

# SILICON OXIDE DIAPHRAGM VALVES AND PUMPS WITH TiNi THIN FILM ACTUATION

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## ABSTRACT

Microns-thick silicon membranes can be formed on a silicon wafer by oxidation and in this process domes are formed that can act as chambers of valves or pumps. Arrays of valves are formed on a single substrate by micromachining. Combining two such domes provides a pump that is self-priming, has no separate check valves, and can deliver nanoliters of high-pressure liquid substance. TiNi thin film is employed as an electrically energized actuator for the valves and pumps.

A family of valves and pumps to meet biotechnology requirements and the needs of implantable medical devices, portable instruments, miniaturized fuel cells, and space vehicles are currently under development.

**Keywords:** Micropump, Microvalve, Shape Memory Alloy, Actuator, Silicon, Membrane, MEMS

## INTRODUCTION

Miniature domes are readily formed by thermal oxidation of thin silicon membranes. This occurs because of the difference in thermal expansion coefficients between Si, ( $4.7 \times 10^{-6}$ ) and SiO<sub>2</sub> ( $2.7 \times 10^{-6}$ ). In cooling from 1100°C (thermal oxidation) to ambient temperature, the oxide layer contracts 0.2 percent more than the silicon layer. The resulting silicon oxide layer is under very high compressive stress, which causes the SiO<sub>2</sub>/ Si/ SiO<sub>2</sub> membrane to bend until the energy of compression of SiO<sub>2</sub> balances the energy of elongation of Si. These 'buckled' composite membranes are very robust. Membranes 10 microns thick and a few mm in diameter do not fracture under several atmospheres pressure. Fatigue life is practically unlimited if the strains are below 0.5 percent.

We have used these domed membranes (diaphragms) to fabricate a variety of miniature valves and pumps. In these devices, the domed SiO<sub>2</sub>/ Si/ SiO<sub>2</sub> diaphragms function as chambers for pumps in which the volume is controlled by modifying the curvature through pressure applied against the convex side of the dome. The wetted surface is silicon oxide, or common glass, which is biocompatible and resistant to many chemicals. Fabrication employs common MEMS processes, which in turn are derived from standard microelectronics processes.

The approximate anticipated displacements and volumes generated by silicon/silicon oxide membranes of various sizes are shown in Table I. This calculation, which is based on modulus of elasticity and ultimate strength of silicon and silicon oxide, shows that these membranes can pump and valve volumes ranging from microliters to nanoliters per stroke at pressures up to several atmospheres. The thickness and diameter of the membranes can be adjusted to accommodate a wide range of pressures and flow rates. Forces, volumes, pressures, displacements, and material compatibility of these buckled membranes are appropriate for use in miniature valves and pumps for medical and biotechnology applications.

*Table I: Calculated displacement and force of silicon/silicon oxide membranes.*

Dimensions	Case 1	Case 2	Case 3
Width W (microns)	250	500	1000
Length L (microns)	250	500	1000
Thickness of Si, T (microns)	10	10	10
Thickness SiO <sub>2</sub> , 2 sides (microns)	3	3	3
Pressure (psi)	120	50	20
Pressure (Kpa)	827	345	138
Pressure (newtons/mm-sq)	0.8	0.3	0.1
Substr.length-change = d-alpha*d-temp*L (microns)	0.5	1.0	2.0
Sagitta of Circle-Arc $h = \sqrt{L^2 - L^2 / 2}$ (microns)	8	16	32
Radius of Circle-arc, microns $R = L^2 / 8h$	988	1976	3953
Volume under dome, microns-cubed = $0.5 * L * w * h$	247053	1976424	15811388
Stroke volume, nanoliters (10e6 micron-cubed)	0	2	16
Max. strain in Si, $\Delta L/L$ $S = \text{Thick}/2R$	0.0051	0.0025	0.0013
Stress due to pressure $S_p = P * R / T$ (kPa)	81765	68137	54510
Stress due to pressure, (lbs/in <sup>2</sup> )	11859	9883	7906
Force req'd of Actuator = Pres.*Area (Newtons)	.0052	0.086	0.138

## METHOD OF FABRICATION

Silicon-on-insulator (SOI) wafers make fabrication of silicon/silicon oxide membranes easy and highly controllable. SOI wafers have silicon membranes of uniform thickness from a fraction of a micron up to tens of microns. Fabrication of domes consists of backside etching on a pattern created by photolithography as shown in Figure 1. These domes are bistable: they buckle to create surfaces that are either concave or convex relative to the wafer front surface.

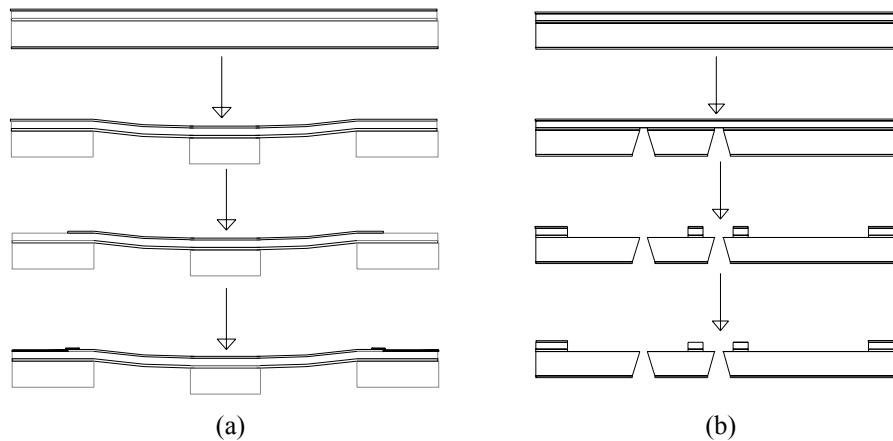
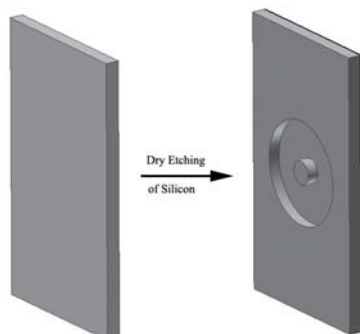


Figure 1: MEMS fabrication of buckled membrane and orifice die for a valve mechanism. (a): Back etching of a window on an SOI wafer and subsequent deposition and patterning of gold layer for eutectic bonding. (b): Front and back etching to produce spacer and inlet (with berm) and outlet orifices. A silicon-on-oxide (SOI) wafer has a layer of silicon oxide approximately 1 micron thick, overlaid with a layer of silicon that can be 10 or more microns thick, covered by a second layer of oxide.

A valve closure is created by joining the diaphragm die face-to-face with an orifice die having inlet and outlet orifices. These two dies are bonded around the periphery by a silicon-gold eutectic: gold is deposited on the mating surfaces, which are pressed together and heated.[1, 2] At 363°C an eutectic forms from silicon and gold, and when cooled it forms a hermetic seal. Figure 2 shows drawings of membrane and orifice die images of the resulting dies.



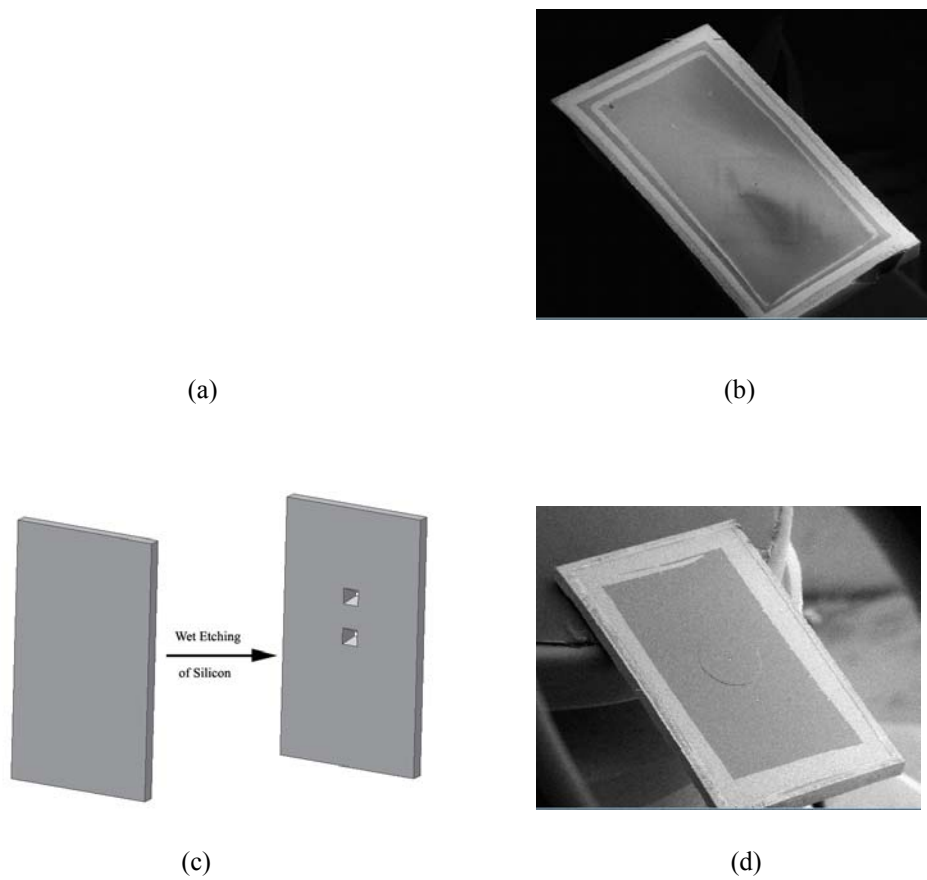


Figure 2: (a) & (b) Silicon/silicon oxide membrane dies made by etching SOI wafers. (c) & (d) Orifice dies with gold patterning for eutectic bonding.

### TiNi-ACTUATED LIQUID VALVE

Actuation can be achieved by depositing a film of TiNi directly on the membrane. Differential thermal contraction of the TiNi layer relative to the silicon/oxide membrane creates a tensile stress in the TiNi when the temperature is increased, for example by Joule heating. This stress is relieved when the transformation to martensite occurs, providing a means of changing the internal volume of the domed membrane. There is a problem with this method in a real device: heat from the TiNi layer is conducted into the fluid.

A separate silicon die carrying a TiNi thin-film actuator [3, 4] can be used to supply the force and displacement to open the valve against a bias spring. Details of such a valve, intended for use in medical and biotechnology applications, are shown in the exploded view drawing of Figure 3.

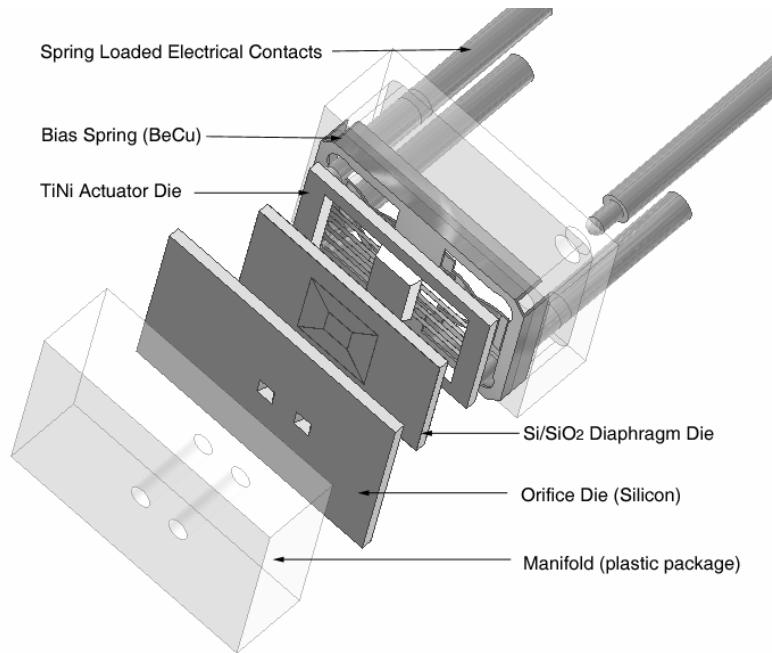


Figure 3: A miniature valve package approximately 1 cm by 0.6 cm by .8 cm encloses orifice and membrane dies that are bonded together, a TiNi thin-film actuator, and a beryllium-copper bias spring. Spring-loaded 'pogo' pins are used for electrical connection. The TiNi thin-film actuator die in the center of the drawing is 5 mm by 8 mm long. It provides displacement of up to 200 microns at a force of up to 0.5 Newtons.

### SILICON OXIDE MEMBRANE PUMP

The original application for the silicon/silicon oxide membrane structure was in arrays of millimeter-sized pumps that operate in unison, driven by changes in pneumatic pressure acting on the membranes. These arrays were intended to aspirate aqueous liquid from 384-well microtiter plates and drive the fluid through a bundle of capillary tubes to deliver samples a few tens of picoliters in volume.

A pump array is illustrated schematically in Figure 4. A linear array of four identical pumps is shown in four possible states. The pumping cycle is accomplished by repeating States #1, #2, and #3.

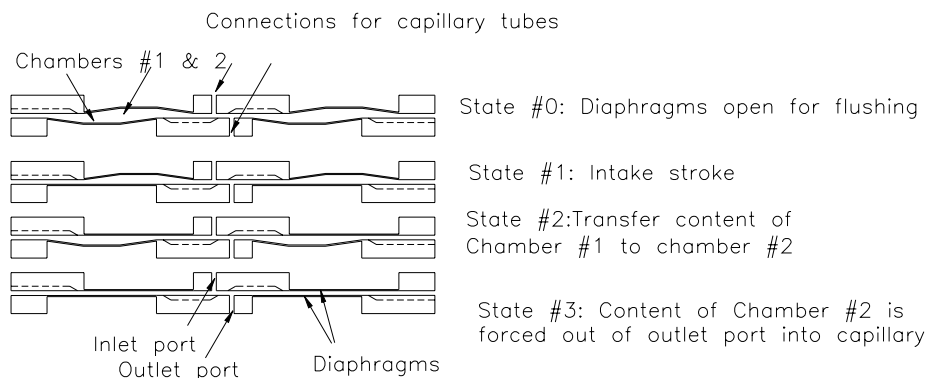


Figure 4: Side view of a rectangular array of micropumps. Pneumatic pressure is applied to the upper and lower surfaces, so that an entire group of membranes are moved (either flattened or buckled) simultaneously for moving fluid in multiple parallel channels.

These three states are shown in more detail in Figure 5. This is a pump with no separate check valves. When spring-loaded or pressed down by pneumatic pressure the membrane/diaphragm flattens against a channel that acts as an input or outlet port and seals it closed. When the holding force is removed by an actuator or by removal of pressure, the membrane bows away from the flat surface to create a volume, and, if the port is connected to a source of liquid, the liquid is drawn into the volume. Figure 6 shows the basic pump module with TiNi thin film actuators on opposing sides to provide the pressure needed to drive the membranes, and Figure 7 illustrates how a pump may be used to aspirate liquid from a microtiter plate for subsequent dispensing in small droplets on a substrate for genomic and proteomic applications. A perspective view of the four silicon (SOI) dies used to fabricate the pump module is shown in Figure 8.

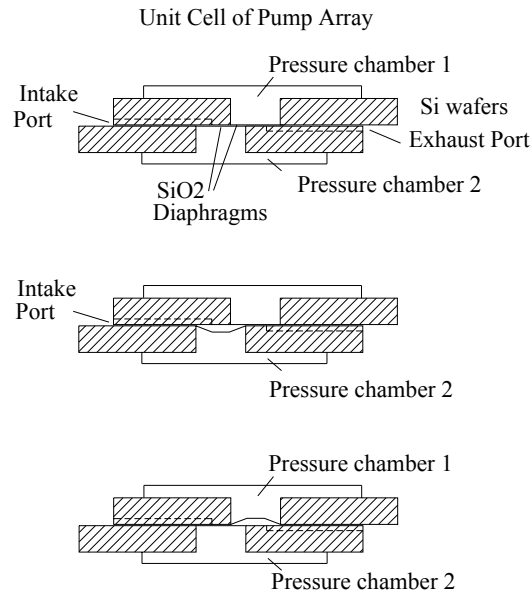


Figure 5: Single module micropump. In the first stage of a cycle, the first chamber is filled from its reservoir. In the second stage of the cycle, the contents of the first chamber are transferred to the second chamber. The second chamber is then emptied into an outlet.

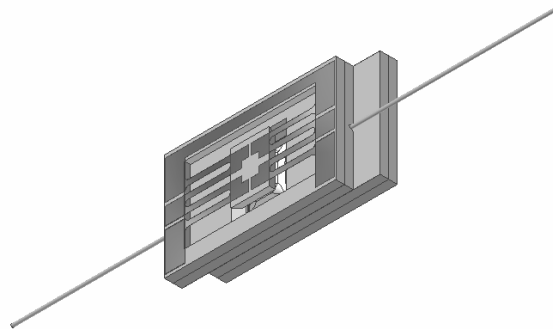


Figure 6: Single module micropump.

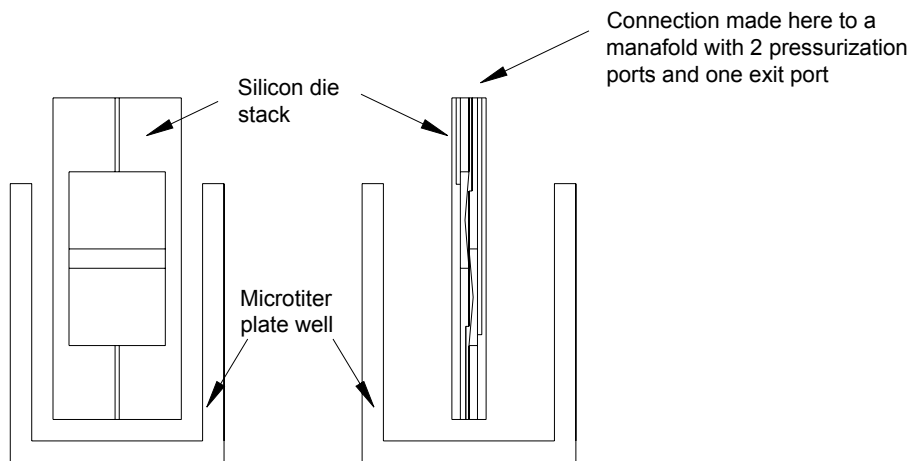


Figure 7: Individual pump module can be turned to move fluid vertically. The lower end of the module is immersed in liquid contained in a microtiter plate well. Sequential pressure changes cause fluid to be introduced at the bottom of the module and pumped out of the top. The top surface is connected to a manifold (not shown) that supplies pressurized air to the pump. A volume of liquid may be pumped into an accumulator within the manifold, and subsequently ejected in small droplets from the bottom of the pump by reversing of the order of pressure changes. An array of such pumps may be used to transfer liquid from wells to microarray biochips. In this application the pump array is attached to a robot arm to move it from its position for aspirating to the position for dispensing.

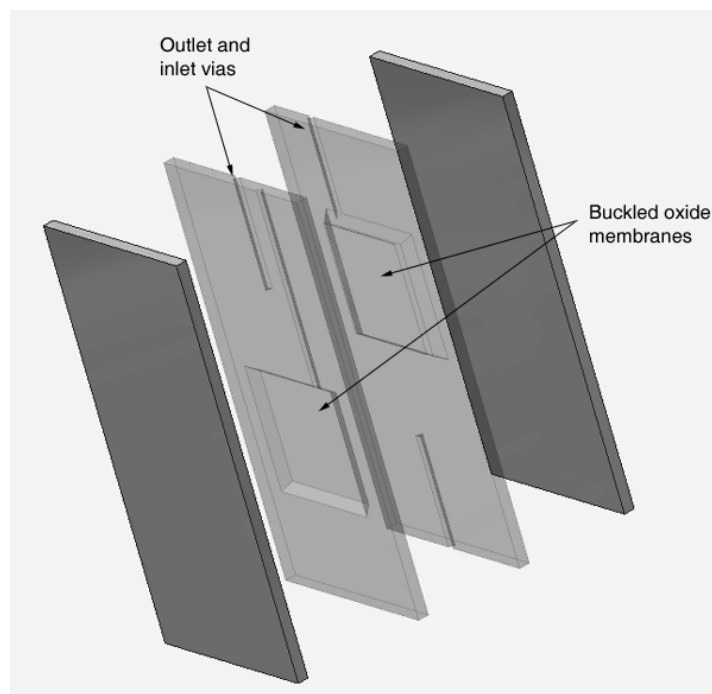
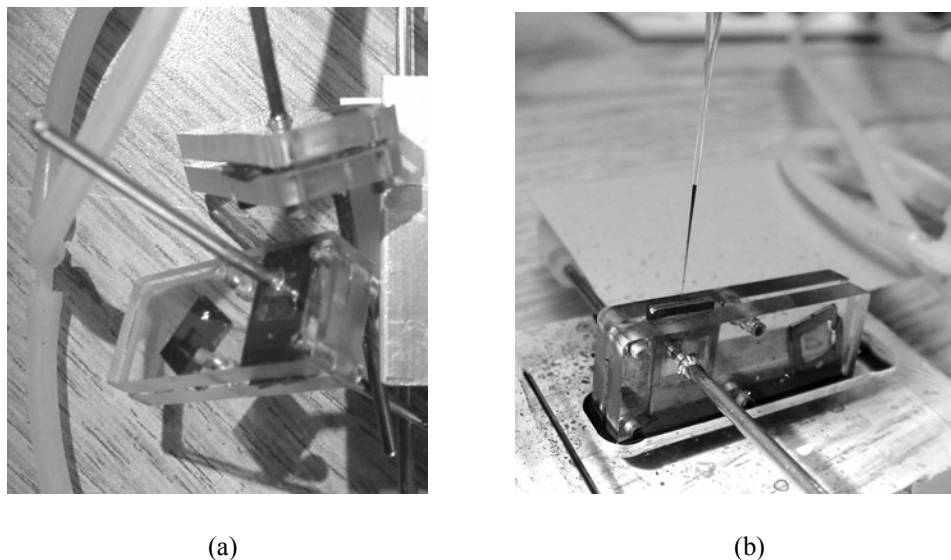


Figure 8: Pump module consists of four layers. The two outer layers are plain, while the two inner layers have been etched to have inlet and outlet vias, oxide membranes, and flow channels to pressurize the volumes behind the membranes.

Functional prototype devices have been made using SiO<sub>2</sub> buckled membranes that deliver 1-10 nanoliters per stroke. These micropumps, shown in photographs in Figures 9(a) and 9(b), are constructed of individual silicon dies with plastic enclosures for proof of concept.

To demonstrate pumping, one edge of the micropump is dipped in water as shown in Figure 9(b). In this view, a capillary tube has been inserted in one end of the silicon die module to show the colored liquid that is being pumped. Air pressure is supplied from a compressor and controlled by a pair of pneumatic solenoid valves. These valves are turned on and off by a microprocessor controller that supplies pulses of electrical power. The pumping action is reversible: the direction and amount of liquid pumped is controlled by varying the duration and sequence of the pulses.



*Figure 9. Two prototype individual micropumps are shown. (a). Each pump module consists of a pair of dark-colored silicon dies approximately 10 mm by 15 mm secured between two transparent plastic blocks. Steel tubes are affixed to the plastic blocks to supply air pressure for controlling the shape of the silicon/silicon oxide membrane to provide a pumping action. (b). One edge of the pump module is dipped in colored liquid and a capillary tube is attached to the other end. As the pump is powered, the liquid from the reservoir is pumped into the capillary tube.*

## RESULTS

Valve prototypes demonstrate leak rates as low as 10<sup>-6</sup> sccm air and flow rates from a low rate of a few ml/day to rates of several ml/min. Valves function at pressures up to 3 atmospheres. The TiNi actuator is a pure resistive load of approximately 20 ohms. The actuator begins to operate at 50 ma and is fully contracted at 120 ma, so the power consumption is between 50 mw and 300 mw, with flow approximately proportional to current in the range. Response times vary by application from a few msec to 100 msec.

## CONCLUSIONS

Buckled silicon oxide membranes and TiNi thin film actuators meet operational requirements for valves and pumps for a variety of applications. In the field of fuel cells, valves and pumps must be compatible with water, air, fuel mixtures, hydrogen, methane, methanol and others. Silicon oxide membranes are compatible with these substances and also satisfy the compatibility requirements of the biotechnology and medical industry. The silicon oxide membrane and thin film actuator module can work at pressures from less than an atmosphere for proteomics and cell biology to several atmospheres for fuel cell and pressurized drug delivery systems.

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