A Self-Controlled Microcontrolled Microvalve

C. A. Rich and K. D. Wise

Center for Integrated Sensors and Circuits, Dept. of EECS, University of Michigan, Ann Arbor, MI 48109-2122

Integrated microvalves are needed for a broad range of semiconductor-processing-related applications. These include precision mass microflow controllers (μFCs) for dry etch systems, miniature gas chromatography systems for real-time monitoring, point-of-use semiconductor process reactant generators, and compact control systems for mini-environments. This paper reports a pneumatically actuated, integrated silicon microvalve, which was developed as a forerunner to an 8b μFC intended for the precision control of semiconductor process gases in the range from 0.1 to 10sccm. The structure was designed to be batch-fabricated and compatible with on-chip thermopneumatic actuation. Assembled single-bit μFC devices achieve the targeted flow rate of 5sccm (determined by an in-line flow channel) at 20psid (1034torr). The valve alone may achieve significantly higher flow rates. The leak rate is 0.08sccm under 26.1psig actuation pressure, and the valve can seal against pressures greater than 29psid (1500torr).

Keywords: microvalve, microflow, mass flow, gas chromatography, dry etch, RIE

INTRODUCTION

Despite the continued advance of MEMS-related technology, development of a high-performance, batch-fabricated, micromachined silicon microvalve for the precision control of fluids remains an elusive goal. Significant research has already progressed in this area, although no single valve structure has been popularly accepted by industry. A brief review of some potential applications demonstrates the relevance of this work to the semiconductor industry in particular.

Gas Metering Applications

Mass flow controllers (MFCs) fulfill the fundamental need of many fabrication processes to introduce precisely metered quantities of fluid reactants into controlled environments. As such, MFCs have a direct impact on process characteristics. For example, oxygen flow in a dry etch (such as RIE) has a substantial impact on process uniformity, anisotropy, selectivity, and aspect-ratio-dependent etching (ARDE) (1)-(3). Note that low-flow process gases, such as oxygen, often require flow rates on the order of 0.1-10sccm or less.

As a reflection of industry consensus, the 1997 National Technology Roadmap for Semiconductors (NTRS) discussion of "critical dimension" (CD) implies an acute need for better gas flow control in the future. The Interconnect Technology Working Group assumes a constant CD tolerance of ±8%, independent of absolute CD (4). Thus, if the etch characteristics of a process are highly dependent on the flow of a certain gas, a flow controlled to within 1-2% accuracy would still consume nearly a fourth of the total tolerance budget. Likewise, the NTRS anticipates that etch selectivity (5) and ARDE (6) will continue to be key issues for future technologies.

Current mass flow control technology, however, may not be adequate to meet future requirements. Commercial MFCs are poorly suited for low-flow semiconductor processing, with accuracies of ±0.1 or even ±1 sccm at low flow ranges. Conventional MFCs also suffer from slow time constants and inherent dead volumes that degrade transient response (7). *Dead volume* refers to the empty cavities in the downstream flow path of an MFC (or stand-alone valve) that must be pressurized with process gas before flow starts and must be depleted before flow will stop. This characteristic is similar to the parasitic capacitance of an electrical circuit and is likewise undesirable.

Embedded Fluid Control Applications

Applications for microvalves embedded in larger systems are also emerging. Of particular interest is the potential for a compact, integrated gas chromatography (GC) module. Micromachined GC (μGC) technology promises performance and portability well beyond present-day macro systems, but complete integration currently awaits a suitable valving system (8)-(11).

The Factory Integration section of the NTRS identifies metrology needs that could potentially be met by a μGC. For example, assessing the effect of factory performance specifications (which would include environmental contamination from gases and volatile organic compounds) on wafer yield would enable a reduction of factory overdesign and process material overspecification (12). Recent research has used GC methods to identify sources of yield-impacting contamination as obscure as HEPA filter potting compounds (13).

In situ process measurement and control, another pillar of the NTRS (14), would benefit greatly from μGC technology as well. GC technology has been pinpointed for the detection of airborne contaminants (15), the analysis of

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process gas impurities (16), and the evaluation of organic content in recycled and/or ultrapure water supplies (17). Some airborne contaminants commonly found in cleanrooms have been shown to substantially degrade deep-UV (DUV) photoresists; effective monitoring of such contaminants is readily achieved with GC technology (18).

Industry has already taken the first steps towards µGC technology. MTI Analytical Instruments, for example, offers a portable GC system that utilizes a silicon micromachined injector and solid-state detector (19). This completely self-contained system measures 15cm × 36cm × 36cm and can operate continuously for 6-8 hours. Detection of 1ppm takes place within 160s. Although not hand-held size, the system nonetheless demonstrates commercial interest in the miniaturization of GC equipment.

The NTRS also anticipates advances in containment that would benefit from microvalves in general. For example, the Front End Processes section recommends point-of-use generation of process reactants to improve efficiency (20). Microvalves and microreaction chambers could facilitate localized generation of such reactants in precisely the quantities required (21), (22). Also of interest, the Factory Integration section foresees the rapid embrace of cluster tools and minienvironments to enhance flexibility, reduce chemical use, and improve contaminant control (23). Such equipment requires compact, inexpensive control devices to restrain the cost of redundancy and to keep machine footprints within reasonable limits.

DESIGN

Considering the breadth of possibilities, it is apparent that the requirements for microvalves may vary greatly; however, a general set of design metrics may be proposed. An ideal microvalve should be *robust, low-power, fast, simply fabricated, useful over a wide pressure and flow range,* and *low-leak.* Although compatibility with corrosive fluids is another concern, nearly all microvalves are micromachined out of silicon, and thus fluid compatibility is dependent on protective films rather than on the fundamental valve structure.

Industry and academia have both shown creativity in the development of stand-alone microvalves, although all of the devices reported so far (to the authors' knowledge) have significant limitations. In-depth reviews of the various technologies available are compiled elsewhere (24), (25). After evaluation of various actuation schemes, *pneumatic* actuation, as a precursor to thermopneumatic actuation, was chosen for development in this research. Pneumatic and thermopneumatic actuation exhibit the potential to perform acceptably by all six microvalve design metrics. Both of these methods rely on gas or vapor pressure to drive a valve diaphragm. (Thermopneumatic valves generate pressure by vaporizing a liquid.) The advantage of these two methods is an ability to generate large forces over a significant distance, since gas molecules will diffuse within a closed cavity to reach pressure equilibrium. The freedom to specify actuator size (by simply scaling the diaphragm), combined with the potential for considerable travel, allows microvalves to be constructed with comparatively high flow conductance. Substantial valve plate travel should also facilitate robust valves with a high tolerance for particulate contamination. Furthermore, actuation pressure is relative to ambient pressure; thus, nearly any inlet pressure may be valved if appropriate ambient and drive pressures are maintained.

Simple pneumatic actuation does require an off-chip compressed-air source, which may or may not be a concern depending on the application. Thermopneumatic actuation (discussed later) should provide a viable solution to this concern, however.

For the sake of simplicity in testing and characterization, the authors decided to initially construct devices consisting of only a single microvalve with an in-line flow channel. While not very useful as a variable flow controller, this simple 1-bit design nonetheless serves as proof-of-concept for a future 8-bit controller. Some design goals were also established for this device:

- *Robustness*: Employ a durable structure that is able to handle 10µm particulates without loss of performance
- *Fabrication*: Use only one silicon and two glass layers; primarily batch-fabricated
- *Leak rate*: Achieve a leak rate of < 0.01sccm at 20psid (1034torr) inlet–to–outlet and 26.1psig actuation pressure
- *Operating range*:
  Pressure: Be able to close against 29psid (1500torr)
  Flow: Conduct ~5sccm at 20psid for the entire µFC; achieve a flow for the valve alone of 1000sccm at 20psid

Figure 1 depicts the developed prototype structure. The corrugated diaphragm in the silicon layer functions as a valve plate and occludes the gas inlet when driven by pneumatic pressure. With no drive pressure applied, the valve is open and gas flows through the precision flow channel etched in the top glass cap, exiting through the gas outlet. The incoming pneumatic drive line is controlled by a 3-way solenoid valve fed from a compressed nitrogen source.

The flow channel and valve seat depths have been set at 20µm to accommodate the passage of large particulates. The remaining dimensions for the flow channel are subsequently sized to generate the anticipated flow rate of ~5sccm. A quasi-empirical model for gas flow rates through the valve structure and flow channel has been developed from the equations of Olsen, Berman, and Steckelmacher (26)-(28). Although their equations assume rectangular channels, an "uncurling" of the circular valve structure allows equivalent rectangular dimensions to be

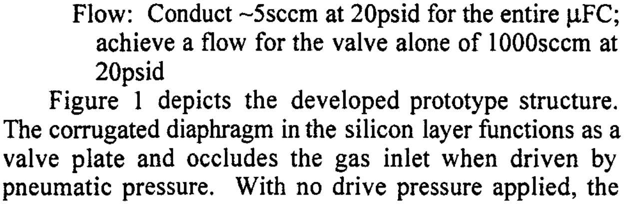


FIGURE 1. Schematic of a pneumatically actuated microvalve.

assigned for modeling purposes.

Design and fabrication of the valve diaphragm and pressure cavity draws upon previous research performed at the University of Michigan by Zhang (29). The overlaid labels of Figure 3 give major dimensions for the valve.

The silicon layer of Figure 1 requires four masks. Front and back deep-boron etch-stops define the diaphragm rim and boss (valve plate), the pressure cavity, and the outlet port edges. Next, an RIE etch defines the top side of the corrugations. Finally, a shallow boron etch-stop defines the corrugation thickness. An EDP etch is then used to bulk micromachine the diaphragm underside, pressure cavity, and gas port. This etch step is also used to separate the dies, since dicing might damage the released diaphragms.

The flow channel and valve seat in the top glass cap are defined by a single mask and etched using an HF-nitric acid etch. A groove is also saw-cut into the bottom glass cap for drive inlet access. After subsequent dicing, the gas inlet and outlet orifices are drilled in the top and bottom caps, respectively, with a diamond bit. Although individually drilled holes are not conducive to batch fabrication, future work may take advantage of batch-compatible ultrasonic glass drilling. Note that the flow channel dimensions, and thus gas flow rates, are completely determined by the separately processed glass cap. Next, the top glass cap is attached by electrostatic bonding, and the bottom glass cap is glued with epoxy (although electrostatic bonding could also be used). Finally, the pneumatic drive tube is inserted into the groove in the bottom glass cap and sealed with epoxy.

Flow and leak rates for the prototype device were expected to be less than 10sccm and as low as 10^{-3} sccm, respectively, depending on drive and inlet pressures. Since no commercial flowmeters were found that could measure such low rates accurately, an indirect approach has been developed: $\Delta P/\Delta t$ measurements for a known volume at the outlet of the device are taken as test data. Using a modified ideal gas law, $\Delta P/\Delta t \cdot V = \Delta n/\Delta t \cdot RT$, with appropriate substitutions, mass flow may then be found.

Zhang's work concluded the lifetime for corrugated diaphragms of similar construction to be at least several hundred thousand cycles without noticeable fatigue. No lifetime tests have yet been performed for the work reported here.

EXPERIMENTAL RESULTS

Final yield after assembly and processing was lower than anticipated, although most problems are understood and can be avoided in the future. The fabricated components were still sufficient, however, to allow the subsequent assembly and testing of several devices. There was also some initial concern that the valve boss would become electrostatically bonded to the top glass cap, thus sealing the valve shut permanently. Fortunately, such problems never occurred and all assembly efforts were successful in this regard. A photo and SEM shots of device structures are shown in Figures 2 and 3.

The circular markers of Figure 4 correspond to measured data for one of the devices tested. None of the valves were tested for maximum unregulated flow, due to difficulty in separating an intact valve from its channel.

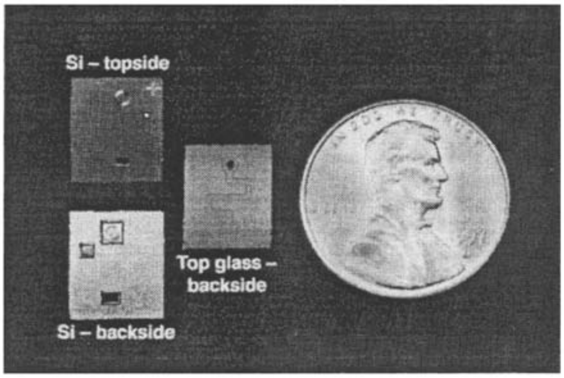
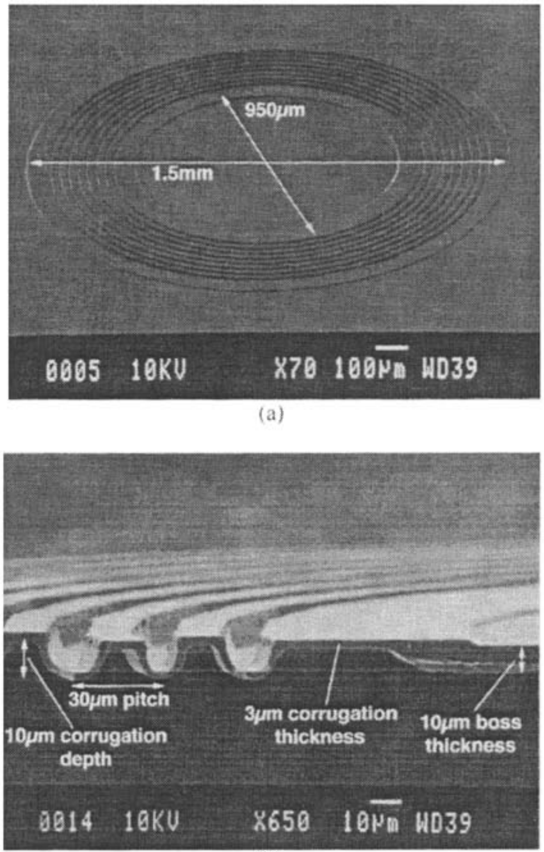


FIGURE 2. Photograph of silicon and top glass cap. Bottom glass cap is not shown.

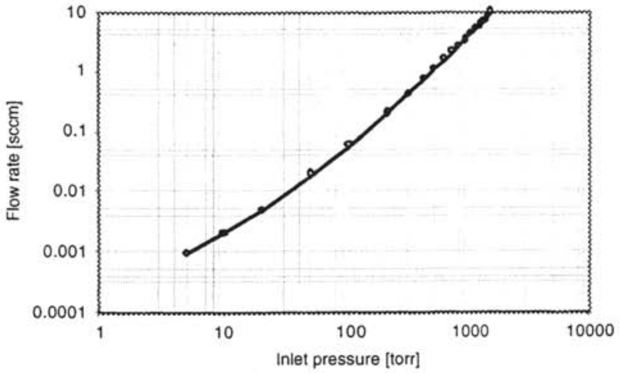


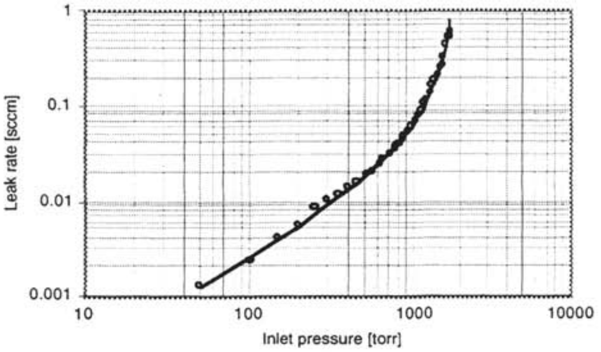
(a)

(b)

FIGURE 3. a) SEM shot of corrugated diaphragm/valve plate. b) Cross-section of corrugations.

During testing, it became apparent that thorough cleaning before the electrostatic bonding step is critical to a good valve seal. Any residue on the valve sealing surfaces degraded the leak rate considerably. It was also found, however, that particulates in the inlet gas stream were largely irrelevant. Valves survived the passage of "dirty" room air, water, alcohol, and other cleaning solvents with


Flow Rate vs. Inlet Pressure for Open Valve


Leak Rate vs. Inlet Pressure for Closed Valve
(Actuation Pressure = 26.1psig)

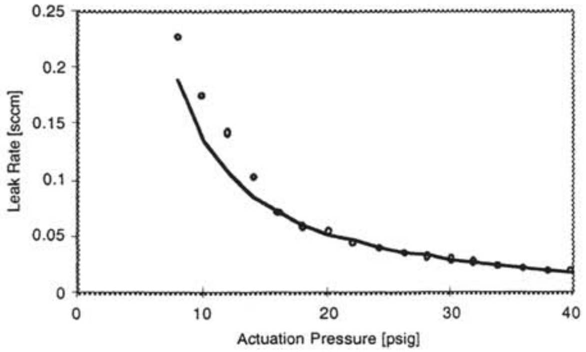

Leak Rate vs. Actuation Pressure for Closed Valve
(Atmospheric Inlet)

FIGURE 4. Flow and leak rate data for the prototype device. All valves were tested with outlets vented to vacuum (10^{-2} Torr). Circles correspond to data points and lines to performance predicted by the model.

little or no change in leak rates. In fact, the passage of liquids *improved* the leak rates slightly for several of the valves, presumably by washing out particulates.

Response time for the valves is largely determined by the relaxation time of drive pressure in the pneumatic drive line, which is dependent on tubing length and diameter. The drive lines were connected to a 3-way solenoid valve by 0.3m of 200μm ID PTFE tubing. The speed of the solenoid valve controlling the drive pressure also influenced the response time. A rough estimate by visual observation indicated a response time of a few tenths of a second.

DISCUSSION & CONCLUSIONS

The solid curves of Figure 4 correspond to the model predictions for the valve under test. Predicted and measured data values match to within a few percent on average. The range of accuracy required for a full μFC implementation would probably require flow channel dimensioning by means of Monte Carlo simulation. This would account for both viscous and molecular flow through the channel.

Additionally, the glass channels of the present device were etched using a wet-etch process, which, although sufficient for testing purposes, is not uniform enough for mass production. An ultrasonic or dry etch process would most likely be required to achieve the precision required for commercial applications.

Leak rates for the valve/μFC combination were less than 0.1sccm through 20psid, with 26.1psig drive pressure. This meets the design target of 0.01sccm only through 5.7psid (300torr); hence, further improvement in valve sealing ability is desirable. Leak rate is determined primarily by the geometry of the closed valve, with little contribution from the flow channel. Thus a larger maximum open flow yields a larger dynamic range. Adjustment of the model to remove the flow channel completely predicts a flow rate at 20psid on the order of 10^4sccm and leak rate of 10^{-1}sccm, giving a potential dynamic range of 100000:1 for a stand-alone valve.

The feasibility of the basic design and process flow developed for the prototype pneumatically actuated valve structure has thus been verified, as demonstrated by the realization of working microvalve/μFC structures. The present design exhibits many of the fluid-handling advantages of a good microvalve, such as low dead volume, low leak rate, high flow rate, and robustness, in spite of its requirement for an external air source.

FUTURE WORK

An 8-bit Microflow Controller

The pneumatically-actuated microvalve is only a proof-of-concept stepping-stone on the way to development of more useful devices. As a first attempt to demonstrate a pneumatic-microvalve-based system, efforts now seek to develop a pneumatically actuated μFC with 8b resolution. Such a flow controller entails routing eight binarily weighted, valved flow channels in parallel. This configuration takes advantage of the long-term stability of micromachined flow channels over proportional valving to develop a μFC with accuracy and repeatability exceeding that of conventional MFCs.

The targeted flow range for the device is 0.05-12.8sccm with 800torr inlet pressure and 26psig actuation pressure. This range slightly overlaps the expected leak rates to allow for future improvements in valve sealing.

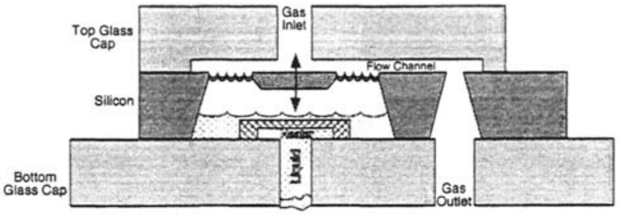


FIGURE 5. Schematic of a thermopneumatically actuated microvalve/μFC.

(One possibility is the deposition of parylene, a soft, conformal polymer, on the valve sealing surfaces.)

Thermopneumatically Actuated Microvalve

It is also desirable in the long term to implement a self-contained microvalve that may be completely regulated by electronic signals. To that end, a *thermo*pneumatically actuated microvalve is depicted in Figure 5.

Thermopneumatic actuation relies upon drive pressure developed by the vaporization of a heated fluid in a sealed cavity. The balance of the structure (diaphragm, flow channel, etc.) shown in Figure 5 is identical to that of the pneumatically actuated μFC. Thermopneumatic actuators previously fabricated at the University of Michigan by Bergstrom, et al. produced nearly 2atm in less than 100ms, with only 20mW of input power (30). Simulations predicted an optimized actuation time of less than 40ms for Bergstrom's elevated heater structure. Such response time and power consumption is competitive with other actuation schemes. Furthermore, the sustained drive pressure of such an actuator falls within a range appropriate for integration with the valve reported herein. Successful thermopneumatic valves of another design have already been demonstrated elsewhere by Zdeblick, et al. (31), (32).

Operating temperature range may be a concern, although a wide variety of actuation liquids with different boiling points are available (24). This may allow adequate freedom to select suitable actuation temperatures for many applications. The author anticipates completion of such a thermopneumatic microvalve as the final step in development of a robust, self-contained, useful microvalve assembly for integration into complete microsystems.

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