

# PARAFFIN ACTUATED SURFACE MICROMACHINED VALVES

E. T. Carlen and C. H. Mastrangelo

Center for Integrated Microsystems  
Department of Electrical Engineering and Computer Science  
University of Michigan, Ann Arbor, MI 48109-2122, USA

## ABSTRACT

A new, active, normally-open blocking microvalve that uses the thermal expansion of a sealed, thin paraffin patch for actuation has been fabricated and tested. The entire structure is batch-fabricated by surface micromachining the actuator and channel materials on top of a single substrate. The paraffin actuated microvalves are suitable for applications requiring many devices on a single die, low processing temperatures, and simple, non-bonded process technology. Gas flow rates in the 0.1-2.0 sccm range have been measured for several devices with actuation powers less than 50mW.

## INTRODUCTION

Over the past decade elaborate microfluidic valves have been constructed based on electrostatic, magnetic, piezoelectric, bimorph and thermopneumatic actuation methods [1]. Because of their complexity, the majority of these devices are made by bonding many thick glass or silicon substrates together, some even requiring external cavity fills. This bulk micromachining construction technique makes the valves large and difficult to integrate with other components in microfluidic systems.

Applications requiring many active valves on a single die are rapidly emerging. Integrated micro gas chromatography and mass spectrometry systems are being developed and will require effective valving devices [2-4]. In addition, microfluidic systems, such as DNA analysis systems [5, 6], require effective microvalves in order to control the transport of samples and reagents throughout different parts of the system. Typically these systems require many, independently operating valves in order to perform complex or parallel functions. Therefore, the actuators must operate independently, have a simple fabrication process and integrate easily with the other system components.

In this paper, we present a simple valve that is easily integrated with other fluidic components on the same die. Key to our simple valve fabrication is the choice of actuation method. Most actuation methods provide either a large deflection but not a large force (or vice-versa); therefore they require complex construction methods that

overcome their short comings (through mechanical advantage schemes for example). Thermopneumatic actuators can provide both large displacements and forces, but their fabrication and integration in large microsystems is cumbersome [7]. Shape memory alloy microactuators also provide both large displacements and forces, but are typically difficult to fabricate [8]. Recently, a new type of microactuator which provides large displacements ( $2 - 10 \mu\text{m}$ ), high actuation force ( $\approx 1\text{N}$ ), fabrication simplicity, and suitability for microfluidic valve applications has been reported [9]. These microactuators are based on the thermal expansion of a sealed, surface micromachined patch of high actuating power paraffin wax film.

## DESIGN

**Material:** The microactuators used for active valves in this paper are based on the thermal expansion of a thin layer of paraffin wax. Paraffin belongs to a family of materials that experience a large volumetric expansion upon melting. Typically a volumetric expansion between 10 - 30% can be reached when heated to temperatures ranging from  $65\text{-}150^\circ\text{C}$ , depending on the melting temperature of the material. Figure 1 shows the pressure-volume-temperature (PVT) characteristics of the paraffin material used for the devices in this paper (bonding wax, Logitech, Ltd. (0CON-175) with a melting temperature of  $75^\circ\text{C}$ ).

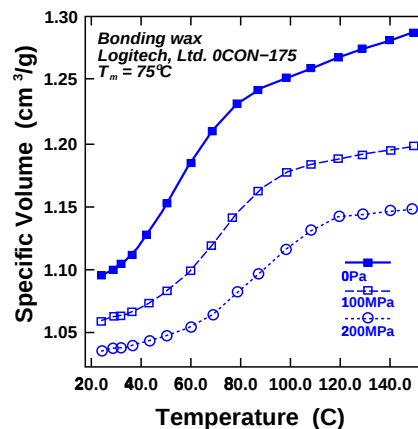


Figure 1: PVT of Logitech 0CON-175 bonding wax

**Actuators:** The microactuator, shown in figure 2, is

based on a 10  $\mu\text{m}$  thick paraffin film (Logitech Ltd., 0CON-175). The paraffin film is sealed with a 3-4  $\mu\text{m}$  thick layer of parylene-C, a flexible diaphragm layer. The heater, located beneath the paraffin is used to melt the paraffin. For a 10  $\mu\text{m}$  thick paraffin film with a 10% volume ex-

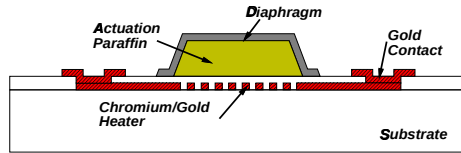


Figure 2: Schematic cross-section of surface micromachined paraffin microactuator

pansion, as shown in figure 1, the center of the diaphragm will deflect vertically about 3  $\mu\text{m}$  in about 20 ms with a power requirement less than 50 mW [9].

**Microvalves:** The blocking microvalve, shown in figure 3, has a 500  $\mu\text{m}$ -diameter inlet hole through the 1 mm thick substrate leading to a reservoir. The reservoir is connected to a 3 mm long capillary channel with a 200  $\times$

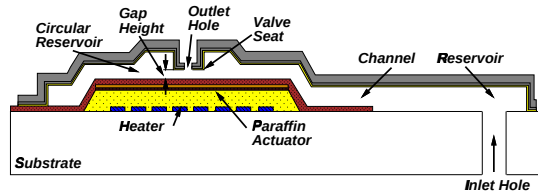


Figure 3: Cross section of blocking microvalve

20  $\mu\text{m}^2$  cross-sectional area. The channel leads to a circular reservoir where the actuator and outlet hole are located. Directly above the actuator is the 100  $\mu\text{m}$ -diameter outlet hole. When the actuator is activated, the diaphragm is deflected vertically pressing the diaphragm layer against the valve seat thus sealing the outlet hole. Since paraffin can provide very large actuation forces, a very good seal is easily attained with low actuation power.

The distance between the diaphragm and the valve seat is the gap height which is determined by the deflection height on the actuator. For this design, the deflection distance was 3  $\mu\text{m}$ , therefore a 2  $\mu\text{m}$  gap height was used, ensuring a good seal between the diaphragm and valve seat. Larger deflection distances are possible using thicker paraffin films, alternative paraffin materials (larger volumetric expansion), or mechanical advantage schemes [9].

Both gas and liquid based valves have been fabricated and differ only in the reservoir and channel structural materials. Pneumatic valves use electroplated nickel structures and the liquid devices have parylene-C structures.

## FABRICATION

**Pneumatic Valves:** The entire blocking microvalve is fabricated using a low temperature process ( $\leq 90^\circ\text{C}$ ) and

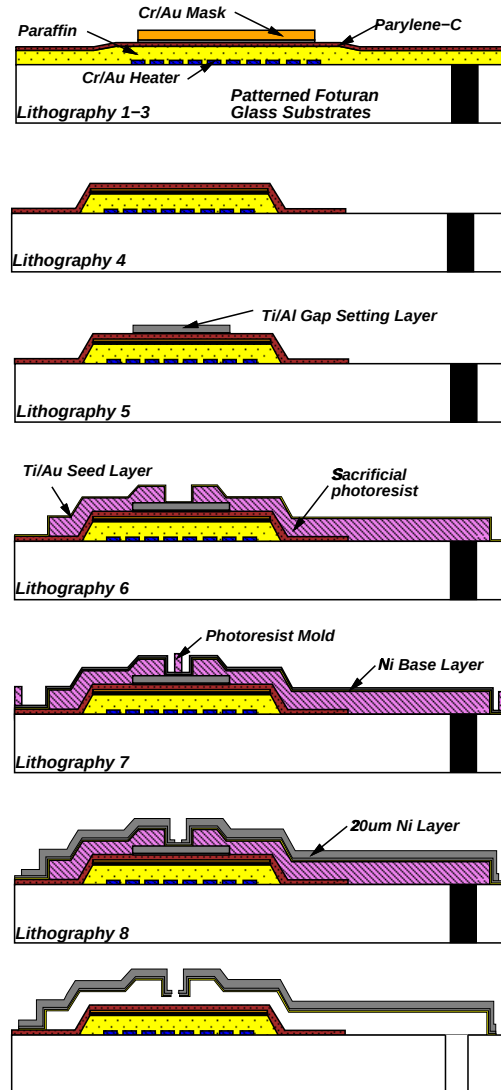


Figure 4: Simplified blocking microvalve fabrication process flow

eight lithography steps. Figure 4 shows a simplified process flow. The fabrication begins by patterning the inlet hole in photodefinable glass substrates (Foturan, Schott Corp.). Patterning the Foturan substrates requires UV (312nm) exposure ( $\approx 2\text{J}/\text{cm}^2$  energy density is required to structurize 1 mm thick substrates) followed by a heat treatment schedule which reaches  $600^\circ\text{C}$  for 1 hour. During the heat treatment, the substrate surfaces become very rough requiring surface refinishing. The substrates are then planarized with a 20  $\mu\text{m}$  calcined aluminum oxide slurry (Logitech, Ltd., 0CON-012) for 1 hour and polished with an alkaline colloidal silica slurry (Logitech, Ltd., SF1 0CON-140) for 1 hour. Next, Cr/Au (500/5000  $\text{\AA}$ ) heaters are evaporated and patterned on the substrate followed by the thermal evaporation of the paraffin. The paraffin evaporation was done in a custom deposition system (pressure:  $5 \times 10^{-6}$  Torr, material temperature:  $150^\circ\text{C}$ , deposition rate: 1000  $\text{\AA}/\text{min}$ ). The paraffin is then selectively patterned using reactive ion etching (RIE: pressure 260 mT,  $\text{CF}_4:\text{O}_2$  20:80 sccm) with a parylene-C/Cr/Au

(5000/500/3000 Å) mask. The patterned paraffin patches are then sealed with a 3 μm thick parylene-C layer. Adhesion to the gold and substrate layers is assisted by a silanation procedure. The parylene-C layer is then etched in an O<sub>2</sub> plasma RIE using a 20 μm thick photoresist mask (Clariant AZ 9260). Fabricated actuators are shown in figure 5.

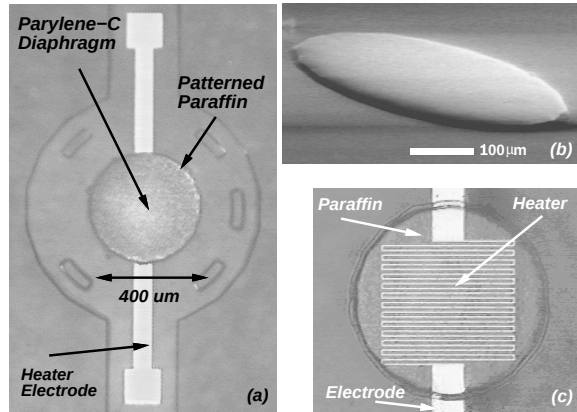


Figure 5: Photographs of paraffin actuators with 9 μm-thick paraffin layer (a) top view of completed actuator (b) SEM of patterned paraffin (c) top view of patterned paraffin with heater clearly visible

Following the completion of the actuator fabrication, the channel and reservoir structures are constructed on top of the actuator. First, a 100 Å/2 μm thick Ti/Al sacrificial gap-setting layer is evaporated and patterned. For this device, a 2 μm gap thickness was chosen based on the actuator characteristics. Next, the sacrificial channel and reservoir areas are formed by spin-casting a 20 μm-thick photoresist (AZ 9260) layer followed by softbake (65°C for 1 hour), exposure (5 mW/cm<sup>2</sup> for 400 sec.), and development (1:4 AZ400K (Clariant):DI H<sub>2</sub>O for 5 mins.). The entire substrate is then sputter coated with a Ti/Au (300/3000 Å) electroplating seed layer. A 3 μm-thick Ni base layer is then electroplated (Barrett-SN nickel sulfamate solution, MacDermid Inc.) onto the seed layer. This is followed by spin-casting, exposure, and development of a thick photoresist (AZ 9260) layer (> 20 μm) forming the channel and reservoir mold. Next, the 20 μm-thick channel structures are electroplated. Following plating, the photoresist mold is removed in acetone and the Ni base is etched with a commercial nickel etchant (Type TFB, Transene Co., Inc.). The Ti/Au seed layer is then wet etched using a commercial Au etchant (GE-8148, Transene Co., Inc.: which does not significantly attack the Ni films) and dilute HF solution (10:1 DI H<sub>2</sub>O:HF(49%)) to remove the Ti layer. The Ti/Al sacrificial spacer layer is then removed using an in-house etching solution (K<sub>4</sub>Fe(CN)<sub>6</sub> · 3H<sub>2</sub>O:NaOH:DI H<sub>2</sub>O). The front side of the substrate is then spin-casted with a thick photoresist (AZ9260) and softbaked at 65°C for 1 hour. The parylene-C on the backside of the substrate (from prior depositions) is removed using O<sub>2</sub> RIE. The Forturan glass substrates are then wet etched (10:1 DI H<sub>2</sub>O: HF(49%))

) forming the inlet holes while at the same time dicing the wafer. Finally, the sacrificial photoresist is removed from the channels of each device in an acetone bath for 5 hours. Figure 6(a) shows an SEM photograph of the valve reservoir surrounding the paraffin actuator where the outlet hole is directly above the actuator. Figure 6(b) shows the reservoir surrounding the inlet hole. Figure 7

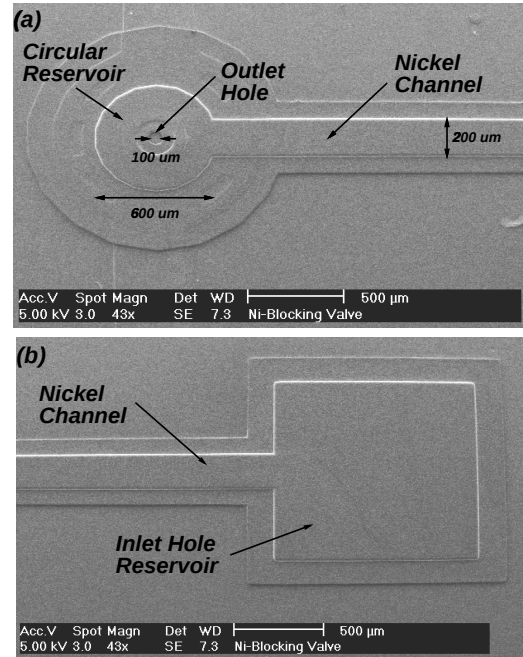


Figure 6: SEM photograph of device (a) circular reservoir with outlet orifice over the paraffin actuator (b) square reservoir covering the inlet hole

is a microscope photograph showing the backside of the fabricated device. Figure 7(a) clearly shows the actuator. Figure 7(b) shows the etched inlet hole through the substrate.

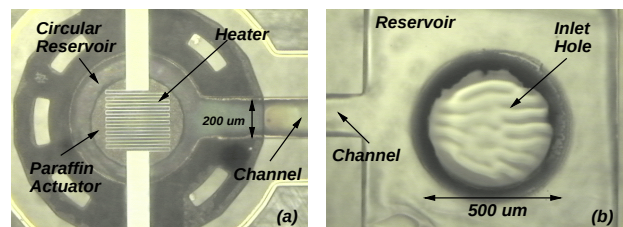


Figure 7: Microscope photograph of pneumatic valve backside showing (a) actuator (b) inlet hole through substrate

**Liquid Valves:** The microactuators for the liquid valves are fabricated using the same process as the pneumatic valves. For the liquid devices, the gap height is set by spin-casting a sacrificial photoresist layer (AZ 9260). Due to the height differences between the substrate and the actuators, the photoresist is thinner on top of the actuator. The photoresist thickness in the channels is 12 μm, while on the top of the actuator it is 6 μm thick. The sacrificial photoresist is then patterned forming the reservoir

and channel areas. Next, a 15  $\mu\text{m}$  thick layer of parylene-C is vacuum deposited. A Ti/Au (500/5000  $\text{\AA}$ ) mask layer is sputter coated on the entire substrate. The Ti/Au mask is patterned and the parylene-C layer is etched in an  $\text{O}_2$  RIE. Figure 8 shows a fabricated liquid (parylene-C channel) valve.

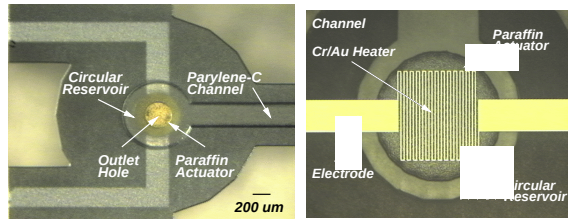


Figure 8: Microscope photograph of liquid microvalve (a) Top side of circular reservoir clearly showing outlet hole and actuator (b) bottom of circular reservoir clearly showing the heater, electrodes, reservoir and channel

**Mass Flow Controller:** A 6-bit micro mass flow controller ( $\mu\text{MFC}$ ) is currently being fabricated using the blocking microvalves as shown in figure 9.

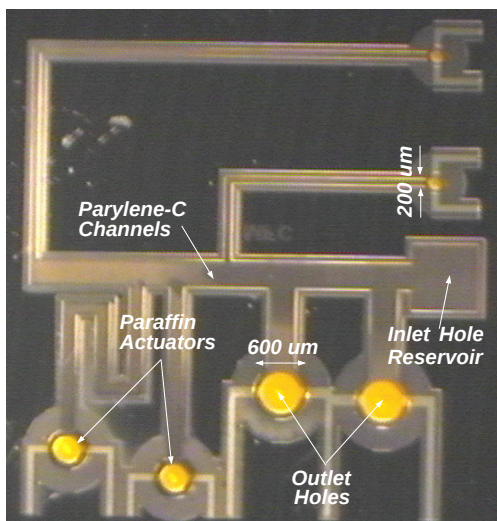


Figure 9: Microscope photograph of 6-bit  $\mu\text{MFC}$

## EXPERIMENTS

**Pneumatic Valves:** The die were mounted on custom made circuit boards (copper clad laminate, kepro circuit systems) with 1 mm-diameter access holes concentric with the inlet holes of the test devices. The die (1.5cm  $\times$  1.5cm) were bonded to the circuit boards with a vacuum epoxy (Kurt J. Lesker Co.). Figure 10 shows an example of a die mounted on the circuit board. The diaphragm center deflection was measured through the outlet hole for various input voltages using a Zygo NewView 5000 non-contact surface profilometer with 100X magnification and a 5  $\mu\text{m}$ , high resolution scan length. Figure 11 shows the center deflection characteristics for the 10 $\mu\text{m}$ -thick paraffin actuator. From figure 11, the actuator has deflected the entire gap height with less than 50

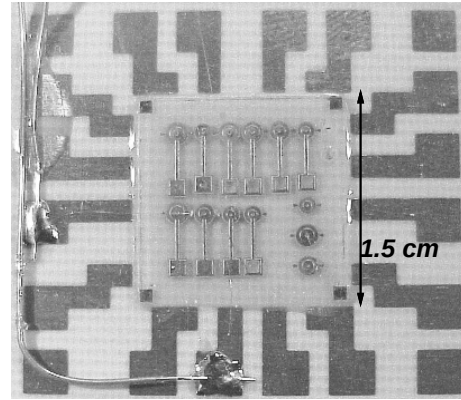


Figure 10: Photograph of die containing ten microvalves bonded to test pc-board

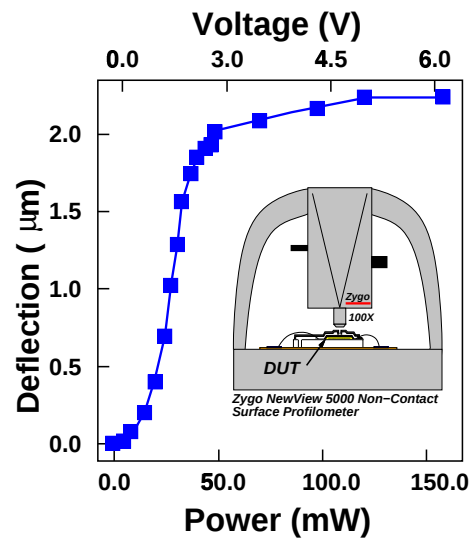


Figure 11: Measured actuator center deflection through the outlet hole with a Zygo NewView 5000 non-contact surface profilometer

mW. The deflection beyond the gap height is due to the soft parylene-C diaphragm pushing into the outlet hole by the molten paraffin.

Since flow and leak rates are expected to be very small, no commercially available flow meters exist in these small ranges. Therefore, an indirect approach has been used:  $\Delta P / \Delta t$  measurements for a known volume at the outlet of the device are taken as test data [10]. Figure 12 shows a schematic of the testing apparatus. The measurement procedure is as follows, initially valves  $v_1$  and  $v_2$  are closed. Valve  $v_2$  is opened ( $v_1$  is still closed) thus evacuating the bottom chamber to about 100 mTorr. Then  $v_2$  is closed and valve  $v_1$  is opened allowing the top chamber to reach the desired pressure (typically 760 Torr). Valve  $v_2$  is then closed. The computer measures the pressure change  $\Delta P$  in the bottom chamber for a period of time  $\Delta t$ . Since the volume of the bottom chamber  $V$  is known, fixed, and assuming the gas is ideal, the mass flow rate  $Q$

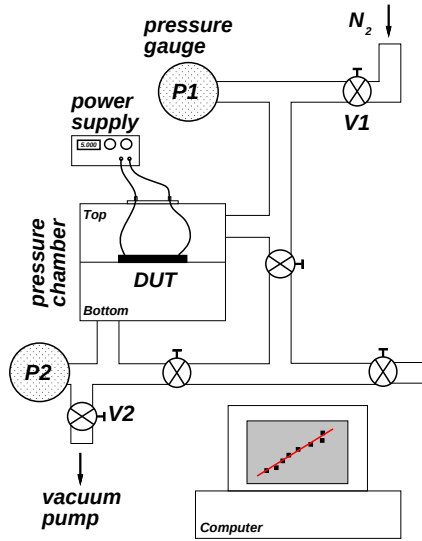


Figure 12: Experimental flow rate measurement apparatus

can be determined as shown in Eq. (1).

$$Q = \left( \frac{\Delta P}{\Delta t} \right) \left( \frac{V}{RT} \right) \left( \frac{M_{N_2}}{\rho_{N_2}} \right), \quad (1)$$

where  $M_{N_2}$  is the molar mass and  $\rho_{N_2}$  the density of nitrogen.

Flow rates were measured for different applied voltages. When the diaphragm is fully deflected the flow rate through the device is the leak rate of the valve. For all measurements, the top side pressure  $P_1$  of the device was maintained at 750 Torr, while the bottom pressure  $P_2$  was maintained in vacuum (about 100 mTorr). Figure 13 shows a measured flow rate through the device as a function of input voltage and power. Measurements indicate

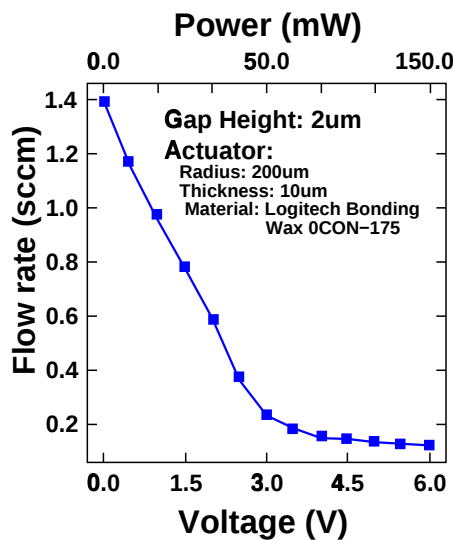


Figure 13: Measured flow rate through device as a function of input voltage and power

flow rates in the 0.1-2.0 sccm range for actuation volt-

ages less than 5 volts while consuming less than 50 mW. The flow rate for this device is determined primarily by the gap layer because its height is an order of magnitude less. Flow rates can be adjusted by simply changing the gap height and actuator deflection height.

## SUMMARY

In this paper, we presented the design, fabrication and testing of an active, normally open blocking microvalve for both pneumatic and liquid applications. The actuation is provided by a paraffin microactuator. The valve is completely fabricated using a simple surface micromachining method with low temperature ( $<90^\circ\text{C}$ ). These valves are particularly valuable when many devices ( $> 10$ ) are required on a single die.

## ACKNOWLEDGMENTS

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