

Electrolysis-Bubble Actuated Gate Valve

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ABSTRACT

The extreme efficiency of producing bubbles via electrolysis of water has been put to use in a new design for an integrated MEMS microvalve. These valves are designed to be part of larger systems in which pumps, mixers and chemical sensors are fabricated simultaneously into one integrated device. The power consumption, flow characteristics and movement of the valve have been observed. The valve design used in this research requires only $4.3 \mu\text{W}$ for actuation. This is more than 4 orders of magnitude less than the power required by similar thermally-actuated valves demonstrated in previous research. With the current design a ratio of 4.7 has been achieved between open and closed flow resistance. In addition, the partially closed valve has demonstrated an intermediate flow resistance, demonstrating the possibility of proportional control.

INTRODUCTION

The microfluidics group at Berkeley is currently developing a system to deliver drugs such as insulin. The system must have integrated fluid control including valves, pumps and mixers. These later two devices also need valves in order to operate. The valves must be fabricated in the same process as the rest of the device and minimize the number of bonding steps in order to make the complete system cost effective. The valves must use as little power as possible so that the entire system can be powered by a small battery. Since the rate of insulin injection needed after meals is about one twentieth the rate needed at other times the goal is to produce a valve with a ratio of open to closed flow resistance of 1 to 20.

Electrochemically generated bubbles have been used previously as low power micro actuators. Neagu [1] used a copper sulfate electrolyte to produce oxygen gas and deflect a membrane. By passing a current through an aqueous solution, Bohm [2],[3] and Jackel [4] have evolved oxygen and hydrogen at the electrodes. Since these gas bubbles are the same temperature as their surroundings, they do not lose any energy through heat loss to the surroundings. As a result, they use quite a bit less energy than thermal bubbles. Bohm [3] and Jakel [4] have also shown bubbles shrinking when in contact with a catalyst. The electrolysis products have a thermodynamic proclivity to revert to water. They remain as gasses only as long as kinetics prevents their reaction. The presence of platinum catalyzes the reaction and allows the gasses to revert to water. This process allows one to control both the creation and destruction of bubbles making them an effective actuator. The addition of energy to the system in the form of heat, or a spark could increase the reaction rate and decrease the cycle time for the actuator.

Previous work [5] demonstrated a micro valve in which vapor bubbles move a free floating silicon gate across a channel. This thermally-actuated microvalve requires as much as 100 mW for actuation. The vast majority of the power is consumed by heat loss

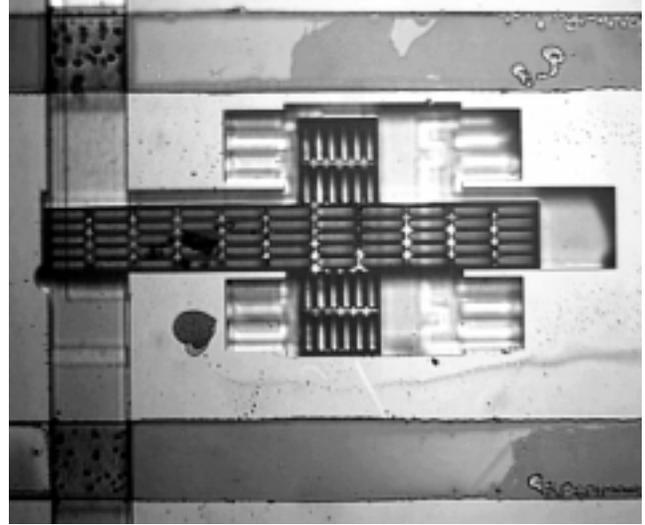


Figure 1. Top view of the gate valve.

to the surroundings. Reducing this power consumption would make the device able to be powered by a small battery for extended periods of time.

Another problem with the valve presented previously was the excessive leakage past the valve. While the gate did move into and block most of the channel and was shown to effect flow, the lack of a proper seal between the valve housing and the cover plate allowed excessive fluid flow past the valve. Providing a better seal would greatly increase the value of the valve.

THEORY

Conventional MEMS electrostatic or thermal actuators are impractical in a fluid environment. Electrostatic actuators will not work in a fluid with mobile ions and thermal actuators require more power in a conductive liquid than they do in air or a vacuum. However, as Lin [6] demonstrated, bubbles can act as large displacement actuators with substantial force. The mixture of hydrogen and oxygen gasses in a $100 \mu\text{m}$ diameter bubble is 617 times less dense than the water from which they are produced. This allows for very large displacements. The pressure inside a bubble is proportional to surface tension σ divided by radius of curvature r .

$$\Delta p = \frac{2\sigma}{r} \quad (1)$$

Thus, at small scales, surface tension forces become much more significant. The force on the moving gate is given by:

$$F = \frac{2\sigma}{r} \times A \quad (2)$$

where A is the cross sectional area of the part of the gate in contact with the actuating bubble.

The power required to create an electrolysis bubble is consumed by three phenomena. First, 4 electrons must be stripped from 2 hydroxide ions and those 4 electrons must be added to 4 hydrogen ions to produce one molecule of oxygen gas and two molecules of hydrogen gas. This requires 1.23 volts or 4.92 eV. Energy is also consumed by the need to move the hydrogen and hydroxide ions to the electrodes where they are being reacted. As the current density is increased the rate at which reactants must be replaced increases and the this portion of the energy consumption increases. In addition, transmitting current through the electrolyte consumes energy. This energy consumption is a function of the resistivity of the electrolytic solution and the geometry of the system.

EXPERIMENTAL DESIGN AND FABRICATION

As in the previous work, each valve used in this experiment consists of a piece of single crystal silicon which can be moved across a channel to create a microscopic gate or pin valve. The basic design of the valve is shown in Figure 1. Bubbles are created at the electrodes on either side of the cross piece which expand until they are able to push the gate. The gate is made of a lattice work of silicon to allow etchants to release it from the substrate.

The electrodes are designed as interdigitated fingers to spread the gasses fairly evenly promoting bubble coalescing and mixing. Increasing the complexity of the array should increase the uniformity of the mixing, however, it also increases the proportion of the bubble generators occupied by the spaces between electrodes. This in turn decreases the total electrode area and as a result, increases current density. As discussed above, increasing current density decreases the efficiency of the electrolysis reaction. A compromise was struck between electrode complexity and current density with four interdigitated electrodes designed to create a well mixed bubble while not compromising total electrode area.

As in the previous work, these valves were fabricated in the Silicon on Epoxy or SOE process. The moving parts of the valve are defined in a thin silicon “device” wafer which is sandwiched between a conventional silicon “handle” wafer and a quartz wafer. The electrodes and leads are fabricated on the handle wafer. The handle wafer is oxidized to insulate the platinum electrodes from the silicon substrate. The platinum is sputtered on with a chrome adhesion layer, over a photoresist lift-off layer. The photoresist is

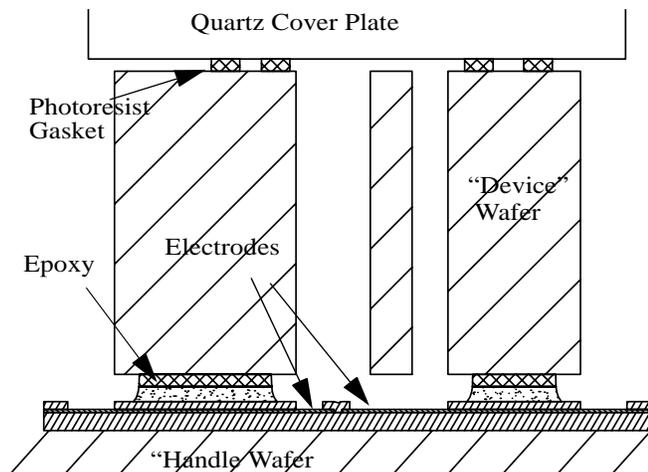


Figure 2. Representative cross section of the SOE process.

removed in acetone to pattern the platinum. The platinum electrodes are covered with a layer of LPCVD silicon dioxide which is patterned to expose only the desired electrode areas. The device wafer is bonded to the handle wafer with Epo-Tek 301 epoxy and the moving pieces are defined by DRIE etching. A sulfuric acid etch releases the valve elements from the epoxy and exposes the electrodes.

Unlike the devices in Papavasiliou [5] the quartz plate over the top of the valves in this paper are actually bonded on with a photoresist gasket to improve sealing. A layer of Olin OCG 825 is spun on a quartz wafer and patterned as a gasket. The quartz and silicon dies are aligned and pressed together. The sandwich is then heated to 115 °C.

Assuming that the sealing of the gasket is perfect, the major source of leakage in the valve will be through the gap between the top of the valve and the cover plate. The thickness of this gap should be the sum of the thickness of the epoxy/photoresist layers removed from underneath the gate and the thickness of the gasket layer. After fabrication, this gap was measured optically to be 12µm. Leakage was estimated by modeling the gap as pousille flow through a rectangular duct. The difference in flow resistance between the open and closed valve was estimated as $4.04 \times 10^{12} Pa \cdot s / m^3$

EXPERIMENTAL RESULTS

Electrolysis bubbles were first created in test channels to characterize their performance. Large polysilicon heaters and platinum electrodes for creating sparks were included in addition to platinum electrode arrays at the bottom of the channels. Bubbles were evolved over the electrodes by passing a current through the water in the channels. The power required to create these bubbles was a function of both the electrode area and generation rate but ranged from around 1 to 10 µwatts.

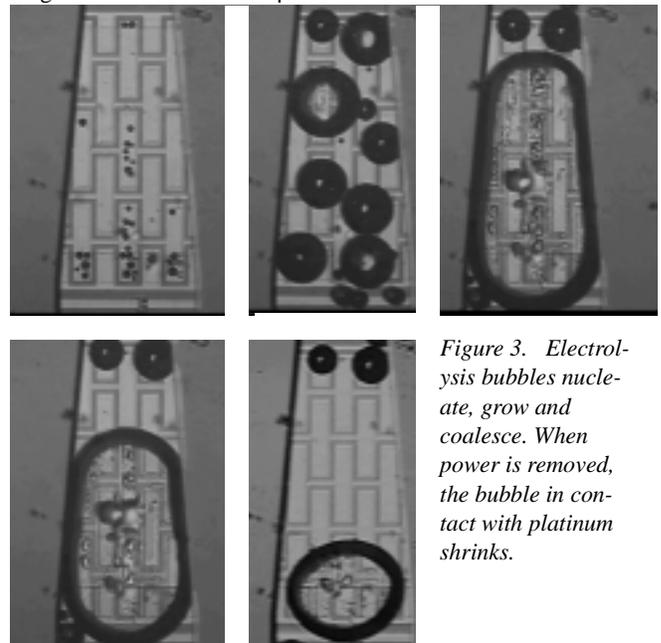


Figure 3. Electrolysis bubbles nucleate, grow and coalesce. When power is removed, the bubble in contact with platinum shrinks.

Three methods were attempted to remove the bubbles. The first method used was merely waiting for the gasses to react and the bubble to shrink. This method although slow, seems effective. Figure 3. shows bubbles generated above electrodes in the test

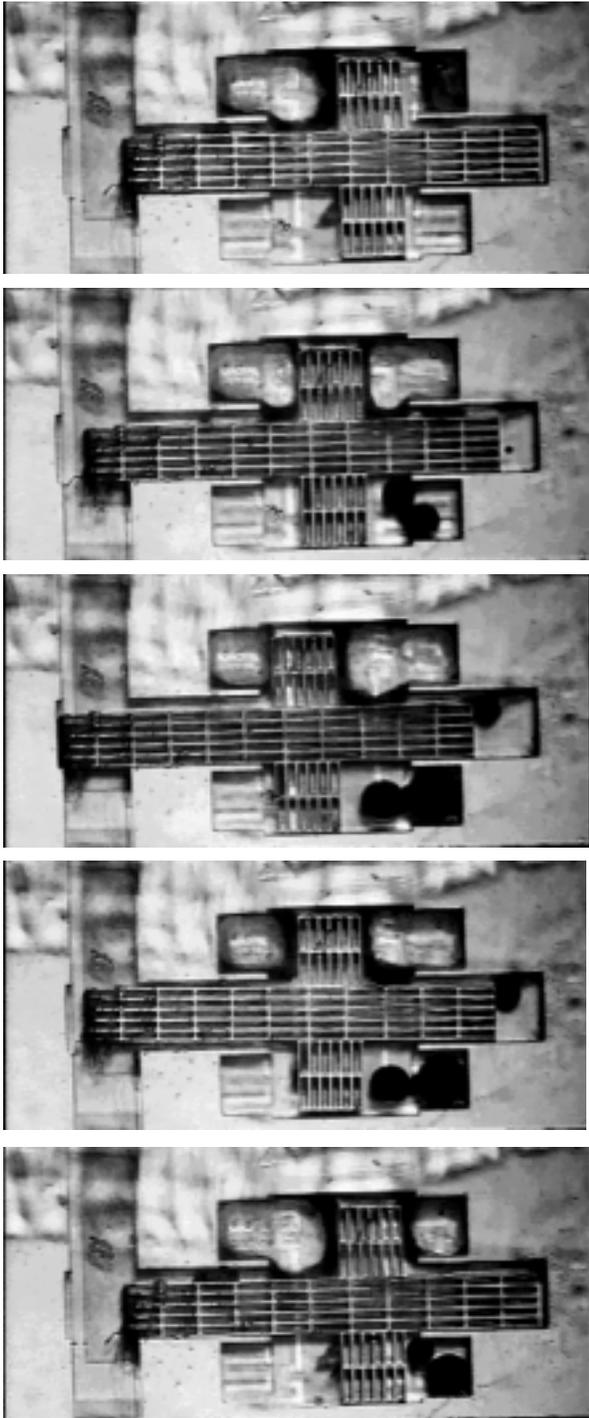


Figure 4. The gate valve in motion. Starting in the open position, bubbles push it closed, then open again

structure. The bubbles grow and coalesce into one larger bubble. After power is removed the bubbles shrink. Notably the small bubbles in the top of the frame shrink much more slowly than the larger bubble. These bubbles are small enough that they do not actually touch the platinum at the bottom of the channels. As a result their shrinkage is due only to dissolution into the fluid. The fact that these bubbles shrink so much more slowly than the bubbles in contact with the platinum indicates that a catalytic reaction is indeed taking place.

Removing the bubbles should proceed more quickly with the addition of energy from either the sparking electrodes or the polysilicon heater. However, the experiments showed that the power input made these methods impractical. In repeated experiments, the bubble shrinks nearly instantaneously when a spark is put across the sparking electrodes, however the bubble invariably does not completely go away. Apparently, the combustion of the bubble which was initiated by the spark is quenched by the rate of heat transfer out of the bubble. Adding heat with the resistor had similarly disappointing results. In addition to increasing the reaction rate, heating the area decreases the solubility of gasses. As a result, heating the area actually swells the bubble with gasses coming out of solution. It was found that cyclic heating can reduce the bubble size but not dramatically faster than simply allowing the reaction to proceed at room temperature.

Creating the actuation bubbles in the test channels requires from 10-50 μJ . In contrast, creating a spark requires around 30 mJ and heating required around 5-20 J. Since both methods require orders of magnitude more power than the initial actuation and do not dramatically improve performance, they were abandoned. It seems that the best way to improve the speed of such actuators may be a method of removing a bubble from the actuation chamber akin to the work done by Evans [8].

Electrolysis bubbles were then incorporated into the microvalve. The movement of the valve is demonstrated in Figure 4. It starts in the fully closed position, moves to the fully open position, and then moves to the fully closed position again. Once the valve has been moved in one direction, the first actuation bubble must be removed in order to move the valve in the opposite direction. Destruction of the bubble through catalytic reaction is a relatively slow process and as a result, one entire cycle, from fully open to fully closed and back to fully open, takes on average around 120 seconds. Carefully regulating the voltage supplied to the two sides of the valve, will allow a controlled bubble on each side of the cross piece to hold the gate in any position allowing proportional control of the flow resistance. Power consumption was measured by regulating voltage and measuring current drawn through the device. Maximum power consumption, while expanding the bubble to push the valve, was measured as 4.3 μW (3.3 V and 1.3 μA). As soon as power is removed, the bubbles begin to shrink due to catalysis of the gasses. Power must be added to maintain the bubbles and hold the valves in place. Electrical input of 2.5 Volts at 0.1 μA has been shown to be sufficient to hold the bubbles in place. The Energizer™ 364, a typical watch battery, contains 106 J [7], approximately enough power to actuate this valve continuously for 285 days or hold the gate in position for 11.2 years.

The flow resistance of the valve was measured by imposing a constant flow rate with a syringe pump and measuring the pressure drop across the die. This measurement was done at several flow rates to create a plot of pressure drop as a function of flow rate. These measurements were performed with the valve open, closed and partially closed. The results of these experiments are shown in Figure 5. The flow resistance of the valve is still several times lower than the expected value, however, there is more than a factor of 5 difference between the open and closed valves. The flow resistance of the closed valve has been improved over the valves shown in previous efforts [5] by a factor of 23. In addition, the flow resistance was measured when the valve was partially closed demonstrating the possibility of proportional control.

The initial actuation bubble must be removed to allow the

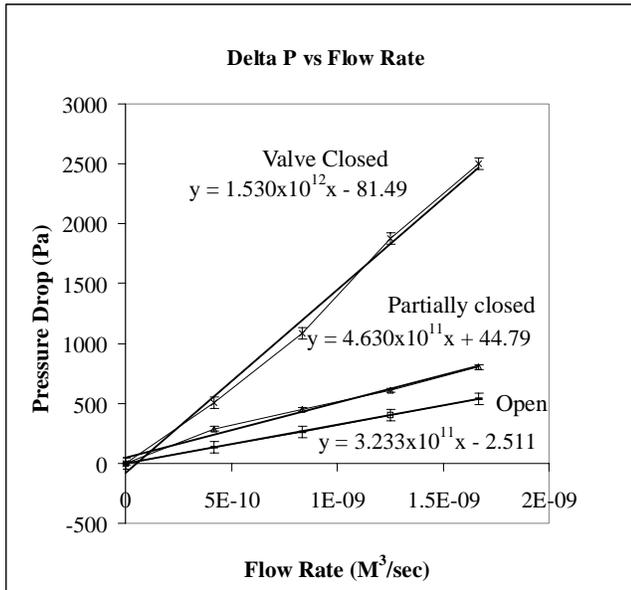


Figure 5. Experimental measurements of flow resistance for the valve fully closed, partially closed, and fully open.

gate to move in the opposite direction. This requires waiting for the relatively slow catalytic reaction process. One operational cycle (fully closed to fully open and back) requires approximately 120 seconds.

The gasket provides this valve with substantially improved sealing. Flow resistance was measured for the entire device including interconnects and channels. The resistance of the device with the valve closed was 4.7 times the resistance with the valve opened. The difference between opened and closed resistance was

Table 1: Valve Data

parameter	value	units
Flow resistance of open valve	1.53×10^{12}	$Pa \cdot s / m^3$
Flow resistance of closed valve	3.23×10^{11}	$Pa \cdot s / m^3$
differential resistance	1.30×10^{12}	$Pa \cdot s / m^3$
predicted differential resistance	4.04×10^{12}	$Pa \cdot s / m^3$
previous differential resistance	4.14×10^{10}	$Pa \cdot s / m^3$
Actuation power consumption	4.3	μW
Actuation Voltage	3.3	Volts
Actuation Current	1.3	μA
Holding power consumption	0.3	μW
Holding voltage	2.5	Volts
Holding current	0.1	μA
Minimum actuation period	120	seconds

37 times as high as that of the previous valve, however, it is still more than a factor of 4 away from the requirement for insulin regulation. However, simply putting 4 or 5 such valves in series would presumably provide sufficient control. In addition to the open and closed positions, the gate was held partially closed and an intermediate flow resistance was measured demonstrating the possibility of variable flow control.

CONCLUSIONS

An extremely low power electrolysis bubble actuated gate valve has been demonstrated. The actuation scheme requires only $4.3 \mu W$ for actuation and $0.3 \mu W$ to hold the valve in place more than 4 orders of magnitude less than similar thermal bubble actuators. The speed of the valve is limited by the need to find a way to remove the bubble quickly. The flow characteristics of the valve have been measured in the opened, closed and partially closed positions, demonstrating the possibility of proportional control. A new photoresist gasket has been shown to greatly reduce leakage around the valve. However, work still needs to be done on decreasing the leakage rate in order to make it commercially viable.

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