SOME SELECTED RESEARCH ITEMS OF THE MICROMECHANICS DEPARTMENT AT MESA

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I. INTRODUCTION

Since the beginning of the eightieth there are activities in micromechanics and silicon micromachining at the University of Twente. With the foundation of the MESA-Research Institute at this university in 1991 the micromechanics department joined as one nine research groups this institute. MESA provides optimal circumstances to conduct research in micro systems technology because of the interdisciplinary character of these activities: In MESA, research is carried out on integrated circuits technology and design, information storage technology, optical sensors and integrated optics, scanning microscopy, chemical sensors and micro analytical chemistry, biosensors and micromechanics. MESA has a clean room facility of ca. 1000 m².

In the micromechanical transducers group research is conducted on device physics, system- and device simulation and fabrication technology of miniature mechanical devices made by use of micromachining (photolithography, wet and dry, isotropic and anisotropic etching, wafer bonding and thin film deposition). Silicon and thin film materials form the basic materials used.

The general interest lies on one hand in mechanical sensors: force, pressure, impact and fluid flow, and on the other in actuators and passive structures.

In the application area special emphasis lies in three fields:

(i) Mechanical sensors, in particular resonating sensors, in which a mechanical structure, usually a micro bridge, is forced in vibration and the resonant frequency is a function of the physical quantities that are to be measured. Resonating micro bridges can be used as strain gauges, replacing piezoresistive strain gauges.

(ii) Components for liquid handling systems with the aim of fabrication of integrated liquid handling systems. In the past, channels, valves, sensors for liquid and gas flow, filters, micropumps, micromixers and micro dosage systems have been developed.

(iii) Actuators, mainly for microrobot purposes.

We have developed structures for resonator sensors able to measure force, pressure and fluid flow. Realisation procedures range from bulk micromachining using anisotropic etching of single crystalline silicon to surface micromachining, using deposition of stress-controlled poly silicon and borosilicate thin films, the latter of which is "sacrificed". This technology enables us to machine vacuum encapsulated resonating micro bridges that can replace the conventional piezoresistive strain gauges with the advantage of much higher sensitivity and precision.
The research resulted in a complete control over both, technology and theory, of the excitation and detection of vibrations of microstructures, including among others electrostatic, piezoelectric, thermal and optical means. We are in a stage of understanding resonant sensors that we are able to design sensor systems for e.g. high precision load cells and (differential) pressure sensors, resonant sensors with optical excitation and detection for application e.g. in hazardous environments, and we know in ins and outs of these systems, including their limits. Resonant silicon sensors will be economically interesting in special situations, e.g. when electrical signals must be avoided, or when high precision instruments are required. In other circumstances we advice to use more conventional principles.

An example of a prototypes of a conventional micro sensor, using piezoresistive strain gages, is a force sensor that has been developed for the measurement of tension gradients in videotapes.

The bibliography on resonant sensors is given in [106 - 119]

The work on pumps and on actuators will be discussed in greater detail in this paper. The application-driven work is supported by research on technology, processes of dry and wet etching, materials science and modelling/simulation of devices and systems. Here we report on some results for reactive ion etching (RIE)

This paper is divided in three major sections: liquid handling, actuators and reactive ion etching.

II. LIQUID HANDLING DEVICES

Based on recent development of components for micro liquid handling (c.f. Gravesen et al [1] and van de Pol et al [2,3] for recent overviews) there is a growing interest in the research on micro-liquid handling systems (MLHS) such as chemical analysis systems, e.g. fluid injection analysis (FIA) see e.g. [4 -11] and electrophoreses systems [12 - 14], micro dosage systems [15 - 16], systems for counting red blood cells [17] and others [18 -22]. The basic components in liquid handling systems are tubes (in MLHS: channels [23,24], passive [25 - 27] and active [28 - 44] valves, actuators for active valves and pumps [45 - 49], flow sensors, e.g. [50 - 70], reaction chambers, filters [71,72] and mixers [73]. Pumps (mainly membrane pumps [27, 48, 74 - 81], but other principles have been suggested as well [29, 82 -86]), passive and active valves are the first systems that have been realised. They comprise several of these elements (actuators, channels, valves). Examples of all these components have been demonstrated in the past few years, and for the realisation of MLHS more complex than pumps all components exit. Here we describe work performed in the micromechanics group at MESA on valves, micropumps, mixers, detectors and dosage systems and we discuss a few possibilities to integrate these and other components to more complex systems.

II.1. HISTORY OF MICROPUMPS IN TWENTE

The work on pumps has some tradition in the micromechanics group at the University of Twente. The research was initiated by Jan Smits, who now is with the University of Boston, in the beginning of the eighties. His work resulted in a peristaltic pump comprising three active valves in line, see fig. 1. Each valve consisted of a camber with inlet and outlet that are both covered by a flexible membrane. By deflecting the membrane an underpressure can be generated.

The actuation was piezoelectric: the membranes were made from two piezoelectric discs. If a potential difference is applied over this pair, one of the discs expand and the other one shrinks with the result that the membrane bends. Smits' work was published not before
Transducers '89 [29], two years after the follow up of this work was presented at Eurosensors 87 by van Lintel, van de Pol and Bouwstra.

Fig. 1. Schematic of Smits' design of a peristaltic pump [29]. The pump cycle is as follows: Membrane (1) is bent, liquid flows into the opening. This state is shown. Next, membrane (2) is bent, again liquid flows into this opening. Then membrane (1) is released, and the valve closes. During this step the liquid under membrane (1) is pushed back. In the next step membrane (3) is activated, liquid flows from the right reservoir into the opening. Now membrane (2) is released, and after this membrane (3). Note that this pump pumps equally in both directions; it is fully symmetric.

Smits' pump was able to pump 100 μl/min with out pressure difference between inlet and outlet, when a voltage of 80 V was connected to the actuator. The pumping rate dropped to zero at a pressure difference of 60 cm water. The pump rate was maximal at an actuation frequency of 15 Hz, and the pump ceased to pump at a frequency above 50 Hz.

The major problem with this design was in our eyes that the pump was leaky: A small pressure difference across the pump resulted in a flow through it, if the actuators were not activated. This is a problem which several pump principles suffer from: Electrohydrodynamic pumps [84 - 86] and pumps that have diffuser nozzles [83]. Without taking additional measures these pumps are obviously not suitable for applications that prohibit any backflow of the pumped liquid such as systems for drug delivery and chemical analysis.

Having these applications in mind, we looked for a principle which makes use of check valves. This investigation led to the design described in van Lintel's paper [27], see fig. 2. Based on this design a pump for medicine delivery is now being commercialised by DEBIOTECH. Van Lintel used a piezo disc glued to a glass membrane; under a voltage difference the piezo disc changes its lateral dimension which results in a bending moment in the bimorph. The original prototype was able to produce a maximum pressure of 100 cm water and had a maximum yield of ca. 10 μl/min at 1 Hz block wave actuation.

Mainly from aesthetic reasons van de Pol suggested an alternative for the piezoelectric actuation which he called thermopneumatic actuation. Piezoelectric discs must be glued to the membrane. An alternative was to grow a thin film of ZnO on the pump membrane, but analysis showed [87] that thin film ZnO is not suitable for pump actuation because the film cannot be grown thick enough. In order to produce the energy required to produce a sufficiently large volume stroke at a given minimum pressure, the piezoelectric material has to have a minimal volume, which is much to large to produce by thin film growth. The principle has been described earlier by Sdeblick at Transducers '87 [28]. By heating a gas or a liquid under its vapour pressure the pressure is increased and a membrane can be deflected. Such an actuator can be realised completely by using silicon technology - in contrast of gluing a piezoelectric disc to a membrane. This pump, when using ambient air as expanding medium in the actuator, was able to produce a maximum pressure of 40 cm water and had a maximum yield of ca. 30 μl/min. Maximum yield occurred at 1 Hz block wave actuation. When using a liquid - vapour equilibrium system the maximum pressure can be much larger ([88], see below).
Fig. 2. Schematic of van Lintels membrane pump [27]. In the centre the pump chamber and the pump membrane is sketched. In the membrane bends down, the right valve opens and the left valve is forced to close; this state is shown. If the membrane is released or bends upwards, the left valve opens. Note that this pump is asymmetric.

Parallel to the development of micropumps we started work on liquid flow sensors realising the great demand for these sensors. From technological point of view a simple scheme for flow sensing is the thermal one, using a central heater and thermal sensors up- and down stream, see e.g. [50,53,55,69]. We soon found that the process we used to fabricate the sensor structure - a Si₃N₄ grid-shaped carrier for the heating elements and temperature sensitive resistors - was suitable also for the fabrication of the resistor that can be used to heat the gas in the pump actuator. Our experience with anodic bonding and machining of Pyrex wafers that stemmed (see below IV.1) from our work on pumps was exploited also for the flow sensor, therefore the integration of the thermopneumatic pump and the flow sensor was at hand. Once having demonstrated the feasibility of integrating several functions of a MLHS on one wafer the progress is obvious: tedious and expensive assembly of micro components can be avoided. The resulting micro dosage system will be described in detail in this paper. It has been described first at MEMS '93 in Ft. Lauderdale, USA [15]. At the same workshop we described a micromixer [73]. This is a key component for MLHS for chemical analysis. In practically all systems of this type, the sample must be diluted in a carrier liquid and/or mixed with one or more reagents. The chemical reaction leads to a compound that has physical properties that make their detection easy. In established FIA-systems the product often absorbs light. Mixing in microsystems is by no means trivial since the Reynolds numbers are of order 1, and no turbulence or vortices can be induced by some means. For mixing only diffusion processes remain which are very slow. This will be discussed in more detail below. Typical flow velocities are of order of 1 cm/s, linear extensions of 1 - 10 cm, so mixing must be completed within one second or faster. The basic idea for sufficiently fast mixing in microsystems was proposed in '92 by R. Miyake from Hitachi, who was then on leave in our group. He proposed to inject the sample liquid into the reagent (e.g. for a FIA) through closely spaced nozzles. Thereby the diffusion length is dramatically decreased, and mixing times of one second and less is possible. Here, the mixer is also described in detail.

Forthcoming work in our group will focus on alternative designs of pumps using cheaper materials than silicon, design of micro pore filters [72], on further integration of components resulting in micro-FIA-systems, modelling of components and systems and development of design strategies for micro liquid handling systems and components.

For the latter modelling of components and systems is of course indispensable. We have good experience with modelling using the language of Bond Graphs, a graphical language especially suitable for modelling lumped elements systems that have degrees of freedom in several energy domains. Unfortunately, the day to day this language is confined to systems control people, and neither known nor appreciated by our colleagues in micro systems technology. In order to take advantage of this language one has to be skilled in using it, but once one has taken this barrier, a graphical model of complex systems is readily composed and implemented in existing software (TUTSIM, CAMAS). The models we have developed are, and will be, described elsewhere [87,89 - 91].
II.2. COMPONENTS

II.2.1. Channels
Channels form the basic element for all micro liquid handling systems. This applies trivially for the interconnections of components, but also for components themselves: A valve is a channel that opens and closes, mixers are assemblies of channels (or, traditionally, just a long channel), in flow sensors heat transfer in channels is observed etc. The cases we are to study here the flow of the liquid is at small Reynolds numbers and small entrance length.

Some authors reported on deviations of the flow resistance from Poisseulle's equations \([23,24]\). In a discussion of this problem in Gravesen's review paper \([1]\), it is stated that the results of different papers are in conflict with each other, and no firm conclusion can be drawn. Urbanek does not give the length of the channels he examined in his experiments. Entrance-phenomena could lead to the observed results. We notoriously observed in our own lab that contamination of liquids by particles, the origin of which is quite unclear, occurs very often. These observations were made thanks to fine pore sieves (2.5 \(\mu\)m) designed and realised by van Rijn \([72]\). Without filtering we never obtained reproducible results, and if they reproduced, they were in accordance with theoretical expectations. Therefore we take these reports with some care.

A notorious problem of micromachined channels stems from the use of anisotropic etching which results in sharp corners. These are potentially regions of very slow streaming, and therefore points where precipitation and trapping of gas bubbles is likely. Both cause serious troubles in MLHS. On the long run therefore it is desirable to develop alternative technologies to machine smooth channels, such as isotropic wet or dry etching. A promising micromachining technology is anodic HF etching \([94 - 96]\) since this method allows precise and reproducible geometry's: etching can be stopped just by switching off the power.

Trapped bubbles in the system greatly change the flow resistance. From our experience it is important to be able to judge if there are bubble in the system and to find them. This was most easily accomplished if all channels are visible. This leads to an important design rule: MLHS should be fabricated such that the ducts are visible. The best was to sandwich a micromachined silicon wafer between two glass wafers.

II.2.2. Valves
A critical design issue is the valve. We can distinguish active and passive valves. A passive valve is a flow dependent obstruction. In active valves an actuator and the valve function are combined in some way; an example for an active valve is seen in fig. 1, in which the membrane itself has two functions: actuation and the moving part to close an orifice. Many alternative concepts for active microvalves are described in the literature \([28 - 44]\).

![Fig. 3 Designs of passive valves. Right: Tiren's design [25] using a cantilever beam over an orifice, left: van Lintel's design [27] using a membrane with a valve seat](image-url)

Several designs were proposed for passive micro valves. Van Lintel \([27]\) used valves sketched in fig 3. Here a circular ring attached to the centre of a flexible membrane is pressed against a flat bottom. Either in the centre of the membrane
surrounded by the ring there is an orifice, or there is an orifice in the bottom plate just beneath the centre of the membrane. The second opening can be realised in the structure containing the membrane or in the bottom plate. In all valve designs we used either a Pyrex wafer as the bottom plate or a silicon wafer covered by a thin sputtered Pyrex film. The surface of the ring was thermal oxide, the ring itself and the membrane were silicon etched from one wafer anisotropically in KOH. The level of the oxide layer exceeds the level of the wafer so that the valve is under a pretension. The silicon wafer and the bottom plate are bonded together using anodic bonding. The oxide ring prevents bonding, and is therefore essential for the production.

Test showed that these valves were tight: We could observe flow of a liquid against the opening of the valve only in those cases that the valves were contaminated by dust particles.

Valves designed by Tirén et al [25] are used in pumps presented by Zengele et al [76,79,20]. In these valve cantilever beams are used in place of membranes with a ring, see fig. 3. The design of these valves is such that the flow resistance is of comparable magnitude as our valves. Also the good properties with respect to leakage are comparable. Similar to van Lintle's valves, the volume flow through the valve is proportional to \((\Delta p)^4\) [25]. A disadvantage of Tirén's valves might be that realisation of a pretension is not so straightforward.

Finally we have to mention the valves Esashi and his co-workers were using in their pumps [18,19]. Their valves are constructed from LPCVD poly silicon and consist of a boss held by four bridges that are anchored at the substrate. As far as we can judge from their published results, these valves were leaky. We cannot judge however if the valve leakage is due to contamination, the material (poly silicon) or the construction.

II.2.3. Flow Sensors

Most micro flow sensors described so far work using the thermal domain: Convection cools a heater and disturbs the temperature distribution close to the heater. An interesting alternative measuring the drag force of the streaming liquid on a flexible element has been described by Gass et al [52]. For our purpose - development of flow sensors that can be easily integrated - a simple technology for the fabrication has the highest priority.

Fig. 4 illustrates the principle of the flow sensor we are using. Similar designs have been described in references [50,55,69]. Three resistors are located in the middle of a flow channel. Heat is dissipated in the middle resistor (H). The resulting temperature distribution is sensed with two temperature sensitive resistors \(T_1,T_2\) located symmetrically up- and downstream with respect to the heater. The heater as well as the sensing resistors are placed on supporting beams which cross the flow channel in the \(x-y\) plane.

An example of a micromachined flow sensor is shown below in fig. 11.

At zero flow rate, no convection exists in the flow channel, and the heat generated by the heater will be transferred only by the axial and tangential diffusion through the fluid and by the conduction through the heater and sensor support to the flow channel walls.

The model for the flow sensor is extensively described elsewhere [53]. The measurement results shown in fig. 5 are qualitatively similar to the model results shown in fig. 6. For low flow-velocities the output signal is linear with \(v\) and at a certain 'turn-over' flow velocity, the output signal reaches a maximum. For a reliable flow signal the sensor has to be used for flow velocities below \(v_f\). In the dosing system, this counts for the momentary flow-velocity.
The liquid flow is measured by its influence on the temperature distribution in the sensor resulting from heat generation in the heater $H$. $T_1$ (upstream) and $T_2$ (downstream) are temperature sensors.

II.2.4. Micro Mixers

Mixing in microsystems is by no means trivial. As we have seen, the Reynolds number is small, and mixing can occur only via diffusion, which is a slow process. Diffusion times increase proportional to the square of the distance $\tau_d = L^2/2Dd$

where $D$ is the diffusion constant of the molecules in the liquid, $d$ represents the dimension of the problem, e.g., $d = 2$ for diffusion in two dimensions. Typically, $D$ is of the order of $10^{-5}$ cm$^2$/s, leading to diffusion times of 1000 s (17 min!) if $L = 1$ mm, which is a typical measure for the width of a channel.

Hence we looked for a micromixer that mixes fast small volumes (of order 1 μl) and which is easily realised by silicon micromachining, in order to facilitate integration in complex systems.

The basic idea of the micro mixer is to decrease the diffusion length required for mixing. A possible geometry is illustrated in fig. 6. The mixer has an area for mixing which is very flat and thin with many micro-nozzles on the bottom. During operation, first, the mixing area is filled with one liquid, and the other liquid is injected into the area through the many micro-nozzles, making many micro-plumes. These plumes increase the contact surface. The nozzles are positioned very closely in rows, 10 - 100 μm apart, reducing drastically the diffusion time. Thus, effective mixing will be performed without any additional driving.

**Fig. 4.** The liquid flow is measured by its influence on the temperature distribution in the sensor resulting from heat generation in the heater $H$. $T_1$ (upstream) and $T_2$ (downstream) are temperature sensors.

**Fig. 5.** Temperature distribution in the middle of the channel as a function of the position $x$. The heater extends from $x = -L$ to $x = +L$. A) $v = 0$, b) $v = 0.001$ m/s, c) $v = 0.01$ m/s

The basic idea of the micro-mixer

The fabrication process of the mixer is simple and described in the literature [73].

**Fig. 6 Basic idea of the micro-mixer**
Instead of a reagent, a fluorescent dye, uranine; \((\text{C}_{20}\text{H}_{10}\text{O}_{5}\text{Na}_2)\) was used because it is easily visible even at low concentrations [17].

Fig. 8 (a) is a microscopic view of the plumes, with a flow rate of 0.57 \(\mu\)l/s and a total of about 0.5 \(\mu\)l of dye. It shows that the dye diffuses into a homogeneous mixture within a few seconds, after the injection stops (at around 0.9 sec.), as the simulation predicts.

Fig. 14(b) shows the boundary between the mixed liquid and the water, 1.5 minutes after dye injection. The boundary can still be seen clearly. This is because diffusion time is great in this case as discussed earlier. This allows the mixing area to be treated as a micro-reaction cell.

II.2.5. Optical Detectors

When one thinks of integrating functions for MLHS's on one wafer, one should select the simplest ones first. Integration of chemical sensors seems to be more difficult than to integrate just an optical window to observe the colour of e.g. reaction products, from which the concentration of a certain chemical species in the sample can be deduced. Below we describe such a detector system. This type of detector was inspired by work of Verpoorte et al [101]. An example for an alternative is described in ref. [102]

The mixing process can be observed conveniently using absorptiometry. Light from an optical fibre enters the mixing area by reflection from the (111) oriented side wall of the channel, and travels between the bottom and the upper reflector. At the other wall, the light is reflected out of the channel and detected by a photo-detector through a second optical fibre. Rhodamine (\((\text{C}_{28}\text{H}_{30}\text{N}_2\text{O}_3\text{HCl})\)) is used as a dye, and an Argon ion laser (514.5nm) is used for illumination, since the main absorption wavelength of Rhodamine is 520 nm [103]. The intensity of the light passing the mixing area decreases due to the absorption by the dye.

Measurement results

Using this set-up, the time to complete mixing is investigated. Fig. 9 shows the
results of measurements when the injection flow rate is 0.75 µl/s, the total volume injected is 0.5 µl, and the injection stops after 0.67 sec. The output signal continuously decreases after the injection stops, and an equilibrium state is reached 1.2 sec. later. This result is in agreement with the flow visualisation, our estimates and our simulations.

![Graph showing experimental result from absorptiometry](image)

**Fig. 9. Experimental result from absorptiometry**

In conclusion, we may state that the detector described here works properly. It is sensitive, simple, and gives reproducible results. The alignment of the optical fibres with respect to the windows is a little tricky, and this may be a bottleneck for large scale production. In later stages of research one should look for possibilities to integrate optical connectors in the system.

### III.2.6. Micro Pumps

Since for applications of the MLHS we are interested mainly in the medical field and in chemical analysis, we concentrate on pumps that make use of check valves. It has two passive valves and a pump chamber connected to a pump membrane. The pump-actuator is thermo-pneumatic to make large volume strokes and simultaneously large pressure build up possible. In the air-chamber, the air is periodically heated by electrical dissipation in the heater resistor. Due to the varying temperature, the air-pressure in the chamber will periodically deflect the pump membrane, which results in the transport of liquid through the pump.

We briefly review the most important results of the model of the pump. As in earlier work the pump is modelled with the bond-graph method which is very convenient if several physical domains are involved [87, 89 - 91]. The pump is divided into three (energetically coupled) sub-systems: a thermal sub-system, a pneumatic sub-system and a hydraulic sub-system.

The thermal sub-system consists of the heater, its mechanical support and the (ideal) gas in the air-chamber. Both, the gas and the support contribute to the heat conduction to the substrate. The heat capacity of the gas is small compared to that of the heater resistor. The lumped element model of the thermal sub-system consists of heat conducting and heat capacitance elements. With the materials used for the heater and the geometry of the air-chamber, a characteristic time for heating and cooling is calculated: $\tau_{th} = 0.05$ s. This time is an important design issue. We found that it is important to minimise the heat capacity of the carrier of the heating element and to minimise the heat conduction from the carrier to the wafer package. Both is simultaneously provided by using a silicon nitride grid suspended in the centre of the cavity. We found earlier that e.g. evaporating the resistor directly on the pump membrane will not work for a pump for liquid because the good heat contact from the membrane to the liquid prevents any heat transport to the gas [46]. This is different for gas pumps, as demonstrated by [81].

The pneumatic sub-system consists of the gas in the air-chamber in combination with the exchange channel with the surroundings and the (movable) pump membrane. The sub-system can be described by a 'pneumatic' characteristic time, which is determined by the volume of the air-chamber and the flow resistance of the exchange channel. For the pump described here, $\tau_p = 30$ s.

The hydraulic sub-system consists of the liquid (with its density and dynamic viscosity) in combination with the pump-chamber, the valves, the liquid channels, the flow sensor and the in- and outlet tubes to the dosing system. Since the valves have non-linear characteristics (see eqs. (9) and (10)), the hydraulic system cannot be described by a single 'hydrolic' relaxation
time. However simulations for the dosing system described here, show a hydraulic relaxation time \( \tau_H \) in the order of 1 s. This time sets the maximum speed with which the pump can be driven.

The pump functions properly only if \( \tau_H < \tau_P \) since otherwise the expanding gas would flow through the exchange channel without deflecting the membrane. \( \tau_P \) sets the minimum speed of the pump. The characteristic time for the actuation, i.e. the time needed to cool or heat the air-chamber, must be larger or equal to the hydraulic time in order not to limit the maximum speed of the pump. This is the case in our design. The dynamics of the pump are limited by the hydraulics and not by the thermal actuation.

II.3. DOSING SYSTEMS

For a correct and reliable dosing of a certain volume of liquid it is necessary that the yield of a pump is independent of the pressure difference over that pump. Pumps are not suitable to directly perform the dose-function of their own. In combination with a flow sensor, and an electronic circuit it is relatively easy to control the yield. The signal of the flow sensor has to be integrated and the speed and accuracy of the sensor have to be sufficient in order to lead to an accurate control of the dose.

Fig. 10. Cross section of dosing system

For the realisation of the dosage system we refer to the literature. The result is shown in the cross-section given in fig. 10.

The pump can be filled straightforward by injecting IPA in the inlet using a syringe. Filling the system with water is accomplished by first filling it with IPA and then switching over to water.

First the flow sensors are characterised. This is done by forcing a flow in the forward direction through the flow sensor by means of a syringe pump. The flow sensors operate in the accurate 'constant power' mode. The temperature measuring resistors \( R_u \) and \( R_d \) are placed in a bridge configuration. A temperature difference between the upstream and downstream sensor is linearly converted to a bridge-output voltage [53]. The measured sensor sensitivities are in the order of 25 \( \mu \)V/K for a heater dissipation of 10 mW using IPA as liquid. The bridge output voltage as function of the liquid flow is given in fig. 13.

The spontaneous flow behaviour of the dosing system is measured with help of the experimental set-up given in fig. 14. If the input pressure is higher than twice the valve 'pre-pressure', liquid will flow through the dosing system. When the syringe pump is programmed at a certain flow, both capillary tubes indicate the pressure difference over the dosing system.
Fig. 12. Optical photograph of the Si-wafer (top face) with four dosing systems next to each other. The glass wafers are not applied yet.

Fig. 13. Flow-sensor output with water as liquid. With a flow channel of 1000 \( \mu \)m \( \times \) 500 \( \mu \)m, a flow velocity \( v = 20 \) mm/s corresponds to a volume flow of 600 \( \mu \)l/min. For water: \( \kappa = 0.6 \) W/mK, \( D = 1.4 \times 10^{-7} \) m\(^2\)/s [53].

The liquid pump is characterised with the help of a square wave generator for the pump-heater, and a high speed digital voltage meter for measuring the flow sensor output. The results on the dynamic pump characteristics of the system are given in fig. 15. For a first characterisation of the pump, the pumping speed is measured with the help of a capillary tube at zero pressure difference between the inlet and the outlet of the dosing system. Results are given in fig. 16.

Fig. 14. Experimental set-up for measuring the spontaneous flow and the pre-pressure of the normally closed valves.

Fig. 15. Actuator-signal, measured liquid flow and the calculated (integration of flow) liquid dose as a function of time. The flow-sensor output is measured every 50 ms.

- static properties of the pump

The passive valves are normally closed. The built-in pressure of the valves is proportional to the bending stiffness of the valve membranes.

For a dosing system described here, with 30 \( \mu \)m thick valve membranes we measured spontaneous flow above 0.01 atm. Below this pressure there was no flow. For a thickness of about 40 \( \mu \)m we measured spontaneous flow above 0.03
atm. These values are in agreement with the expected ones.

*dynamic properties of the pump*

The 'thermal' relaxation time is measured using the temperature dependence of the pump-heater resistor. We measured $\tau_{th} \approx 0.1$ s. With the pump-heater continuously activated, the 'pneumatic' relaxation is estimated from the pump-membrane displacement as a function of time: $\tau_p = 15$ s. Fig. 15 shows that the pump stroke with an input power of 5 W is about 0.9 µl. From the flow sensor signal we see that the hydraulic relaxation of the dosing system is within 1 s. After a pump cycle some liquid flows back into the pump body (see also [74]).

At low pump frequencies the hydraulic system relaxes within one cycle, so the pump stroke is constant and the pump yield is linear with the pump frequency (see fig. 17). At higher frequencies there is not enough time for hydraulic relaxation and the yield decreases. At a fixed pump frequency, the pump speed is almost linear with the heating power $P$ (fig. 17). The offset in the curve is due to the back-flow or 'dead' volume of the pump. At high powers the curve is saturates.

The pressure built-up of the pump is limited by the maximum pressure built-up in the actuator chamber. With a liquid-vapour equilibrium the pressure can be increased dramatically, with comparable temperature elevations. This was experimentally verified with a pump with a broken pump membrane. With sufficient input power the alcohol that now penetrates the actuation chamber boils, giving rise to an actuation pressure large enough to pump against 1/3 atm.

**II.4. OUTLOOK FOR FURTHER INTEGRATION**

Fig. 35 gives an art impression of a Fluid Injection Analysis system in the possibly most simple case. A reagent and a sample are brought together in well defined amounts by using two dosage systems as described above. These liquid are mixed in a micromixer and transferred to an optical detector unit. In view of the simple processes to realise the mixer the integration with dosage systems should be straightforward, and we hope that we shall be able to publish results of our current work on FIA's soon [104].
The system shown in Fig. 18 is not complete yet. First of all, the sample usually needs some pre-treatment, at least it must be filtered since small particles jam the valves. Further, precipitation would be fatal for the system. Means of cleaning (by rinsing) must be developed, and perhaps integrated. This would mean that one needs more functions on the wafer. At least, the sample carrying part should have a connection to enable rinsing; this part could consist merely of a normally closed valve or, preferentially an active valve.

We have described a concept of horizontal integration. The co-operation between the Neuchatel micromechanics group directed by N.F. de Rooij and Ciba Geigi led to a concept of vertical integration [11], where the components were realised in different wafers and stacked in order to assemble a system. The wafers were not bonded but mechanically fixed.

A third concept would be to start from a backplate that contains channels for the connection of components [105], similar to a printed circuit board. It is necessary then to develop standards for the inlets and outlets of the components. If this concept turns out to be successful it would offer great flexibility to build up MLHS in a simple and economically feasible way.

The horizontal integration certainly is the less flexible way, but, if successful, the cheapest way for large scale production, since there is no need for assembly. The demonstration of the dosage system makes us optimistic.

III. ACTUATORS

Our research on pump actuators has been described in chapter II. Here we concentrate on a new electrostatic actuator that we call an "active joint". The structure we shall describe is capable of delivering large forces (if use can be made of PZT with a dielectric constant of 1000, the force can be of the order of mN) combined with large deflections.

Fig. 19. Schematic of an active joint. The bilayer on top of this structure is attached to the left of the bottom electrode and free to move (by bending) above the electrode. Top: inactivated; bottom: activated.

The idea is based on recent work of Branebjerg and Gravesen[25], which is closely related to work of Sato et al. [32] and Ohnstein et al. [30]. The first description can be found in [120]. The actuation is electrostatic. The structure consists of two capacitor plates one of which or both are bend. A voltage difference between these plates results in a force which bends the plates to each other. This is sketched in fig. 19.

In first order we can describe the actuator in the following way:

We assume that the actuator is partially collapsed to a length $l$. The electrical field is strongest in the volume of this collapsed part. We neglect the remaining field. The electrostatic energy in this volume is given by

$$W_{es} = \frac{1}{2} \varepsilon \varepsilon_0 A V^2$$

where $\varepsilon$ and $\varepsilon_0$ are the relative and absolute dielectric constants, $d$ is the distance between the electrodes, $A$ their area in contact, and $V$ is the voltage between the plates. $A = b'l$ is the area of rectangular overlapping capacitor plates. The work
necessary to bend a cantilever beam is given by
\[ W_{el} = -\frac{E h^3 b l}{24 R^2} \]

Where \( E \) is the effective Young's modulus of the cantilever beam material (which may be a stratified structure), \( h \) its thickness and \( b \) its width. \( R \) is the radius of curvature in the initial state. From the requirement
\[ \frac{\partial W}{\partial l} = 0 \]

with \( W \) the total energy we find the equilibrium value for \( l \). It turns out that \( l \) drops out of this equation, and accordingly there is a critical voltage at which the beam switches from the inactivated state to the activated state. The critical voltage is given by
\[ V_c^2 = \frac{E h^3 d}{12 R^2 e_0 \left( \varepsilon_{\text{gap}} + \varepsilon_{\text{die}} \right)} \]

where we assumed \( b = b' \).

The switching property of this actuator can be circumvented by letting one of the parameters depend on \( l \), e.g. \( b', b, h \) or \( R \). Under these circumstances it is possible to control the position of the tip.

Using micromachining a convenient way to realise such an active joint is the following: After evaporation of a bottom electrode (e.g. Ti/W) and deposition of an insulating layer (160 nm PECVD SiO\(_2\)) a very thin sacrificial layer (50 nm) of fluorocarbon is deposited in a RIE chamber [121, 122]. This layer is patterned (RIE, O\(_2\)) and a thick Al layer (1 \( \mu \)m) is evaporated followed by 50 nm Cr. The tensile stress in the Cr layer is such that released Cr/Al cantilever beams curl upwards with a radius of curvature of approximately 160 \( \mu \)m. The beams are patterned by lift off. Due to the poor adhesion to the fluorocarbon they tend to release spontaneously, however the reproducibility of this procedure is insufficient. The FC layer therefore is removed in an oxygen plasma. Redeposition of FC, in order to refill the gap of 50 nm is straightforward. The procedure results in a structure shown in fig. 20.

If the air gap is taken into account for the critical voltage, we have
\[ V_c^2 = \frac{E h^3 d}{12 R^2 e_0 \left( \varepsilon_{\text{gap}} + \varepsilon_{\text{die}} \right)} \]

**Fig. 20. Active joints as produced here.**

Top before sacrificial layer etching of the fluorocarbon layer, bottom after removal of the FC layer. The beam spontaneously bends up due to the stress gradient in the bimorph.

The subscripts gap and die refer to the air gap and the dielectric film, respectively.

Young's modulus of Al is \( 7 \times 10^{10} \) Pa, and \( \varepsilon_{\text{SiO}_2} = 3.8 \). This leads to a critical voltage of 52 V, while we observed 45 V, in satisfactory agreement with the model. However, there is a trend of larger \( V_c \) for long cantilever beams.

We are now in a stage of characterising the actuators with respect of dynamics and the force these actuators actually can deliver. A few examples of fabricated actuators are shown in fig. 21.

Possible applications of the actuators can be found in micro positioning, and arms and legs of micro robots.
The invention of silicon surface micromachining was very promising with respect to microrobotics. Surface micromachining made possible the design and fabrication of microstructures and micro devices of much greater variety and with much greater freedom than the more traditional bulk micromachining. The latter relies on anisotropic wet chemical etching of silicon (and quartz). Possible shapes obtained using this technology are governed by the slowly etching planes (in silicon: \{111\} and in certain cases \{001\} and \{110\}). With the help of surface micromachining structures could be made such as gear trains, cranks, tweezers, x-y-stages and finally linear and rotating electrostatic and magnetic motors. In interviews and outside scientific papers researchers began to envision microrobots sent inside human bodies for diagnosis and repair.

However it soon became clear that the structures are too thin for use in systems that are of interest today. Two alternative approaches to resolve the problem - realisation of thick microstructures with great design freedom - are being carried out: The LIGA technique, conventional lithography and dry etching.

One of the basic steps in the LIGA technique is the lithographic step. Visible or ultraviolet light has a wavelength too large to allow precise pattern transfer into a thick photoresist layer due to diffraction. In the LIGA technique use is made of X-rays with a wavelength close to 1 Å. Structure heights of 1 cm have been demonstrated with aspect ratios of (in ideal cases) much better that 1:1000 and a surface roughness of the side walls of a few 10's of a μm.

Once the structure is defined in photoresist, replicas can be fabricated by electroplating and moulding.

The first LIGA-step is very expensive. Perhaps the LIGA technology will be restricted therefore to either very large scale fabrication or to special systems and devices that cannot be fabricated by an alternative technology. These are devices in which the combination of large structural height, smooth sidewalls, large aspect ratios and precise definition is required. We can imagine that for a large part of the potential market volume for microsystems the requirements are less stringent. This
opens the way for alternatives for LIGA, alternatives that are sometimes called "poor men LIGA", because the expensive X-ray lithographic step is circumvented. The two technologies to do this are conventional photolithography of thick (30 -50 μm) photoresist films, with poor of sidewalls roughness, and poor aspect ratio, and dry etching, with reasonable sidewalls roughness aspect ratio. The latter has been demonstrated e.g. by Noel McDonnald and his co-workers using chlorine and bromine gases and by Esashi and his co-workers using fluorine gases at cryogenic temperatures combined with magnet fields (to increase the plasma density).

Here we demonstrate the possibility of using fluorine gases that are considerably less toxic than chlorine and bromine at room temperature in a conventional RIE apparatus, to dry etch silicon with aspect ratios up to 10 (and possibly more), sidewalls roughness less than 100 nm, with etch rates of more than 1 μm/min.

Furthermore, we come up with a method to find easily and quickly the conditions for anisotropic etching with vertical side walls, which we call the "black silicon method".

III.1. Experimental
We used 3" <100> oriented p-silicon wafers and a RIE systems from Plasmafab. Although we tried several mask materials we found a sufficiently large selectivity of the silicon etch rate to a silicon dioxide etchrate, which is then a suitable mask material. On other materials we shall report elsewhere [123].

III.2. Results
There are basically two modes of dry etching that result in anisotropy. Generally the anisotropy is due to an anisotropy of the medium that etches, in contrary to anisotropic wet chemical etching, where the anisotropy stems form the material being etched (monocrystals). The anisotropy in reactive ion etching (RIE) is due to the ions that are accelerated in the dark space between plasma and the substrate. The etch result finally is a complex interplay of the impinging ions and uncharged species (the important ones are radicals which are produced by collisions between accelerated electrons and molecules in the plasma) that diffuse to the substrate. Very critical is the energy of the impinging ions.

If this energy is too large, everything is etched by a physical process (sputtering). There are mainly two annoying consequences: First the selectivity with respect to the mask material becomes poor and the height of the structures that can be etched is controlled by the selectivity times the thickness of the mask. Secondly, mask material is sputtered and redeposited, a process that leads to in micro masking. Under certain conditions, the result is so called micro-grass, see fig. 22.

If the ion energy is too small, the etch profile tends to become isotropic because the chemical processes dominate the etch process.

Fig. 22. Micrograss giving rise to black silicon

The ion energy can be controlled by the pressure in the chamber. It is essentially the mean free path that controls the ion energy, therefore the gases in the chamber and their concentration in a mixture play a role. Since concentration and its gradients at the substrate are controlled also by hydrodynamics of the streaming gases, conditions for a particular etch profile do not reproduce easily when transferring the recipe to a different apparatus.

We carried out experiments in a mixture of SF₆, CHF₃ and O₂. The parameter space in which we looked for conditions of
vertical walls consists of the flow of these gases, the total pressure and the plasma power. We found that in a surface of constant SF$_6$ - flow, pressure and power, which is then a plane spanned by CHF$_3$ and O$_2$ - flow, there is a line along which we get vertical walls. This line extrapolates to a small O$_2$ - flow and a very small CHF$_3$ - flow. It appears that this line exists in all those planes, i.e. one should be able to find this line irrespective of the SF$_6$ flow, pressure and power (in the limits that are covered by our experiments).

The interesting point is that this line is rather easily found. On the O$_2$ side of the line the profile is positively tapered, on the other side it is negatively tapered. Micro masking will have no effect on this side since micro masks will be readily underetched. On the contrary on the O$_2$ side: micromasking will result in grass, see fig. 22. Grass is immediately recognised because it results in black silicon. Thus: If one sees black silicon one is very close to the optimum conditions: add a little CHF$_3$ and one will get vertical walls.

in fig.'s 23 - 25 we give a few example of structures we made.

The mechanism of anisotropic etching is probably the result of a passivation of the sidewalls which are not hit by ions, and which is etched very slowly therefore. More details will be given elsewhere [123].

Fig. 23. RIE vertical walls

Fig. 24. RIE etched released x-y stage driven by comb drive actuators

Fig. 25. A tip of micro tweezers

V. ACKNOWLEDGEMENTS
The work presented in this paper is the result of work of many researchers. On liquid handing the following persons must be mentioned: Frans van de Pol, Theo Lammerink, Ryo Miyake (from Hitachi), Joost van Kuijk and Niel Tas. On actuators: Leif Schmidt (from Uppsala University), Erwin Berenschot, Bob Groenewoud and Marc Beusting. On dry etching Henry Jansen, Rob Lechtenberg, Meit de Boer and Vincent Spiering. For general discussions I want to acknowledge Jan Fluitman, Han Gardeniers, Job Elders, Michiel Hamberg, Edwin Smulders, Bob Haverkort, Gert-Jan Burger, Gui Chenqun, and Dick Ekkelkamp.
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