a chamber containing nitrogen [3.18]. The moderate deposition rate of 16 nm/min has led to chose a membrane thickness of 1 μm , which was also tested in the past for free standing beams [3.18-3.20]. The Young's modulus of Ta-Si-N was measured with an x-ray method at the Neu Technikum Buchs [3.21]. The Poisson coefficient value of 0.3 is generally adopted for ductile materials such as metals when no measured value is available.
- Orifice diameter: 10 μm represents the limit for patterning the surface insulation layers with good size reproducibility (section 4.1). The bottom electrode has a hole diameter of 20 μm , which is the surface where the gas pressure is applied when the valve is closed (Figure 3.6).

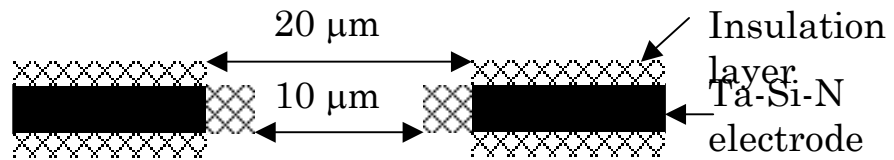


Figure 3.6: Close up of the orifice. The bottom Ta-Si-N electrode has an orifice diameter larger than the insulation layer.

- Gap height: Since the membrane should not limit the flow, it is preferable to have a high gap. The limit can be calculated with equation (3.15), which gives a gap of 2.5 μm for an orifice diameter of 10 μm . The need for a high spring constant favors as well a large membrane deflection. The technologically dictated maximum value of gap height is related to the thickness of the sacrificial layer (Chapter 4) and the capacity of Ta-Si-N to cover a step. It was chosen to be 1.5 μm to ensure the quality of the beam anchor.

3.4.2. Variables

The variables to be optimised are the horizontal dimensions of the membrane, namely width, length and central membrane diameter. The central membrane surface has essentially two

properties: it is the part of the membrane that is most efficient for electrostatic attraction, and it determines in part the quality of the valve seal. Since no data were found concerning gas leakage dependency on the membrane surface in a similar configuration, the dimension of the central membrane is chosen 10 times larger than the central orifice. Therefore, the variables to be optimised are the length and width of the beams, which define the spring in the model.

3.4.3. Link between variables

For linking the variables, the physical model is placed in two critical situations. The first situation is when the membrane is displaced about one third of the gap distance at the pull-in voltage. This will be used to dimension the membrane arms. The second situation corresponds to the maximum gas pressure that the membrane can withstand when actuated at the pull-in voltage, without considering leakage. This will serve to ensure that the pull-in voltage is sufficient to control large pressures.

Actuation voltage

As explained in section 2.3, the pull-in voltage corresponds to the frontier between the stable and unstable domains. At this limit, the mechanical force F_m balances the electrostatic force F_{el} , and the slopes relative to the displacement of the two are equal (3.17),(3.19). Solving the system of equations (3.18),(3.19) gives the pull-in conditions (3.20),(3.21). At pull-in, the membrane has displaced one third of the initial gap d_{g0} (3.20).

$$|F_{me}| = |F_{el}| \quad (3.17)$$

$$\frac{16Ewh^3}{l^3}(d_{g0} - d_g) = \frac{\epsilon_0 \pi d_m^2 U^2}{8d_g^2} \quad (3.18)$$

$$\left| \frac{\partial F_{me}}{\partial d_g} \right| = \left| \frac{\partial F_{el}}{\partial d_g} \right| \quad (3.19)$$

$$\Rightarrow d_{g_pull-in} = \frac{2}{3}d_{g0} \quad (3.20)$$

$$\Rightarrow U_{pull-in} = U_p = \frac{2}{3} \sqrt{\frac{2kd_{g0}^3}{3\varepsilon_0 S_m}} = \frac{16}{3d_m} \sqrt{\frac{2Ewh^3d_{g0}^3}{3\pi\varepsilon_0 l^3}} \quad (3.21)$$

Equation (3.21) shows the pull-in voltage geometrical dependency on the width w and the length l of the membrane arms.

Maximum gas pressure

The threshold gas pressure that reopens the valve when actuated at the pull-in voltage is calculated. At this limit, the sum of the forces on the closed membrane equals zero (3.22),(3.23). Therefore, based on the size of the orifice and the thickness of the insulation layer, the maximum pressure is estimated by taking orifice size and insulation layer thickness into consideration (3.24). As stated in section 3.3.3, the gas pressure is applied only in the central surface that is not in contact with the bottom electrode, which means that leakage is neglected.

$$\sum F^{membrane} = F_{me} + F_{el} + F_{ga} = 0 \quad (3.22)$$

$$\frac{-16Ewh^3}{l^3}d_g + \frac{\varepsilon_0\varepsilon_{ri}\pi d_m^2 U_p^2}{8d_i^2} + \frac{-\pi d_h^2}{4}\Delta p_{\max} = 0 \quad (3.23)$$

$$\Delta p_{\max} = \frac{-64d_g Ewh^3}{\pi d_h^2 l^3} + \frac{\varepsilon_0\varepsilon_{ri}d_m^2 U_p^2}{2d_h^2 d_i^2} \quad (3.24)$$

3.4.4. Optimisation procedure

The goal is to optimise the membrane stiffness and gap in order to avoid sticking during valve operation. Unfortunately, since sticking behavior is not a well-defined behavior [3.22], dimensions of the beams will be adapted in order to have a pull-in voltage close to 10 V, without internal stress. This chosen

value is situated in the low-middle range of published MEMS devices based on electrostatic membrane actuation [3.22,3.23].

Since it was planned to develop arrays of valve based on a basic valve membrane, the size is minimized. To ensure a good sealing when actuated, the central membrane should touch completely its seal when actuated, and therefore the minimum beam length l was chosen at 2.5 times the central membrane diameter d_m , based on simulations of a microrelay [3.24]. This results in a beam width w of $44\text{ }\mu\text{m}$ for a pull-in voltage of 11.4 V .

3.4.5. Basic valve dimensions and characteristics

The final dimensions of the basic membrane are as follow:

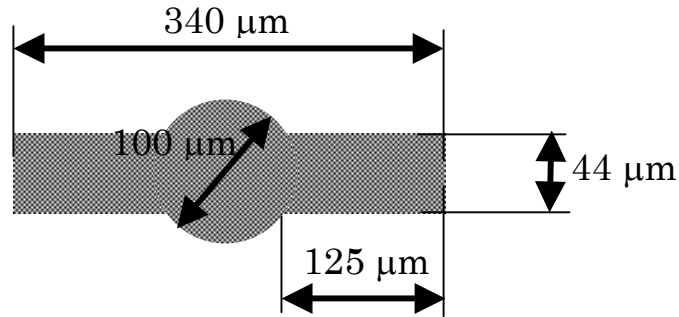


Figure 3.7: Top view of the basic membrane showing final dimensions

Its corresponding characteristics based on the proposed model are given in Table 3.2.

Table 3.2: Calculated valve characteristics based on the simplified model.

Pull-in voltage U_p	11.4 V
Maximum gas dif. pressure at pull-in voltage Δp_{max} withstood by the closed membrane	8.2 bar (2500 Å Si_3N_4)
N_2 flow rate Q at 2 bar inlet pressure	1.64 ml/min (without glass cover)

3.4.6. Model validity

Summary of approximations

Equations (3.21) and (3.24) for pull-in voltage and maximum operating pressure, respectively, include many approximations that are inherent to the model, method of calculation or the uncertainty of some parameters.

- Central membrane defined as rigid => increases calculated U_p
- Electrostatic force only active under the central membrane => increases calculated U_p
- No mechanical stress in the layer considered => reduces calculated U_p
- Spring force considered linear
- Effect of Poisson's coefficient neglected for calculating spring stiffness
- No gas flow effect on the open membrane taken into account
- Since no gas flow effect is taken into account, the model is not relevant for calculating the commutation times
- No roughness of the surfaces taken into account for calculating the leakage
- No charge accumulation on the insulation layer considered
- The thickness of the insulation layer neglected for calculation of pull-in voltage
- No electrical breakdown considered in the gas during switching

The three first approximations are considered of major influence on the result, with the first two increasing the calculated result, while the assumption of absence of stress decreases it. Therefore, a crosscheck with other methods of calculation will determine which approximation dominates, and allow evaluation of the validity of the dimensioning.

Crosscheck with semi-analytical formula

P.M. Osterberg et al. developed semi-analytical formulas describing the pull-in voltage of beams having constant rectangular sections [3.23]. He parameterised a general pull-in analytical formula with 2-D FEM simulations and obtained a semi-analytical formula (3.25) containing parameters based on simulation.

$$U_p = \sqrt{\frac{\gamma_1 S}{\epsilon_0 l^2 D(\gamma_1, k, l) \left[1 + \gamma_3 \frac{d_{g0}}{w} \right]}} \quad (3.25)$$

With the parameters valid for equation (3.25):

$$D = 1 + \frac{2\{1 - \cosh(\gamma_2 kl/2)\}}{(\gamma_2 kl/2) \sinh(\gamma_2 kl/2)}, \gamma_{1,2,3} = 2.79, 0.97, 0.42$$

$$k = \sqrt{\frac{12S}{B}}, S = \tilde{\sigma} h d_{g0}^3, B = \tilde{E} h^3 d_{g0}^3$$

and the physical variables valid for equation (3.25):

equivalent Young's modulus: $\tilde{E} = E/(1 - \nu^2)$

equivalent stress: $\tilde{\sigma} = \sigma_0(1 - \nu)$

This equation (3.25) allows the tensile stress of the Ta-Si-N layer to be taken into account, which is in the range of 50 to 100 MPa after annealing [3.21]. Unfortunately, the calculation is applicable only to beams of constant rectangular section. Therefore, the geometrical approximation that has to be made in our case will have a large impact on the result. More precisely, this approximation means that the membrane is considered to have a constant width of 44 μm . The effect will be to over estimate pull-in voltage U_p .

Table 3.3: Calculated valve characteristics based on the semi-analytical model, assuming a membrane of constant width.

	Pull-in voltage {V}
No stress	17.2
50 MPa tensile stress	32.5

100 MPa tensile stress	46.0
------------------------	------

Table 3.3 shows that without stress, the pull-in voltage is larger than the one calculated based on the simplified model (section 3.3)($U_p=11.4$ V). This can be attributed to the geometric simplification.

The second observation concerns the large effect of tensile stress on U_p . Apparently tensile stress can almost triple the value of pull-in voltage. Such a large effect of tensile stress was also observed by P. Osterberg [3.23].

Crosscheck by finite element simulation (FEM)

The number of approximations included in the previous analytical calculations of the pull-in voltage U_p underlines the need to compare these calculations with FEM simulations. These simulations conducted on a PC with CFDRC software (CFD Research Corporation) were based on the same material properties and geometrical dimensions as the analytical calculations.

U_p was found to be 7 V under conditions of zero internal stress, which is smaller than that calculated using on the simplified model presented. This can be attributed mainly to the two most important model approximations: 1) the electrostatic force was considered only under the central membrane and 2) the central membrane was assumed to be rigid. The relatively good agreement of the analytical and FEM simulation results, which amounts to less than a factor 2 difference, lead to the conclusion that FEM simulations are the best estimation of reality.

Further FEM simulations were conducted to calculate the pull-in voltage U_p of the basic valve with a tensile stress of 100 MPa (Figure 3.8, Figure 3.9). The pull-in value obtained is 24.5 V. From a leakage perspective, it is interesting to note that the membrane actuated at a voltage just higher than pull-in contacts the ground electrode over a large surface.

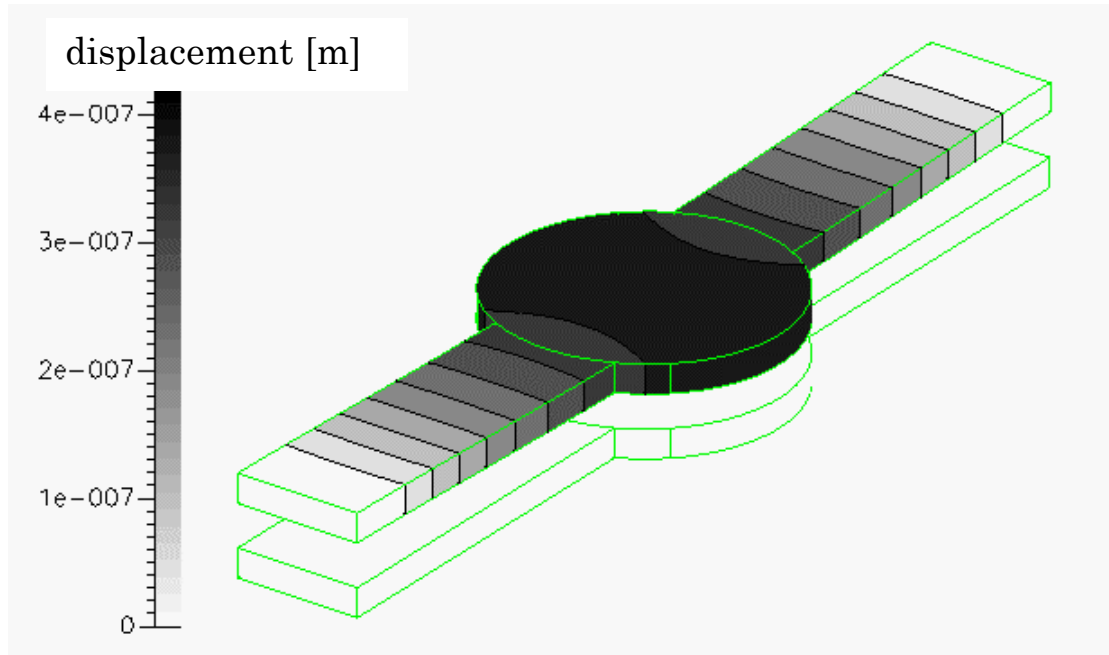


Figure 3.8: FEM simulation showing the displacement of a Ta-Si-N membrane actuated at 24 V, just below the pull-in voltage. A tensile stress of 100 MPa is taken into account.

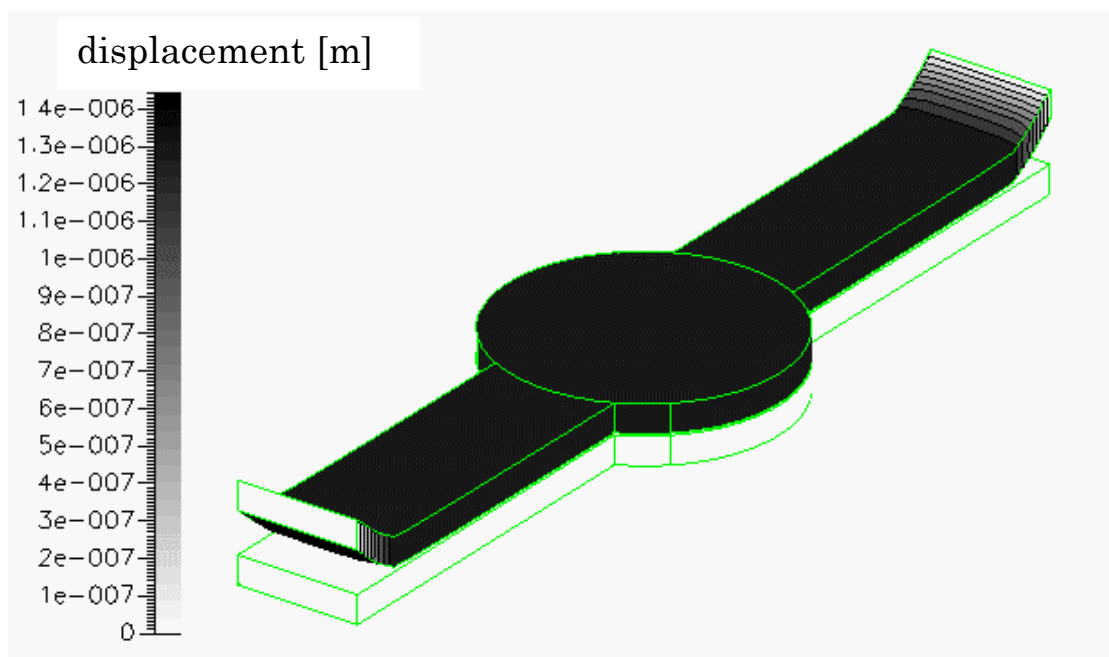


Figure 3.9: FEM simulation showing the displacement of a Ta-Si-N membrane actuated at 25 V, just higher than the pull-in voltage.

Discussion: comparison of results for different calculation methods

Table 3.4: Summary of the valve pull-in voltages based on different methods of calculation.

	$U_p \{V\}$ Without stress	$U_p \{V\}$ 100 MPa stress
Simplified model	11.4	----
Semi-analytical	17.2	46.0
FEM simulations	7.0	24.5

Table 3.4 shows that the pull-in voltage calculated using the simplified model is about a factor two smaller than the one based on FEM simulations which took tensile stress into account. This difference is mainly attributed to the tensile stress that is not taken into account in the analytical calculation based on the simplified model proposed, and which is the dominating approximation. The semi-analytical calculation, which assumes tensile stress, is in agreement with FEM simulations as far as the large influence of the stress in the Ta-Si-N layer is concerned. However, the difference in absolute values obtained can be explained by the geometrical approximation of the semi-analytical model. Therefore, FEM simulation should be the most accurate of the three calculation methods.

Since an actuation voltage of 25 V is considered acceptable, the membrane dimensions based on the simplified analytical model proposed will be kept. The dimensions of the fabricated membrane valve are those calculated in section 3.4.5.

3.5. Different membranes based on the basic shape

Different designs based on the basic membrane shape were tested in order to optimize some characteristics such as actuation voltage or flow resistance. In order to prevent sticking, these different designs have the same membrane mechanical repelling force in the center hole area as the basic valve.

3.5.1. “T”-shaped membrane

The “T”-shaped membrane shown in Figure 3.9, was developed to reduce the actuation voltage. Having a low actuation voltage is very useful for reducing the risks of electrical breakdown in the insulation layer and for simplifying the electronics. The approach chosen to achieve a reduced actuation voltage involved changing the shape of the valve membrane. Since the electrostatic force is proportional to the inverse of the square of the gap distance, the gap between the membrane and the lower electrode should be as small as possible to reduce the actuation voltage. However, a small gap limits the mechanical repelling force of the membrane and therefore increases the risk of sticking. To reconcile these two contradictory characteristics, a double-clamped “T”-shaped membrane with an additional beam was developed (Figure 3.10).

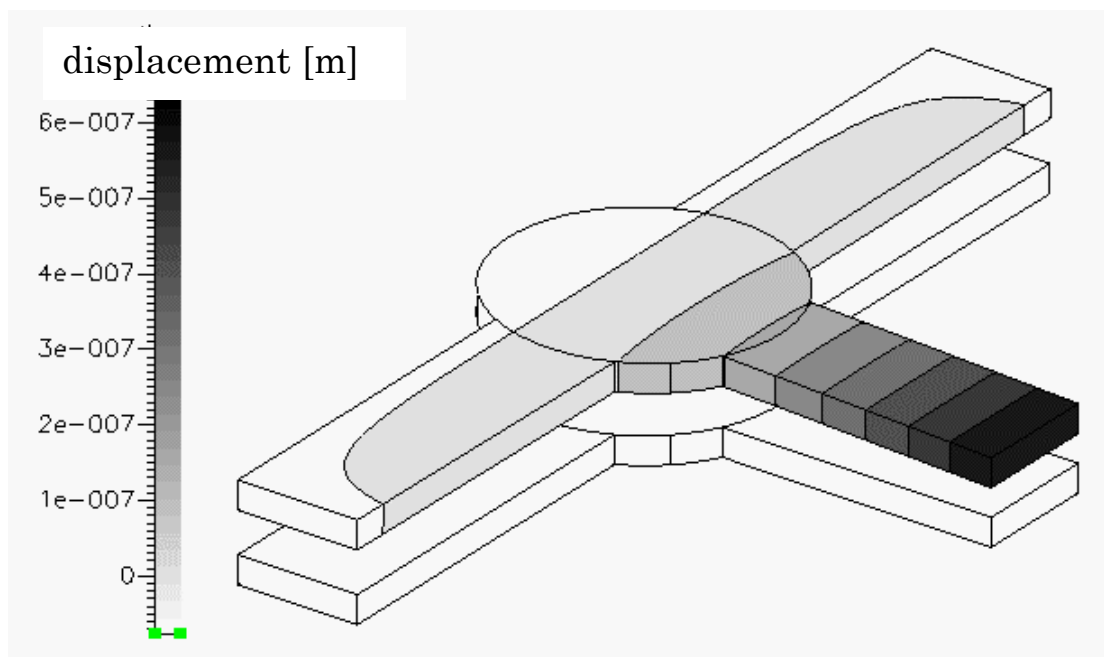


Figure 3.10: FEM simulation showing the displacement of a “T”-shaped membrane actuated at 7 V, just below pull-in voltage. A tensile stress of 100 MPa was taken into account. The additional beam measures 100 μm length.

At the beginning of a voltage actuation cycle, the end of the additional beam is attracted first and touches the lower electrode. This local gap reduction pulls down the rest of the membrane.

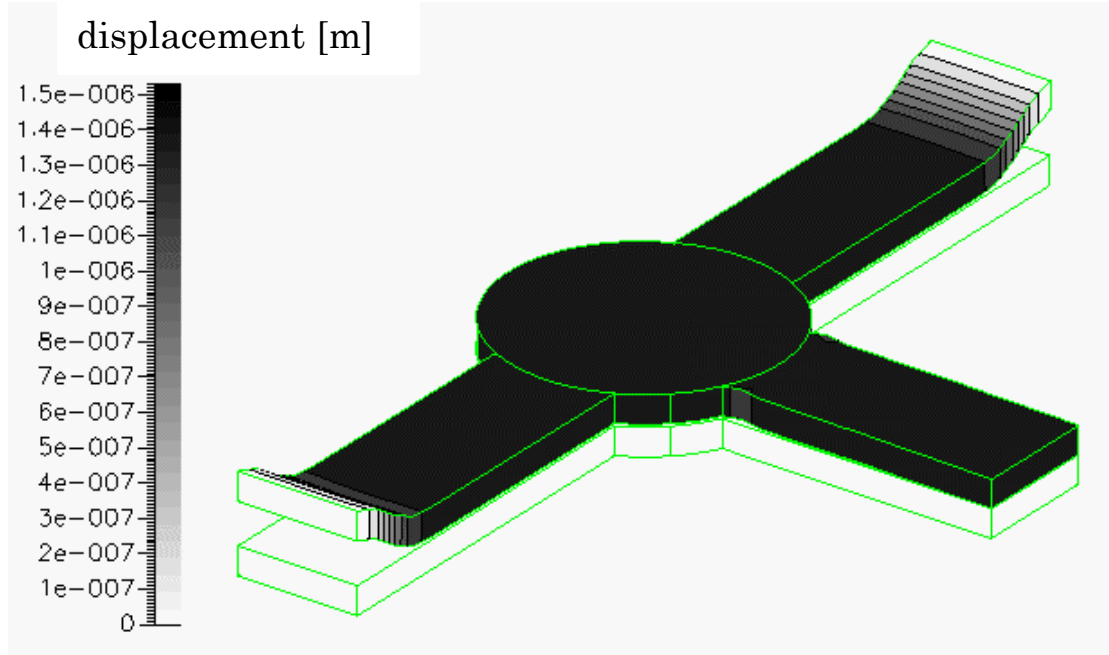


Figure 3.11: FEM simulation showing the displacement of a “T”-shaped membrane actuated at 8 V, slightly greater than pull-in voltage.

It should also be noted that, when actuated, the membrane completely touches the lower electrode (Figure 3.11); this is essential for obtaining a good seal. The additional transverse beam increases drastically the electrostatic pull-down force during an actuation cycle. This is obtained without reducing the mechanical pull-up force in the membrane area on top of the orifice. A strong pull-up force is essential to avoid sticking at the beginning of the opening cycle.

Table 3.5: Comparison of the basic valve and “T”-shaped valve pull-in voltages.

	Basic valve	“T”-shaped valve
U_p {V}	24.5	7.5
100 MPa stress		

Finite element comparison between the basic valve and the T-shaped valve shows a clear reduction by a factor of 3 of the pull-in voltage (Table 3.5). This concept of “T”-shaped membrane could be applicable to other MEMS devices to reduce the actuation voltage where sticking should be avoided only in the central area. For instance, an application can be radio frequency (RF) electrical switches.

3.5.2. High-flow membranes

For some applications, it can be essential to have a larger flow. Existing electrostatic surface-micromachined MEMS valves have been designed for higher flow rates but can only operate at pressures below 0.15 bar [3.5-3.7], which is due to the relatively large orifices used. In the case of the Ta-Si-N valve, the approach adopted was to drastically increase operating flow rates while keeping the switching behavior of the basic valve. Making many valves work in parallel will multiply flow rate by the number of valves, while having the characteristics of the basic valve. Due to the small membrane size of $340\text{ }\mu\text{m} \times 100\text{ }\mu\text{m}$, it is possible to fit many valve in a limited area. It is also possible to actuate each valve in a valve array independently to modulate the flow, as reported by Robertson et al. [3.7]. However, actuating all valves at the same time in pulse width modulation (PWM) is simpler for electrical connections and should give a smooth linear flow response. Therefore, all valves should be electrically connected.

In an area of 1 mm^2 , 20 valves were arranged in order to interrupt the same main flow (Figure 3.12). They were mechanically linked, and therefore all moved at the same time, even though the actuation slope was not steep. The first one that reached pull-in pulled down the others.

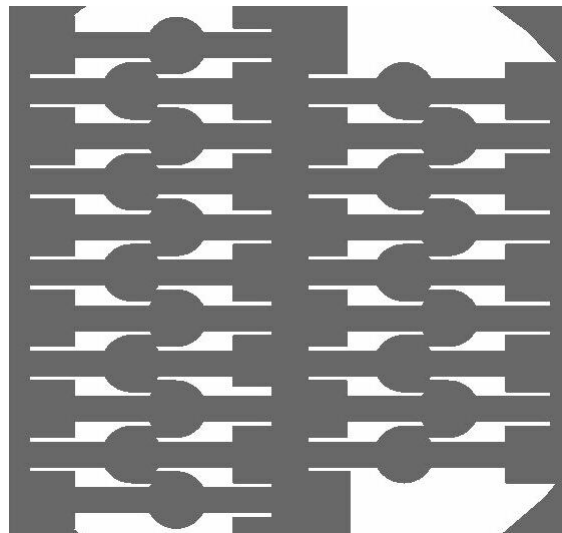


Figure 3.12: Top view of 20 valves membrane working in parallel. Since they are all electrically and mechanically linked, they all move at the same time. The surface area occupied by 20 valves was only 1 mm².

3.6. Conclusion

First, a basic valve was designed according to a proposed, simplified physical model. The model enabled the shape of the membranes to be dimensioned so as to optimize valve sealing and high-pressure handling capability. The model was compared with other methods of calculation, namely semi-analytical approach published by Osterberg et al. [3.23] and FEM simulations. The sufficient agreement between different calculation methods allowed valves to be fabricated with calculated dimensions. The large effect of tensile stress in the Ta-Si-N membrane on the pull-in voltage was demonstrated as well. To optimize characteristics such as pull-in voltage or operating flow rate, different types of valve based on the basic valve design were developed. A simulated reduction by a factor 3 of the pull-in voltage was reached with an asymmetric membrane that bends in flexion and torsion. Another approach investigated was to make many basic valves work in parallel on the same chip to increase operating flow rates. The advantage compared to a single valve with a larger orifice is its capability of handling large pressures.

3.7. References

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Chapter 4

TaSiN Electrostatic Gas Valve

Fabrication

The fabrication challenge was to combine surface and bulk micromachining in order to etch a 10 μm orifice under a freestanding Ta-Si-N membrane that is isolated from the substrate, while ensuring fluidic as well as electrical connections. Different techniques of surface micromachining, such as wet and dry etching, were used for shaping deposited layers, while deep reactive-ion etching (DRIE) was chosen to etch the orifice in the silicon substrate.

The approach adopted was to fabricate a first version of the valve using standard fabrication steps, and then to develop subsequent versions, which were modified to some extent. This reduction of the fabrication complexity was pursued in order to improve the reliability of the valve.

The realization of valve fluidic and electrical connections was linked to chip shape and had to be considered in the development of the fabrication process. The packaging of the valve evolved from the first version, which used a ceramic PCB to the final version, where the silicon chip can be placed directly in a sealed container.

Three different fabrication runs have allowed fabricating three valve versions. The main evolution is due to packaging modifications, which allowed a drastic reduction in the number of fabrication steps. Changing the sacrificial layer from oxide,

used in the first version, to aluminum was essential to demonstrate the suitability of Ta-Si-N for MEMS applications.

The DRIE capability of our facility has undergone some major improvements over the last few years. In 1999, the aspect ratio limit was etching 50 μm holes through a silicon wafer. In 2001, it has become possible to etch holes five times smaller. This improvement has allowed simplification of the fabrication process of the valves and increasing yield.

The main difficulty of the valve fabrication is creating a 10 μm orifice under the valve membrane, while retaining the largest possible bottom electrode surface. Attracting the membrane on to a large surface close to the central orifice is essential to guarantee a tight seal and high gas pressure handling capability (Chapter 3).

4.1. First version: Pyrex-glass-bonded

In the first fabricated version, an attempt was made to develop a process compatible for both, polysilicon and Ta-Si-N as membrane material, in order to be able to compare them. Therefore, all layers deposited before the last one, which was polysilicon, had to be able to withstand the temperature of 1050 $^{\circ}\text{C}$, which was used for deposition and diffusion doping of polysilicon. For this reason, SiO_2 was chosen for the sacrificial layer.

In 1998, the smallest orifice that could be obtained by DRIE was 50 μm . Since this was too large for the valve characteristics desired, a bottom electrode was fabricated in doped polysilicon. This electrode has two purposes: it reduced the size of the orifice and maximized the surface over which the electrostatic force was applied.

The silicon chip was covered with an anodically bonded Pyrex glass chip that had a small channel to conduct gas flow. This technique allows placement of both the inlet and outlet on the backside of the chip, thereby separating electrical connections on the topside from the fluidic connections on the bottom (Figure 4.1).

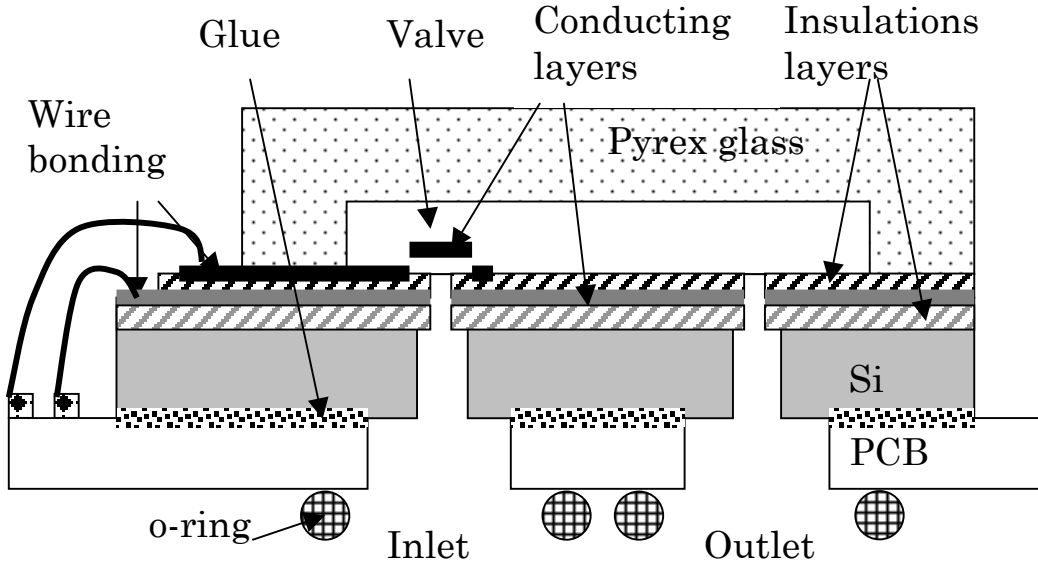


Figure 4.1: Schematic showing the first fabricated version valve of the valve having both gas inlet and outlet on the backside while the electric connections are on the topside. The different materials making up the layers are defined in section 4.1.1.

The consequence for the chip fabrication process was that the Pyrex had to be in contact with a layer compatible with anodic bonding, such as silicon, silicon dioxide, or silicon nitride.

4.1.1. Process

The fabrication process of this version included more than 30 steps and used eight masks. A condensed of the process flow is reported here underlining the critical aspects (Figure 4.2-Figure 4.7). This is followed by a more detailed description in the text.

 : thermal SiO₂

 : LPCVD Si₃N₄

 : phosphorus doped CVD SiO₂

 : silicon wafer

 : phosphorus doped polysilicon

 : Ta-Si-N

 : Pyrex glass

 : Au

(i)

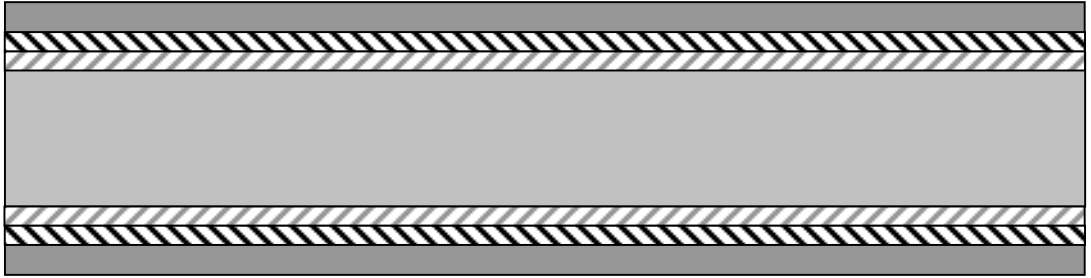


Figure 4.2: Schematic showing the first thin film layers deposited on a double-sides polished a silicon wafer.

(ii)

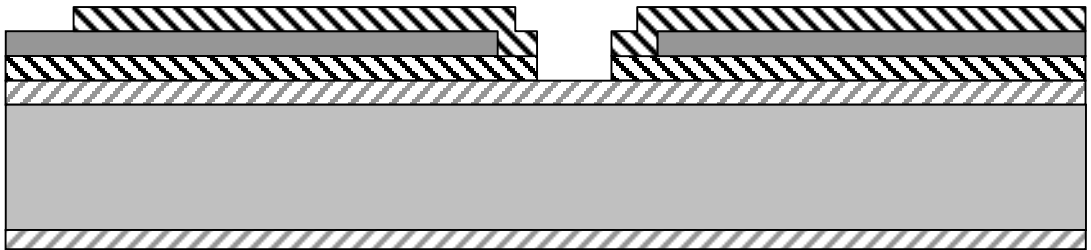


Figure 4.3: View of the plasma-structured 10 μm diameter orifice, after deposition of a Si_3N_4 LPCVD layer. Backside polysilicon and Si_3N_4 have been removed.

(iii)

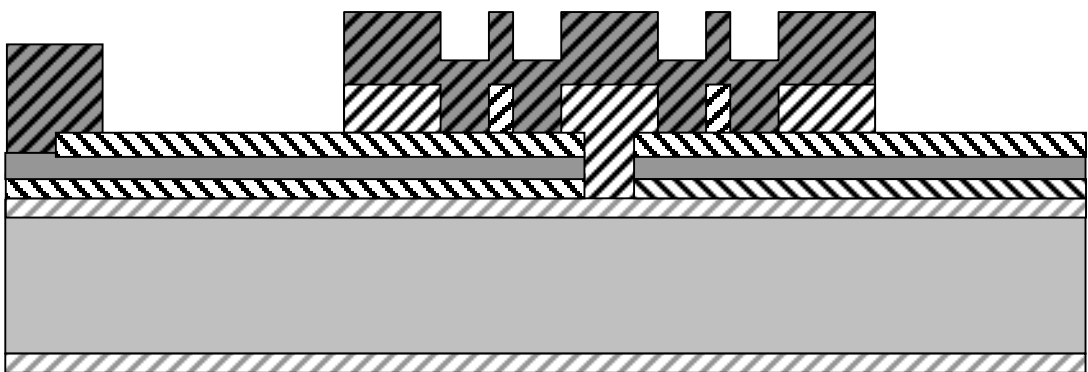


Figure 4.4: Deposition of two sacrificial layers and subsequent structuring. The first layer is a standard CVD oxide, and the second a silicon oxide doped with phosphorus. The orifice is simplified here, as well as on the next drawings.

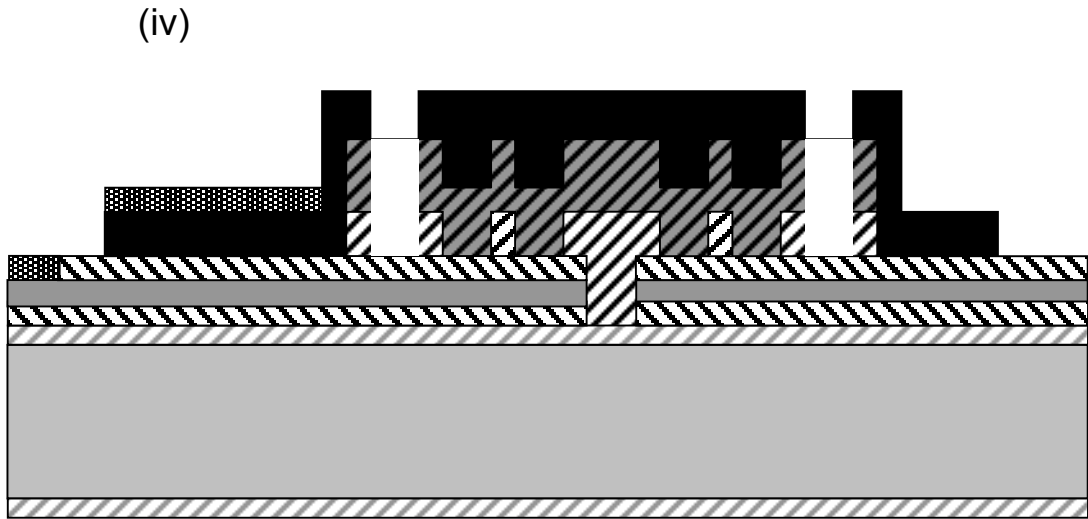


Figure 4.5: The membrane layer is deposited and plasma structured; this layer was either Ta-Si-N as shown here or polysilicon. The sacrificial layer is then partially etched, and a Cr-Au layer is structured by lift-off in the contact area.

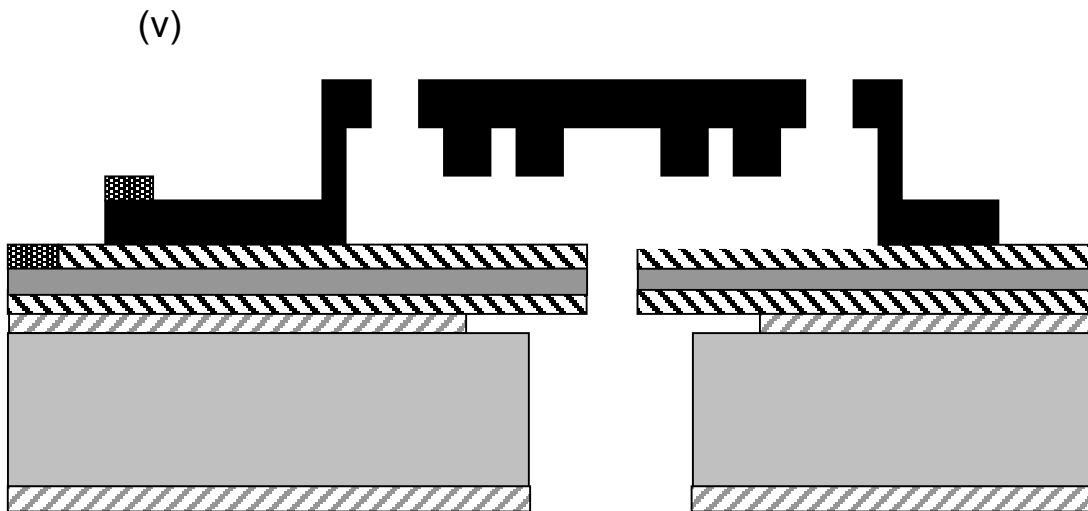


Figure 4.6: A 50 μm hole is etched from the backside with a DRIE process, and the sacrificial layers are removed by wet etching in BHF.

(vi)

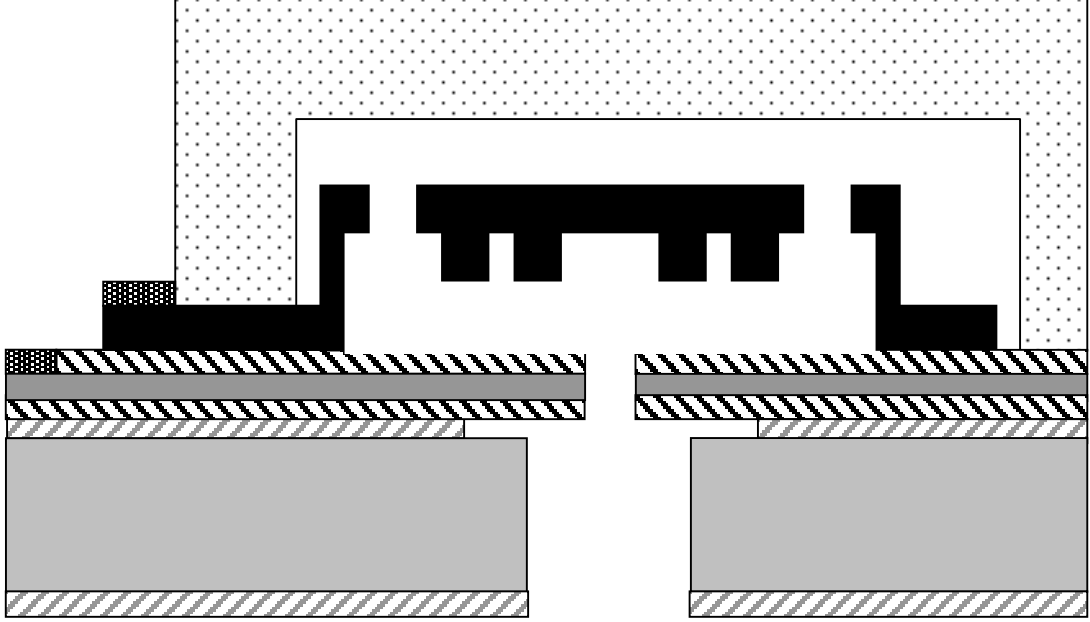


Figure 4.7: Pyrex glass is etched with a DRIE process or HF, and anodically bonded onto the silicon wafer.

In Figure 4.2 a $5000 \text{ \AA}$ silicon dioxide layer is first thermally grown on a $390 \text{ }\mu\text{m}$ thick silicon substrate for passivation, followed by deposition of $2500 \text{ \AA}$ LPCVD silicon nitride layer for electrical insulation in the orifice area. A $1 \text{ }\mu\text{m}$ polysilicon layer is then deposited, and phosphorus ion implanted to reduce its resistivity. The role of this last layer is two-fold: it serves as bottom electrode and minimizes the orifice size to $10 \text{ }\mu\text{m}$.

Figure 4.3 shows a close-up view of the orifice after deposition of a second $2500 \text{ \AA}$ LPCVD silicon nitride layer, used as insulation between the moving membrane and the bottom electrode. The orifice measures $10 \text{ }\mu\text{m}$ in diameter, while the bottom polysilicon electrode has a central hole of $20 \text{ }\mu\text{m}$ diameter. This configuration, close to the technological limit, maximizes the efficiency of bottom electrode. A contact to the polysilicon has been opened.

Then, two CVD SiO_2 sacrificial layers are deposited in order to create an anti-adhesion structure on the membrane (Figure 4.4) [4.1,4.2]; the total height of these layers is $1.5 \text{ }\mu\text{m}$. The

bottom electrode electrical contacts are covered with phosphorus doped silicon oxide in order to create a RIE etch-stop for structuring the membrane layer.

Figure 4.5 illustrates the deposition and plasma structuring of the membrane layer, which can be either Ta-Si-N or polysilicon. The amorphous $\text{Ta}_{49}\text{-Si}_{14}\text{-N}_{37}$ layer is deposited by reactive sputtering of a $\text{Ta}_5\text{-Si}_3$ target in a plasma Ar/N_2 filled chamber. Once deposited at 100 °C, the stress in the Ta-Si-N layer is compressive, but annealing at 450 °C allows conversion of this stress into a tensile stress of about 100 MPa [4.3]. For doubly clamped freestanding structures, it is essential to avoid compressive stress. The patterning of Ta-Si-N was performed in SF_6/O_2 plasma using a photoresist mask. For wire bonding, gold is deposited and structured by lift-off in the contact area.

DRIE is used for etching the 50 μm orifice through the 390 μm thick silicon wafer (Figure 4.6). The wafer is then diced into chips, and the sacrificial layers are removed by wet etching in a BHF bath after dicing. For avoiding adhesion, an anti-stiction process based on sublimation of cyclohexane is used.

Pyrex Glass chips having 10 to 60 μm cavities etched with a DRIE process or BHF are anodically bonded on the silicon chips (Figure 4.7).

4.1.2. Chip fabrication results

The good performance reached of the DRIE resulted in 50 μm cylindrical holes having an aspect ratio of 8 (Figure 4.8). (The chip was broken at the orifice level to take the picture.) An under-etching of 20 μm at the end of the attack, which can be observed in the second picture on the right, is acceptable, since the flow is mainly limited by the 10 μm Si_3N_4 orifice. Since the etching process was optimised for high-aspect-ratio, the surface roughness is not perfect at the end of the etching process.

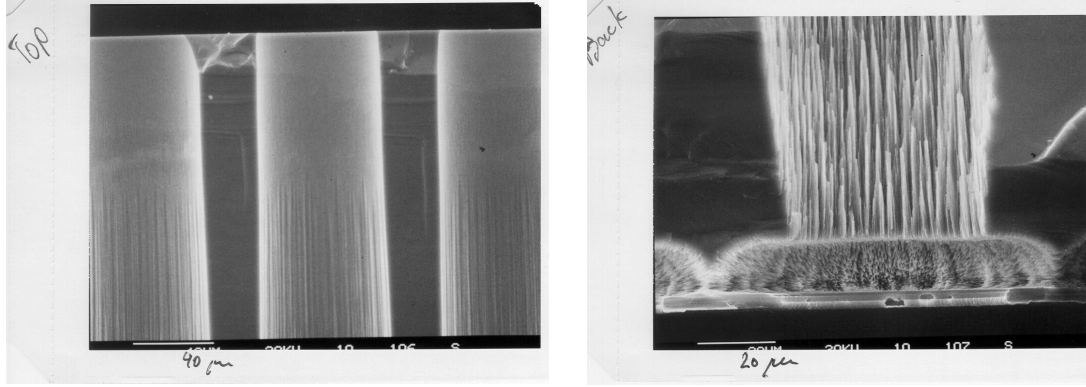


Figure 4.8: SEM views of 50 μm , cylindrical, high-aspect-ratio hole made by DRIE. The left picture shows the beginning of the attack, while the other one shows the end of the etching process.

A distinction has to be made between valves based on Ta-Si-N membranes and those on polysilicon membranes as discussed below.

Ta-Si-N membrane

Different types of valves with Ta-Si-N membranes were fabricated according to the process presented in section 4.1.1 (Figure 4.9). Unfortunately, the step-coverage of the SiO_2 CVD sacrificial layer by Ta-Si-N was not sufficient, and most of the membrane anchors broke (Figure 4.10). This was unexpected, since previously reported freestanding Ta-Si-N beams were shaped on top of aluminum [4.4] and SiO_2 sacrificial layer as well [4.5]. However, previous freestanding Ta-Si-N beam shaped on SiO_2 had thinner sacrificial layers than those patterned on aluminum. This problem of step coverage is not fully understood, but could be linked to charge accumulation at the edges of the insulation layer during deposition.

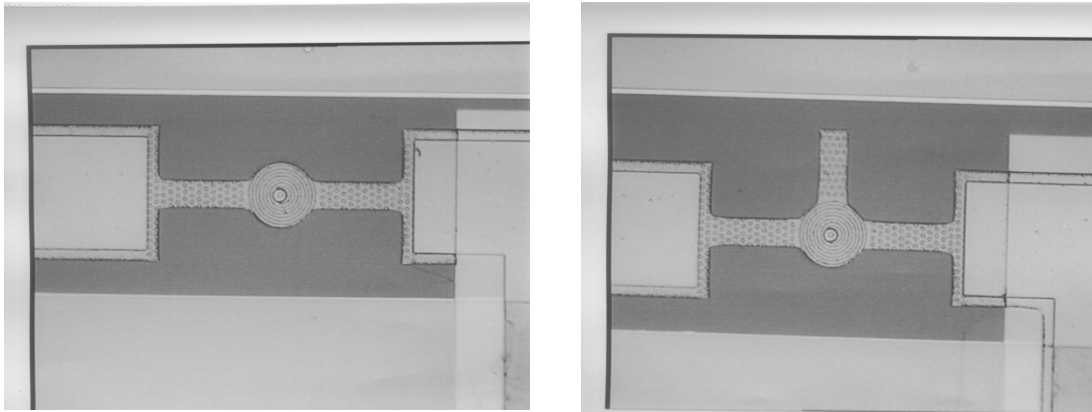


Figure 4.9: Ta-Si-N basic and “T”-shaped valve fabricated according to the process described in section 4.1.1.

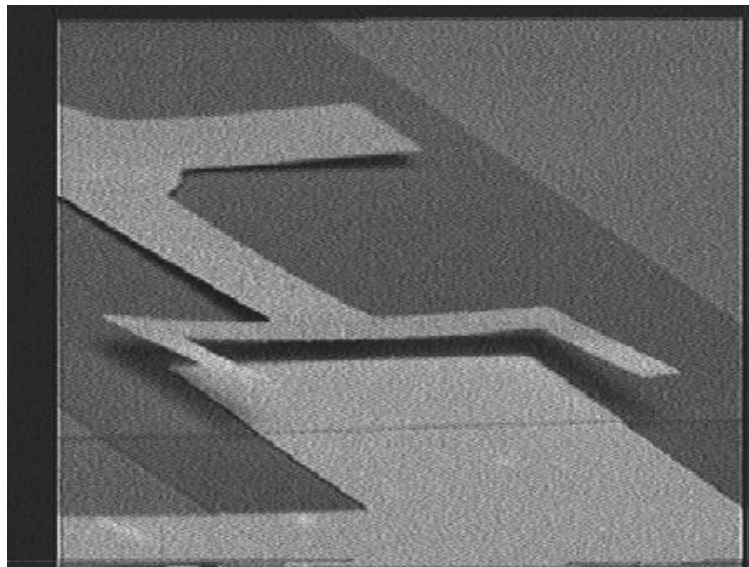


Figure 4.10: SEM view showing the detachment of the membrane in the anchor area. The clamping arms are clearly floating freely in air.

Nevertheless, to be able to test valves, on one wafer, the fabrication was slightly modified. Just before releasing the membrane, an aluminum layer was deposited on top of Ta-Si-N and structured with the mask used for Ta-Si-N to reinforce the membrane anchor. The valve fabricated with this modified process was successfully tested (section 5.3) and provided the impetus for further developments. Since an SiO_2 sacrificial layer was used only to have a fabrication process compatible

with polysilicon, it was replaced by aluminum in later valve versions, to promote better step coverage.

Polysilicon membrane

The fabrication of phosphorus-doped polysilicon membranes presented a major problem, as it was found that this material could not withstand the SiO_2 sacrificial layer etching time of one hour in BHF. The solution would be to use HF instead, but it is more hazardous procedure for personal safety. Unfortunately, chips were destroyed when using BHF, and as a consequence, the polysilicon process was abandoned.

4.1.3. Packaging

Chip packaging had to address two important issues, namely: valve encapsulation with Pyrex glass bonding and electrical and fluidic connections of the valve to a container. Over the course of this work, the packaging has evolved from the concept of a valve chip glued onto a PCB and connected by wire bonding, towards reduction of the use of glue and contacting with contact-pins.

Valve encapsulation

The valves were covered with a Pyrex glass chip that had etched channels to redirect gas flow down to the outlet orifice. The first option taken was to batch process glass bonding [4.6]. This allowed the chips of an entire wafer to be bonded at the same time and then diced them. However, it was found that none of the chips had a movable membrane after this stage. Since the dicing saw is cooled with water, one explanation for this could be that water entered the valve cavity during dicing, resulting in membrane stiction as the valve was dried.

Therefore, the option of bonding chip by chip was investigated. Since in this case, the silicon devices had to be liberated chip-by-chip, this prolongs the fabrication process.

Fortunately, this approach was successful, and many working valves were encapsulated this way (Figure 4.11).

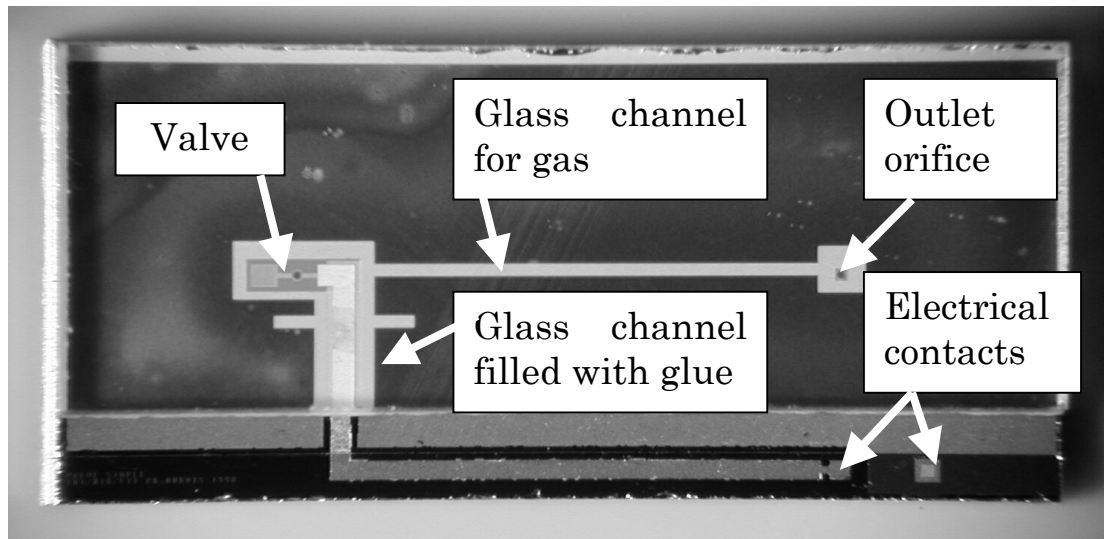


Figure 4.11: Photo picture of an HF-etched Pyrex glass chip and bonded onto the silicon valve. The bright areas are channels in the glass. The chip measures $8.5 \times 4.0 \text{ mm}^2$

However, it was found that some valves experienced membrane stiction during the anodic process. Because anodic bonding requires high voltages of about 1000 V, charges might accumulate on the deposited layers and cause membrane adhesion.

Since the Ta-Si-N protruded from the polysilicon surface, a channel in the glass was etched to prevent contact with Ta-Si-N/polysilicon along the electrical tracks (Figure 4.11). To ensure tightness, this channel had to be filled with glue using capillary forces. Nevertheless, even if it is possible to let the glue enter the channel without touching the valve membrane, the process had a low yield due to the difficulty of the manipulations.

PCB- based packaging concept

In the first concept, the valve chip having a Pyrex glass cover was glued onto a PCB and electrically connected with wire bonding. The chip was placed in a PVC box, in which gas tightness was achieved using o-rings (Figure 4.12). Pyrex glass

bonding, gluing and wire bonding proved to be very tricky steps that were time consuming.

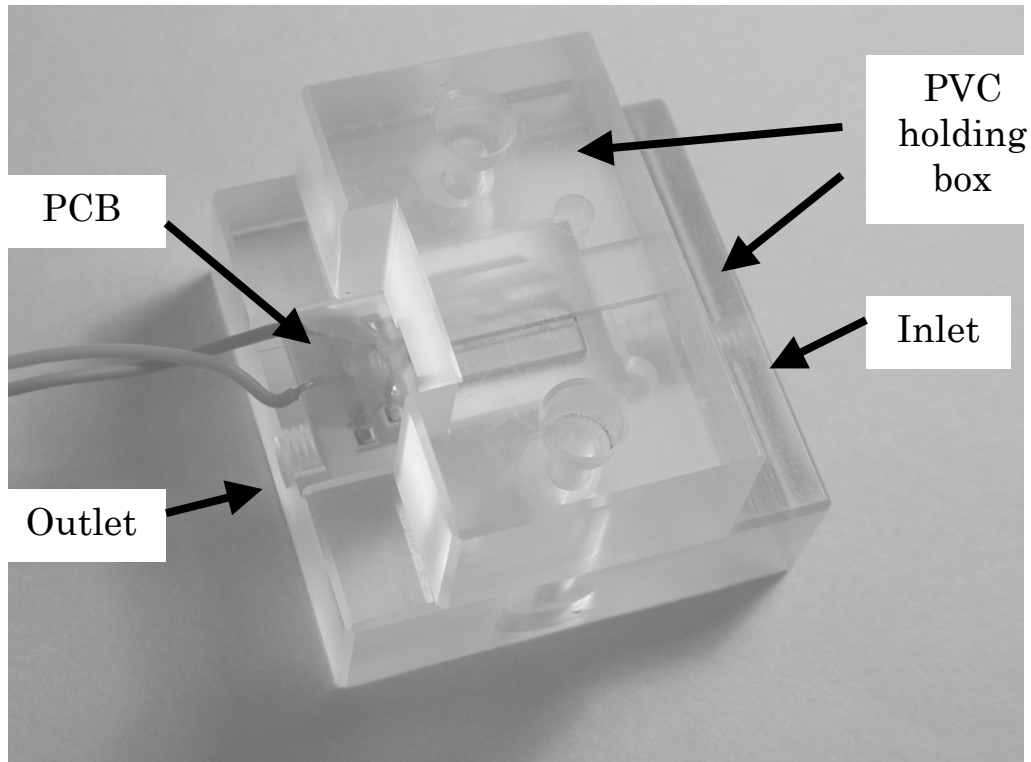


Figure 4.12: Picture showing the first packaging concept.

Glue was used for three different purposes: (1) to fill a channel in the glass, (2) to fix the chip onto the PCB and (3) to encapsulate wire bonding. The properties of the glues have to be adapted to these different problems, and therefore different glue types were tested. It was found that when tightness is important, in the glass channel and between the chip and PCB, epoxy glue (H77, Polyscience) was most suitable. To protect wire bonding, a different epoxy glue (fast Araldit) was used.

Figure 4.13 presents the valve chip glued and wire-bonded on its PCB support, which has two holes in it, one under the valve and the other one under the outlet silicon orifice.

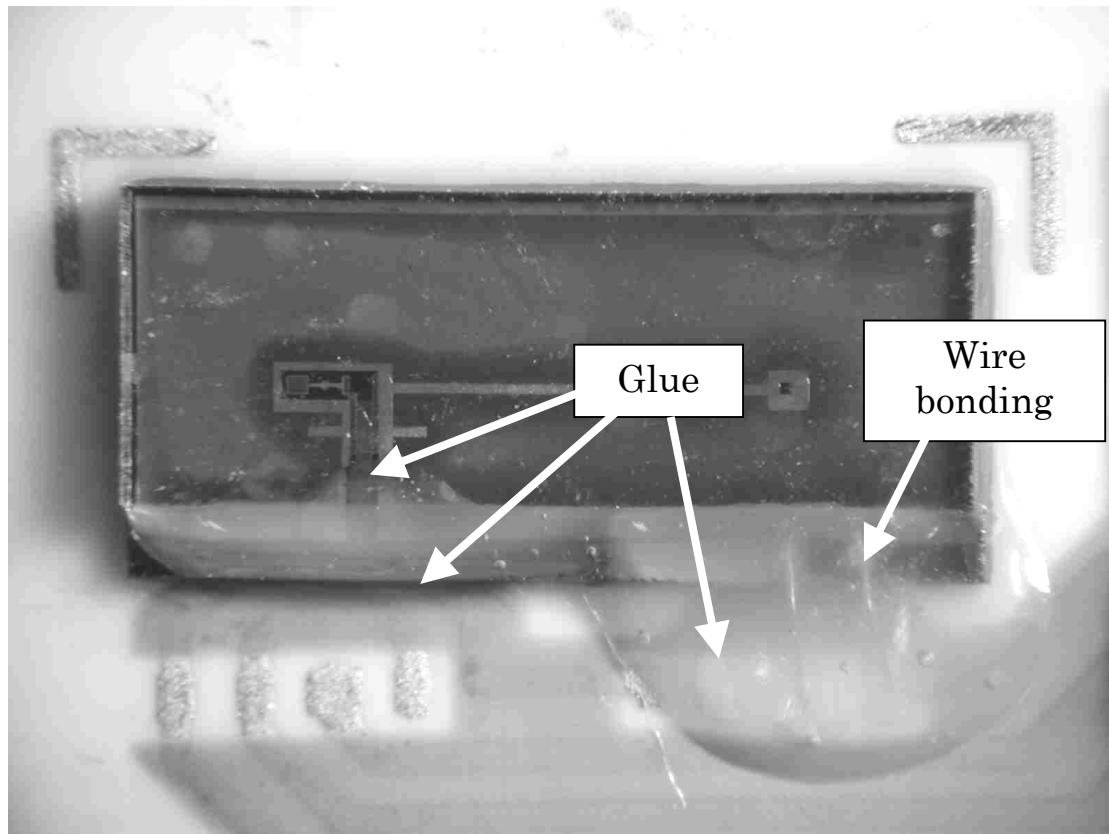


Figure 4.13: Top view of the valve chip glued onto its PCB support and wire-bonded.

This packaging concept was not ideal, since it is time consuming and had a low yield due to the difficulty of the various manual steps. Here, a modified packaging concept was developed, which kept the Pyrex glass anodic bonding step, but no longer depended on gluing to a PCB or wire bonding.

Contact-pin-based packaging concept

In the first packaging concept, a PCB was used to electrically contact the silicon valve chip by wire bonding. Eliminating the PCB and contacting directly the valve by spring contact-pins instead obviated the two difficult steps of gluing and wire bonding (Figure 4.14). The fluidic tightness was achieved the same way as in the first version using o-rings. Valves were successfully tested in this container.

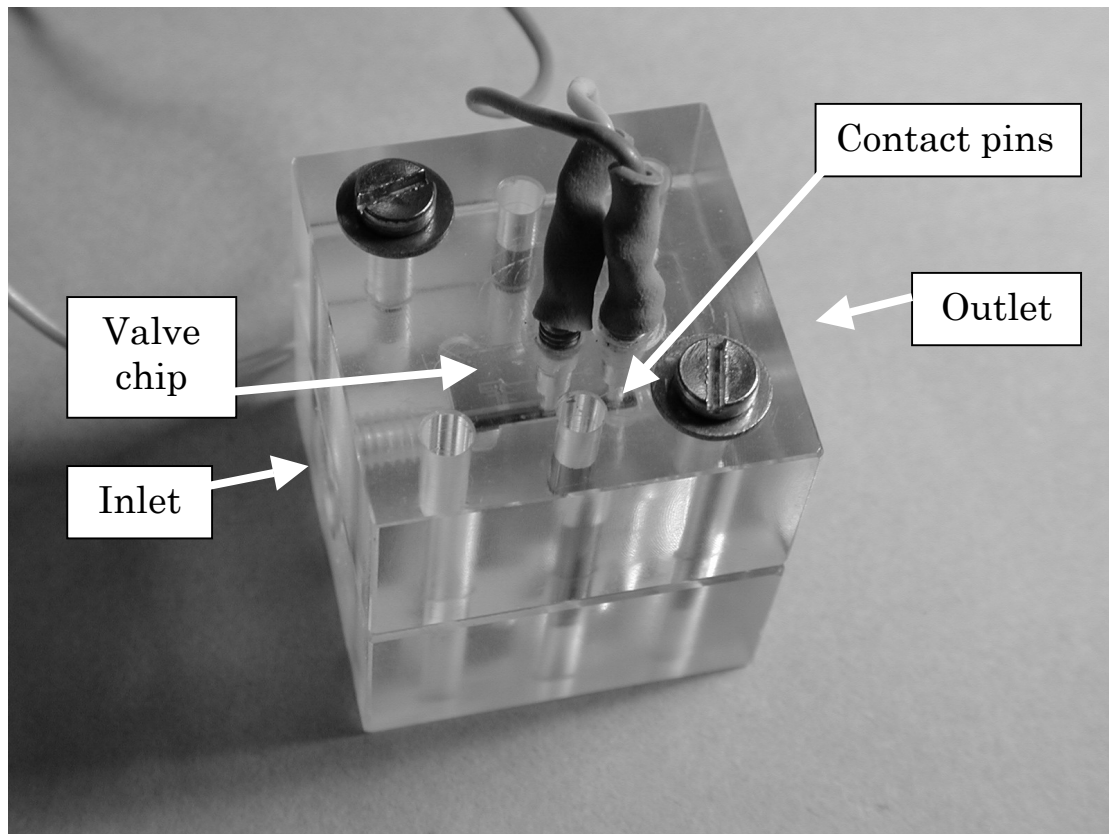


Figure 4.14: Photo showing the valve, encapsulated with a glass cover and packaged in a small holding PVC container having spring contact-pins.

4.1.4. First version conclusion

Though the first fabrication process did not have a high yield, it worked well enough to produce functioning Ta-Si-N membrane-based valve once an additional aluminum membrane layer had been deposited to anchor Ta-Si-N membrane. It was not possible to carry the polysilicon valve through to the end of the processing, due to undesired etching of the polysilicon in BHF. It appeared that using SiO_2 as a sacrificial layer increased the tendency of the Ta-Si-N anchor to detach. This layer was therefore replaced by aluminum in later developments.

This first valve version encapsulated with Pyrex glass was tested in two different types of packaging container box. With the first one, it was necessary to glue the chip onto a PCB and wire bond the electrical connections. In the second approach, the electrical contacts were made by contact-pins and no PCB

was required. The second container was more reliable than the first one, mainly because of the reduced use of glue.

In fact, glass Pyrex bonding was necessary to protect valve membranes because of the use of glue. However, since the use of contact-pins proved to be easier, subsequent developments did not necessitate the use of glass anodic bonding for packaging.

4.2. Second version : double Ta-Si-N layers

The developmental goal of this version was to solve the main problems encountered in the first version, which were Ta-Si-N membrane anchor detachment and complexity of the fabrication. In order to limit the number of materials, it was decided to use two layers of 1 μm Ta-Si-N as the electrodes.

The packaging was also simplified to eliminate the need of anodic bonding, wire bonding and gluing. The valve chips were directly placed in an adapted container that had the inlet in the bottom and the outlet in the top (Figure 4.15). The electrical contacts were made with contact pins. This configuration without a PCB reduced the size of the valve chip by a factor 2 to yield outer chip dimensions of 4.4 mm x 4.4 mm.

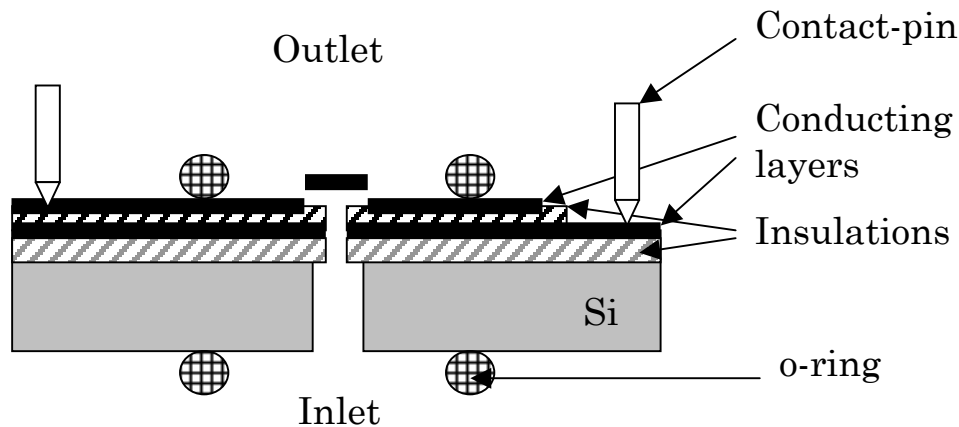


Figure 4.15: Schematic showing the second valve version having gas inlet on the backside and gas outlet on the topside. Contact pins are used to establish electrical contact to the conducting layers.

4.2.1. Process

Compared to the first version (section 4.1.1), polysilicon is replaced by Ta-Si-N, the SiO_2 sacrificial layer is replaced by aluminum, and nitride and gold depositions are omitted. Moreover, the insulation layer over the bottom electrode is CVD SiO_2 . The number of masks has been reduced from 8 to 5.

To start, the fabrication process, a 5000 Å thermal oxide is grown on a 390 µm thick silicon wafer. A 1 µm Ta-Si-N layer is then sputtered on the oxide and plasma-structured. For the electrical insulation layer, a 2500 Å CVD silicon oxide layer is deposited and shaped by BHF wet etching (Figure 4.16). A single aluminum sacrificial layer of 1.5 µm is deposited by e-beam evaporation and patterned in an aluminum-etching bath. This is followed by the deposition and plasma-structuring of a final 1 µm Ta-Si-N layer for the membrane (Figure 4.17). A 50 µm hole is etched in the silicon substrate from the back using DRIE. The wafer is then diced into valve chips. After dicing, final removal of the aluminum layer with isotropic etching in an Al-etch solution liberates the membranes (Figure 4.18).

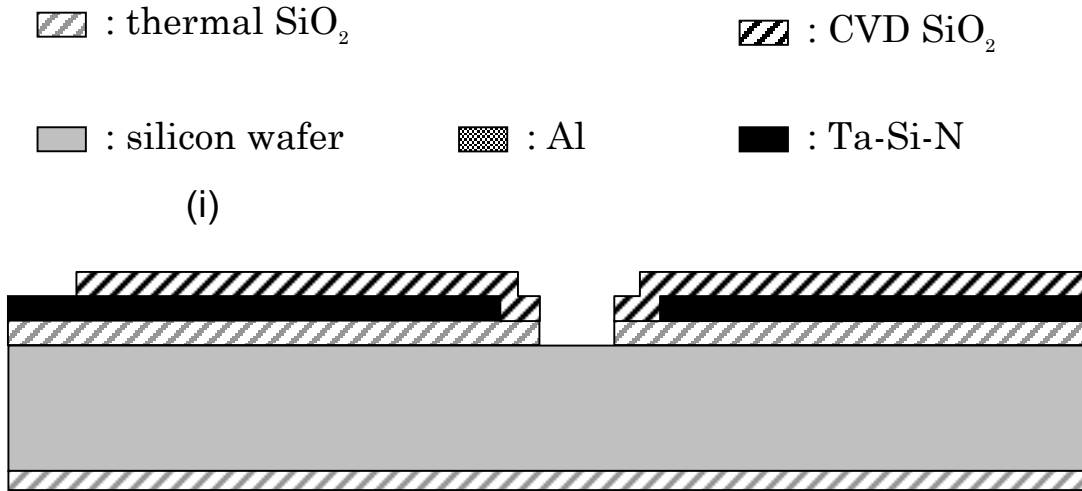


Figure 4.16: Schematic showing the first thin film formation and structuring steps.

(ii)

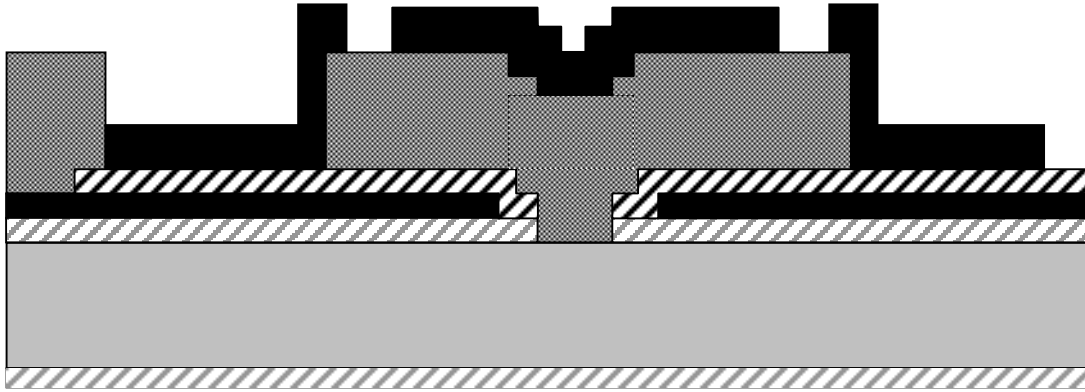


Figure 4.17: The Ta-Si-N membrane is sputtered and patterned on top of a unique aluminum sacrificial layer.

(iii)

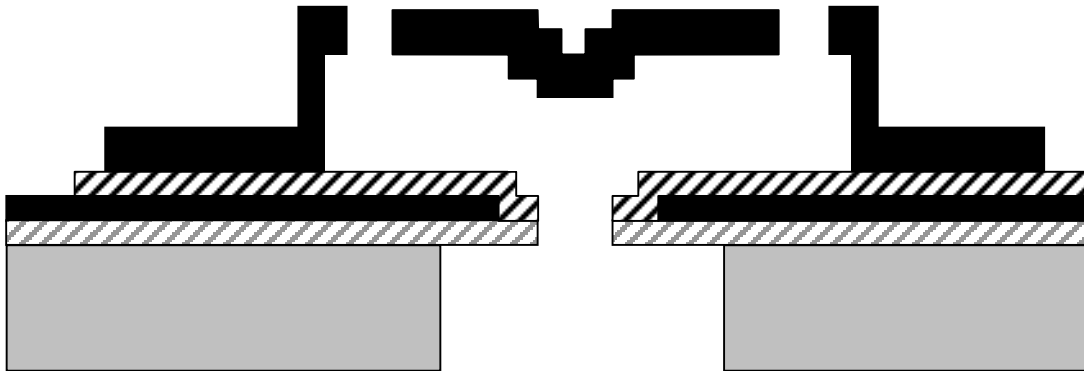


Figure 4.18: A 50 μm hole is etched from the backside with a DRIE process, and the sacrificial layer is removed by wet etching.

4.2.2. Chip fabrication results

Membrane

Different types of valve membranes were successfully fabricated according to this process and tested (section 5.3), (Figure 4.19, Figure 4.20, Figure 4.21, Figure 4.22).

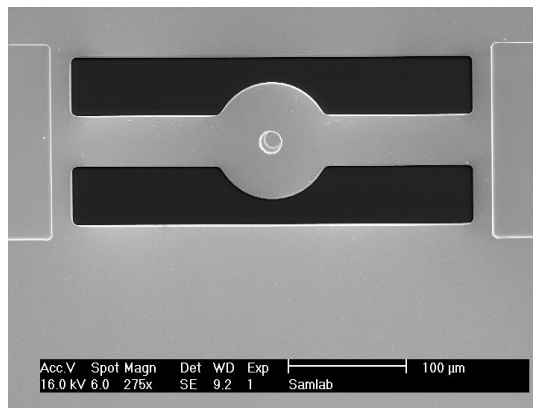


Figure 4.19: Basic valve

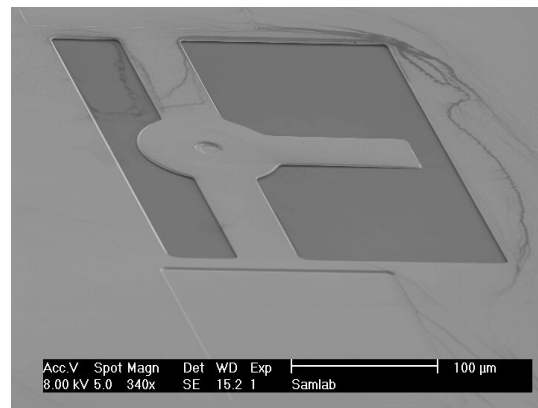


Figure 4.20: "T"-shaped valve valve

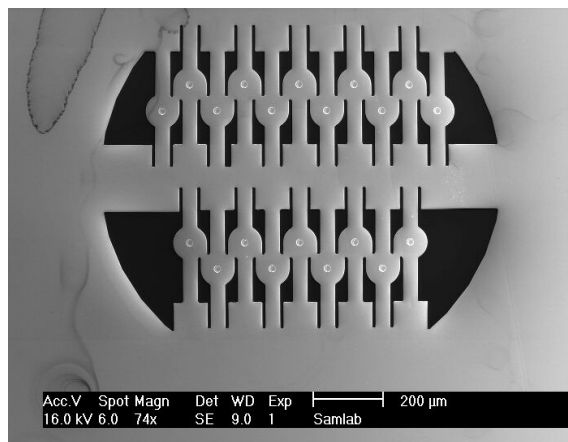


Figure 4.21: An array of 20 parallel valves.

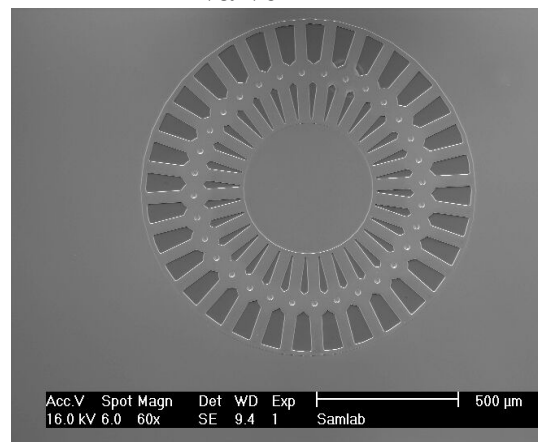


Figure 4.22: An array of 32 parallel valves .

The anchor detachment problem was not observed anymore, and the Ta-Si-N layer never broke mechanically during valve actuation or flow circulation at below 2 bar differential pressures (Figure 4.23).

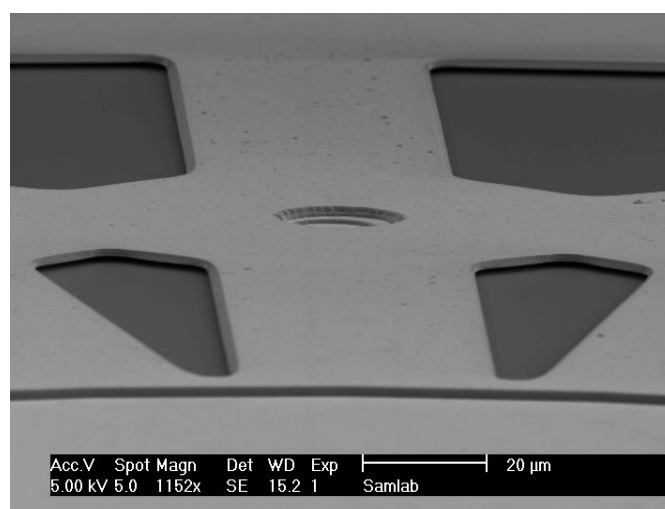


Figure 4.23 SEM view showing the anchor of the valve membrane.

Stiction

In this version, membrane stiction was not a problem once the chips were released and dried, even though no anti-adhesion bumps were created in the lower membrane surface.

Insulation

The electrical insulation between the electrodes is the most sensitive part of the device. During testing, random occurrence of breakdown led to destruction of the valve membrane due to an increase of current, usually in the orifice area (Figure 4.24). Such behavior could occur even at voltages below 40 V, and for most devices around 70 V.

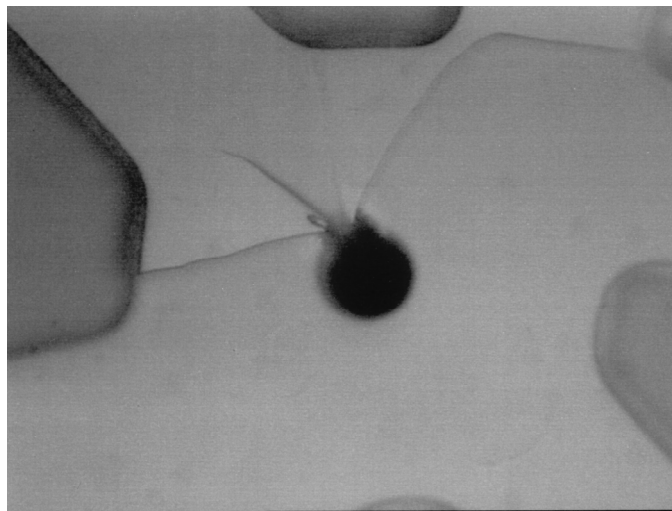


Figure 4.24 Photo showing how electrical breakdown resulted in the destruction of the center of a Ta-Si-N membrane.

Since the distance separating the two electrodes through the gas is much longer than through the insulation layer, the electrical breakdown occurred in the SiO_2 insulation layer. It is likely that the CVD SiO_2 insulation layer had pinholes that favour this phenomenon, especially on the sidewalls of the Ta-Si-N in the orifice area where the oxide was thinner. One solution would be to replace this insulation by a thicker, better quality material such as LPCVD SiO_2 or Si_3N_4 . In the first process however, LPCVD nitride exhibited cracks, and therefore

SiO_2 is preferred. Unfortunately, LPCVD SiO_2 is not available in our lab.

Therefore, another solution was to eliminate the bottom Ta-Si-N layer, and use a thermal oxide for the sacrificial layer, which is the best insulation layer available. However, such a process change necessitated 10 μm hole to be etched by DRIE instead of 50 μm , if valve characteristics were to be preserved. It was only in 2001 that DRIE performance had reached a sufficient level to fabricate a valve based on this process. Details of this process change will be discussed in section 4.3.

4.2.3. Packaging

The packaging of this valve was quite simple, with the valve chip being sandwiched in between the two parts of the container (Figure 4.25). The inlet was in the bottom half, and the outlet was in the top half of the container. Contact pins were used to establish electrical contact to the two Ta-Si-N layers.

The container was a 2.7 cm^3 cube having an aluminum bottom contacted to the ground to avoid charge accumulation in the silicon part of the chip. The top part was made in Plexiglas.

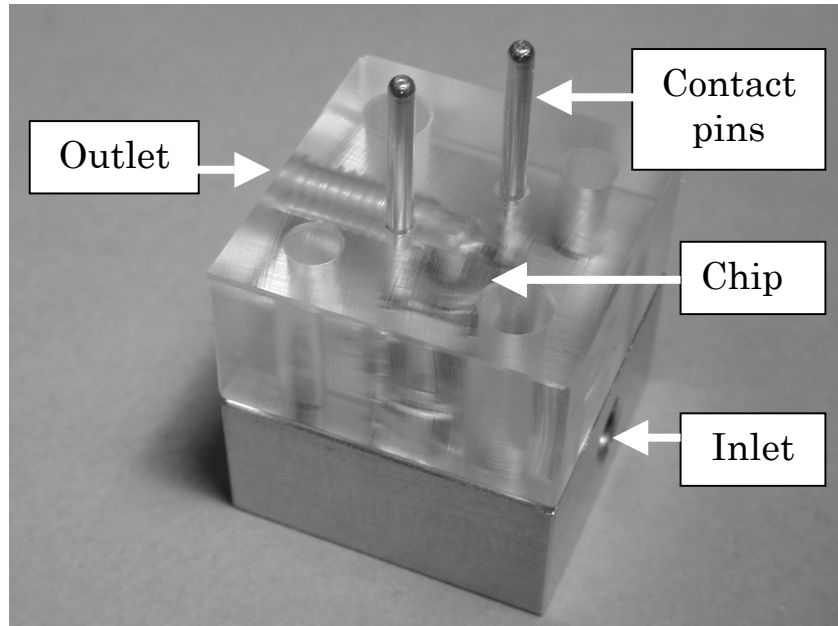


Figure 4.25 Double-Ta-Si-N-layer valve container for packaging. The side of the cube measures 14 mm.

4.2.4. Conclusion

Many working valves were fabricated according to this process based on a double Ta-Si-N layer. Replacing the SiO₂ sacrificial layer with aluminum solved the membrane anchor detachment problem, and allowed many changes in the fabrication process, which ultimately simplified it. However, since this process optimised for Ta-Si-N was not compatible with polysilicon due to the use of aluminum, a way of comparing the two materials for the valve membrane was not found.

Testing showed that the quality of the CVD SiO₂ insulation layer was not sufficient. One solution to this problem would be to eliminate the bottom Ta-Si-N layer, and making smaller diameter DRIE holes while using a thermal oxide as insulation layer. The absence of a glass cover allowed for smaller, simplified packaging, in which valve chips could be introduced and replaced directly without gluing.

4.3. Third version: 10 μm silicon orifice

The main advantage of this process was that it used a high-quality, thermal-silicon-oxide insulation layer, which is known to contain no pinholes [4.7]. Due to the possibility of etching reduced-diameter 10 μm orifices right through a 300 μm -thick wafer, the process was shortened to only three masks and three different thin film layers. The packaging approach was the same as that used in the second version.

4.3.1. Process

To begin the fabrication process, a 5000 Å thermal oxide was grown on a 300 μm -thick-silicon wafer (Figure 4.26). A single aluminum sacrificial layer of 1.5 μm was deposited by e-beam evaporation and patterned in an aluminum-etch bath. This was followed by the deposition, annealing at 470 °C, and structuring of a 1 μm Ta-Si-N layer for the membrane. A 10 μm hole was etched in the silicon substrate from the back using DRIE (Figure 4.27); this etching performance has recently become

possible due to progress made in DRIE technology. The backside SiO_2 was then removed, as well as the SiO_2 in the bottom of the holes. Finally the wafer was diced into valve chips, and the sacrificial layer was removed after dicing (Figure 4.28).

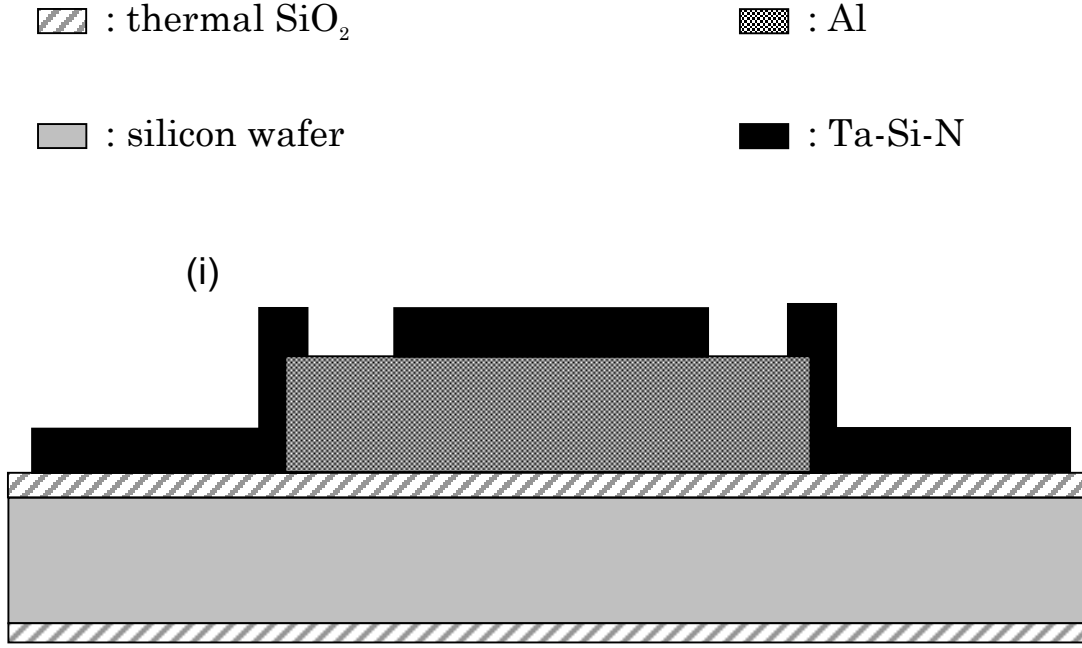


Figure 4.26: Schematic showing the first steps involving thin film formation, deposition and structuring.

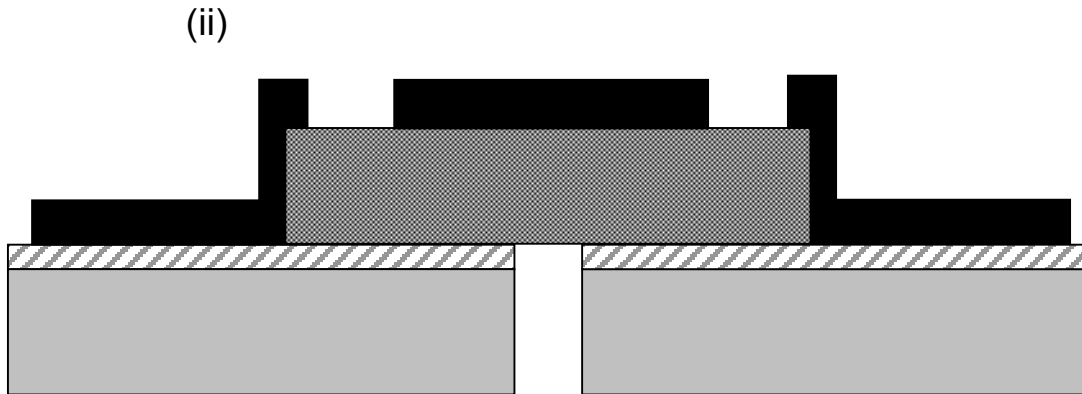


Figure 4.27: A 10 μm hole is etched from the backside with a DRIE process, and the SiO_2 is removed with BHF on the back side as well as in the bottom of the orifice.

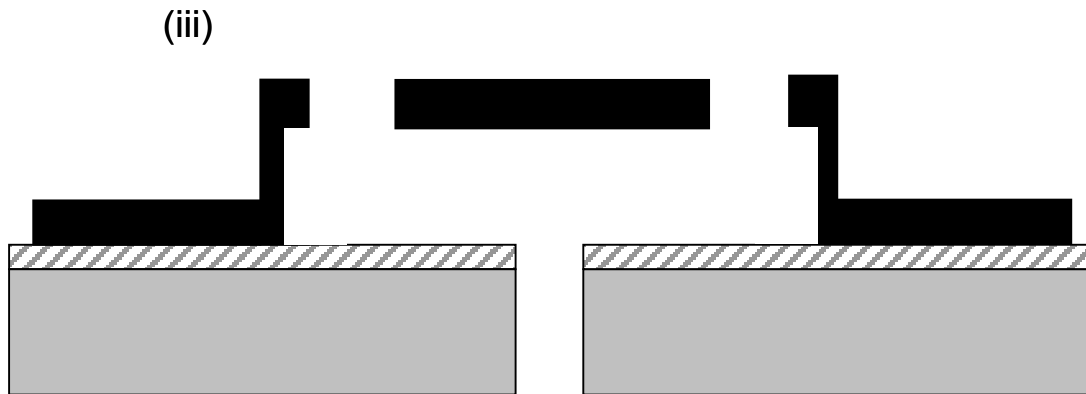


Figure 4.28: Finally, chips are diced and released. In the package container, the electrical contact on silicon is made from the backside.

4.3.2. Fabrication results

Different types of valve membranes were successfully fabricated according to this process and tested (Figure 4.29, Figure 4.30, Figure 4.31, Figure 4.32).

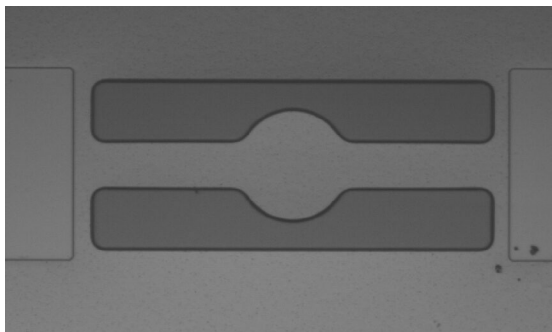


Figure 4.29: Basic valve

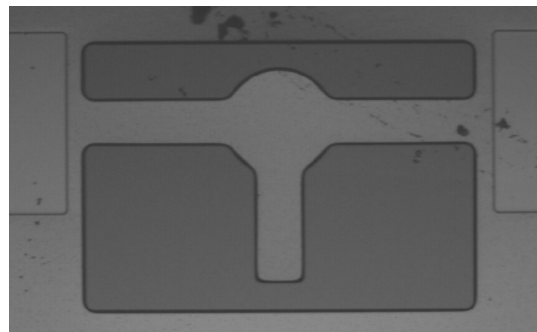


Figure 4.30: "T"-shaped valve

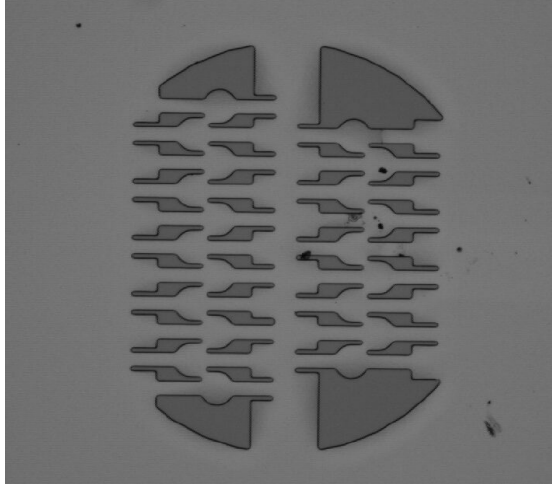


Figure 4.31: Array of 20 parallel valves.

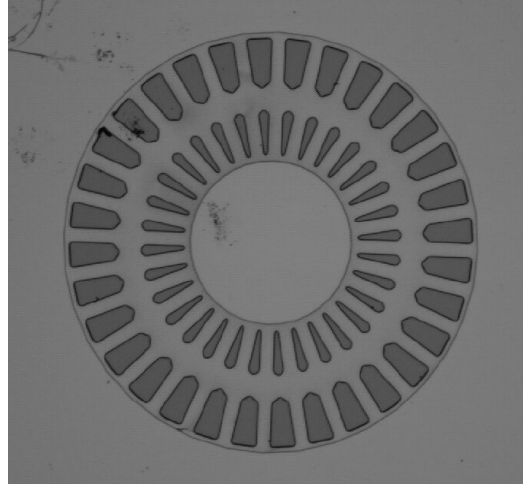


Figure 4.32: Array of 32 parallel valves.

In contrast, to the membranes fabricated with the other processes, there was no depression in the central region, which contacts the orifice.

Insulation layer

As will be explained in section 5.7.2, no breakdown was observed in the thermal oxide insulation layer. Therefore, the reliability of the device was improved by changing of the insulation layer from a CVD oxide to a thermal oxide as used in this version.

The increase in thickness of the insulation layer from 2500 Å in the previous process to 5000 Å have had the side effect of more than doubling the actuation voltage (section 5.3). In future developments, thinner oxide layers will need to be investigated.

Orifice

DRIE of a 10 µm diameter orifice through the wafer is the main difficulty of the process. At the present time, the uniformity of the etching is not perfect; the diameter of the orifices close to the membrane vary between 0-12 µm. On a wafer, about 70 % of the holes are in the range of 8 to 12 µm diameter.

4.3.3. Conclusion

Many working valves were fabricated according to this process. This concept based on 10 μm orifices and using SiO_2 as sacrificial layer, has enhanced the reliability of the valves and reduced the complexity of the fabrication to only three masks. In order to achieve better uniformity, improvements are required in the DRIE processing. To reduce the actuation voltage, future developments should use thinner oxide layers.

4.4. Conclusion and outlook

Different types of working valves were fabricated according to three different processes. The process evolution, enabled by technological advances, resulted in reduced complexity of the fabrication and increased reliability of the valves. The first process, based on eight masks, was very difficult to conduct to the end, even for a few chips, while the last process used only three masks to obtain a yield above 50 %.

Comparison of polysilicon membranes with Ta-Si-N membranes necessitated a process that caused Ta-Si-N membrane anchor detachment. This aspect of the study was abandoned. The packaging evolved from one using glue, Pyrex bonding and wire bonding, to a container where the chips are directly held and connected. In future developments, reducing the thickness of the thermal oxide insulation layer to minimise the actuation voltage should be investigated.

4.5. References

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Chapter 5

TaSiN Electrostatic Gas Valve Characterization and Testing

The purpose of the characterization and testing carried out is to observe valve behavior in order to (1) validate the models used for dimensioning, (2) to be able to identify the causes of eventual problems and (3) to define guidelines for optimization. Since for all valve versions membrane shapes and gap heights were kept identical, the general behavior are similar. The differences between versions are mainly due to differing insulation layers and orifices diameters.

In order to characterize the valves, flow, leakage, actuation voltage and switching times were measured and compared with calculated. Breakdown risk was also evaluated, and long-term measurements realized. Valve characteristics were also compared to electrostatic microvalves reported in the literature.

5.1. Flow

The open flow of single valves and valves working in parallel was measured and compared with the flow model used for valve design.

5.1.1. Single valve

First, the nitrogen flow of the three valve versions was measured with a Bronkhörst flow meter placed after the valve (Figure 5.1). Since in the first version, a glass channel linked two orifices in these valves (section 4.1), the flow was much more restricted than for a single orifice.

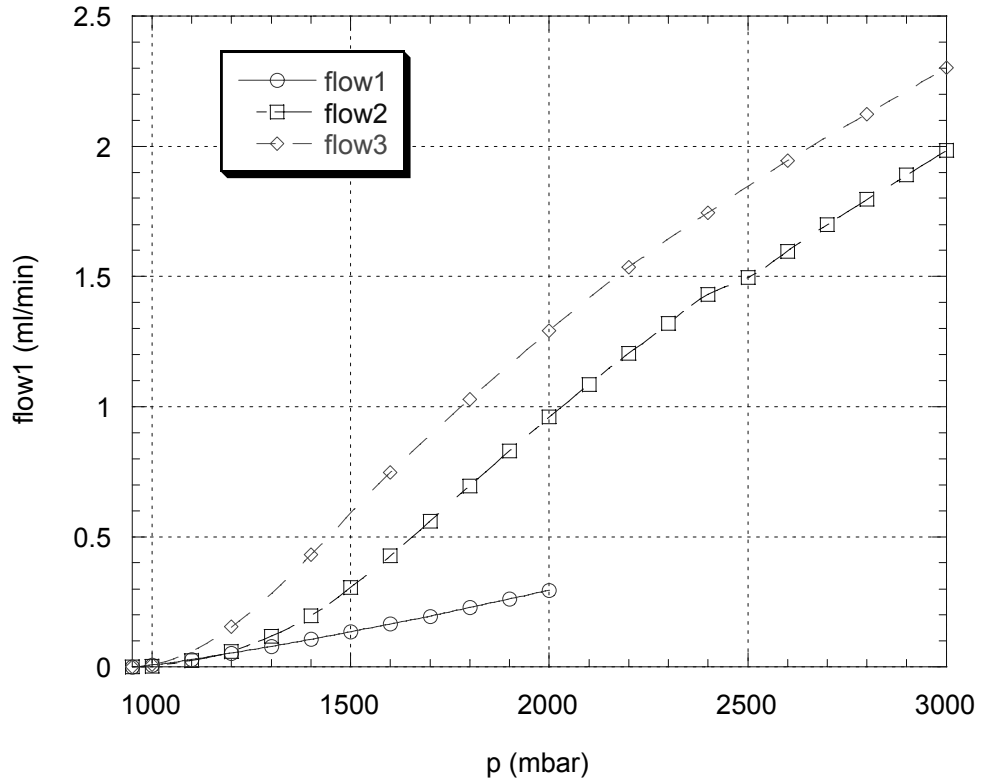


Figure 5.1: Flow vs. pressure for the three different valve versions. Flow 1 was the data set obtained for 1st version valves, flow2 for 2nd version valves, and flow 3 for 3rd version valves.

Gas flow calculation

As explained in appendix A, an open valve is comparable to a nozzle having no viscosity effect considered within its inner channel. For nozzles, James et al. have linked isentropic flow theory to formulas containing only measurable parameters such as pressure and flow rate [5.1].

Two domains can be distinguished: the sonic or choked domain flow and the subsonic domain. For pressure ratios higher than 1.9, the flow rate does not depend on the outlet pressure p_{ou} (5.1), while for the subsonic domain, the calculation is more complex (5.2).

As James et al. have stated, it is possible to introduce a parameter α in their formula to take into account fluid viscosity effect due to the change of cross section around the nozzle. This parameter has to be measured. Since the valve has simple orifice geometry, data provided by the fluidic component manufacturer Lee Company is used to determine this coefficient, which is contained in their parameters. However, this valve manufacturer using a different way of calculation [5.2], the formulas he proposed had to be derived from the isentropic theoretical formulas from James et al. by introducing slight approximations valid only for diatomic gases (5.3)(5.4). The head loss coefficient α being not explicitly given by the valve manufacturer, it is calculated by comparing the flow rates of the two formulas (5.1)(5.3), at choked flow for nitrogen.

For the sonic or choked flow domain, the formula for volume flow rate from James et al.:

$$Q = \alpha \frac{\pi d_h^2 \sqrt{3}}{4 \rho^2 \sqrt{\frac{\gamma R T}{M}}} p_{in} \left(1 - \left(\frac{2}{\gamma + 1} \right)^{\gamma/(\gamma-1)} \right) \quad (5.1)$$

For subsonic domain, the formula for volume flow rate from James et al.:

$$Q = \alpha \frac{\pi d_h^2 \sqrt{3}}{4 \rho^2 \sqrt{\frac{\gamma R T}{M}}} \sqrt{\frac{p_{in} - p_{out}}{p_{in}} \left(\frac{2\gamma}{3} - \frac{p_{in} - p_{out}}{p_{in}} \right)} \quad (5.2)$$

For sonic domain or choked flow, the formula for volume flow rate from Lee Company, containing

the approximation $\left(\frac{2}{\gamma + 1} \right)^{\gamma/(\gamma-1)} = 0.466 \approx 0.5$ for

$$(5.3)$$

diatomic gases ($\gamma=1.4$): $Q = \alpha \frac{\pi d_h^2 \sqrt{3}}{8 \rho^2 \sqrt{\frac{\gamma R T}{M}}} p_{in}$

For subsonic domain from Lee Company, the formula for volume flow rate containing the approximation $\frac{2\gamma}{3} = 0.933 \approx 1$ for diatomic gases

$$(\gamma=1.4): Q = \alpha \frac{\pi d_h^2 \sqrt{3}}{4 \rho^2 \sqrt{\frac{\gamma R T}{M}}} \sqrt{p_{ou}(p_{in} - p_{ou})} \quad (5.4)$$

For N_2 : $\rho=1.25 \text{ kg/m}^3$ (at $293 \text{ }^\circ\text{K}$), $\gamma=1.4$, $M=28 \text{ g/mol}$, $R=8.31 \text{ J/(mol }^\circ\text{K)}$

The head loss coefficient α extracted from the data provided by the valve manufacturer Lee Company is 0.83 for nitrogen. Numerical comparison between the theoretical formulas of James et al. and those provided by the valve manufacturer showed a matching below 5 % for a $10 \text{ }\mu\text{m}$ diameter nozzle d_h with inlet pressure p_{in} ranging from 1 to 3 bar and ambient outlet pressure p_{ou} , and volume flow rate below 3 ml/min .

Comparison between measurement and calculation

Comparing real valve flow of the third version and calculated flow with the theoretical formula for $10 \text{ }\mu\text{m}$ orifice diameter reveals a divergence, especially at low pressures (Figure 5.2).

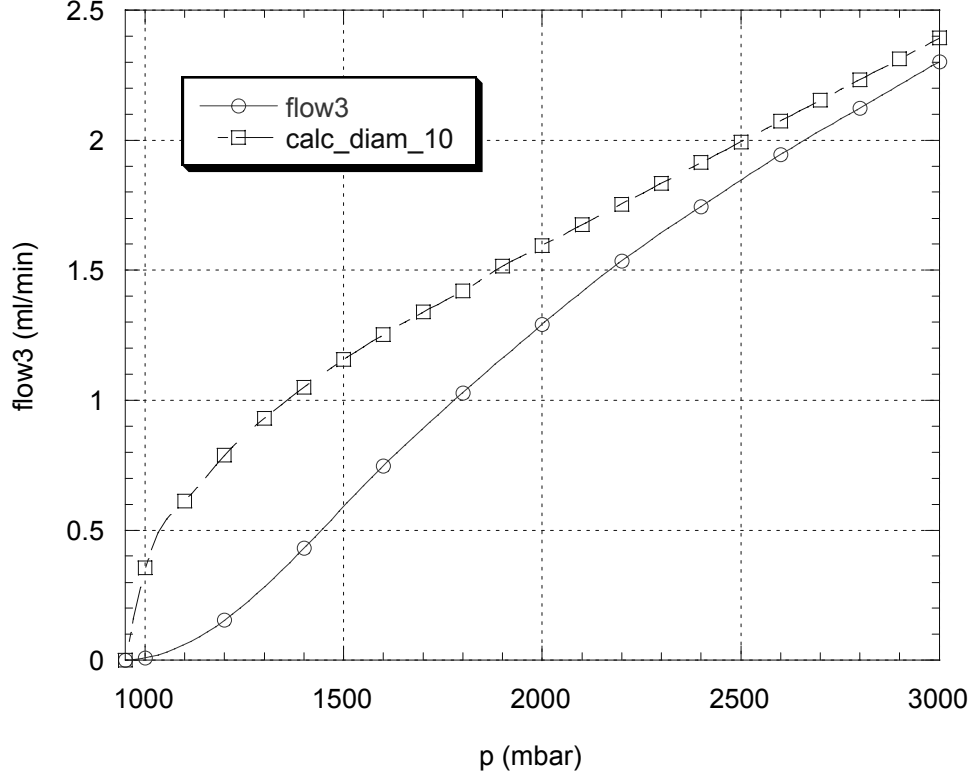


Figure 5.2: Flow vs. inlet pressure: Comparison of measured and calculated values for the third valve version. The outlet pressure is 0.95 bar. The comparison is made for a calculated diameter of 10 μm . The measuring errors are smaller than the plotted dots.

A flow measurement without membrane over top of the orifice provides a much better match with calculation (Figure 5.3). Nevertheless, even if reduced by the absence of membrane, a small divergence higher at low pressures is attributed to the simplifications of the isentropic flow model that does not take into account viscosity within the orifice channel, which is more influential at low pressures (appendix A). This viscosity effect is more present in the third valve version where the thin channel is longer than for the others.

Apparently, the membrane limits the flow significantly at low flow rates. The reason for a larger influence of the membrane at low pressures comes from variations of gap height

due to gas dynamic pressure, which makes the membrane deflect away for large flows.

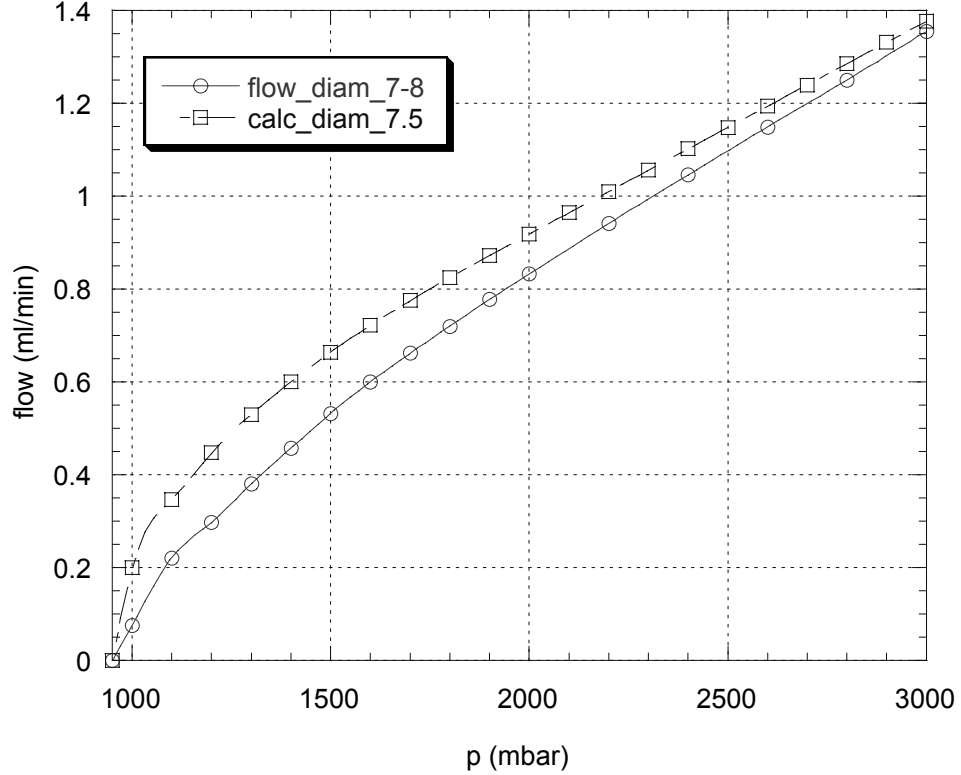


Figure 5.3: Flow vs. inlet pressure: Comparison of measured and calculated values for the third valve version without membrane on top. The outlet pressure is 0.95 bar. The orifice was not perfectly circular, but was within the range of 7 to 8 μm diameter. The comparison is made for a calculated diameter of 7.5 μm . The measuring errors are smaller than the plotted dots.

The membrane upward displacement f_y (5.8) due to gas dynamic force F_{ga} can be derived from equations (5.5),(5.6)[5.3]. At the equilibrium, the gas force F_{ga} compensates the mechanical spring force F_{me} (5.7). In section 3.3.4, the frontier between high and low influence of the membrane gap height was estimated at 2.5 μm for a 10 μm diameter orifice, which, based on the

membrane displacement calculation, corresponds to a flow rate Q of 2.26 ml/min (5.8).

$$F_{ga} = \frac{\rho_{ga} Q^2}{S_h} \quad (5.5)$$

$$F_{me} = \frac{16Ewh^3}{l^3} f_y \quad (5.6)$$

$$F_{me} = F_{ga} \quad (5.7)$$

$$f_y = \frac{\rho_{ga} Q^2 l^3}{4\pi d_h^2 Ewh^3} \Rightarrow Q = 2d_h \sqrt{\frac{\pi Ewh^3 f_y}{\rho_{ga} l^3}} \quad (5.8)$$

Therefore, for flow rates Q above 2.3 ml/min, the open membrane does not limit the flow significantly, and the nozzle flow model used gives good results. For reduced orifice diameter, the flow rate model limit diminishes.

5.1.2. Valve array

For the second valve version, measurements show that the flow rate of a 20 parallel array of valve, is a little bit higher than that of a single valve multiplied by 20 (Figure 5.4). However, since the space between valves is about 40 times the membrane size, the higher flow rate is attributed to orifice diameter variations. Furthermore, N.Vandelli et al. measured in a valve array configuration a total flow close to that of a single valve multiplied by the number of valves [5.4].

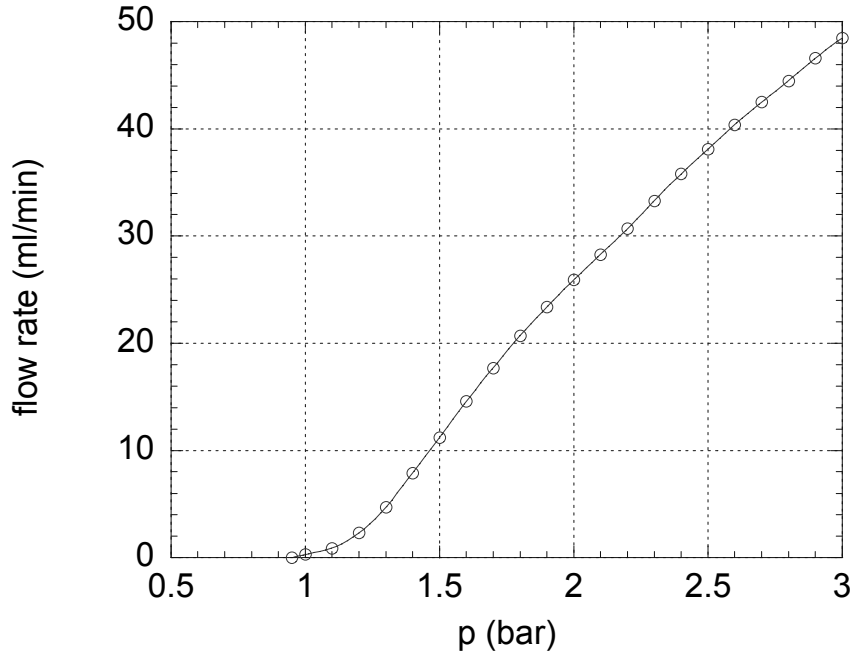


Figure 5.4: Flow vs. inlet pressure: N_2 flow rate through 20 valves in parallel. This chip is tested in the same container used for single valve. The outlet pressure is 0.95 bar. The measuring errors are smaller than the plotted dots.

5.2. Test set-up electrical schematic

The simplified electrical schematic of the valve mounted on its support is represented in Figure 5.5. This schematic is essentially a low-pass filter, which cut-off frequency depends on the value of the dominating components. The purpose of this calculation is to establish the worst case, which is the slowest cut-off frequency. The values of the components related to the valve version are given in Table 5.1. Beside the parasitic capacitance and the contact resistances, the values are calculated based on material properties and chip dimensions. The parasitic capacitance and the contact resistance of Ta-Si-N were measured with a parameter analyzer. The contact resistance of doped silicon on aluminum is calculated based on a

doping concentration of 10^{15} impurities per cm^3 , which corresponds to the silicon wafers used [5.5].

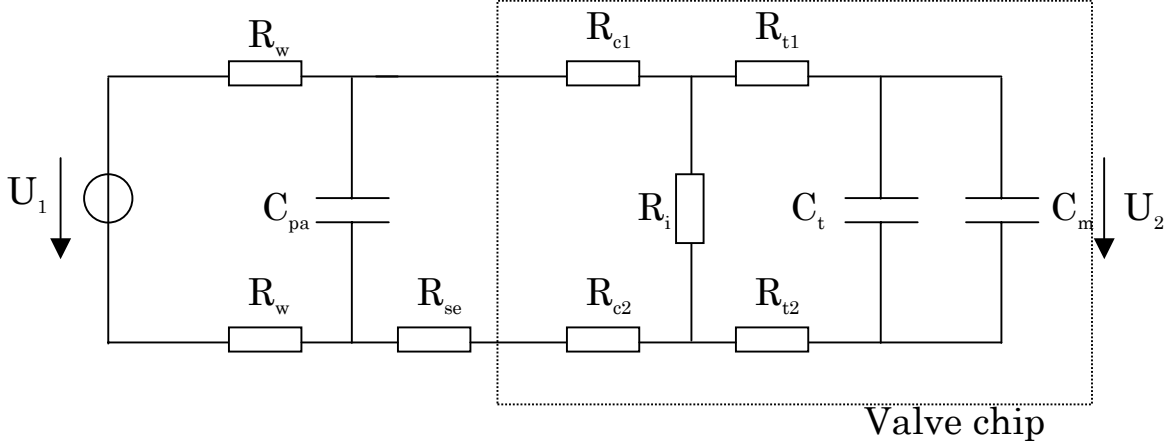


Figure 5.5: Valve simplified electrical schematic.

Table 5.1: Value of the electrical resistors and capacitances.

	Version 2	Version 3
R_i , insulation resist.	$> 10 \text{ M}\Omega$	$> 100 \text{ M}\Omega$
R_w , wire resist.	$< 2 \Omega$	$< 2 \Omega$
R_{c1} , contact resist.	$< 100 \Omega$	$< 100 \Omega$
R_{c2} , contact resist.	$< 100 \Omega$	$< 4 \text{ k}\Omega$
R_{t1} , track resist.	$< 10 \Omega$	$< 10 \Omega$
R_{t2} , track resist.	$< 10 \Omega$	$< 5 \Omega$
R_{se} , security resist.	$1 \text{ k}\Omega$	$1 \text{ k}\Omega$
C_{pa} , parasitic capa.	$< 5 \text{ nF}$	$< 5 \text{ nF}$
C_t , track capa.	$< 1.5 \text{ nF}$	$< 0.8 \text{ nF}$
C_m , membrane capa.	$< 5 \text{ pF}$	$< 3 \text{ pF}$

In the worst case, which is version 3, the time constant of this low pass filter is below $4 \mu\text{s}$.

Since the switching speed of the valve membrane is about 25 times slower due to fluid mechanics reason (section 5.6), this electrical low-pass filter can be neglected when considering the valve actuation. Therefore, the source voltage U_1 is considered equal to the voltage applied on the membrane U_2 .

5.3. Actuation voltage

Since the valve was designed to work as an on/off switch, the actuation voltage is an essential parameter for describing valve behavior. It was measured by means of an optical set-up (section 5.6) under no-flow conditions, and by flow measurements when gas was applied.

The actuation voltages of the second and third valve versions were measured for different membrane designs, with and without flow. Without flow, the actuation voltage is the pull-in voltage U_p (Table 5.2). One sample of the first version modified with an additional stiffening aluminum layer was actuated properly, but its short lifetime, due probably to electrical breakdown, did not allow for further measurements.

Table 5.2: Measured pull-in voltages.

U_p {V}	Version 2	Version 3
Basic valve	23±1	27±1
“T-shape”	10±1	20±1

Table 5.3: Actuation voltages at 2 bar inlet N₂ gas pressure and 1 bar outlet pressure.

Actuation voltage {V}	Version 2	Version 3
Basic valve	60-70	130-150
“T-shape”	30-40	120-140
20 valve in //	40-50	120-150

First, the pull-in voltages U_p can be compared with simulated values (section 3.4.6). In the second valve version, the agreement of valve pull-in voltages with their corresponding simulated values validated the FEM model used for calculation. For the third version, the measured pull-in voltages are higher, especially the one of for “T-shape” valve. Two factors are responsible: (1) thicker insulation layer and (2) higher tensile stress in the membrane layer due to annealing at 470 °C

instead of 450 °C (section 4.3.1). The thick insulation increases the actuation voltage of the “T-shape” valve more substantially than in the other design because the transverse beam pulls down the rest of the membrane by touching the insulation layer below pull-in voltage.

Under flow conditions, all actuation voltages were much higher than expected based on the design model, which means that the maximum pressure handling capability was lower than that described by equation (3.24)(section 3.4.3). The reason for this behavior was the action of both dynamic and static gas pressure on the membrane during the closing process, due to flow circulation and gas squeeze film effects. Since thicker insulation layers show much higher actuation voltages, the most critical situation with respect to valve actuation occurred when the membrane was quasi closed. It is likely that the membrane somehow bounced back on a thin gas film when actuated between pull-in and measured actuation voltages under flow conditions. Unfortunately, this nonlinear situation involving mechanics, electrostatics and fluidics would be very difficult to simulate, even with a multi-physics FEM solver. The problem is mainly the fluid flow that crosses fluid domains when the membrane closes. Therefore, to verify the assumption of the bounce back phenomenon, future studies should focus its determination under different flow conditions.

However, to reduce the actuation voltage in a significant manner, the insulation layer should be as thin as possible while avoiding electrical breakdown.

5.4. Leakage

For the second version, the leakage was measured with a Bronkhörst flow meter for a 20-valve array. For the third version, a bubble flow meter was used to test a single valve (Table 5.4). The actuation voltage used for testing the second version was 50 V, and for the third, 140 V.

Table 5.4: Leak-rate for 1 bar differential pressure.

	Version 2	Version 3
Leak-rate	< 0.6 $\mu\text{l}/\text{min}$	< 10 $\mu\text{l}/\text{min}$
Open/close flow rate ratio	> 1600	> 130

It appears that the leakage of the third version is about 10 times higher than that of the second version. Since the electrical field was even higher for third version testing, this result is surprising. One factor of influence could be that the silicon dioxide roughness was smaller in the second version, due to deposition on top of a Ta-Si-N layer, which has a surface roughness less than to 20 nm [5.6]. A second factor of influence could be reduction of the electrical field due to charge accumulation at the SiO_2 surface. V. Filip et al. showed that this phenomenon is more pronounced in thicker oxides having high resistivity [5.7].

Measurements showed that increasing the actuation voltage, or keeping the membrane closed for a few minutes reduced leakage. One explanation could be that the pressure keeping the membrane closed diminished temporarily the roughness of the surfaces in contact [5.8].

Therefore, the insulation layer has a significant effect on valve sealing. In future development, thinner oxide layers should be investigated.

5.5. Pulse width modulation (PWM)

For varying the flow rate, the valve can be actuated in pulse width modulation (PWM), which gives linear flow rate response at switching frequencies below 1 kHz (Figure 5.6). Some dependence of the PWM flow rate response to the actuation frequency can be observed (Figure 5.7).

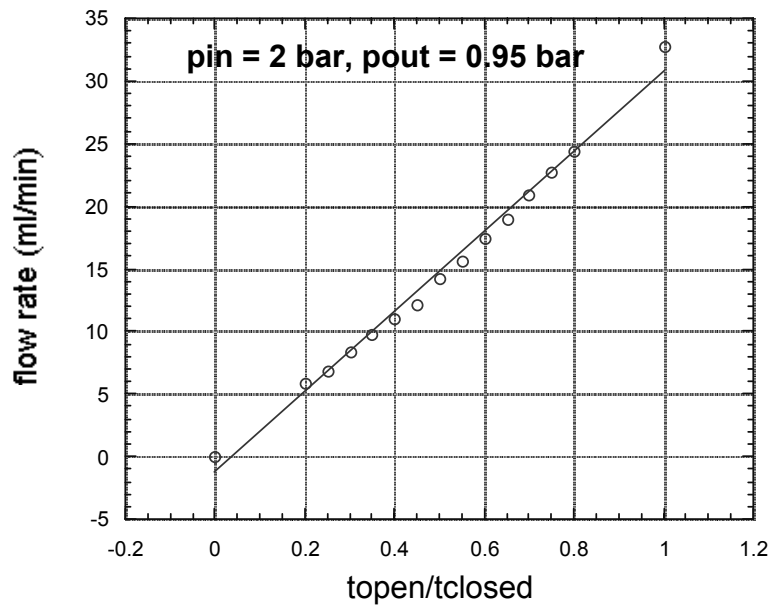


Figure 5.6: Nitrogen flow rate of 20 valves vs. actuation duty cycle at 100 Hz modulation frequency.

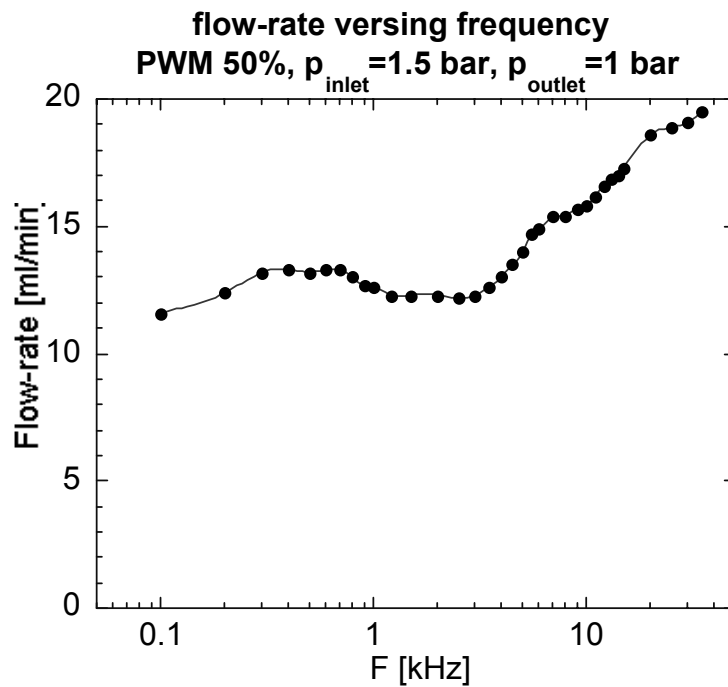


Figure 5.7: Flow rate dependence on frequency during actuation in PWM at open/close time ratio of 50 % (open flow rate = 25.9 ml/min).

Since the valve is normally open, the tendency of the valve to deliver higher flow rates at modulation frequencies over 5 kHz was expected to be related to the switching times of the valve membrane.

In a normal utilization of the valve in PWM, the commutation frequency would be kept constant at a frequency below 5 kHz, and variations of the duty cycle could be used either to vary flow rate or to deliver constant flow rate under variable differential pressure. The ideal situation would be to place the valve actuated in PWM in a feed back loop having a flow sensor and a controller.

5.6. Switching times

The optical set-up built to observe the vertical displacement of the membrane was based on an optical fiber directing a laser beam toward the top of the moving membrane. Half of the reflected beam was detected by a high-speed photodiode (Figure 5.8).

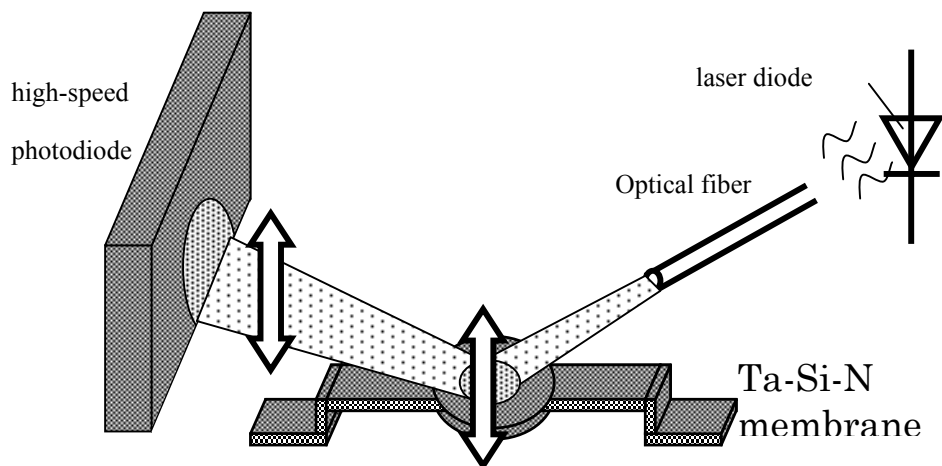


Figure 5.8: Schematic view of the optical set-up for measurement of the valve membrane motion.

First, the switching behavior of the basic valve was observed under ambient pressure and no-flow conditions. At a voltage of

50 V, the valve closed in 80 μs , and opened in 100 μs (Figure 5.9, Figure 5.10). Because the opening time in vacuum estimated by FEM simulations was about 1 μs and the electrical time constant is below 2 μs (section 5.2), the slower membrane displacement speed observed results from gas effects.

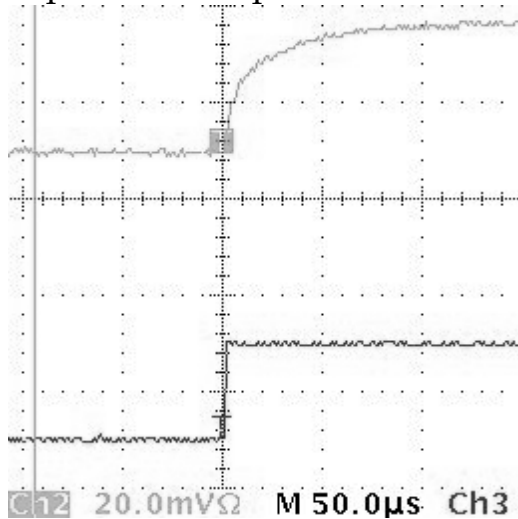


Figure 5.9: Step closing response of the basic valve membrane under 1 bar ambient pressure (no flow). At a voltage of 50 V, the valve closes in 80 μs .

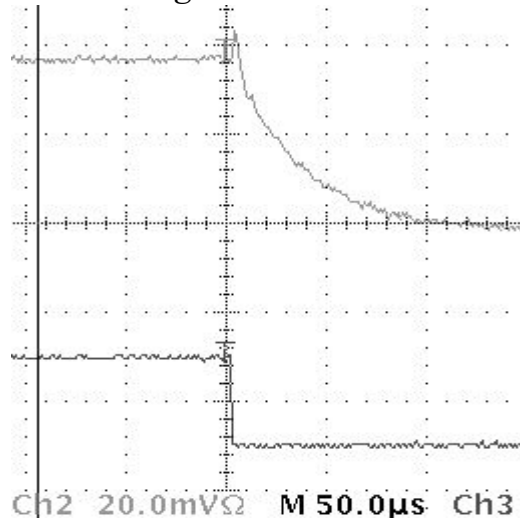


Figure 5.10: Step opening response of the basic valve membrane under 1 bar ambient pressure (no flow). The opening time is 100 μs .

Since gas appeared to have a major influence on the membrane motion, gas flow was introduced into the testing system. For the same actuation conditions, and under 0.4 bar differential pressure, closing took only 10 μs longer than when there was no flow, and opening took the same amount of time. For the “T”-shaped valve, the observations were similar though the actuation voltage was lower. At 1 kHz actuation frequency and under 2 bar differential pressure, the switching time represents almost 20 % of the period (Figure 5.11). Because the switching times are quite symmetrical, they start to have a major influence on the flow rate only when they do not allow a complete opening and closing of the membrane. This happens at 5 kHz, when the membrane moves around an intermediate position without completely closing the orifice. These observations matched previous flow rate measurements in PWM with a 50 % duty cycle, where the response became highly dependent on frequency at values above 5 kHz. Therefore, the

maximum actuation frequency is about 1 kHz, in order to guarantee a linear flow response while varying the duty cycle.

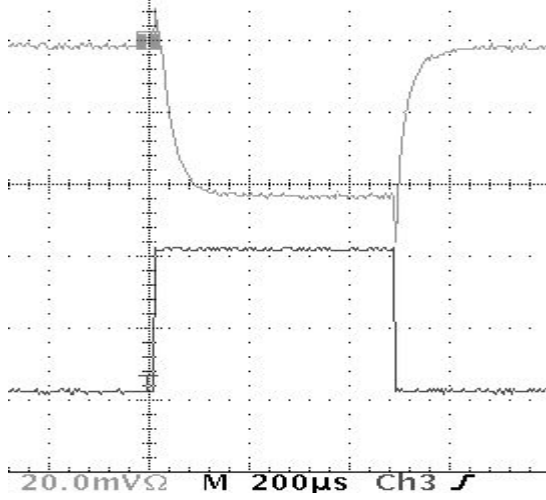


Figure 5.11: Step closing/opening response of the “T”-shaped valve membrane under 2 bar differential pressure, at a voltage of 50 V, and an actuation frequency of 1 kHz.

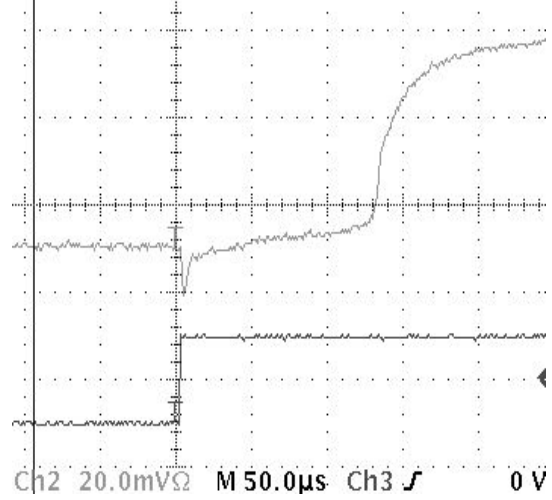


Figure 5.12: Step closing response of the basic valve membrane under 0.48 bar differential pressure at 50 V. This differential pressure represents the limit of actuation for this voltage. A delay, varying from one cycle to the other, was observed. In this figure, the delay is 130 μ s.

It was also observed that for a defined actuation voltage, a maximum differential pressure exists over which the membrane did not respond to the actuation signal. Just at this limit, the motion of the membrane was inconstant; most of the time, the collapse was delayed (Figure 5.12).

Squeeze film effect, which is dependant on the static gas pressure, slows down membrane displacement compare to a vacuum situation. Therefore, the switching time is dependent on the pressure around the membrane during its motion. Observations of the switching times at a fixed actuation voltage, appeared to be independent of the gas flow up to a threshold level where delays were observed. Therefore, the outlet pressure should influence more the switching time than the flow rate. To verify this hypothesis, the membrane motion would have to be calculated based on squeeze film effect without flow influence. To do so, the differential equation can be established by using the spring constant k calculated with the static physical model and introducing the squeeze film effect $b(y)$ dependent on the fluid viscosity η (5.9),(5.10) [5.9].

$$m\ddot{y} + b(y)\dot{y} + ky = \frac{\varepsilon_0 S_m}{2(d_{g0} - y)^2} U^2 \quad (5.9)$$

$$\text{With: } b(y) = \frac{6\eta d_m^4}{\pi^4 (d_{g0} - y)^3} = \frac{b1}{(d_{g0} - y)^3} \quad (5.10)$$

$$m\ddot{y} + \frac{b1}{(d_{g0} - y)^3} \dot{y} + ky = \frac{c}{(d_{g0} - y)^2} U^2 \quad (5.11)$$

However, since the motion differential equation is second order highly non-linear (5.11), no analytical solution can be found and numerical solution is most difficult since it cannot be linearized due to the large displacement. Therefore, the switching time calculation would have to be done by numerical iterative calculation procedure, such as three physics domains FEM simulations, which will not be realized due to the large amount of time that they necessitate.

As expected (section 5.1), the static position of the membrane was higher with flow than without flow, but the experimental set-up did not allow quantification of the displacement.

5.7. Risk of failure

Evaluating risk of failure is a complex procedure that is usually not the goal of a research project. Therefore, only two experiments were carried out. One was a long-term test, while the other looked at electrical breakdown.

5.7.1. Valve lifetime

At 1 bar differential pressure, one second version “T-shape” valve was tested for 10^9 cycles at 5 kHz. No sign of failure appeared. Nevertheless, such a cycle number represents only 116 days at a working frequency of 100Hz. Further testing would have to be conducted on different samples to fully characterize the reliability of the valve.

5.7.2. Breakdown risk

Short term testing has shown that the main risk of failure is electrical breakdown.

In gas

The Paschen law describes the threshold conditions over which gas ionization due to an applied electrical field occurs, and predicts that for gaps below 1 micron at ambient pressure, the electrical breakdown in air rises to values above 600 V [5.10]. However, this law is not valid for gaps below 4 microns when at least one of the electrodes is metal [5.11]. The Paschen curve does not take into account electron field emission, which is thought to be dominant for metals at reduced gap [5.12,5.13,5.14]. T. Ono and M. Esashi have measured the breakdown voltage for silicon and metal electrodes [5.12,5.14]. Their experiment shows that the breakdown voltage with silicon electrodes is very close to the Paschen curve, while that for metal electrodes is much lower below 4 microns (Figure 5.13).

T.Ono et al.

Figure 5.13: Breakdown voltages measured by T.Ono and M.Esashi at different gaps for metal electrodes and silicon electrodes [5.12,5.14].

Concerning the microvalve, the highest breakdown risk in gas is for the third valve version in the orifice area, where the Ta-Si-N membrane is separated from silicon only by 0.5 μm , the thickness of the insulation layer (section 4.3). In this case, the materials involved are silicon and Ta-Si-N. Unfortunately, the Ta-Si-N electron field emission coefficient is unknown.

During testing, no gas electrical breakdown occurred in the orifice area of the third valve version at voltages below 150 V. Due to voltage source limitations at 155 V, the breakdown limit was not measured accurately, but a few instances of breakdown seem to have occurred at slightly over 150 V.

In the insulation layer

For the second valve version, breakdown occurred many times in the insulation layer during testing, usually at around 70 V (4.2.2).

For the third valve version, breakdown did not occurred in the insulation layer.

5.8. Electrical consumption

The electrical consumption is dependant on to two factors, capacitive charge switching and conduction in the dielectric. When the dielectric is a thermal oxide, its high resistivity allows current leakage to be neglected in it [5.15]. Therefore, the power dissipated by the actuation of a valve in PWM is dependent on the switching frequency (Equation (5.12)).

$$P_{el} = freqCU^2 \quad (5.12)$$

For the third valve version mounted in its container, a capacity of 1.5 nF was measured. At a commutation frequency of 100Hz and an actuation voltage of 140 V, the consumption is 3 mW. It

has to be mentioned that these losses do not occur in the valve itself, but in the resistors that bring current to the electrodes.

5.9. Valve characteristics: comparison with publications

Compared to published electrostatic gas valves based on surface micromachining, the valve proposed switches flows under higher pressure, while its flow per single valve is relatively small (Table 5.5), [5.4,5.17,5.18].

Table 5.5: Comparison with published performances of other electrostatic valves. The two columns on the right hand side are for electrostatic bulk silicon valves, while the others are surface-micromachined.

	TaSiN [5.16]	Ohns [5.18]	Robe [5.17]	Vand [5.4]	Yoba [5.19]	Scha [5.21]
Max dif. pressure {bar}	2	0.15	0.13	0.14	0.3	16
Max flow per valve {ml/min}	2.5	(150)	1	6	80	1000
Max flow per array {ml/min}	77	-	-	150	-	-
Actuation voltage {V}	35-150	30	45-60	250	70	200
Switching time {ms}	0.1	-	-	-	-	1
Leak ratio	>130	38	10	20	80	200
Orifice diam. { μm }	10	43	-	100	100- 200	-
PWM	yes	(yes)	no	no	no	yes
Array	20/32	no	no	25	no	no

However, the concept of making many valves work in parallel on the same chip showed that it is possible to link both high pressure handling capability and large flow rates. For instance, in a surface area of 4 mm^2 , 100 Ta-Si-N valves can work in parallel to switch a flow of 1000 ml/min under 2 bar differential pressure. To our knowledge, there is only one published gas electrostatic microvalve that exhibits high pressure handling capability and large flow rate, but the fabrication is based on bulk micromaching, and the valve can work only in a limited range of pressures [5.20,5.21].

5.10. Conclusion

These measurements have shown the exceptional working characteristics of the Ta-Si-N valve compared to published electrostatic microvalves. The valve works in two different

modes of actuation: as an on/off gas switch or in pulse width modulation (PWM) to obtain a linear flow response. The Ta-Si-N membrane can switch flow under 2 bar differential pressure, and deliver a flow rate of 45 ml/min in a 20 valve parallel configuration. If the parallel concept is extended, a flow rate of 1 l/min could flow through 400 valves arranged on a surface of 4 mm².

Depending on the thickness of the insulation layer and the shape of the membrane, the actuation voltage under flow conditions varied from 30 V to 140 V. For thin oxides, the “T-shape” membrane exhibited a factor 2 reduction of the actuation voltage.

Different valve versions have shown that the best reliability could be achieved with thermal silicon oxide. Future research should test thinner insulation layers that might reduce significantly actuation voltage and valve leakage. The insulation layer thickness limit of the third valve version depends on electrical breakdown in gas. To optimize the valve characteristics and quantify the influence of electron field emission, studies could be conducted to measure the electrical breakdown of Ta-Si-N electrodes in gas at reduced gaps.

5.11. References

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Chapter 6

Sliding Microvalve/tribometer

Introduction

The research presented in Chapter 6 to 9 of this Ph.D. thesis addresses the development, fabrication and testing of a micromachined, active silicon microtribometer/valve designed for friction studies of diamond-like-carbon (DLC) coated chips and liquid handling.

6.1. Motivation

Studies are being conducted to develop silicon-based sliding valves to eventually replace metallic rotating sliding valves used by the company Hamilton AG in order to improve lifetime (Figure 6.1, Figure 6.2). In the presence of liquids such as human serum that have tendency to agglutinate, sliding valves behave much better than other types of valve [6.1]. Attempts to prolong valve lifetime have focused on the deposition of hard thin-film layers, such as diamond-like-carbon (DLC), on the contacting silicon surfaces [6.2]. DLC is known for its low wear and friction properties, but since a large variety of DLC layers exist, many tests have to be realized to select the layer most adapted to a specific application [6.3].

Unfortunately, most existing tribometers such as the pin-on-disk tribometer are not suited for observing the wear of flat areas moving in a reciprocating manner [6.4-6.5]. A previously fabricated tribometer exhibited friction in the guiding area that could induce imprecisely controlled motion [6.1]. Therefore, a micromachined silicon microtribometer that can also work as a three-way valve and does not exhibit friction in the guiding area was developed. This study reports on the development and testing of a sliding silicon microtribometer/valve for liquids.

Many microsystems devices employ contacting flat surfaces, but few of them have sliding motion [6.6,6.7]. One of the reasons may be that low-wear coatings have not been broadly studied to date [6.8]. Testing thin film layers with the proposed microtribometer should facilitate the choice of adapted coatings for MEMS applications involving a sliding oscillating motion. To our knowledge, only one silicon-based sliding microvalve for liquids has been reported in the literature [6.9].

6.2. Scope of the Research

The microtribometer/valve fabricated was designed to observe wear of removable flat silicon test inserts, coated with thin-film layers such as DLC and moving in an oscillating manner relative to each other, the actuation being achieved by an external linear motor. For observing the low wear of DLC layers in a reasonable amount of time, high oscillating speed is essential and can be achieved by reducing the mass in motion [6.10]. Because silicon has an advantage over other materials because of the invariance of its mechanical stiffness over time [6.11], silicon mechanical deformation will be used to produce a force normal to the test chips. The fabricated silicon microtribometer/valve reached oscillating frequencies of 10 Hz while applying a normal force on the test inserts up to 10 N, maximum amplitude of motion of 1.5 mm.



Figure 6.1 Hamilton AG three-way actual mechanical microvalve measuring about 10 cm³.

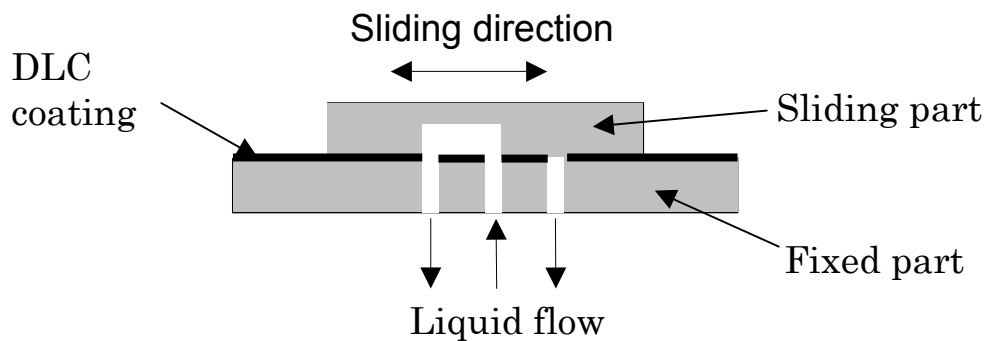


Figure 6.2 Principle of the silicon based sliding microvalve

This study also presents tribological observations of thin-film, DLC-coated test chips aged with the microtribometer. In many MEMS applications where contacting flat surfaces slide over each other, certain thin-film coatings can reduce friction and the resulting wear of the contacting surfaces [6.2]. The tribological situation encountered in MEMS devices with back-and-forth motion, such as sliding valves or guiding devices, is different from those with continuous motion, due mainly to the deposition of extracted particles on the pathway [6.12,6.13]. Previous studies conducted at the Neu Technikum Buchs (Buchs, Switzerland) have shown that DLC films can be used to reduce wear and friction, but may suffer from adhesion problems, depending on the deposition and testing conditions [6.1]. Therefore, many tests have to be done rapidly over a large number of cycles to develop reliable DLC layers for a specific application. With respect to fluid circulation in the test chips,

only leakage tests were realized. However, a new project focused on valve development is ongoing.

6.3. Tribometer principle

A tribometer is a measuring device that quantifies friction of two surfaces moving relative to each other. The main parameters of interest are the wear and friction coefficient under different conditions such as humidity, temperature, or lubricating fluid [6.4]. Since for a reciprocating tribometer the positioning and speed v oscillates, the lower the mass in motion m the faster it can move, as shown in Figure 6.3 and equation (6.1). A normal force F_N and a tangent force F_t are applied to the moving element of the tribometer. A friction force F_f opposed to the displacement is measured directly or indirectly.

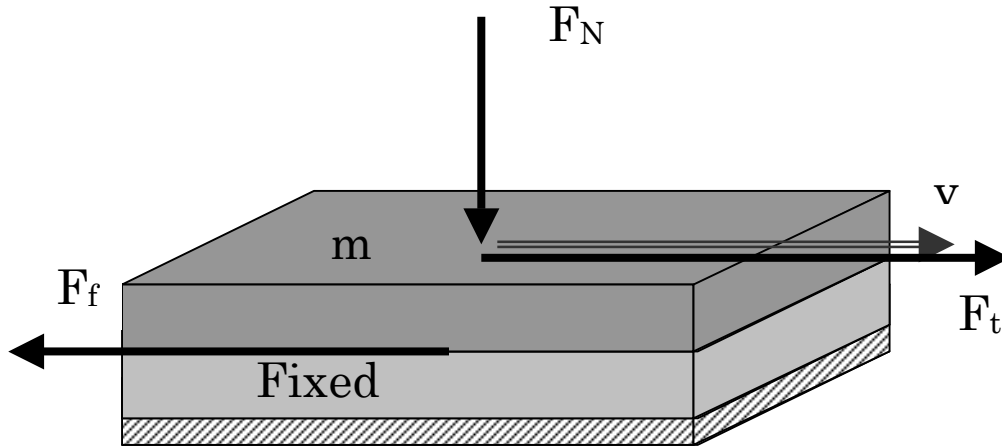


Figure 6.3 Drawing showing the way the fixed part and the moving part of a tribometer interact.

$$v(t) = v_0 + \int_{t_0}^t \frac{F(t)_t - F_f(F_N, \mu(t, v, x))}{m} dt \quad (6.1)$$

Since wear is mostly dependent on the number of cycles accomplished, achieving high oscillating frequencies means being able to age test chips in a short time [6.14]. Because test chips coated with layers such as DLC are used for their low

wear, the high-speed capability of this small mass tribometer would enable comparison of more layer types and enhance our knowledge on the adequacy of coatings layers for specific applications.

For making reliable observations of friction force, speed and position, some conditions have to be fulfilled. The friction force has to be related only to the friction of the test chips, and there should be no stick-slip effect in non-measured areas to influence the speed of the moving body. Furthermore, the normal force should be constant and independent of position, speed and acceleration of the tribometer.

6.4. Tribometer downscaling

In 1998, a large-size metallic tribometer/valve was fabricated and tested using DLC-coated silicon chips (Figure 6.4) [6.1].

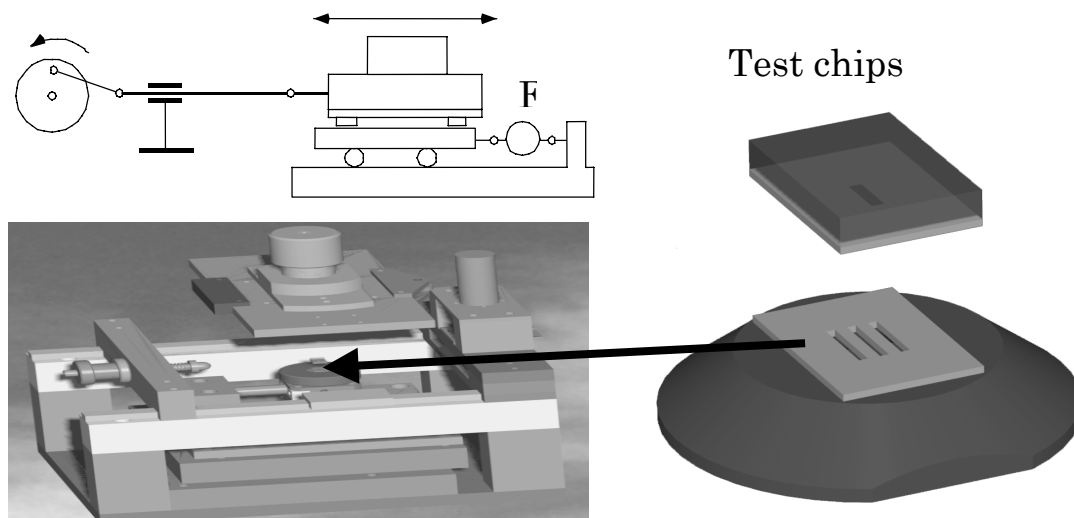


Figure 6.4 Schematics of the 1998 large-size tribometer measuring about 20 cm length.

The DLC testing was promising, but due to the tribometer's lack of accuracy, it was not possible to obtain reliable tribological measurements [6.1]. The problems of this large tribometer were:

- Stick-slip effect due to guiding of the moving part
- Slowness due to the heavy mass and mechanical resonance limited the number of tests (maximum frequency of 2 Hz)

- Friction not limited to the measured sections of the device
- Determination of position was not accurate enough
- No measurement of the acceleration or speed
- No feedback loop

The main undesired characteristics were stick/slip effect due to friction in the undesired area and slow operating speed related to the heavy mass in motion. For measuring tribological properties accurately, these critical points have to be taken into account:

- The friction force measured should be related only to the surfaces to be characterized
- Speed of the surface in contact should be as constant/defined as it can be (no stick-slip effect on other contact surfaces)
- Position and speed of the surface in contact should be measured accurately.
- The normal force should be constant and independent of position, speed and acceleration.

Therefore, a reduced-mass microtribometer/valve that does not exhibit peripheral friction was developed. This silicon microtribometer is actuated externally by a feedback linear motor, while the position, speed and acceleration measurements are external as well. An external gauge measures the actuation force, which is related to the friction by subtracting the acceleration force. Though the forces are in the Newton range, we call it a microtribometer since the fabrication of this tribometer relies on bulk micromachining of silicon. The advantages of a silicon tribometer compared to a large-size tribometer are numerous, with high-speed capability and the absence of friction at the sides being just two benefits gained (Table 6.1).

Table 6.1: Comparison between the large-size metallic tribometer developed in 1998 and the silicon tribometer presented.

	Large size tribo.	Silicon tribometer
Speed	 Big mass	 Low mass
F_f measuring	 On static body	 O.k. if feed-back
Speed measuring	 Can be added	 Close to the surf
Friction area	 Fric. at the sides	 No side friction
Packaging	 Chips bonded	 No intern.stress
Close to real valve?	 Very different	 Could be used
Complexity	 High	 Low

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Chapter 7

Sliding Microvalve/tribometer Design

The design of the two silicon elements comprising the microtribometer is based on high-aspect-ratio silicon beams, extremely soft in one direction for bending up to 0.375 mm, and very stiff in the other one for producing a normal force up to 10 N. To achieve accurate guiding while avoiding friction on non-tested areas, the device relies on the superposition of two layers of thin silicon beams linked together at the sides by screws (Figure 7.1). To our knowledge, such a structure based on a multitude of thin parallel bending beams has never been reported in literature (Figure 7.2). These two layers each hold a coated insert that is thicker than the layers, so that they bend vertically to generate a normal force (Figure 7.3). To accomodate liquid flow, channels are etched in the inserts.

The design process focuses on analytical calculation and FEM simulation in order to achieve the performance desired, while minimizing failure risk such as breaking or buckling. The first microvalve/tribometer fabricated was based on a simplified model, while the second one took into consideration the complex mechanical behavior of buckling. The FEM simulations realized with Ansys and ProMechanica software were conducted by A. Enzler and S. vonGunten at the Neu Technikum Buchs.

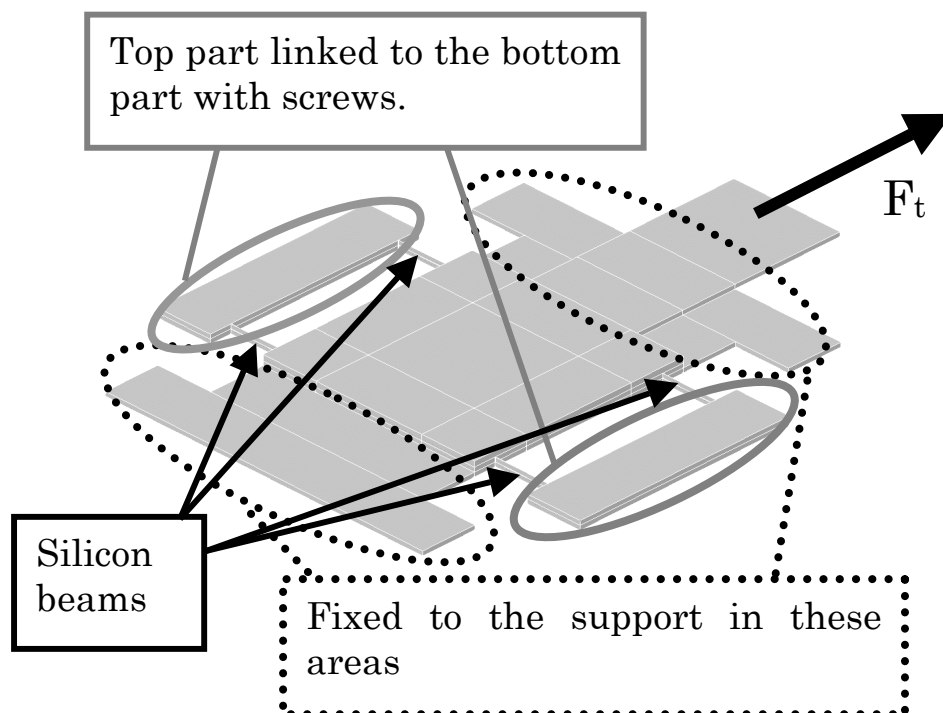


Figure 7.1 Schematic of the two microtribometer components. The upper one moves back and forth horizontally, while the lower one is fixed. In reality, there are a few hundred rows of beam instead of 2.



Figure 7.2 Schematic close-up view of the beam structure. Hundred of high-aspect-ratio beams are arranged in parallel.

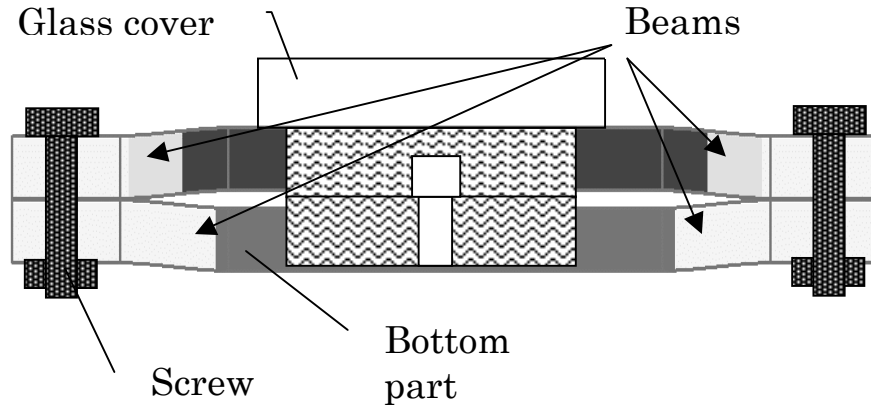


Figure 7.3 The vertical bending of the beams generates a normal force on the test chips.

7.1. High-aspect-ratio beams

For bending mechanical beams, the spring stiffness k is proportional to the geometrical moment of inertia I_G (7.2) [7.1]. This result is valid for simple clamped or double clamped beams.

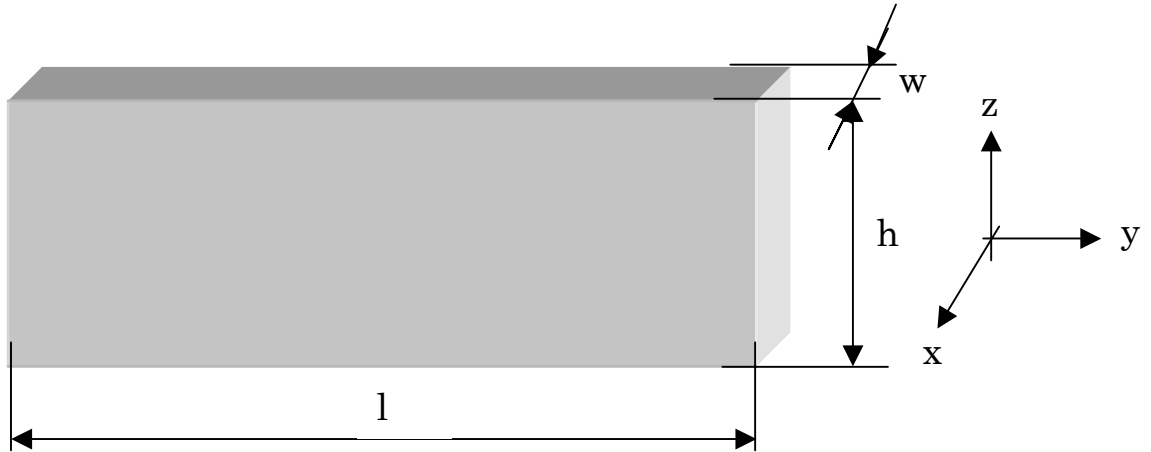


Figure 7.4 Drawing showing beam dimensions

$$F_z = k_z f_z \quad (7.1)$$

$$k_z \propto I_{Gz} = \frac{wh^3}{12} \quad (7.2)$$

Therefore, for rectangular beams bending in two orthogonal directions with the same mode, the stiffness ratio can be calculated by inverting width and height of one of the geometrical moment of inertia (7.3),(7.4).

$$k_x \propto I_{Gx} = \frac{hw^3}{12} \quad (7.3)$$

$$\frac{k_z}{k_x} = \frac{I_{Gz}}{I_{Gx}} = \left(\frac{h}{w}\right)^2 = aspect_ratio^2 \quad (7.4)$$

Since the stiffness ratio is proportional to the square of the aspect ratio, it is possible to have beams much softer in one direction than the other. For instance, DRIE can yield an aspect ratio of 20, which will correspond to beams 400 times softer in one direction than the other.

The maximum horizontal and vertical displacement can be calculated for a defined admissible stress σ_{adm} . The stresses due to horizontal F_x and vertical forces F_z are established [7.1].

$$\sigma_{yx} = \frac{1}{4} \frac{F_x l}{I_{Gx}} \frac{w}{2} \quad (7.5)$$

$$\sigma_{yz} = \frac{1}{4} \frac{F_z l}{I_{Gz}} \frac{h}{2} \quad (7.6)$$

Equation (7.1) is substituted in equations (7.5),(7.6) to obtain the stress σ dependency on displacement f (7.7),(7.8).

$$\sigma_{yx} \propto \frac{I_{Gx} f_x w}{I_{Gx}} = f_x w \quad (7.7)$$

$$\sigma_{yz} \propto \frac{I_{Gz} f_z h}{I_{Gz}} = f_z h \quad (7.8)$$

For $\sigma_{yx} = \sigma_{yz} = \sigma_{adm}$, when the beam is displaced alternatively horizontally and vertically, which corresponds to independent stresses, the displacement ratio is calculated. The displacement ratio is proportional to the inverse of the aspect ratio (7.9).

$$\frac{(f_z)_{max}}{(f_x)_{max}} = \frac{w}{h} = \frac{1}{aspect_ratio} \quad (7.9)$$

Therefore, for a defined strain, the forces ratio corresponds to the aspect ratio (7.10).

$$\frac{F_z}{F_x} = \frac{k_z(f_z)_{\max}}{k_x(f_x)_{\max}} = \text{aspect_ratio} \quad (7.10)$$

Concerning the microvalve/tribometer, a total horizontal displacement of 1.5 mm is needed for fluidic reasons (section 7.4.1), while a few tens of microns will be adopted for vertical displacement. Therefore, with high-aspect-ratio beams, it is possible to allow large displacements in one direction, while in the corresponding orthogonal direction a large force can be produced.

7.2. Calculation of first version

This first version was calculated analytically, and the results obtained compared with FEM simulations. Finally, the dimensions are established by taking into account the two approaches.

7.2.1. Analytical optimization

For this first version, the goal was to optimize beam length, width and number, in order to minimize tribometer size and risk of failure due to breaking when operated.

The beam deformation corresponds to the deflection of a double-fixed beam whose length measures twice the length of a real beam [7.1] (Figure 7.5).

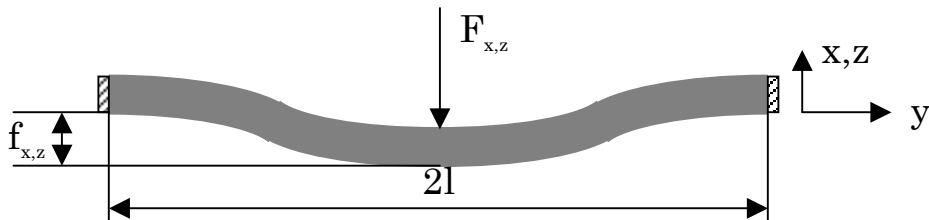


Figure 7.5 Deflection of a double-fixed beam measuring twice the length of a real beam.

The first consideration concerns the beam width and number. Since the geometrical moment of inertia of one beam in the

vertical direction is proportional to its width w , for n parallel beams and defined vertical force F_N and displacement f_z , the equivalent total beam width w_{tot} is defined, but the beam width w depends on the number of beams n [7.1] (7.11),(7.12).

$$f_z = \frac{F_N l^3}{24 E n I_z} = \frac{F_N l^3}{2 E n w h^3} = \frac{F_N l^3}{2 E w_{tot} h^3} \quad (7.11)$$

$$\text{For } f_z, F_N: \text{ constant} \quad (7.12)$$

$$\Rightarrow w_{tot} = w n \text{ is defined}$$

Therefore the beam width w is chosen based on other considerations. In the horizontal direction, the displacement being fixed at 1.5 mm for test chips, reducing the beam width reduces the horizontal force as well, which is useful for obtaining improved actuation (7.13).

$$F_{x_tot} = \frac{2 f_x E n h w^3}{l^3} = \frac{2 f_x E w_{tot} h w^2}{l^3} \propto w^2 \quad (7.13)$$

For this reason, the beam width was chosen at the smallest acceptable technological value of 20 μm . The height corresponds to the wafer thickness of 390 μm . Two parameters have to be optimized: beam length l and number n . For non-independent stresses at beams base corner, the expression of the total stress in the y direction links them (7.17) [7.1].

$$\text{General expression: } \sigma = \frac{M_{stress}}{I_G} \frac{h}{2} \quad (7.14)$$

$$\text{In this case: } M_{stress} = \frac{F_{x,z} l}{4} \quad (7.15)$$

$$\text{Therefore: } \sigma_{yz} \propto \frac{3 F_z l}{2 n w h^2} \quad (7.16)$$

$$\sigma_y = \sigma_{yx} + \sigma_{yz} = \frac{3 f_x E w}{l^2} + \frac{3 F_z l}{2 n w h^2} \quad (7.17)$$

Therefore expression for n , the number of beams can be obtain by rearranging (7.17), which gives (7.18):

$$n = \frac{\frac{3 F_z}{2 w h^2} l^3}{\sigma_y l^2 - 3 f_x E w} \quad (7.18)$$

The 1st derivation of (7.18) as a function of beam length l , exhibits a minimum (7.19),(7.20):

$$\frac{\partial n}{\partial l} = 0 \quad (7.19)$$

$$\text{So that: } \Rightarrow l_{opt} = 3 \sqrt{\frac{f_{x\max} E w}{\sigma_y}} \quad (7.20)$$

A minimum beam width also minimizes the optimum beam length.

7.2.2. Numerical optimization based on analytical calculation

A few conditions have to be defined:

- $\sigma_{\text{dim}}=200$ MPa : acceptable maximum stress (including a security factor of 4) [7.2]
- $E= 130$ GPa : Young's modulus of monocrystal silicon for the planes <100> [7.3]
- $F_{N\max}=10$ N : maximum vertical normal force for all beams
- $w_0=20$ μm : beam width
- $h=390$ μm : beam height, corresponds to wafer thickness
- $f_{x\max}=1.5\text{mm}/4=0.375$ mm maximum horizontal displacement (factor 4 due to double layers moving back and forth)
- $\alpha_k=1.41$: geometrical coefficient due to radius of 10 μm at beam basis [7.4]

Since silicon is a brittle material, the acceptable maximum stress σ_{dim} , including a security factor of 4, is based on experience, but it is not a well-defined value; the theoretical stress at rupture is 7 GPa [7.5]. The coefficient α_k takes into account stress concentration at beam base, due to a geometrical radius not taken into account in the simulations because a coarse mesh was used.

Therefore, the maximum stress in the “y” direction is (7.21):

$$\sigma_{y_adm} = \frac{\sigma_{\text{dim}}}{\alpha_k} \quad (7.21)$$

For a defined stress and beam width, the number of beams can be plotted as a function of their length based on equation (7.18), (Figure 7.6). At $\sigma_{\text{dim}}=200$ MPa, the minimum beam length is 7.9 mm, for a beam row number of 410. The corresponding horizontal force at a maximum total displacement of 0.75 mm is 0.26 N, and at a 10 N normal force, the vertical displacement of one beam is $38.5 \mu\text{m}$.

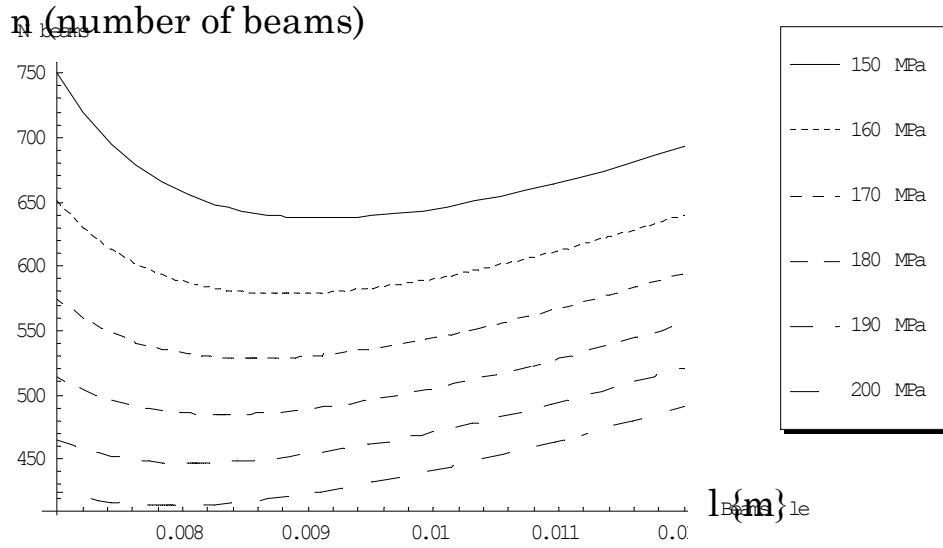


Figure 7.6 Number of beams vs. their length at different maximum acceptable stresses calculated using analytical equation (7.18).

7.2.3. FEM simulations

Finite element simulations (FEM) were used to validate the analytical calculation and estimate the normal force fluctuation under horizontal displacement.

Comparison

The analytical calculation is compared with FEM simulations realized on different software.

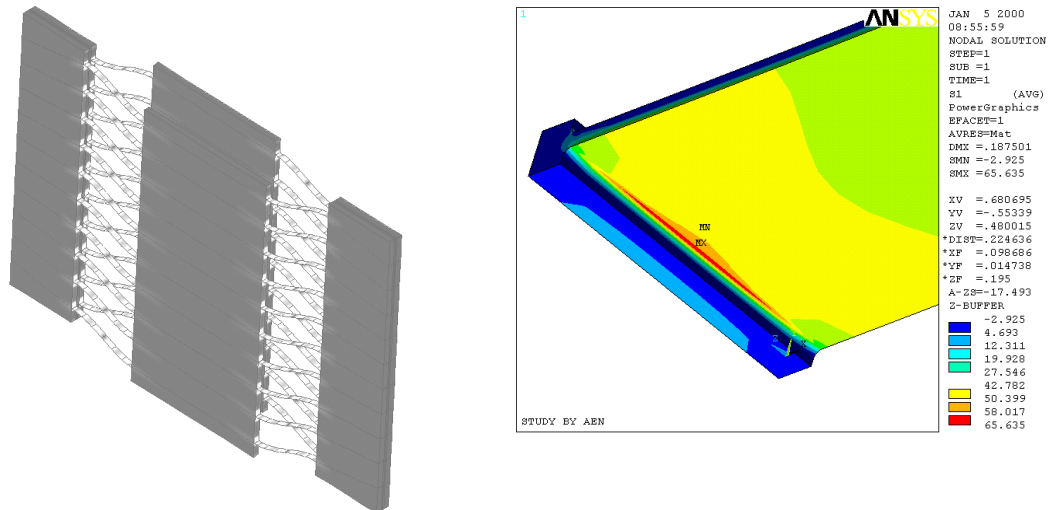


Figure 7.7 FEM models of the silicon microtribometer/valve realized with ProMechanica (left) and ANSYS (right) under a horizontal displacement of 0.75 mm.

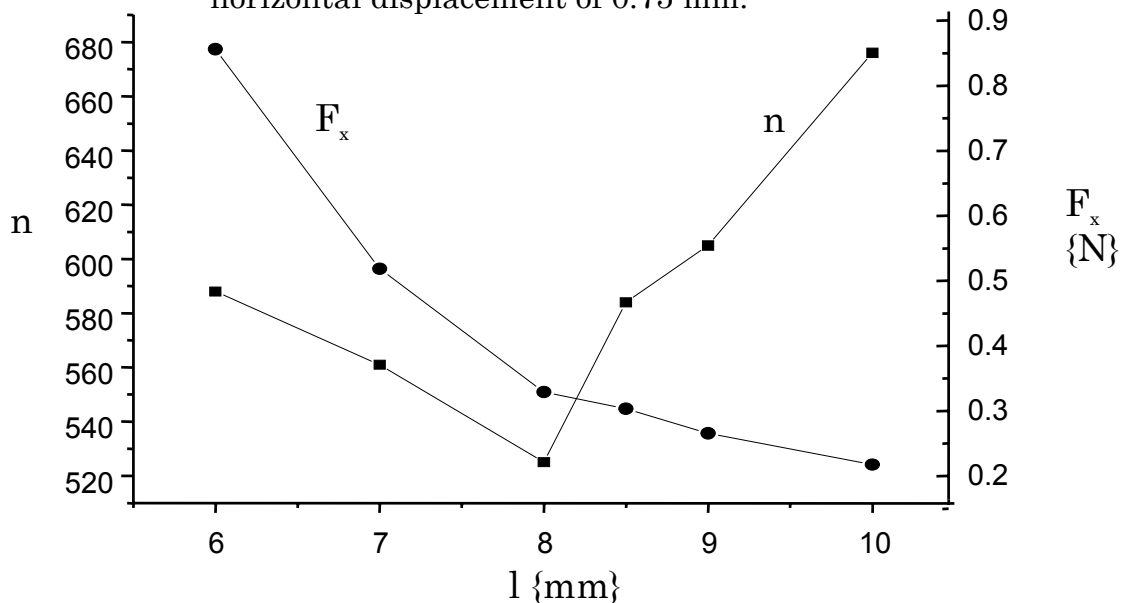


Figure 7.8 Results of simulations realized on Pro/mechanica software for an allowable stress of 200 MPa.

The minimum beam number corresponds to a beam length of 8 mm, which correlates well with the 7.9 mm value obtained based on the analytical model. However, the simulated beam number of 525 is higher ($n=410$ for analytical model). A more accurate simulation realized with Ansys software showed that stress values calculated in ProMechanica were overestimated by about 30 %, most likely due to the use of a coarse mesh.

As a general remark, calculated or simulated forces or displacements are very accurate, with less than 5 %, but

calculated or simulated stress or strains have a much larger uncertainty. This can be understood by the fact that a localized high stress would not induce large displacements.

Thus, to ensure a successful operation, the first microtribometer/valve version will be fabricated with 500 beam rows, each beam measuring 8 mm length by 20 μm width. To achieve a normal force of 10 N, the vertical displacement of the central plate of the tribometer was 70 μm .

Normal force fluctuation

In the analytical calculation, the forces due to horizontal and vertical displacement were considered independent, but simulations revealed a reduction in normal force of 16 % when the top half of the device was displaced horizontally (Figure 7.9). This behavior is attributed to a limited twisting of the beams and was therefore dependent on the third power of the beam width as lateral buckling is (7.25).

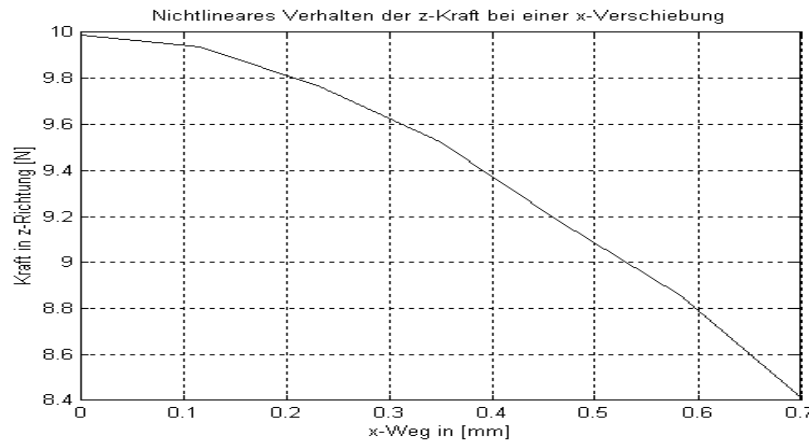


Figure 7.9 Fluctuation of the normal force when the top half of the tribometer is displaced horizontally.

7.3. Second version

Since the first version fabricated exhibited two problems, one related to fabrication difficulties due to the extreme beam thinness, the other due to a buckling behavior during operation. Hence, a modification of the design was undertaken (section 9.2.2). The beams could be reinforced in two different ways,

either by adding transverse beams or fabricating thicker beams. FEM simulations made it possible to decide between the two options.

7.3.1. Buckling scaling law

Buckling is a bifurcative behavior, with a material structure under stress deforming in one manner up to a certain point, and then suddenly changing its deformed shape into a buckling mode [7.4]. Since a structure deforms in a way that presents the lowest stored energy, a structure can have different buckling mode shapes that could be stable under defined boundary conditions.

With respect to the tribometer beams, buckling is due to torsion. Beams twist at a strain threshold, inducing a displacement along the x axis of one of their side and reducing the normal force as a result.

For beams under a momentum load, the differential equation that describes torsion of angle φ is (7.22)[7.4]:

$$\varphi''(y) + \frac{M_k^2}{EI_{Gx}GI_t} \varphi(y) = 0 \quad (7.22)$$

Where the geometrical moment of inertia in the x

direction is: $I_{Gx} = \frac{hw^3}{12}$

And the torsion moment of inertia : $I_t = \frac{hw^3}{3}$

The torsion modulus being related to the Poisson's

coefficient: $G = \frac{E}{2(1+\nu)}$.

The critical momentum (7.24) can be calculated for a deformation function of type (7.23) [7.4].

$$\varphi(y) = C \sin\left(\frac{\pi y}{l}\right) \quad (7.23)$$

$$M_k = \frac{\pi}{l} \sqrt{EI_{Gx}GI_t} \propto w^3 \quad (7.24)$$

Therefore, the buckling is influenced by the third power of the width. For a load applied at the extremity, the critical force is dependant on the inverse of the square beam length (7.25).

$$F_k \propto l^{-2}, w^3 \quad (7.25)$$

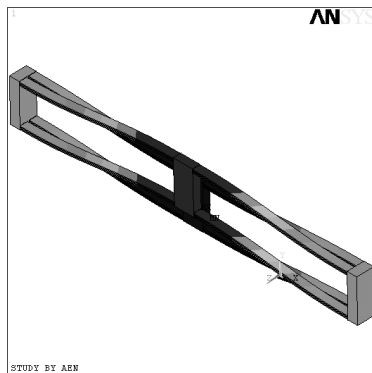
For the tribometer, since the load application is complex due to displacement of beam extremities and the appearance of structure modes, simulations have to be conducted to calculate the critical buckling force. Nevertheless, the critical buckling force scaling laws will be useful for resizing tribometer beams in order to avoid buckling.

7.3.2. Buckling simulations

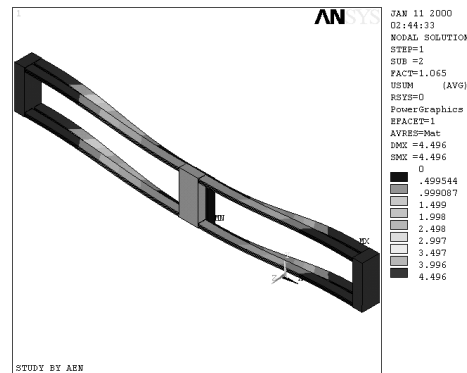
Buckling FEM simulations, done with Ansys software, were realized in order to resize a second tribometer version to avoid buckling. Increasing the critical buckling load value had the side effect of increasing the force in the x direction as well. Two options were considered, namely increasing the beam width or connecting beams traverse to the beam row, inter-beams.

Without inter-beams

First, simulations of the first version led to the correlation of observed instabilities with buckling behavior (Figure 7.10).



a)



b)

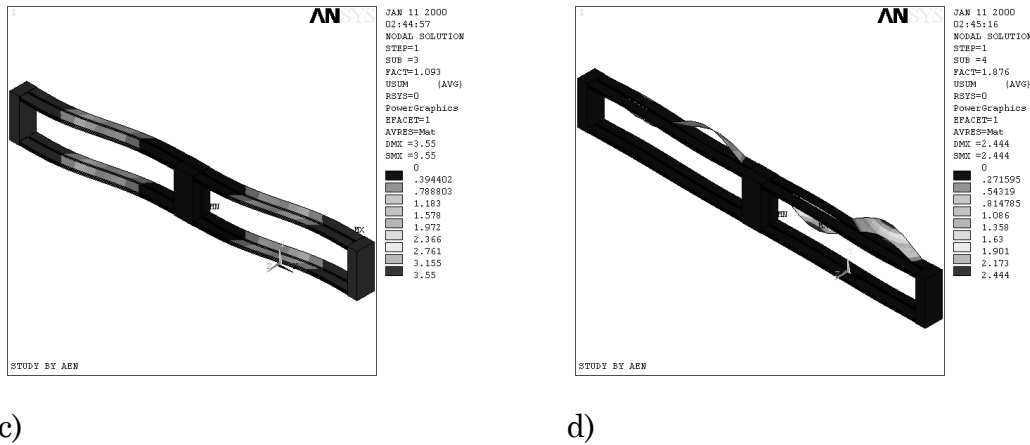


Figure 7.10 Buckling simulations of the beams of the first microtribometer/valve realized with ANSYS software. The first buckling mode occurs at 8.7 N (a), the second at 10.7 N (b), the third at 10.9 N (c), and the fourth at 18.8 N (d).

The first simulated buckling mode is at 8.7 N, which corresponds to a displacement of 57 μm . Observations have shown that on the real device, the first instability occurs at 38 μm displacement (section 8.1.2). The difference is attributed to the real beam section shape being thinner and conical.

In this design, a displacement of 0.75 mm induced a horizontal force of 0.3 N, which resulted in a maximum stress of 176 MPa. Knowing the scaling law of buckling beams and the critical value for buckling of the tribometer beams, it is possible to change the beam width in order to avoid buckling at normal force below 10 N.

With inter-beams

A design having inter-beams is also simulated (Figure 7.11). In this case, a displacement of 0.75 mm induces a horizontal force of 1.2 N, which results in a maximum stress of 265 MPa. The buckling occurs at 24.3 N.

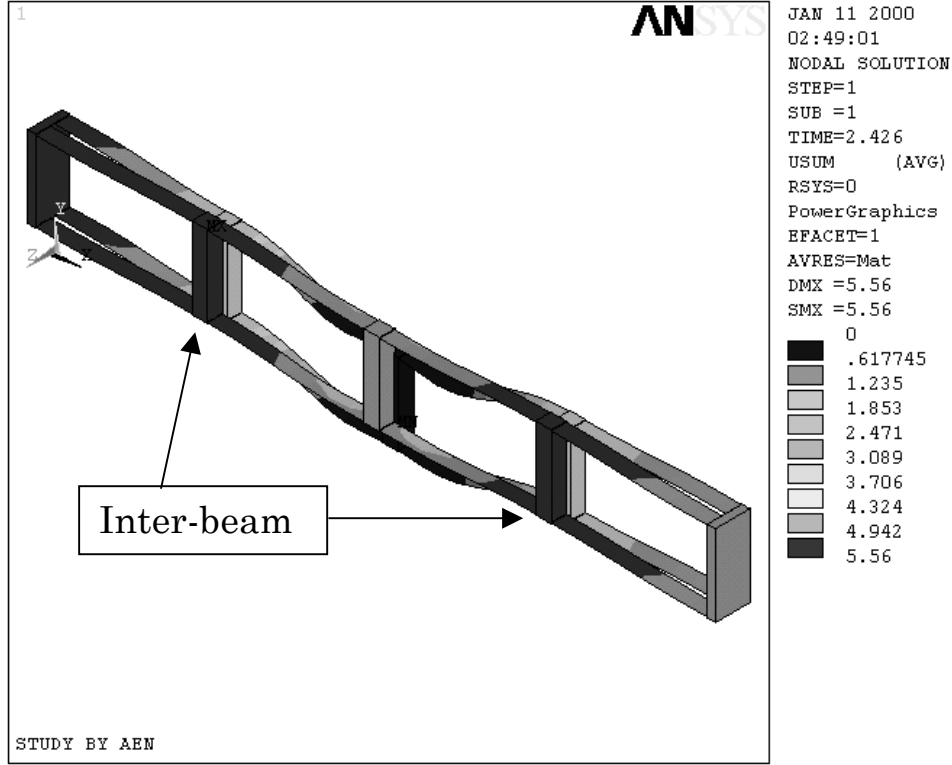


Figure 7.11 FEM simulations of the first buckling mode with one inter-beam on each side.

7.3.3. Design comparison

Based on scaling laws and FEM simulations results, a variety of designs can be considered.

Summary of scaling laws:

1) Beam number:

- $n = n_0 \frac{w_0}{w}$, $n_0=500$ (see(7.12))

2) Horizontal force:

- $F_x = F_{x0} \left(\frac{w}{w_0} \right)^2$, $F_{x0}=0.3$ N (see (7.13))

- $F_x = F_{x0}(N+1)^2$, N being the number of inter-beam,
 $F_{x0}=0.3$ N: without inter beams (see (7.13))

3) Stress in the y direction:

- $\sigma_y = \sigma_{yxo} \frac{w}{w_0} + \sigma_{yz0}$, $\sigma_{y_{x0}}=45.7$ MPa, $\sigma_{y_{z0}}=83.2$ MPa, from
simul. (see(7.17))
- $\sigma_y = \sigma_{yxo}(N+1) + \sigma_{yz0}$

4) Critical force for buckling:

- $F_k = F_{k0} \left(\frac{w}{w_0} \right)^3$, $F_{k0} = 8.7$ N, from simul. (see(7.25))
- $F_k = F_{k0} \frac{(N+1)^2}{4}$, $F_{k0_inter-beam} = 24$ N, from simul. (see(7.25))

Comparison and second version dimensions

For a compound horizontal and vertical displacement, at a comparable stress of about 265 MPa, the option with larger beams represents a lower horizontal force and a higher critical buckling force (Table 7.1). Therefore, increasing beam width was the best approach to optimize device performance.

Table 7.1: Comparison of the beam design having inter-beams a) and the one having larger beams b). The two highlighted lines exhibit a similar stress of 265 MPa.

a)				b)				
N	F_x {N}	σ_y {MPa}	F_k {N}	w { μm }	n	F_x {N}	σ_y {MPa}	F_k {N}
1	1.2	265	24	20	500	0.3	176	8.7
2	2.7	330	55	30	335	0.7	234	29.4
3	4.8	395	97	35	287	0.9	263	46.6

The option of increasing beam width also has the advantage of reducing normal force fluctuation, which is due to stable beam twisting (7.26).

$$\Delta F_x = \Delta F_{x0} \left(\frac{w_0}{w} \right)^3 = \Delta F_x \frac{F_{k0}}{F_k} \quad (7.26)$$

To limit normal force fluctuation to 0.4 N, the beam width of the second version was chosen at 35 μm , while their length and height were kept identical. The total number of beam rows was 280 instead of 500, as in the first version.

The drawback of this second version compared to the first one was an increase in the horizontal force. However, the constancy of the Young's modulus of silicon over time means the horizontal spring force can be taken into account for calculating the friction force.

It should be mentioned that the maximum stress will be higher than the allowed value of 200 MPa. Nevertheless, the rupture occurring around 800 MPa, there is still a significant operating margin. The risk of breaking thin beams during chip handling was more significant than that due to operation (section 8.2.1).

7.4. Flow rate and tightness of seal

Flow rate and tightness of seal are two independent characteristics of a valve. The flow rate depends on the size and shape of the channels, while the tightness is related essentially to the moving surfaces in contact. The demonstrator was

developed to test DLC coatings in order to reduce wear and to have good sealing properties. Therefore, flow rate considerations were not a priority, though a model is proposed to calculate the liquid flow associated with the sliding valve.

7.4.1. Flow rate model

A simplified model is proposed to approximate liquid flow in a sliding valve. This model does not take into account viscosity, which is not dominant for short restrictions (short narrow channels) under large differential pressure.

For non-compressible fluids of density ρ flowing through restrictions, knowing the differential pressure Δp , the liquid flow rate is defined by the change of section, Bernouilli's equation (7.27), and the fluid internal viscosity, the Poiseuille's law (7.28) [7.6].

$$Q_B = \frac{\pi d_c^2}{4} \sqrt{\frac{2\Delta p}{\rho}} \quad (7.27)$$

$$Q_P = \Delta p \frac{\pi d_c^4}{128\eta l_c} \quad (7.28)$$

When the narrowest channel is short, the change of cross-section restricts more the flow than the viscosity within the valve channels (appendix A). The critical dimensions between long and short channels can be calculated by equaling flow rates of the two laws (7.29).

$$\left(\frac{d_c^2}{l_c} \right)_{crit} = 32\eta \sqrt{\frac{2}{\Delta p \rho}} \quad (7.29)$$

Concerning the valve, the orifice is not round, but if a worst case is considered with a 0.5 mm channel diameter at 0.25 bar differential pressure Δp , the frontier between long and short channel is 28 mm for water, which is well beyond the length of the valve channel.

Therefore, calculating flow rate based on the smallest restriction in the valve channels while neglecting their length and viscosity is relatively accurate for water. For 2 bar differential pressure, the maximum operating condition, the

water flow rate of the valve is 1.2 l/min, based on the Bernoulli equation (7.27) and a cross-section of 1 mm². For such a flow rate, the horizontal displacement of a sliding valve has to be about 1.5 mm to switch the flow. This flow resistance places the microvalve/tribometer in the same operating range as the metallic valve presently used by Hamilton AG [7.7].

7.4.2. Tightness

To obtain a tight seal, two conditions have to be fulfilled. First, for hard surfaces, the roughness has to be minimum, to avoid leakage. Secondly, the surfaces have to be maintained in contact without having a fluid film in between. The leakage due to roughness is difficult to model, due to the extremely small size of the flow paths. However, the minimum normal force pressure that has to be applied to keep surfaces in contact can be calculated.

The valve leak testing procedure of Hamilton AG involves the introduction of 3 bar air differential pressure into the device without flow, and measurement of the pressure decay over time. Measurements realized on chips fabricated for an older model of tribometer/valve test bench, showed a threshold normal force, below which leakage rose drastically (section 9.3.2).

If the leakage flow is assumed to behave like flow in nanochannels, the Knudsen number will be very large, and therefore, the pressure gradient constant [7.8]. Based on this assumption, a method for calculating the critical normal force F_{Ncrit} is proposed (7.30).

$$F_{Ncrit} = \Delta p \left(A_{cavity} + \frac{A_{surf_contact}}{b} \right) \quad (7.30)$$

The influence of orifice and contact surface shape is characterized by b , which is larger than 2. For instance, for a circular cavity/orifice surface A_{cavity} and a circular contact surface $A_{surf_contact}$, b equals 3, and for the valve, b should be between 2 and 3. Leak measurements realized validate the critical force estimation method (section 9.3.2).

Table 7.2: Normal critical force estimated for an old tribometer whose leakage was rate tested (section 9.3.2), and for the tribometer under consideration in this study. Values are calculated for $b=2$.

	A_{cavity} {mm ² }	$A_{\text{surf contact}}$ {mm ² }	F_{Ncrit} {N}
Old tribo	8	80	<14.4
New tribo	13.26	38.6	<9.8

Table 7.2 shows that the normal critical force of the proposed microtribometer/valve is below 10 N. This underlines the necessity of dimensioning the device for 10 N normal force if valve tightness is to be guaranteed.

7.5. Conclusion

Based on analytical calculations and FEM simulations, two versions of the microtribometer/valve were dimensioned. For the first version, no buckling behavior was taken into account and the tribometer was unstable at normal forces below 10 N. However, for the second version, the limit of buckling was increased to above 10 N with a large margin for error. This had the additional advantage of reducing the normal force fluctuation to 0.4 N. The drawback was an increase of the horizontal mechanical spring force from 0.3 N to 0.9 N. However, the constancy of the Young's modulus for silicon over time allows this force to be taken into account when calculating the friction force. A flow model was proposed, and valve tightness taken into account to estimate the minimum normal force, which is below 10 N.

7.6. References

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Chapter 8

Sliding Microvalve/tribometer Fabrication

The fabrication of the two parts of the tribometer was based on deep reactive-ion etching (DRIE) of silicon beams having an aspect ratio higher than 10. To vary the normal force, removable test inserts having different thicknesses were fabricated and coated with DLC thin-film layers.

Two versions of the tribometer's main parts were micromachined with a similar process. The first one had beams of 20 μm width on the mask, while the second had larger beams of 35 μm width to facilitate fabrication and improve performances.

The top and bottom coated inserts were not processed the same way. The cavities of the top insert were etched with anisotropic wet etching, while the vertical channels of the bottom inserts are etched with a DRIE process. Both inserts were thinned down with anisotropic etching.

An aluminum support, having channels for fluidic connection, was designed and fabricated to hold the microtribometer/valve with removable glue for testing. When mounted on this support, the two main parts of the microtribometer/valve were screwed together to produce the normal force on the coated test chips.

8.1. Microtribometer/valve main part

The principle part of the microtribometer/valve guides test inserts and produces a normal force on them. Its fabrication relied on high-aspect-ratio DRIE [8.1,8.2].

8.1.1. Process

The fabrication is quite simple to describe since it is a one-step lithography process. However, etching structures of aspect ratio up to 20 through a 390 μm thick silicon wafer relies on optimization of lithography and etching [8.2]. Two pairs of tribometer parts are processed on a single wafer.

▨ : photoresist

■ : silicon wafer

■ : Al

(i)

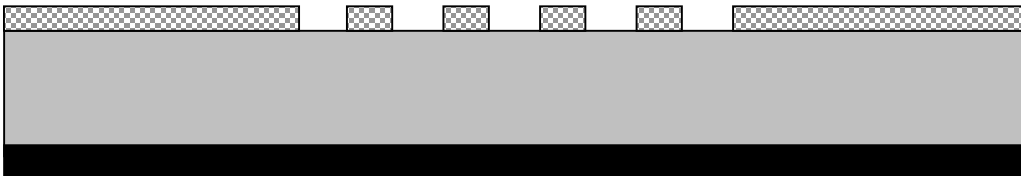


Figure 8.1 Evaporation of 1 μm aluminum onto the backside and lithography in 12 μm thick AZ-4562 photoresist.

(ii)

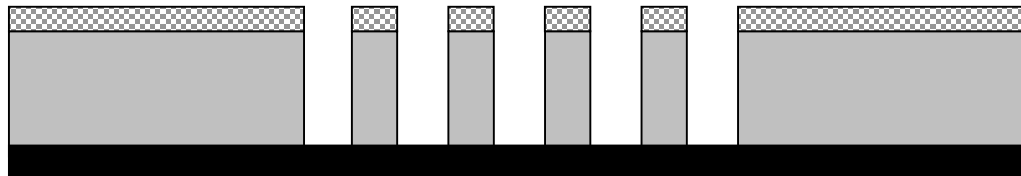


Figure 8.2 DRIE of silicon through the 390 μm thick wafer.

(iii)



Figure 8.3 Plasma O₂ photoresist removing, and etching of aluminum to free the structures.

First, a 1 μm aluminum layer is evaporated on the backside (Figure 8.1). The utility of an aluminum layer on the backside is twofold: 1) it almost eliminates under etching at the end of the etching process and 2) it supports the beams once the etching process is over [8.3,8.4].

A 12 μm thick photoresist is spin-coated on the topside to serve as mask during the entire etching process. A combination of thinner photoresist and SiO₂ was as well tested, but the stress in the oxide resulted in some broken beams at the end of the etching process. Since the lithography parameters were adapted to this process, they are mentioned here. AZ-4562 photoresist is spun at 1700 rpm and pre-baked three times on a hot plate at 100°C for 1 minute, with cool down times of 2 minutes in between. Then, the photoresist is exposed with 150 mJ/cm² energy. The development is done in AZ400 at a concentration of 1:3 (AZ400:H₂O) for 90 s. Finally, the postbake is started first, like the pre-bake on a hot plate at 100°C for 1 minute and to finished in an oven at 125°C for 30 minutes.

The structures are then etched through the wafer with a DRIE process (Figure 8.2). Since the beam etching presented different difficulties than structures fabricated in the past, parameter optimization had to be carried out to achieve good verticality and uniformity [8.2].

Finally, photoresist is removed in an O₂ plasma followed by aluminum removal by wet etching (Figure 8.3). After this step, the microvalve/tribometer parts are held on the wafer by little beams designed to be broken manually when the structures are to be freed from the wafers. To hold the inserts, a glass cover is anodically bonded onto the top part of the device [8.5].

8.1.2. Fabrication results

The different beams widths of the two versions led different etching results. The second version was easier to process than the first, due to reduced fragility of the beams.

First version

The first version with 20 μm beam widths on the mask, was difficult to process, essentially due to non-uniformity of the DRIE etching. The first attempts with a mask composed of SiO_2 and photoresist induced too much stress in the beams at the end of the process, breaking them. With a mask composed exclusively of photoresist and an aluminum layer on the backside, it was possible to process a few samples (Figure 8.4, Figure 8.5).

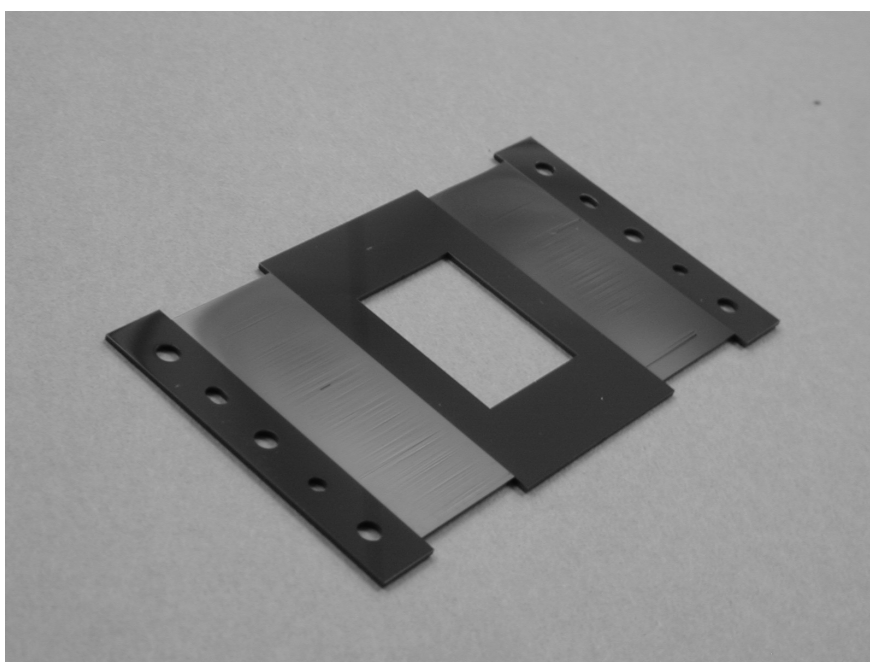


Figure 8.4 Photo showing the main bottom part of the microtribometer/valve which measured 39.5 x 22 mm² and had 500 beams rows.

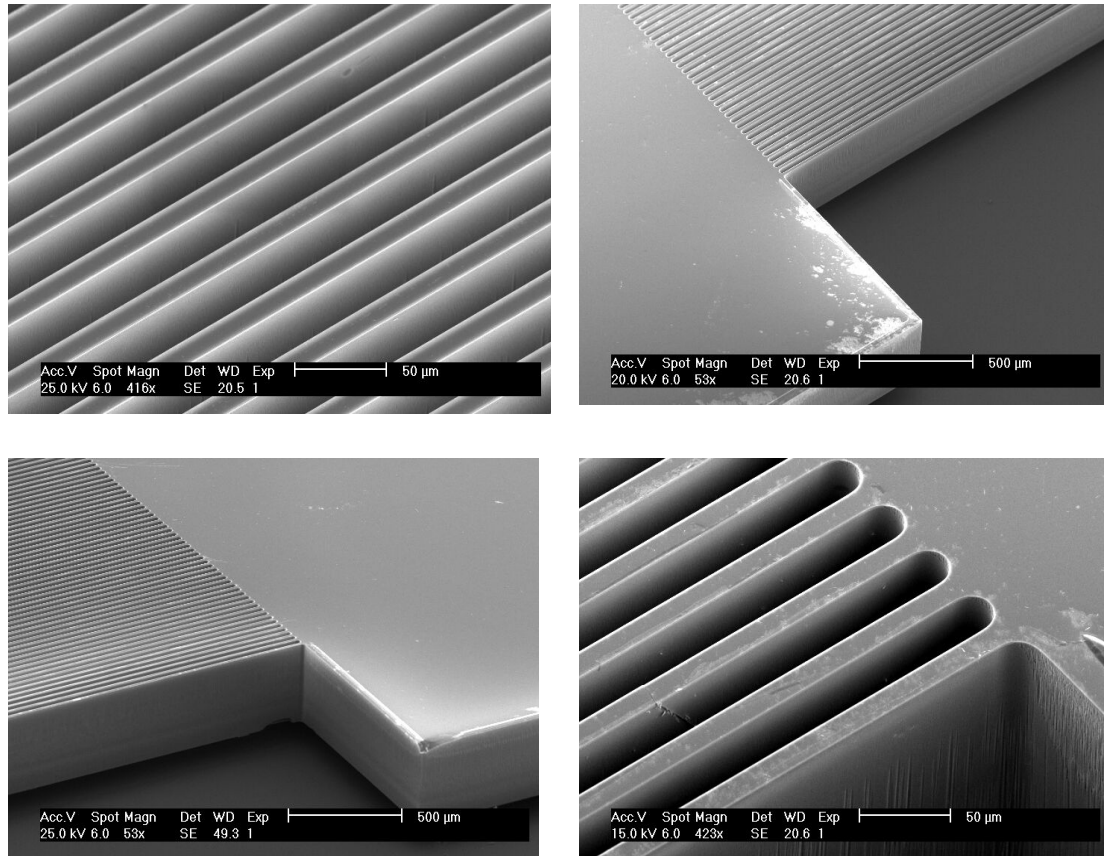


Figure 8.5 SEM views of the first version beams successfully fabricated on this sample.

Nevertheless, depending on the etching parameters, some samples had beams which were thinner than others, and which broke very easily or were unstable laterally (Figure 8.6, Figure 8.7). The SEM Figure 8.8 of broken beams shows that their widths varied as a function of height.

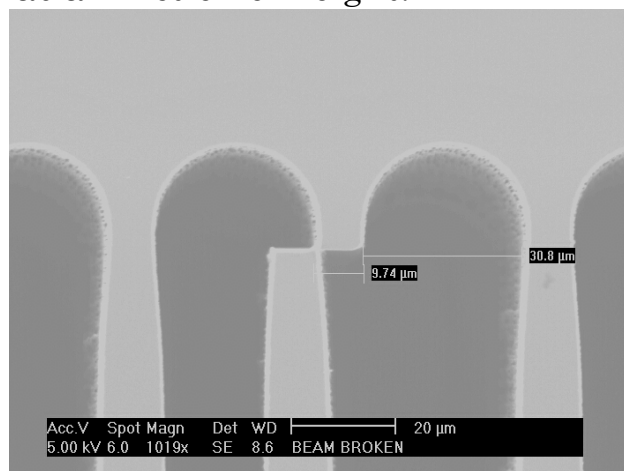


Figure 8.6 SEM view of a beam which broke at its thinnest point of 10 μm width.

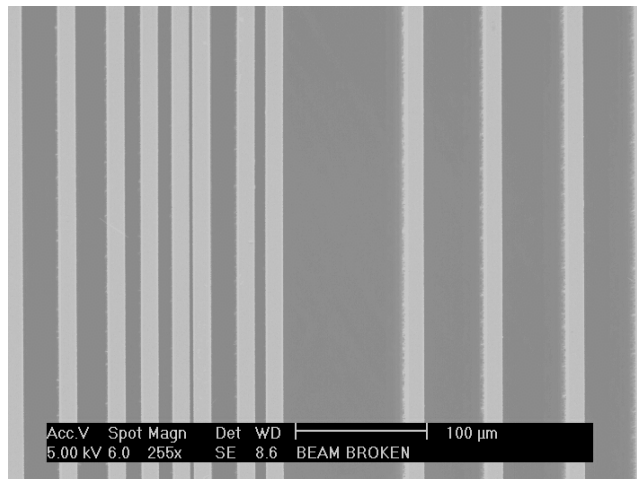


Figure 8.7 SEM view of laterally unstable beams, due to their thinness.

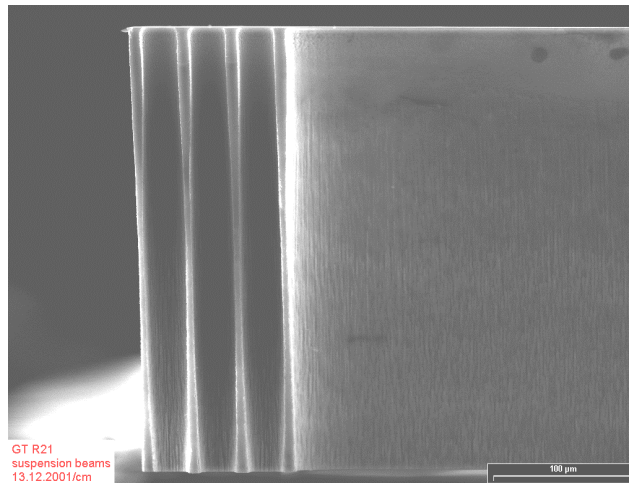


Figure 8.8 SEM view of a broken beam that shows a narrowing at approximately beam half-height.

Therefore, actual beams were thinner than the size on the mask, principally due to photoresist mask etching during the DRIE process. The beam thickness was in the range of 3 to 19 μm , depending on the position on the wafer and the DRIE parameters.

Second version

The second version with 35 μm beam thickness on the mask was much easier to process, essentially due to the reduced fragility of the beams. DRIE, realised with a photoresist mask

and aluminum layer on the backside, was successful for all devices (Figure 8.9, Figure 8.10).

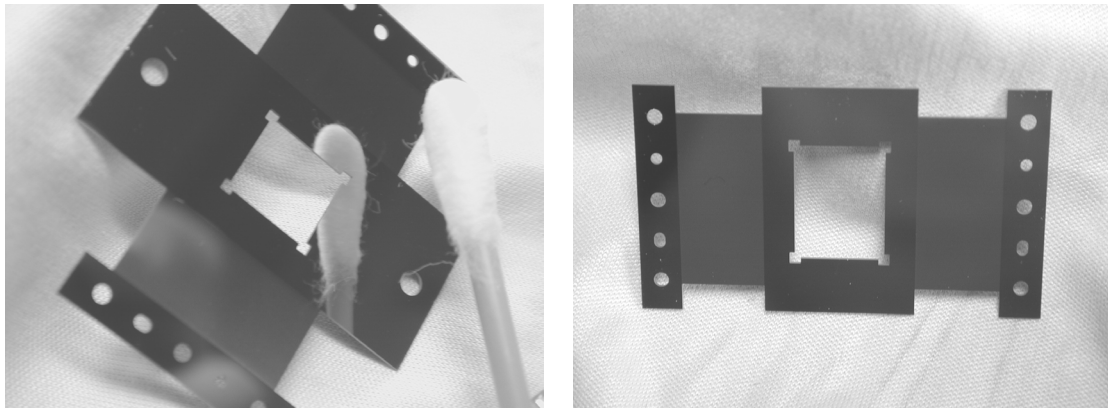


Figure 8.9 Top and bottom main parts of the microtribometer/valve body, having 280 beams rows.

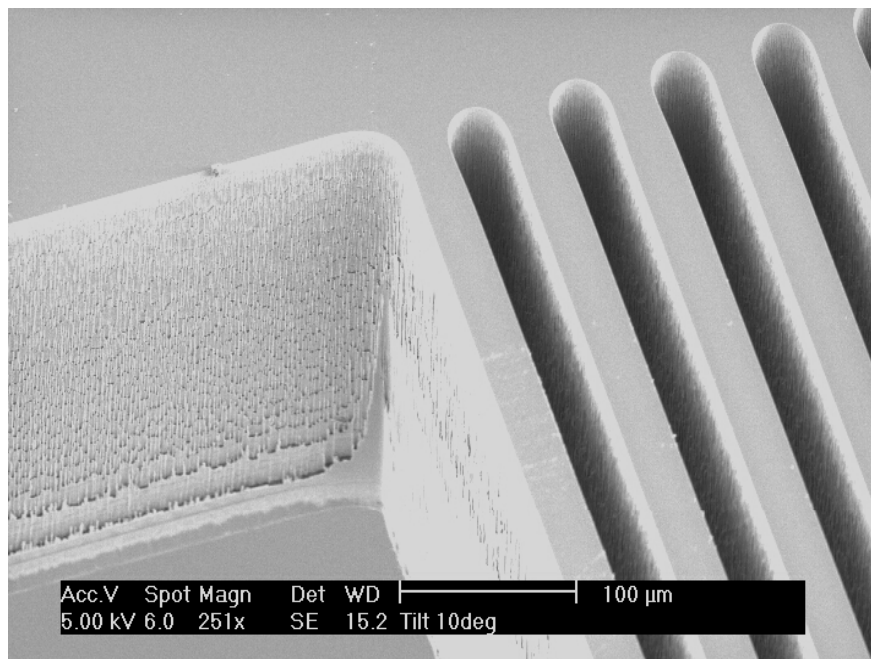


Figure 8.10 SEM view of beams fabricated according to the second version process.

A conical beam shape and a reduction of the beam width, essentially due to mask etching, will lower the stiffness of the beams (Figure 8.11, Figure 8.12).

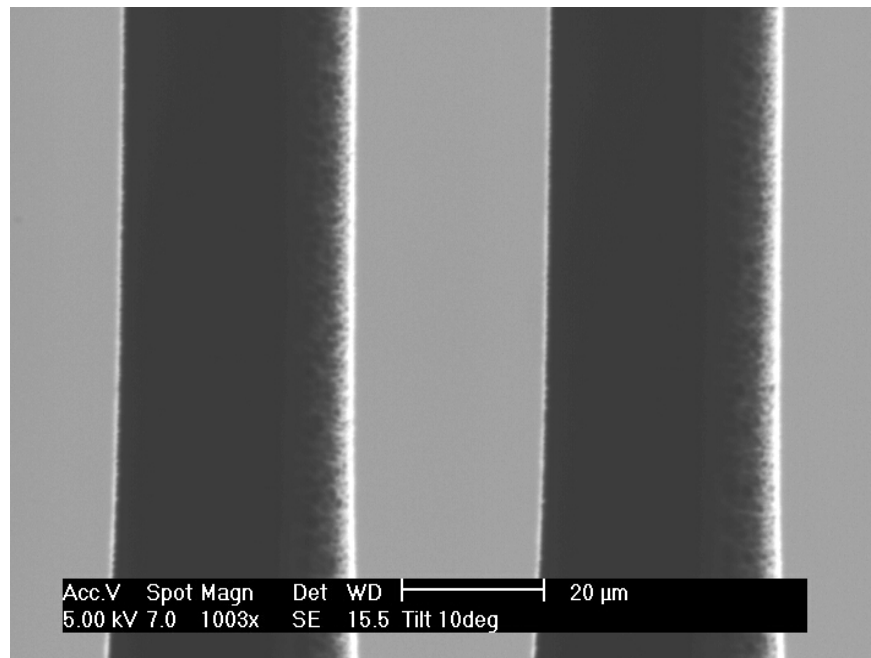


Figure 8.11 SEM view showing a beam thickness of 28 μm on the topside.

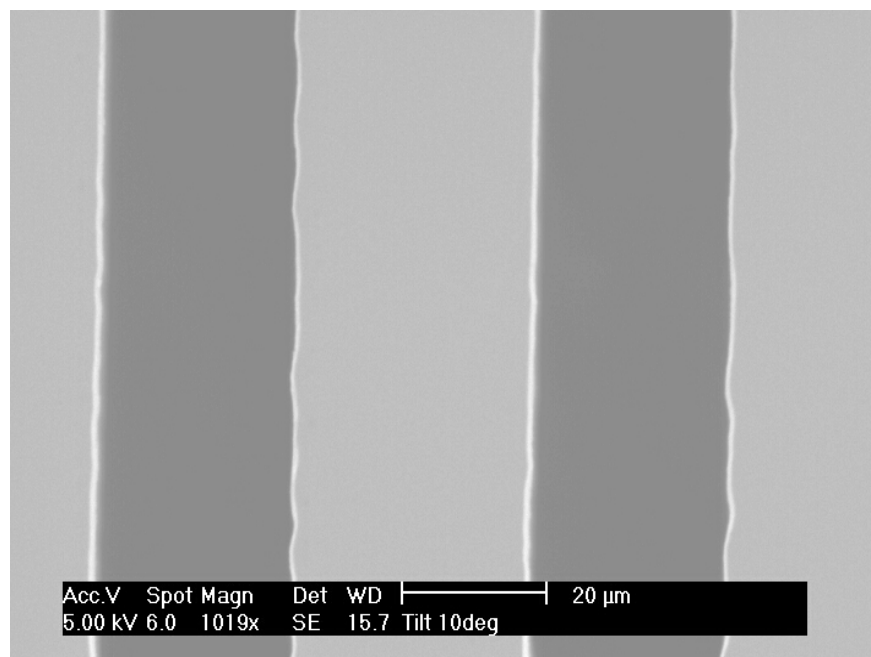


Figure 8.12 SEM view showing a beam thickness of 32 μm on the backside.

8.2. Test chips

Test chips were removable silicon inserts coated with DLC layers for tribological or flow measurements. Their processes relied on bulk micromachining with wet and dry etching.

8.2.1. Process

The insert fabrication was developed based on a previous process of test inserts for a large-size tribometer [8.6]. The fabrication of the top inserts used only KOH wet etching, while the bottom chip process was based on a combination of wet anisotropic etching and DRIE.

Top chip

First, a 5000 Å thermal SiO₂ was grown on both sides of the wafer, and patterned in BHF in order to create a mask for KOH wet etching (Figure 8.13). The backside oxide was removed in BHF to enable thinning of the wafer by wet etching.

A cavity was then etched in KOH, and the wafer thinned down at the same time (Figure 8.14). The final thickness depended on the desired normal force when mounted in the main part of the tribometer/valve. Therefore, cavity height varied, but since fluidics were first considered only for valve tightness measurements, it was not critical. However, doing the etching of the cavity separately from the thinning would allow control of the cavity height.

Finally, before sawing the chips, Sulzer Innotec deposited a DLC layer with a proprietary process (Figure 8.15).

▨ : photoresist

■ : silicon wafer

■ : DLC

▨ : thermal SiO₂



Figure 8.13 A 5000 Å thermal SiO₂ mask is patterned on the topside.

(ii)



Figure 8.14 A KOH solution thins the wafer from 525 μm to thicknesses within the range of 410 to 450 μm .

(iii)



Figure 8.15 The silicon oxide is removed, and a PVD DLC layer is deposited on the topside. This process is proprietary.

Bottom chip

First, a 5000 $\text{\AA}$ thermal SiO_2 is grown on both sides of the wafer, and removed on the backside in BHF in order to protect the topside for thinning the wafer in KOH (Figure 8.16, Figure 8.17). A mask for DRIE is then patterned, and channels are etched through (Figure 8.18). Finally, before dicing the chips, a DLC layer is deposited with a confidential process (Figure 8.19).

(i)



Figure 8.16 A 5000 $\text{\AA}$ thermal SiO_2 mask is grown on both sides, to be removed from the backside in a subsequent step.

(ii)



Figure 8.17 The silicon wafer is thinned down to 420 μm .

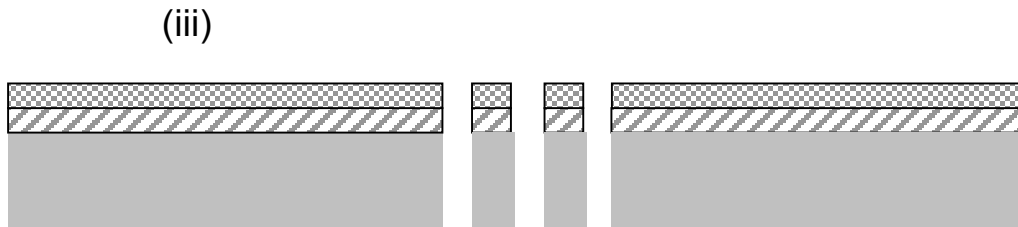


Figure 8.18 Patterned layers of 10 μm thick photoresist AZ-4562 and SiO_2 serve as mask for DRIE.

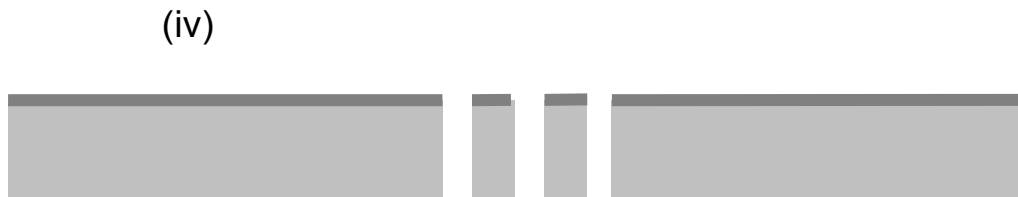


Figure 8.19 The DRIE mask is removed and a PVD DLC layer is deposited.

8.2.2. Fabrication results

The fabrication did not present any major difficulty. The thinning of the wafer in KOH, which is not very common, gave good uniformity and low surface roughness. Different types of DLC layers were deposited by Sulzer Innotec.

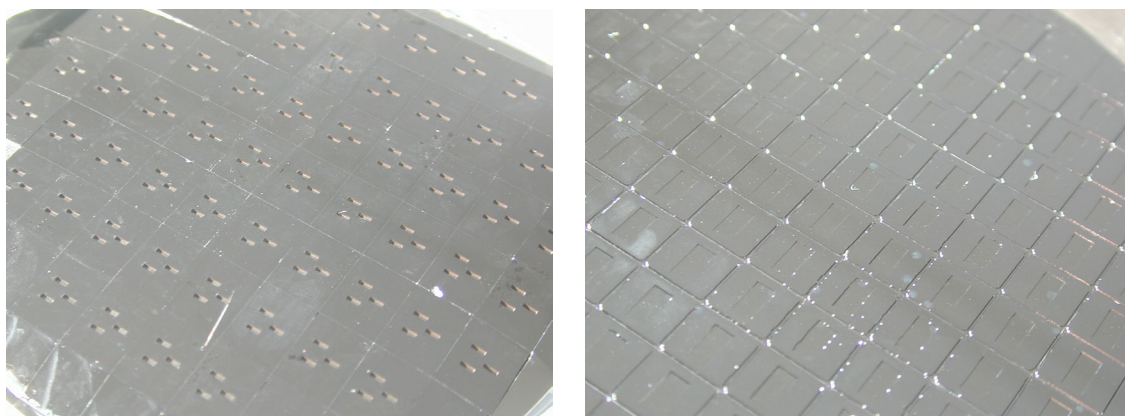


Figure 8.20 Photos of the bottom and top chips coated with DLC layers. The bottom inserts measured: 8.49 mm x 10.99 mm and the top insert, 7.49 mm x 8.49 mm.

8.3. Support

The microtribometer/valve was made to be mounted on an aluminum support that held the bottom main part and chip fixed, while letting the top main part and chip move freely, actuated by an external motor (Figure 8.21). This aluminum support connected the fluid flow to the valve.

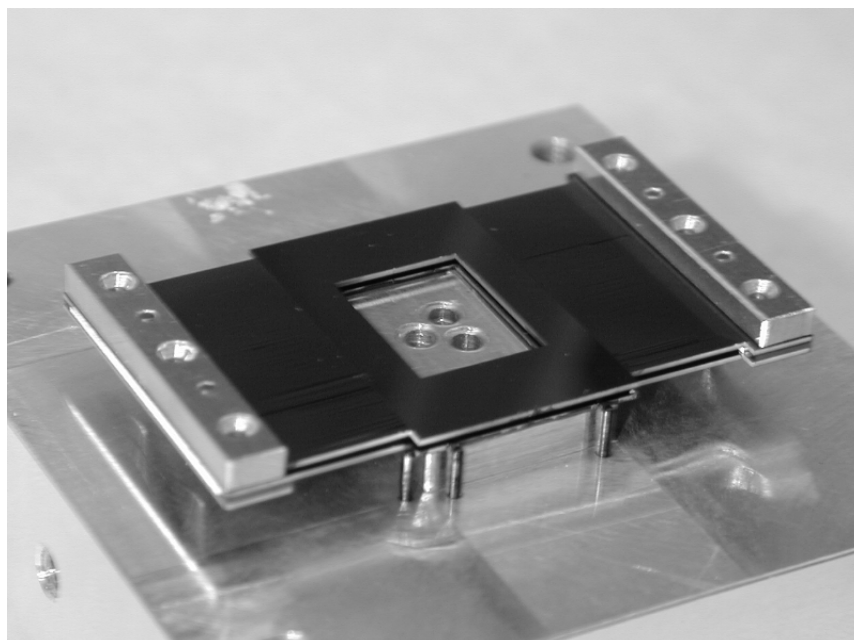


Figure 8.21 The microtribometer/valve mounted on its support, without the test chips. The three circular orifices in the center connect the fluid flow to the valve.

The mounting presented a few challenges. First, the bottom main part was fixed onto the aluminum support with glue that melts when heated over 60 °C (Crystal Bond), (Figure 8.22). Therefore, many microtribometer/valve could be used and removed without damaging them.

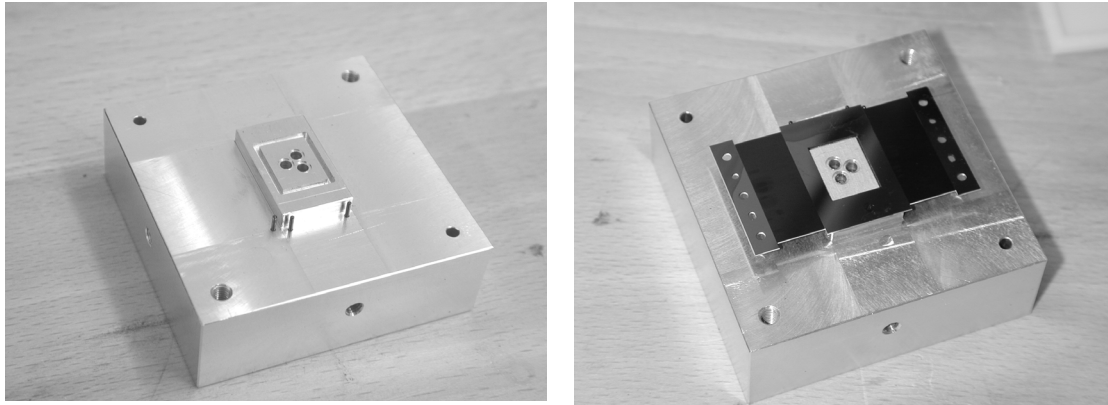


Figure 8.22 Photos showing the bottom main part glued on its aluminum support.

Removable DLC-coated test chips could then be placed in the rectangular cavities, and normal pressure applied on them by screwing together the two silicon main parts (Figure 8.23). This process of screwing was a little bit difficult to realize, but on the second version of tribometer/valve it did not pose any problem, due to the reduced fragility of the beams.

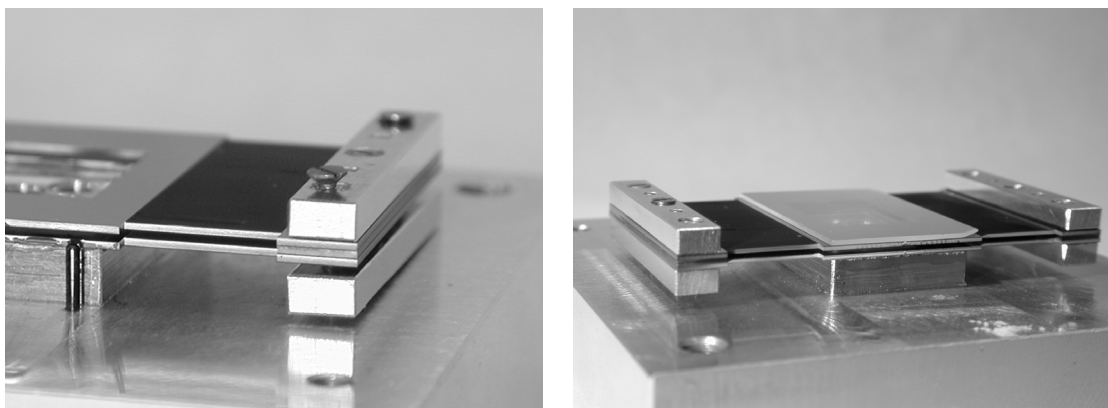


Figure 8.23 Pictures of the two main parts tight together by screws.

Finally, the top silicon part was linked to a linear motor for actuation by gluing a metallic plate to the silicon top part (Figure 8.24).

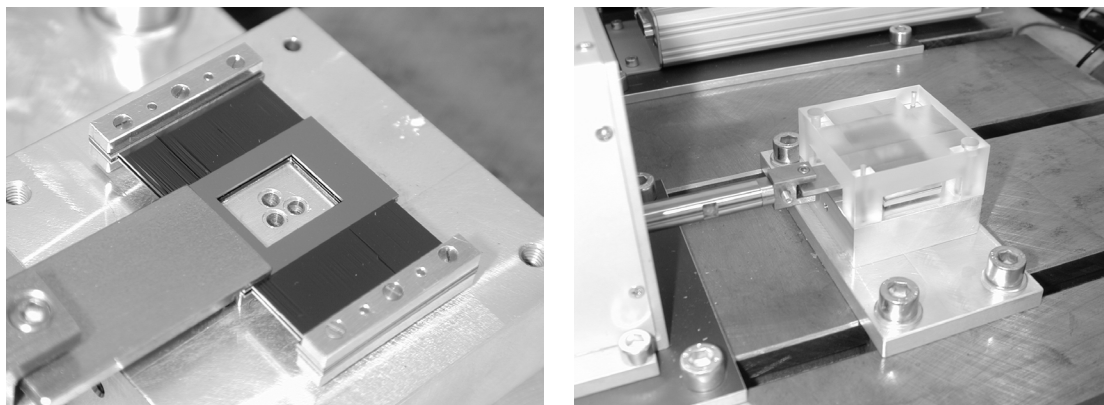


Figure 8.24 Pictures of the link to a linear motor.

This type of mounting allows replacement of chips without using glue if the main part of the tribometer is kept. Chips with different beam widths were aged with microtribometers/valves mounted on this aluminum support [8.7].

8.4. Conclusion

Two versions of the main part of the microtribometer/valve were fabricated with different beam widths. The first version, based on 20 μm beams, was difficult to process right to the end, due to the fragility of the beams. The second process, on the other hand, based on 35 μm beams was successful for all devices. Beam thinning of about 5 μm was observed.

Many chips coated with DLC thin-film layers were micromachined successfully. The process based on wafer thinning in KOH did not present any particular problems. The mounting method of gluing onto an aluminum support and screwing the silicon parts was satisfactory.

8.5. References

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Chapter 9

Sliding Microvalve/tribometer

Testing and Characterization

The measurements reported in this chapter were realized at the Neu Technikum Buchs, mostly by S. von Gunten [9.1]. For testing, an electrical feed back linear motor made by the company SMAC (California) that measured displacement, speed and acceleration actuated the microtribometer/valve mounted on its aluminum support (Figure 9.1).

The first microtribometer/valve version was tested with DLC coated chips for more than one million cycles [9.2,9.3]. Observation of unstable behavior in the first version led to the development of a second version that avoided beam buckling (section 7.3). The mechanical stability of the second version was verified.

Three DLC-coated pairs of chips were aged for more than one hundred thousand cycles each. Surface scratches due to wear were analyzed by SEM and AFM measurements. Based on published DLC tribological studies and analyses, a basic explanation of the low DLC wear is proposed. However, the complexity of the processes involved will necessitate much more testing to be able to choose the coating most adapted to a specific application.

For time reasons, the characterization was focused on dry friction without introducing fluids in the system. However, previous tightness measurements made on a large size

tribometer/valve are compared with the calculated minimum normal force (section 7.4.2).

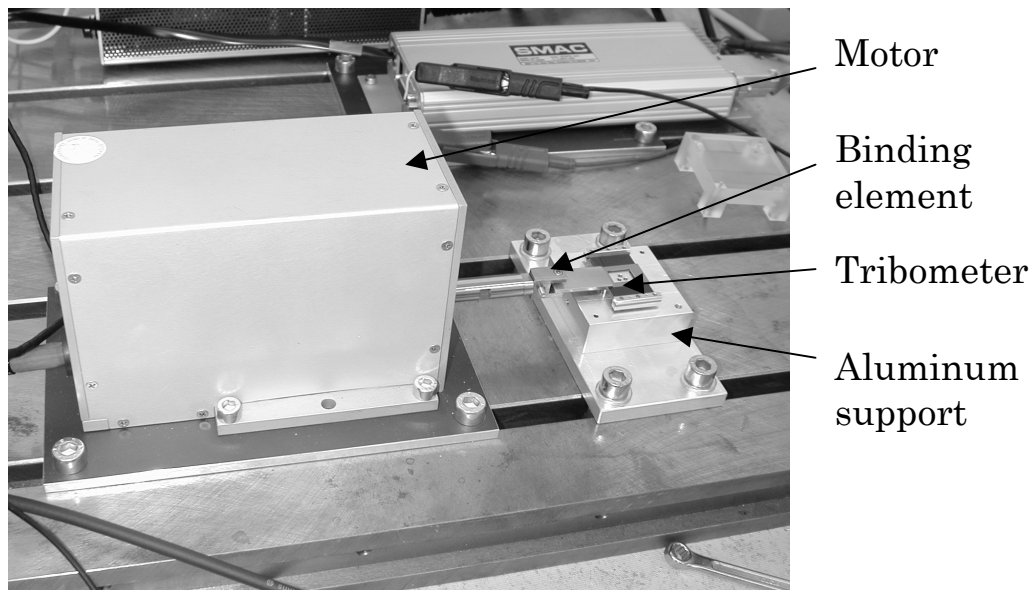


Figure 9.1 Photo of the microtribometer/valve mounted on its aluminum support and actuated by a linear motor from the company SMAC (California).

9.1. Testing set-up

An electrical linear motor made by the company SMAC (California) was fed back by an electronic controller, which took into account measured parameters of electrical current, speed, position, and force (Figure 9.2). A PC using a LabView program (National Instruments) sent the motion parameters to the electronic controller by a serial communication.

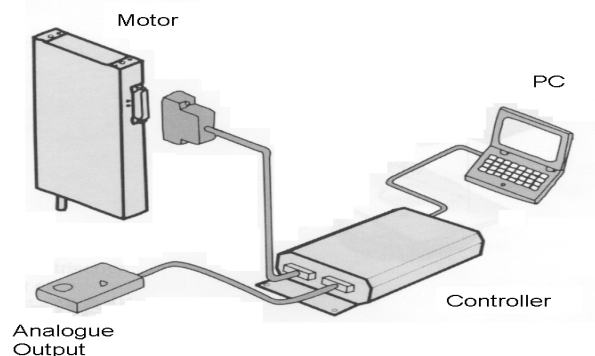


Figure 9.2 Linear motor controller made by the company SMAC (California).

A binding element allowing slight misalignment linked the motor shaft to the microtribometer/valve (Figure 9.3).

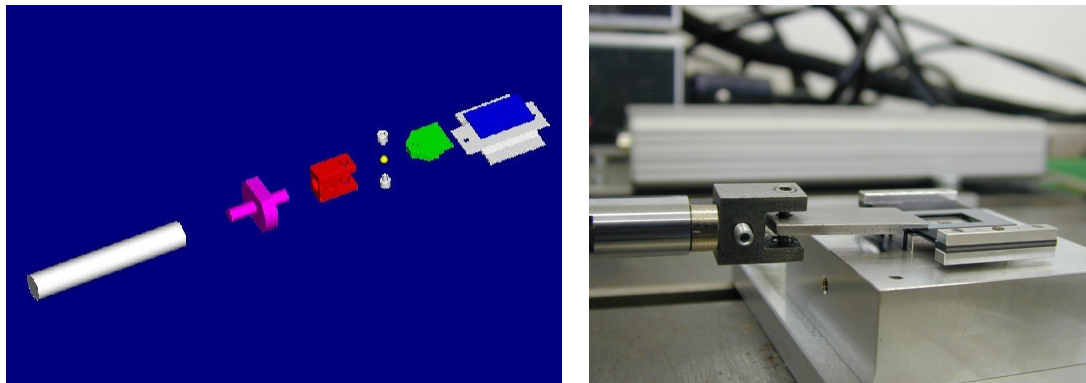


Figure 9.3 Mechanical linking element.

The control system of this motor did not allow reaching oscillating frequencies higher than 10 Hz, and above 2 Hz, instabilities were observed. The reason of this behavior is most likely the position incremental encoder built-in the motor, that delivers a pulse square instead of a pulse triangular positioning signal.

During testing, the normal force was calculated based on the measured thickness of test chips sandwiched in between the two microtribometer/valve elements. To do so, the results of FEM simulations were used and scaled down to the real beam thickness.

9.2. Microvalve/tribometer main parts

The microtribometer/valve body behaved very well when placed in conditions that did not allow mechanical beam instabilities to occur. A beam width that was too small explained the first version instabilities, which were avoided in the second version with wider beams.

9.2.1. Behavior under actuation

The behavior of the main body of the tribometer/valve under actuation was completely satisfying. A total horizontal displacement of 1.5 mm was reached as expected, and no beam breaking occurred when operated. The first version was tested over one million cycles with a maximum normal force of 4.3 N to avoid instabilities, while with the second version just stability verification for a few oscillations was realized.

9.2.2. Instabilities

Observation of the first version microvalve/tribometer mounted on its aluminum support, with test inserts that induced vertical beam displacement, can reveal instabilities. This behavior occurred for vertical displacements larger than a threshold value of 38 μm for one of the tested samples. Knowing the scaling laws (section 7.3.3), a theoretical average beam width was calculated based on the assumption that the instability was due to buckling (9.1),(9.2),(9.3).

Defined quantities:

- $f_{z \text{ instability}} = f_{z \text{ ins}} = 38 \mu\text{m}$
- For $b_0 = 20 \mu\text{m}$: $F_{\text{buckling simulated}} = F_{k \text{ sim}} = 8.7 \text{ N}$ (7.3.2)
- For $b_0 = 20 \mu\text{m}$: $F_{z \text{ max}} = 10 \text{ N}$, $f_{z \text{ max}} = 70 \mu\text{m}$ (7.2.3)

$$F_{z \text{ ins}} = \frac{f_{z \text{ ins}}}{f_{z \text{ max}}} \frac{w}{w_0} F_{z \text{ max}} \quad (9.1)$$

$$F_{k \text{ mea}} = \left(\frac{w}{w_0} \right)^3 F_{k \text{ sim}} \quad (9.2)$$

Assumption: $F_{z \text{ ins}} = F_{k \text{ mea}}$

$$w = w_0 \sqrt{\frac{f_{z \text{ ins}}}{f_{z \text{ max}}} \frac{F_{z \text{ max}}}{F_{k \text{ sim}}}} = 15.8 \mu\text{m} \quad (9.3)$$

If the instability was due to buckling, the average beam width is 16 μm . Since this value matches SEM observations of beam

width reduction due to etching (section 8.1.2), the buckling explanation of the instabilities can be considered correct.

With the second version, no instabilities were observed, and vertical displacements of up to 80 μm were tested, which corresponds to a normal force of 9.6 N for a measured beam width of 30 μm (section 8.1.2). However, at the maximum nominal vertical displacement of 70 μm for which the microtribometer/valve was designed, the allowable normal force is 8.4 N. Nevertheless, the large margin of error means the expected breaking limit is much higher.

9.3. DLC coated chips

Three chips were aged with the first version silicon microtribometer/valve under normal forces ranging from 2.4 N to 4.3 N without flow. The valve tightness was not tested for these chips, but interesting results obtained with the old large size tribometer are compared with the minimum normal force calculation.

9.3.1. Tribological study

The tests were done for a range of forces below the critical force at which the first buckling mode had previously appeared. No buckling modes were observed. The system was very stable for the duration of the tests. No fluid was introduced in the system.

Table 9.1: Three first tests realized for different normal load and cycles number.

Test #	f_z { μm }	F_N {N}	Number of cycles
1	38	4.3 -1.7	105'500
2	21	2.4 -0.9	147'370
3	21	2.4 -0.9	344'000

Table 9.1 lists the test conditions for three different DLC-coated chips. For a beam width of 16 μm , the normal force is calculated based on simulations, taking into account the force reduction for horizontal displacement. The friction resulted in the

appearance of scratches as can be seen on the SEM views (Figure 9.4).

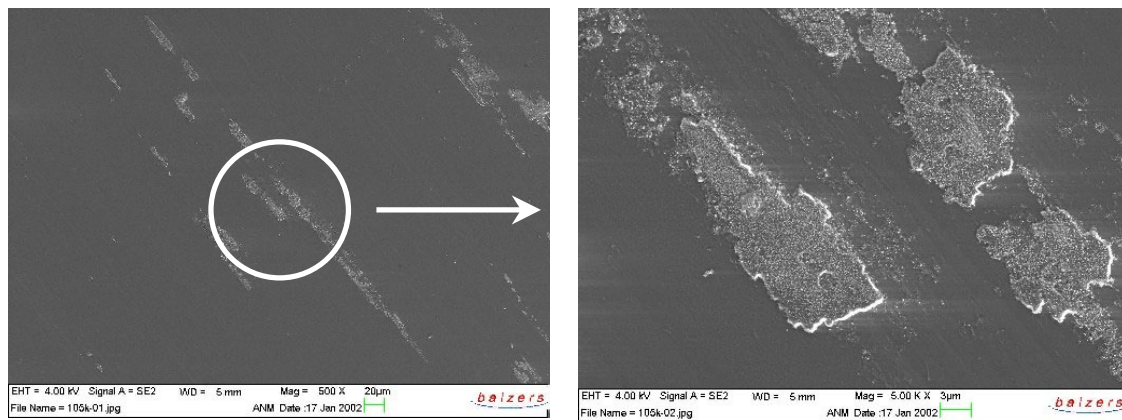


Figure 9.4 SEM views of scratches apparition on sample n°1.

AFM measurements of scratches depths in different locations showed that this parameter was smaller than the thickness of the DLC layer (Figure 9.5). The localized nature of these scratches could be related to free particles or imperfect flatness of the surfaces. These scratches reveal extraction of DLC debris, probably due to the brittle character of such layers and their high compressible stress [9.4]. The appearance of ridges close to the scratches suggests a re-deposition of the material extracted.

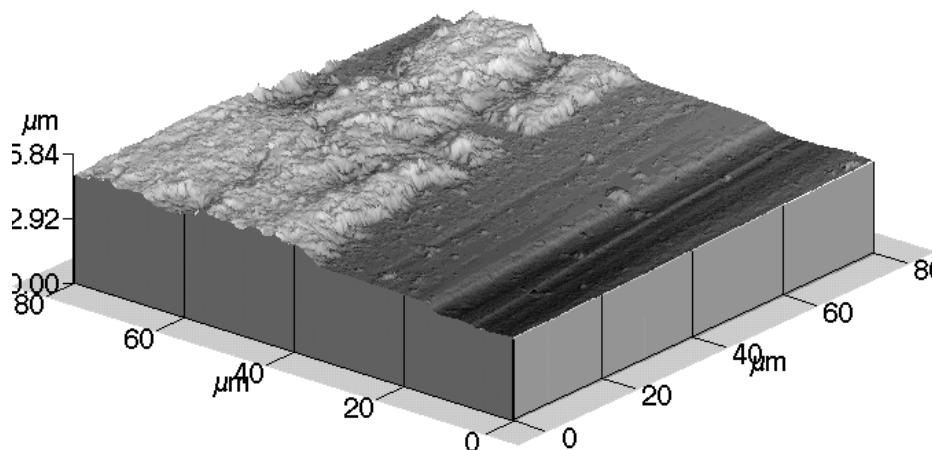


Figure 9.5 AFM of a scratch area on sample n°1. The original surface is surrounded by a scratch (on the right) and a ridge (on the left).

Measurements of sample n°3 showed scratches with same depth as sample n°1 aged by a factor 3 less. The scratch apparition is dominant at the beginning of the aging process and then tends to stabilize.

A proposed explanation of the DLC aging process and scratch development observed could be that imperfect surface flatness or external dust particles between surfaces cause local heating at the beginning of friction tests [9.5,9.6]. For high speed and pressure, the heating can be sufficient to result in the transformation from diamond to graphite, or from sp^3 to sp^2 C forms, to occur [9.7]. This would yield a transfer film in between the two surfaces [9.4]. The little ridges observed close to the scratches would then be due to deposition and agglutination of material that could be graphite [9.5].

However, the literature shows that friction and wear of two identical DLC-coated surfaces is highly dependent on the DLC conditions of deposition and film composition [9.8]. The humidity is also a factor that can completely modify the friction coefficient [9.8]. The complexity of DLC friction will necessitate studies on many more samples to be able to eventually have a general understanding of the different physical phenomenon involved.

9.3.2. Valve tightness

Tightness measurements were only realized on test chips inserted in the previous large-size tribometer. However, since these measurements were used to validate the normal pressure calculation method, they are reported here (section 7.4.2).

The test procedure of Hamilton AG involved placing the three-way valve in one position and pressurizing the valve with 3 bar differential air pressure. The system was closed and after 6 seconds, the pressure decay measured [9.1]. The tightness of the valve was qualified by this procedure as good to sufficient [9.1].

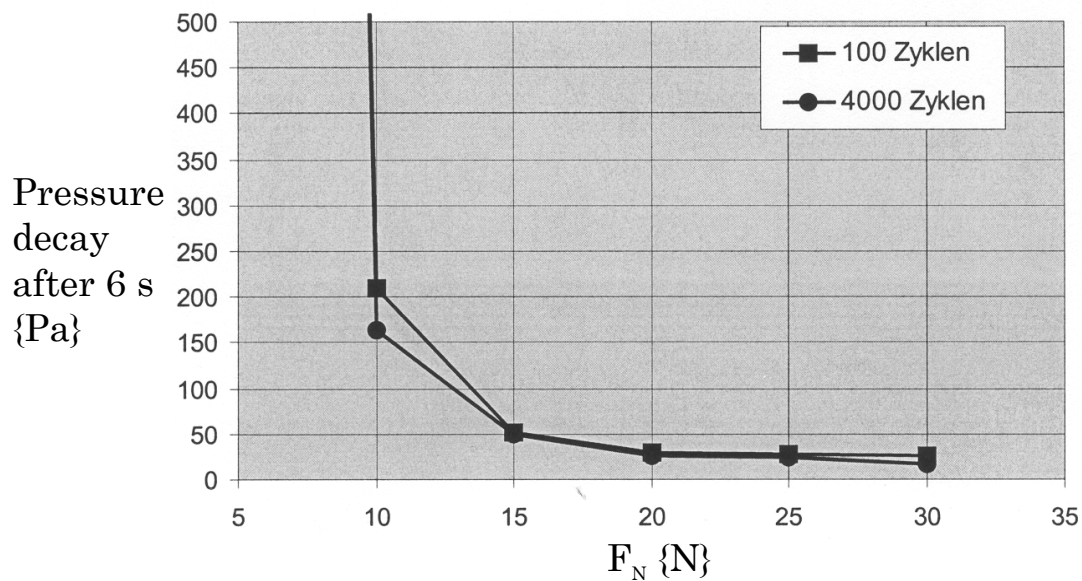


Figure 9.6 Graphic showing the pressure reduction after 6 seconds of tightness tests at different normal forces [9.9].

Measurements realized after short and long aging show that for normal forces lower than 15 N, the leakage increases drastically (Figure 9.6) [9.9]. This validates the calculation made in section 7.4.2, where the minimum pressure was calculated by considering a constant gas pressure gradient between the two contacting surfaces. Apparently, gas leakage occurs everywhere between the two surfaces, due to surface roughness or particles extracted from the material in friction. However, for some reason recent measurements did not exhibit such a clear normal force threshold value [9.1]. This different behavior could be due to free particles, humidity or flatness.

9.4. Conclusion

A silicon microtribometer designed using an analytical calculation and FEM simulations was successfully tested. This device can accurately guide DLC-coated silicon test chips over a distance of 1.5 mm, while producing a normal force of up to 9.6 N. In the second version with wider beams, the beam instabilities observed previously were suppressed. With the first version microtribometer/valve body, more than 1 million cycles were realized.

DLC-coated inserts were submitted to a few hundred thousand oscillations under normal forces in the range of 2.4 to 4.3 N. Micro-scratches appeared after a few thousand cycles, but did not evolve much for the rest of the test. Observation with SEM and AFM showed that material was not only removed, but re-deposition occurred in some areas close to the scratches. Since these scratches are less deep than the thickness of the DLC coatings, such thin films are suitable for reducing the wear of contacting flat silicon surfaces sliding over each other. Further studies of friction of DLC coatings on a larger number of samples would help develop an understanding of the mechanism of scratch evolution over time, and ultimately help in the selection of the most adapted coating for a specific application.

The tightness of the valve was qualified by the Hamilton AG testing procedure to be between good and sufficient. This result is encouraging for developing a demonstrator valve using the concept of flat DLC-coated silicon chips sliding in a reciprocating manner.

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Chapter 10

Conclusion

Two microvalve demonstrators were designed, fabricated and characterized. One is an electrostatic Ta-Si-N valve for gases, and the other is a sliding silicon valve for liquids that works also as a tribometer. The first was fully tested as a valve, while the second was mostly tested as a tribometer.

Different working high-speed Ta-Si-N-based gas valves made using different designs and processes were tested. The basic valve version was capable of switching gas flows under 2 bar differential pressure, and could work as an on/off switch or be actuated in pulse-width modulation (PWM). The actuation voltage ranged from 70 to 140 V, depending on the fabrication process. Based on this basic valve, design investigation was realized to reduce actuation voltage and increase flow rate. A factor 2 measured reduction of the actuation voltage under flow conditions was reached with an asymmetric membrane that bends in flexion and torsion. Another approach which proved satisfactory was to make many valve orifices work in parallel on the same chip. The flow rate of a single orifice would be multiplied by the number of orifices to reach a flow rate of 45 ml/min under 2 bar nitrogen gas differential pressure for 20 valves in parallel. If the parallel concept is extended, a flow rate of 1 l/min could flow through 400 valves arranged on a surface of 4 mm².

Different fabrication processes have made it possible to improve the reliability of the Ta-Si-N microvalve and simplifying the packaging. The first valve versions exhibited electrical breakdown in the insulation layer. In the last version, made with a higher quality, thicker insulation layer, this phenomenon never occurred. However, this thicker oxide required an increase of the actuation voltage and most likely resulted in more leakage around valve seat. Future research should focus on thinner insulation layers that might reduce the actuation voltage significantly. When using an aluminum sacrificial layer, the Ta-Si-N membrane never suffered from lack of reliability when operating. To our knowledge, this valve is the first MEMS working device using Ta-Si-N as a mechanical material.

A silicon microtribometer/valve designed using analytical calculation and FEM simulations was successfully fabricated and tested. This device can accurately guide DLC-coated silicon test chips over a distance of 1.5 mm, while producing a normal force of up to 9.6 N. FEM simulations of lateral buckling allowed development of a second version microtribometer/valve, in which previously observed instabilities were suppressed. Different test chips were aged with this microtribometer for over one hundred thousand cycles, each with a normal force up to 4.3 N.

Tribological studies were conducted with DLC-coated inserts using different normal forces and cycle numbers. The most important observation enabled by the tribometer involved the evolution of coating wear over time. Micro-scratches, which could indicate wear in the DLC coatings, appeared after a few thousand cycles, but did not change much for at least a further few hundred thousand cycles. Since these scratches are less deep than the thickness of the DLC coatings, such thin films are suitable for reducing the wear of contacting flat silicon surfaces sliding over each other.

Further studies of friction on DLC coatings using a larger number of samples should help develop an understanding of the mechanism of scratch evolution over time. Testing will have to be conducted to show the adequacy of this sliding microvalve to fluidic applications involving human serum.

These developments of valve demonstrators have revealed the possibility of using thin film layers or coatings, such as

tantalum silicon nitride (Ta-Si-N) or diamond-like carbon (DLC) to enhance the performances of microvalves.

Appendix A

Boundary between nozzles and channels

The absolute flow rate limit of a fluid flowing through a change of cross-sectional area is given by inertial transformation, which is defined by Bernoulli's equation for incompressible fluids [10.1] and isentropic transformation for compressible fluids [10.2]. However, in addition, friction within the fluid can dominate under some geometrical and pressure conditions. The purpose of this calculation is to determine the boundary conditions under which viscosity has to be taken into account within the channel of the orifice. For long channels, viscosity determines flow rate [10.3], while for ideal nozzles it is inertial transformation [10.1].

Viscosity can be easily understood by considering internal friction occurring within the fluid. For laminar flow in a channel of length l_c , Poiseuille's law shows that, speed flow v_p in a channel of diameter d_c is proportional to the differential pressure Δp for constant η (10.1). For non-laminar flow, the calculation is more complex and will not be studied here; in the small channels encountered in microsystems, the Reynold's numbers are usually much lower than the limit of 2300 [10.4].

$$v_p = \Delta p \frac{d_c^2}{32\eta l_c} \quad (10.1)$$

Bernouilli's equation (10.2), which expresses the conservation of energy for a perfect laminar fluid, shows that for a defined volume dV , the sum of the internal energy, given by the product of pressure by the volume $p dV$ and the dynamic energy $\frac{1}{2} \rho v^2 dV$ remains constant. In this case, the fluid is characterized by its density ρ and velocity v .

$$dV(\frac{1}{2} \rho v^2 + p) = \text{constant} \quad (10.2)$$

This equation shows that if the potential energy along the path of a fluid stream drops, the potential energy is converted to inertial energy. When the conversion is total, meaning that no potential energy remains compared to the lowest possible level of energy in the system, the maximum fluid speed v_B can be calculated (10.3).

$$v_B = \sqrt{\frac{2\Delta p}{\rho}} \quad (10.3)$$

This is the fluid velocity going through a nozzle placed in between two reservoirs of defined pressure $\Delta p = p_{in} - p_{ou}$, which is independent of the geometry of the orifice in first approximation [10.1].

Therefore, depending on the pressure drop and fluid viscosity, the limit of validity of these equations (10.1),(10.3) can be calculated. Equating velocity flow defined by Poiseuille's law and the one defined by Bernouilli's law gives the critical conditions for viscosity consideration within the channel of the orifice.

$$v_P = v_B$$

$$\left(\frac{d_c^2}{l_c} \right)_{crit} = 32\eta \sqrt{\frac{2}{\Delta p \rho}} \quad (10.4)$$

At low pressures, the viscosity effect dominates, while at large pressures it is inertial transformation (Figure 10.1).

A.1. Gas

For gases, the pressure variation induces changes in density which become important at large flow velocities. Equation (10.3) is valid up to half the speed of sound, which corresponds to pressure variations up to 0.25 bar [10.5]; at higher velocities, formula (10.3) has to be replaced by isentropic formulas for gases [10.6].

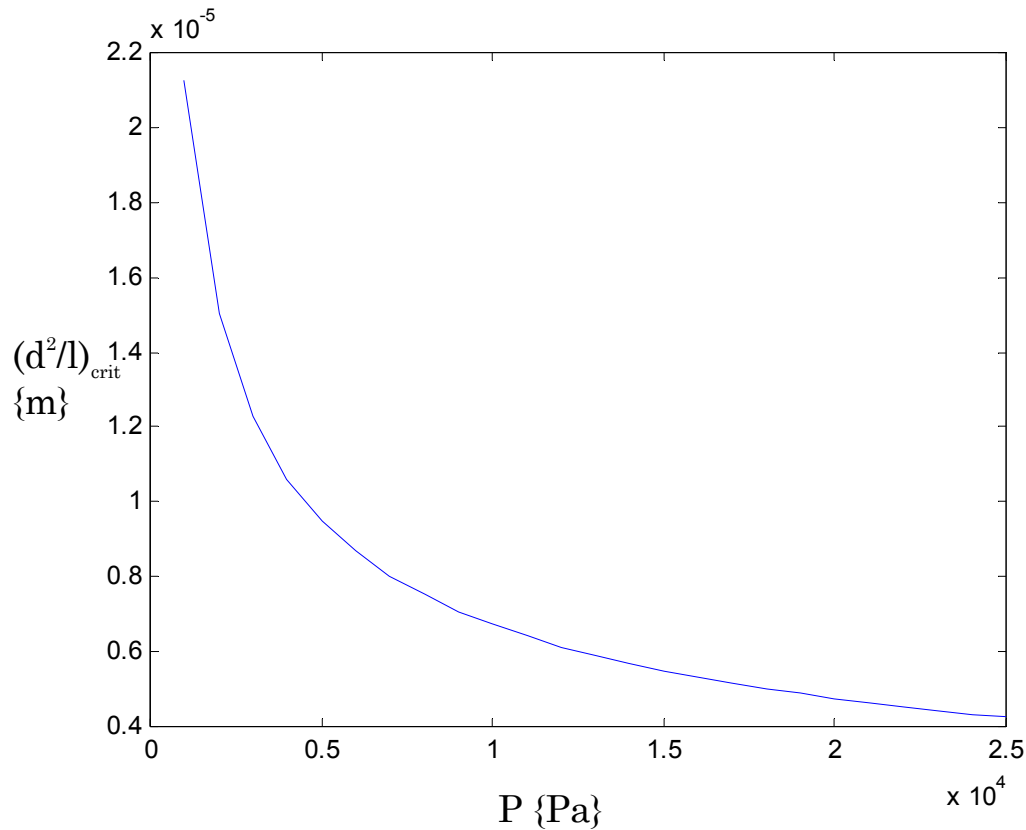


Figure 10.1 Graph showing the critical geometrical channel/orifice conditions for nitrogen flow rate calculations. At conditions below the curve, a calculation not taking into account viscosity within the channel is not accurate.

This graph shows that the Bernoulli's or isentropic flow models overestimate flow rates at low pressures for non-ideal nozzles. For the gas valve, if the average diameter of the orifice channel measures 20 μm , at a differential pressure of 0.25 bar, the critical channel length is 94 μm . Therefore, the first and second valve versions can be simplified to nozzles. The third valve having a 300 μm length conical channel ending on a 10 μm diameter orifice, limits the flow more than a nozzle at low pressures, due to fluid viscosity.

This calculation of the frontier between two calculation methods gives an estimate of the validity of the flow model used for the valve. Some aspects, such as viscosity in the area close to the cross-sectional change, slip flow and turbulence at low Reynold's number were neglected. Only FEM simulations can give accurate calculated flow rates valid over the entire pressure range.

A.2. Liquid

For liquids whose density remains constant due tot their incompressibility, the validation of Bernouilli's equation is not dependent on pressure.

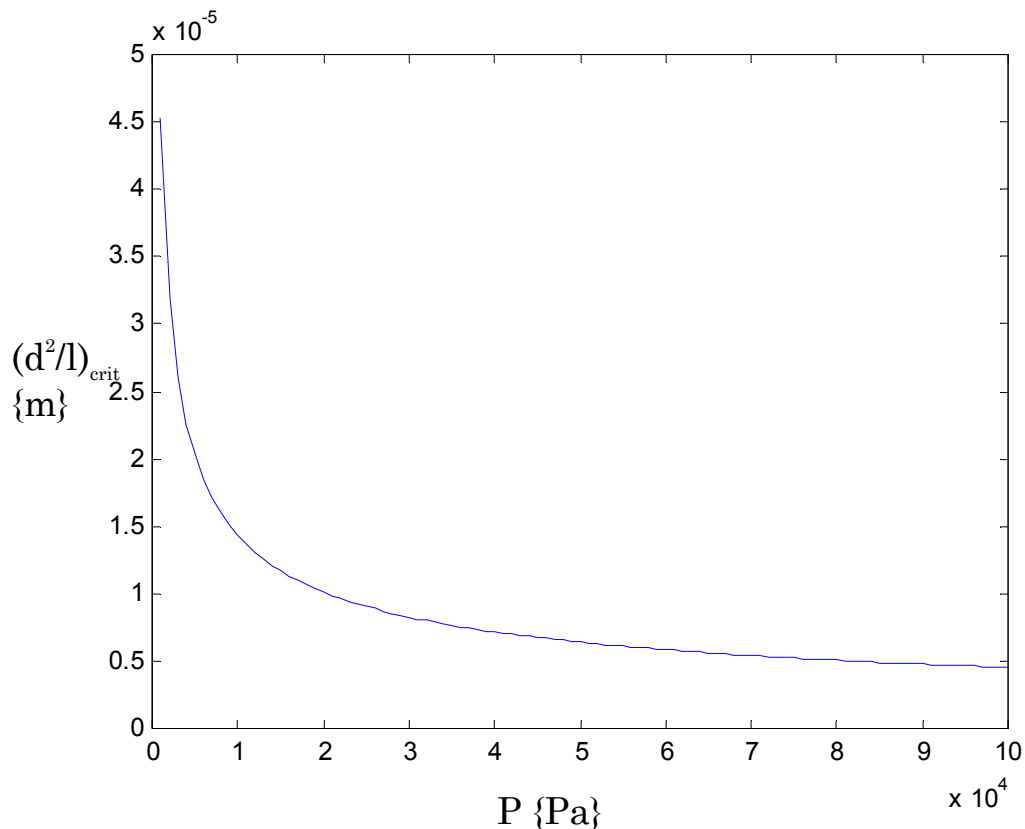


Figure 10.2 Graph showing the critical geometrical channel/orifice conditions for water flow rate calculations. At conditions below the curve, a calculation not taking into account viscosity within the channel is not accurate.

For the liquid valve, if the average diameter of the orifice channel measures 0.5 mm, at a differential pressure of 0.25 bar, the critical channel length is 28 mm for water. Therefore, the

liquid valve fabricated, having a channel length shorter than 5 mm behaves like a nozzle.

A.3. References

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