

CONTAMINATION REDUCTION USING MEMS-BASED, HIGH-PRECISION MASS FLOW CONTROLLERS

Albert K. Henning¹, James M. Harris¹, Ben Hertzler², Ron Pearlstein²

¹Redwood Microsystems, Inc.

959 Hamilton Avenue, Menlo Park, CA 94025

²Air Products and Chemical, Inc.

7201 Hamilton Boulevard

Allentown, PA 18195-1501

Biography

Albert K. Henning received the A.B. and A.M. degrees from Dartmouth College in Physics in 1977 and 1979, respectively. From 1979 to 1982 he was a device physicist with Intel in Santa Clara, CA. From 1982 to 1987 he was a research assistant at Stanford University, receiving the Ph.D. (E.E.) in 1987. From 1987 to 1995, he was Assistant and Associate Professor of Engineering Science at Dartmouth College. He spent portions of the 1993-94 academic year on sabbatical, as a Visiting Scientist in the Microstructures Technology Laboratory at MIT. In 1996 he joined Redwood Microsystems, where he has served as Program Manager, Wafer Fab Manager, and Director of Technology. He has published over fifty journal and proceedings articles, has received one U.S. patent, and has several patent applications in process. He is a member of Sigma Xi, ASEE, and IEEE.

Abstract

Microfabrication technology has been, over the past twenty years, applied increasingly to the research and development of microelectromechanical systems (MEMS). In the arena of semiconductor gas and liquid distribution and control, MEMS devices include pressure-based mass flow controllers (PMFCs). A significant challenge in the use of MEMS components in this arena is the identification and qualification of MEMS specific materials in the wetted path of the process gas or liquid. In particular, the silicon used in microvalves, sensors, and orifices is in contact

with the process gas or liquid. Contamination reduction through the use of MEMS-based gas distribution devices is expected to be substantial. For use in ultraclean processes, PMFCs must demonstrate their contamination-free nature. In this work, the operation principles and performance advantages of MEMS-based PMFCs, relative to thermal mass flow controllers, are discussed briefly. Subsequently, we present data on dry-down time, particle generation, and corrosion resistance of these PMFCs for a variety of semiconductor-grade gases. The use of corrosion-resistant coatings for the MEMS-based components will also be discussed.

Introduction

Semiconductor processing for integrated circuits manufacturing requires high-precision mass flow control of gases and liquids. Applications of such mass flow control include liquid- and gas-phase processes, for etching and deposition of insulator, metal, and silicon thin films, as well as control of source liquids and gases for ion implantation. These applications impact directly the yield and reliability of semiconductor products. Additional applications, such as control of helium for precision wafer chuck cooling in etch processes, affect yield and reliability less directly, though no less importantly.

Thermal mass flow controllers (TMFCs) have been the means of choice for many of these applications [1], though large-scale pressure-based mass flow controllers (PMFCs) have found use in control of gases for ion implantation [2]. The use of MEMS-

based microvalves for flow control [3], and for use in integrated mass flow controllers [4, 5], has also been discussed elsewhere. No application of such MEMS-based flow control has been made previously to the realm of semiconductor processing, however.

MEMS-Based PMFC Operation Principles

The general principles of pressure-based mass flow control, incorporating MEMS components such as microvalves [6] and pressure sensors [7], has been detailed elsewhere [8-10]. The important features will be summarized here.

As with TMFCs, the flow through the PMFC must be determined from sensor measurements. In this instance, however, the pressure upstream and downstream of a flow element (whether valve or orifice) is related to a calibrated flow model, in order to measure the flow. For gas flow, if there is no viscous loss, then the compressible, subsonic flow model (which holds for either the proportional microvalve or the flow orifice in Figure 1) can be expressed as given in Equation (1) [11]. δ is a parameter related solely to the ratio γ of specific heats (at constant pressure and volume) for the particular gas under control. R is the universal gas constant divided by the molecular weight of the gas. C_d is the coefficient of gas discharge for the flow element. P is the pressure either into, or out of, the flow element. T is the fluid temperature.

$$\dot{m} = \frac{P_{in}}{\sqrt{RT}} C_d A \left(\frac{P_{out}}{P_{in}} \right)^{1/\gamma} \delta(\gamma) \sqrt{\left(\frac{P_{in}}{P_{out}} \right)^{\gamma-1} - 1} \quad (1)$$

Sonic flow for both the valve and the orifice is given in Equation (2). α is a parameter similar to δ . Flow in the microvalve rarely enters the sonic regime. However, the flow area of the valve must be determined either using a loss coefficient model [8], or some other means to relate the structural parameters (inlet area, and membrane-to-inlet gap) to the effective area. On the other hand, flow in the orifice is nearly always sonic; hence, it is most frequently termed the ‘critical flow orifice’. The valve and orifice have different values of C_d , usually between 0.7 and 0.9.

$$\dot{m} = P_{in} C_d A \frac{\alpha(\gamma)}{\sqrt{RT}} \quad (2)$$

Liquid flow for the PMFC is given in Equation (3). C_l is the coefficient of liquid discharge for the flow element. β is the ratio of the orifice or valve inlet diameter to upstream plumbing diameter.

$$\dot{m} = C_l A \sqrt{\frac{2\rho(P_{in} - P_{out})}{1 - \beta^4}} \quad (3)$$

The PMFC is represented schematically in Figure 1. A ceramic or metal substrate provides a modular package for the thermopneumatically-actuated microvalve, a flow orifice, two pressure sensors, and a temperature sensor. Specifications for the PMFCs are shown in Table I. PMFCs have demonstrated maximum flow rates from 1 sccm to 2 slpm. Higher flow rates are also possible.

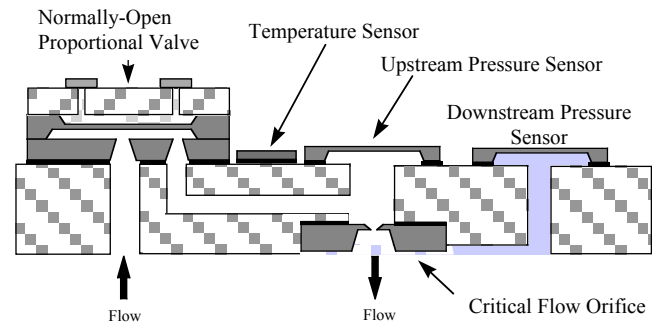


Figure 1: Schematic representation of the PMFC.

Fluid Media:	Gases/Liquids
Maximum Flow Rates:	1, 10, 100, 1000 sccm/ccm
Turndown Ratio:	5:1 (sonic); > 20:1 (subsonic)
Accuracy:	± 1% of reading
Repeatability:	± 0.2% of reading
Resolution:	± 0.2% of reading
Response Time:	1000 ms typical
Inlet Pressure Range:	20 to 50 psig
Maximum Outlet Pressure:	200 Torr
Temperature Range:	0 to 50°C
Power Consumption:	1.5 W typical
Dimensions:	106 mm x 32 mm x 25 mm (with S.S. manifold)

Table I: Specification for PMFC.

Figure 2 diagrams the system behavior, including feedback. In the following discussion, ‘CO’ refers to the flow orifice, and stands for ‘critical orifice’, even though the flow is not critical for liquids, and can be sub-sonic for gases. ‘NO’ refers to the normally-open microvalve. The flow area of the CO is a constant. The effective flow area of the NO is proportional to the NO membrane stroke, which itself is governed by the power supplied to the microvalve. For gas flow through the CO in the

sonic regime, the mass flow is linearly proportional to the absolute pressure upstream of the CO, as shown in Equation (2). The CO thus sets the flow range, consistent with the PMFC specification. Since the CO and NO devices are in series, the intersection of the flow models for each element determines both the sensed pressure P_x , and the mass flow. This principle is shown in Figure 3, where the PMFC module inlet pressure is 50 psia, and the module outlet pressure is 200 Torr. As the NO changes from 100 percent flow to lesser values of flow, the intersection of the NO and CO flow curves falls (the value of P_x falls), and the PMFC flow decreases.

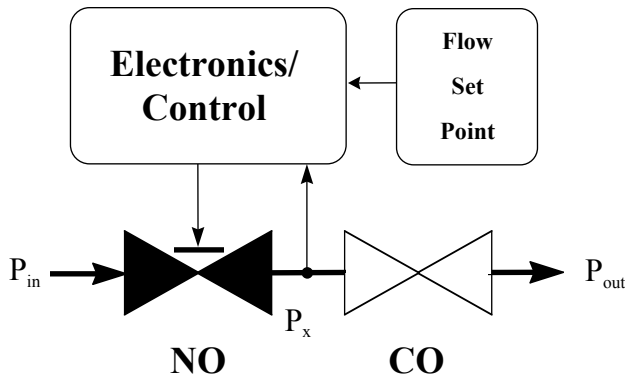


Figure 2: Schematic representation of the compressible flow model for the series combination of a normally-open proportional valve, and a critical orifice.

The CO also sets the flow resolution, as shown in Figure 4 for sonic flow. Taking the derivative of Equation (2) with respect to pressure upstream of the orifice creates the relationship between flow resolution and pressure resolution shown in this figure. For a given pressure sensor resolution and CO area, the minimum flow resolution becomes known. Thus, the CO determines not only the PMFC flow range, but also the minimum flow resolution.

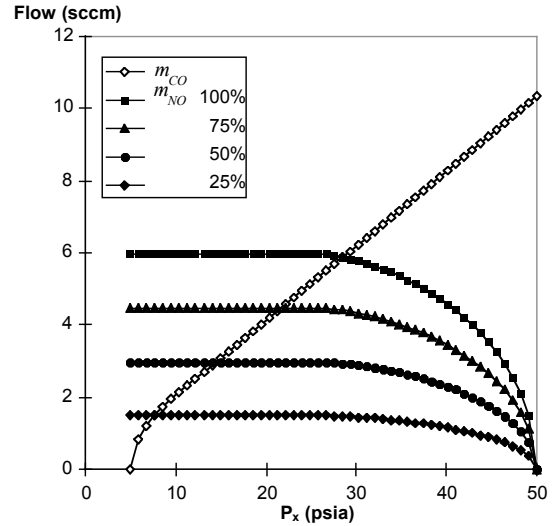


Figure 3: Flow model for a 5 sccm gas PMFC.

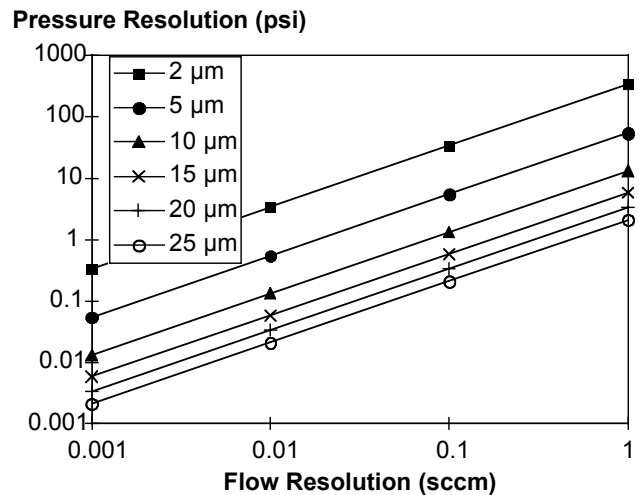


Figure 4: Pressure resolution required to achieve a given flow resolution, versus CO hydraulic diameter.

Implementation and Structure

Important to this work are those aspects of MEMS-based PMFCs related to improvement of the process yield and device reliability of microfabricated integrated circuits. As shown in Table II, a number of attributes of PMFCs may affect IC process yield and device reliability. These attributes derive in turn from both the performance characteristics of PMFCs, as well as their own intrinsic reliability. Also shown in these tables are some of the typical means for assessing the impact of any mass flow controller on IC yield and reliability.

	Attributes Which Affect IC Yield & Reliability	Means to Assess Impact on IC Yield & Reliability
PMFC Performance Characteristics	<ul style="list-style-type: none"> Flow Accuracy (for etch, deposition, or wafer chuck cooling applications) Flow Repeatability 	<ul style="list-style-type: none"> Absolute film thickness; absolute etch rate; abs. wafer chuck temperature Run-to-run parameter variations
PMFC Reliability Characteristics	<ul style="list-style-type: none"> Single crystal silicon diaphragm (no elastomer valve seals) Small internal volumes Small internal wetted surface area Internal surface finishes Minimum wetted materials list; non-ferrous wetted materials; materials compatibility 	<ul style="list-style-type: none"> MTTF for PMFC valve; corrosion resistance to process fluid Particle generation Dry-down time Metal contamination

Table II: Attributes of PMFCs which address the contamination requirements of IC manufacture.

From a performance perspective, IC manufacturers seek increasing accuracy and repeatability in depositing films of decreasing thickness; etching these films; and controlling the temperature of etch and deposition tool wafer chucks during these processes. The control of process gases and liquids is thus critical to the achievement of these goals. TMFCs have limitations which present barriers to such achievement. PMFCs, however, offer excellent accuracy and repeatability, when compared to TMFCs through direct flow measurements, as will be shown shortly. In the end, however, the measure of performance lies with the thickness of deposited films, the rate at which films are etched, and the precision with which wafer temperature is controlled. Currently, Redwood's PMFCs are being evaluated in a SEMATECH sponsored study at HP-Corvallis. Results will be presented as they become available.

From a contamination perspective, PMFCs must not corrode or erode in the presence of IC process gases or liquids. Nor may they generate particles, or create damage or nonuniformities in the etch or deposition processes which lead to process yield loss or device reliability shortfalls. As summarized in Table II, PMFCs are expected to have long MTTF, a result of materials compatibility and the stability of single crystal silicon as a mechanical material in the PMFCs flow control microvalve. Corrosion resistance is imparted to the PMFC design by a combination of a short list of wetted materials, each of which is chemically resistant to corrosion in its own right, while additionally having an outstanding surface finish, and minimal contact surface area. Finally, particle generation and dry-down times are expected to be superior, a consequence of both the corrosion resistance of the wetted materials, as well as the small internal dead volumes and contact surface areas employed.

In this work, two PMFC package, or module, schemes have been investigated. The first, or

prototype, package consists of an alumina ceramic. The bottom interface of the ceramic is machined smooth to provide a viable interface to the Chemraz™ seal rings between the module and the stainless steel manifold. Valve and sensor die are mounted to the top surface of the module, using a Teflon-like polymer as the die attach material. A laser-welded lid encapsulates the valve and sensor die, providing containment against burst pressures, and ensuring the gas or liquid under control does not escape to the environment during a high pressure event.

In the second, or production, package, the ceramic is replaced by Alloy 42 plated with 0.0001" of nickel. The Chemraz™ seal rings are replaced with nickel "C-seals".

In terms of contamination reduction, then, demonstration of materials compatibility between the PMFC materials, and the gases or liquids under control, must be made. The list of wetted materials unique to the PMFC is short:

- silicon (in valve and pressure sensor die)
- die attach material (Teflon-like properties)
- substrate (alumina or nickel)
- stainless steel manifold

As discussed further below, we emphasize that, for the great majority of gases and liquids encountered in semiconductor processing, these materials are essentially inert, and will not corrode or degrade. For those cases where additional corrosion resistance is demanded, coatings of nickel or silicon carbide (SiC) may be applied.

Performance

Some representative performance curves are shown in the following figures. While these results are for 10 sccm PMFCs flowing nitrogen, available (maximum) flow rates range from 1 sccm up to 2000 sccm using pressure-based techniques.

Figures 5-8 show measurements performed on a 10 sccm PMFC, as well as comparable TMFCs. The tests were performed under the SEMATECH specifications SEMASPEC #92071221 B-STD. The test system is based on a calibrated laminar flow element secondary standard, which is itself calibrated to a high-precision, rate-of-rise primary standard [13]. The figures demonstrate the PMFC has superior performance characteristics when juxtaposed with comparable TMFCs. It also has

adequate response time for semiconductor process equipment applications.

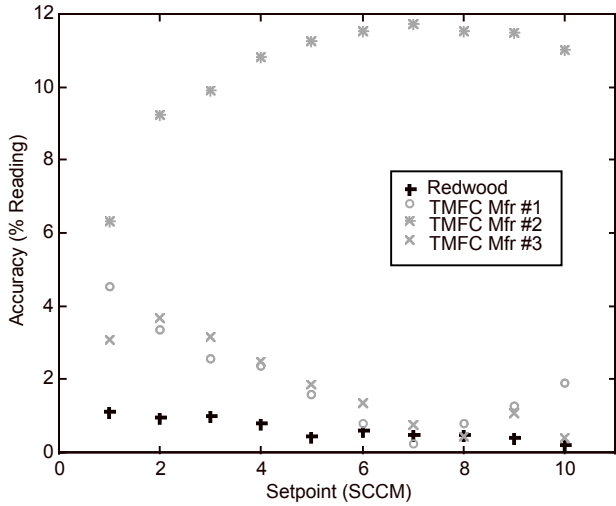


Figure 5: MFC accuracy comparisons for PMFC (this work) and TMFCs (other units).

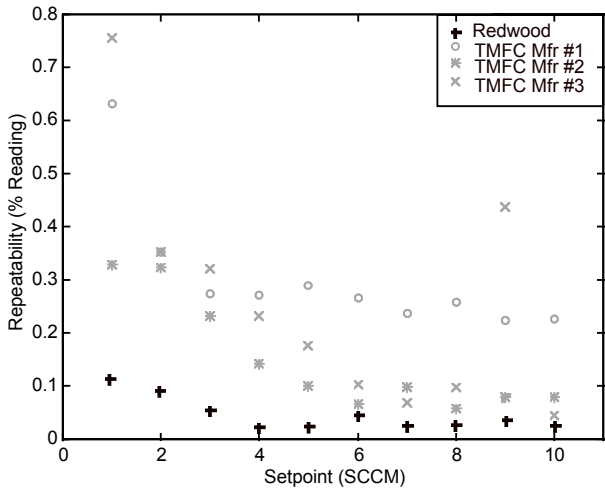


Figure 6: MFC repeatability comparisons for PMFC (this work) and TMFCs (other units).

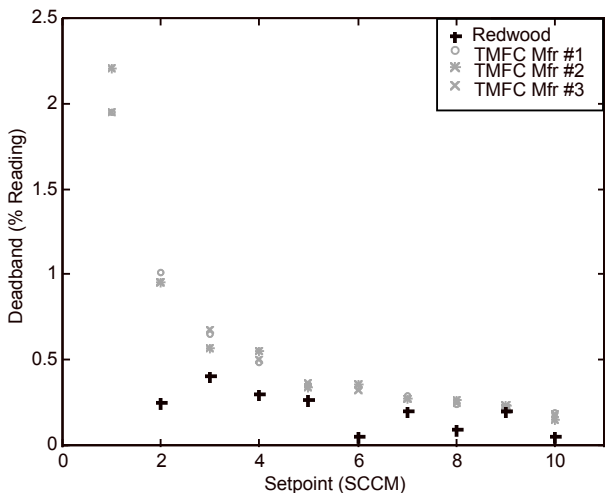


Figure 7: MFC deadband (resolution) comparisons for PMFC (this work) and TMFCs (other units).

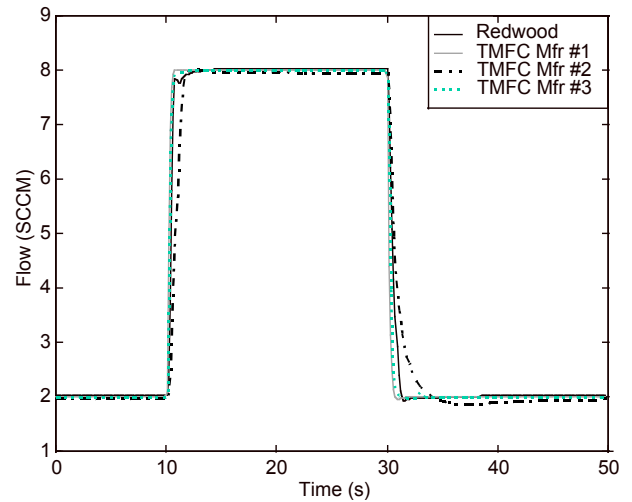


Figure 8: MFC response time comparisons for PMFC (this work) and TMFCs (other units).

Contamination Reduction: Previous Work

While the PMFCs explored here are the first micromachined flow controllers designed specifically for semiconductor processes, previous work has established the viability of the PMFC wetted materials upon exposure to the range of liquid and gaseous chemicals used in semiconductor manufacture.

PMFCs can be specified with several different choices of wetted materials depending upon the chemical resistance required. In the standard product, silicon, alumina or nickel and a proprietary PTFE type polymer are in the wetted path. The corrosion resistant product has a silicon carbide type protective layer over the silicon. In either configuration a variety of manifolds and O-ring sealing materials can be specified, such as stainless steel or nickel or a corrosion resistant material such as Chemraz™, Kalrez™, Kel-F™ or Teflon™. The corrosion resistance of the alumina or nickel package is excellent and well known; the properties of the proprietary polymer approximate Teflon™ from a chemical resistance standpoint, is also excellent [14], as discussed in more detail below.

The corrosion resistance of silicon is quite good by itself. As has been documented extensively in the literature [15-19] very few chemicals attack silicon

actively. In general, no organic solvents or organic acids will attack silicon or alumina. Conversely, several organic and inorganic bases will attack silicon in specific temperature and concentration ranges [15].

The halogens represent a special class of materials. Aqueous or gaseous mixtures of HF, HCl, HBr or HI will in general not etch silicon. However mild pitting of the silicon may be observed in HBr or HI under certain limited conditions [19].

It is well documented that atomic fluorine will attack silicon at room temperature; for instance if the process gas is XeF₂. Interestingly, atomic fluorine will not attack SiO₂; even native SiO₂ on silicon is sufficient to stop the reaction [15]. Regardless, the corrosion resistant MFC is recommended for use with XeF₂.

Chlorine gas or atomic chlorine have not been reported to etch silicon without the assistance of a plasma. Only in combination with fluoride ions, (M)F⁻, have strong oxidizing mineral acids, e.g. HNO₃, been reported to etch silicon. As is well known, silicon is impervious to aqua regia (HCl + HNO₃) and Piranha (H₂SO₄ + H₂O₂) cleaning solutions. The PMFC in this work, with the nickel package, is well suited for processes requiring HF, vapor or liquid, as well as HCl or chlorine. Solutions containing HNO₃ must be avoided when nickel is present.

The moisture content of the gas stream can be a critical factor in the corrosion resistance of most materials. Typical stainless steel passivity is strongly dependent upon the amount of H₂O present, declining sharply as water content increases above 1 ppm in the presence of halides [20-22]. However, silicon corrosion resistance is virtually unaffected by moisture content. With the specific exceptions of ammonia and atomic F, silicon will be unaffected by the gas and the relative water content of the stream, even into the liquid state. In the case of ammonia vapor or fluid

streams, there may be a mild pitting of the silicon surface [19].

It has been reported [23, 24] that Nickel can promote the dissociation of certain hydrides, e.g. SiH₄, at temperatures as low as 50°C. In addition, there is reason [24] not to use nickel with carbon monoxide (CO).

In applying PMFCs for liquid control, high pH fluids (higher than 9) require some precautions. Mixed acids containing an oxidizing component and a reducing component require corrosion-resistant coatings; obvious examples of mixed acids are HF plus any of the oxidizing acids, e.g. HNO₃. Organic bases with properties similar to KOH are incompatible with silicon micromachined devices. Nickel and some stainless steels will not be acceptable in some of these mineral bases and various mixtures of acids. Ceramic will be acceptable except in solutions containing HF or strong bases. The literature on use of organic acids is sketchy; caution should be used here as well. All of the materials in the system, including the manifold and the piping must be considered when looking at corrosion resistance.

When its natural properties are not enough, thin film coatings may be used to enhance silicon's corrosion resistance [25]. Silicon nitride provides excellent resistance to many process gases, though it is still vulnerable to fluorine-based etch chemistries [26]. In the MEMS community, SiC films have received the greatest scrutiny in the past four years [27]. Compared to silicon, SiC provides increased mechanical hardness, increased chemical resistance, high thermal conductivity, electrical insulation, and a low coefficient of friction [28]. These attributes are especially important in and beneficial for flow control applications for IC microfabrication.

Silicon has shown excellent erosion resistance in fluidic applications [28]. SiC was used to coat microfabricated silicon atomizers for fuel atomizer applications (a more difficult environment for

erosion when compared to that found in semiconductor process gas and liquid control). Erosion tests were performed using a Military Standard Mil-C-7024D Type II test fluid, for 30 hrs, with 150 psia inlet pressures, at a flow rate of 500 cm³/min, with controlled 'spiking' of the liquid flow with 0-5 μm sized hard grit. No degradation in a 560 nm SiC coating was observed, nor was the flow affected substantially.

Particles may be generated via a variety of mechanisms [12]. The principle generation mechanisms are:

- Gas-to-particle conversion, occurring in the dead volume of MFCs, typically under stagnant flow conditions;
- Evaporation-condensation, occurring most frequently due to sidewall contact between the reactants and the MFC wetted materials;
- Chemical disintegration, or corrosion, caused by long-term interaction between process fluids and MFC materials;
- Mechanical disintegration, caused by long-term, part-to-part contact;
- Erosion, caused by the mechanical interaction between process fluids and MFC materials.

Evaporation-condensation particle generation mechanisms are expected to be most difficult for liquid processes, especially where organic precursors (for instance, in copper interconnect deposition processes) may be easily volatilized at relatively low temperatures.

Contamination Reduction: Expectations for MEMS-Based PMFCs

The important material and structural features of MEMS-based PMFCs have been shown schematically in Figure 1. These features lead to expectations for process yield and device reliability improvements, relative to the TMFC. *Small size:* As a direct replacement for conventional TMFCs, the PMFC with steel manifold is roughly one inch by 2 inches by 3 inches high, including electronics. In module form only, the size decreases by more than a factor of two. If remote electronics are utilized, the vertical dimension shrinks to one-half inch. *Higher performance:* The use of 16 bit A/D for the pressure sensor, and 16 bit D/A for the valve driver, enables very high accuracy and resolution, which exceeds the SEMATECH specifications. *Materials compatibility:* The

wetted path in the PMFC is comprised of silicon, alumina (ceramic) or nickel, appropriate die attach materials, and the stainless steel manifold. As such, it facilitates flow of all semiconductor processing fluids, save those which contain atomic fluorine, or other materials which etch silicon. *Lowered defect generation:* MFC-generated particles are understood to derive from the number of sealing surfaces, the internal surface roughness, and the internal volume. Compared to TMFCs, the number of seals is reduced, and the internal dead volume is decreased by a factor of two to ten (see Table III).

Previous work with silicon microvalves explored the materials compatibility of the valves, their die attach materials, and their ceramic packages with mixtures of the fluorine-based refrigerant R-134a and lubricants [29]. The ceramic packages had a composition and surface finish identical to those used in PMFCs. None of these materials suffered measurable degradation upon exposures for three-to-six months, at elevated temperatures.

The reductions in volume and internal surface area are expected to be instrumental in reducing generated particles, and reducing the exposure to possible corrosion. A ten-fold reduction in internal volume, and in contact surface area, will reduce the likelihood for particle generation, or corrosion, by a similar factor.

The flow rates through these reduced-size devices also lead to expectations for improvements in particles generated by mechanical interaction between the process fluids and the PMFCs flow passages. For instance, the diameter of the approach tube to the critical flow orifice is 1 mm, while the orifice diameter varies from 1 μm to 318 μm (for 1 sccm to 2000 sccm flow rates). As has been shown in earlier work [12], Reynolds numbers below 6000 generate less than 1 particle per cubic foot of process gas for a 0.030" stainless steel tube. Given that PMFC Reynolds numbers reach a maximum of 6000 in the critical orifice (for 2000 sccm flow rates), and are much less than this value elsewhere in the system (and for lower flow rates), the PMFC is expected to generate far less than 1 particle per cubic foot of process gas.

	# of Welds	# of Seals	Internal Volume	
1990	10	0	38 cc	
1995	6	8	31 cc	
1996	3	12	22 cc	
1997	0	10	2 cc	Redwood Phase I
1998	0	0	<1 cc	Redwood Phase II

Table III: Functional comparison of present and future integrated gas sticks/panels (after [30]).

The contamination properties of silicon diaphragm pressure sensors have been discussed elsewhere [31]. In essence, such pressure sensors are robust in most harsh environments, except when the silicon itself is subjected to chemical attack, as discussed in the previous section.

Critical orifices have been studied in terms of their effect on particles. Orifices may trap or generate particles, or they may be particle-neutral [12]. The use of silicon as the orifice material creates a tough surface which resists both particle trapping (which creates the opportunity for subsequent particle re-generation) and particle generation.

The effects of stainless steel used in ultra-high purity gas distribution systems on contamination have been well-documented [32-36]. Materials compatibility, in the form of chemical or corrosion resistance, is an important consideration. PMFCs use standard stainless steel manifolds, though opportunities exist to reduce exposure to even this minimal amount of metal.

In most MFCs, metal contamination and generation of metal-based particles is also a frequent occurrence. Surface finish is an important component of resistance to this form of contamination. Surface finish for the PMFC ceramic package is 16 Ra, while that of the nickel package is 20 Ra. Improvements in surface finish are available through commercial machine shops, especially for the nickel package (down to 5 Ra).

Results: PMFC Corrosion Resistance

In testing at Redwood, no reaction of either the silicon or the polymeric die attach material was noted subsequent to exposure to a 50% aqueous HBr solution after 45 days immersion.

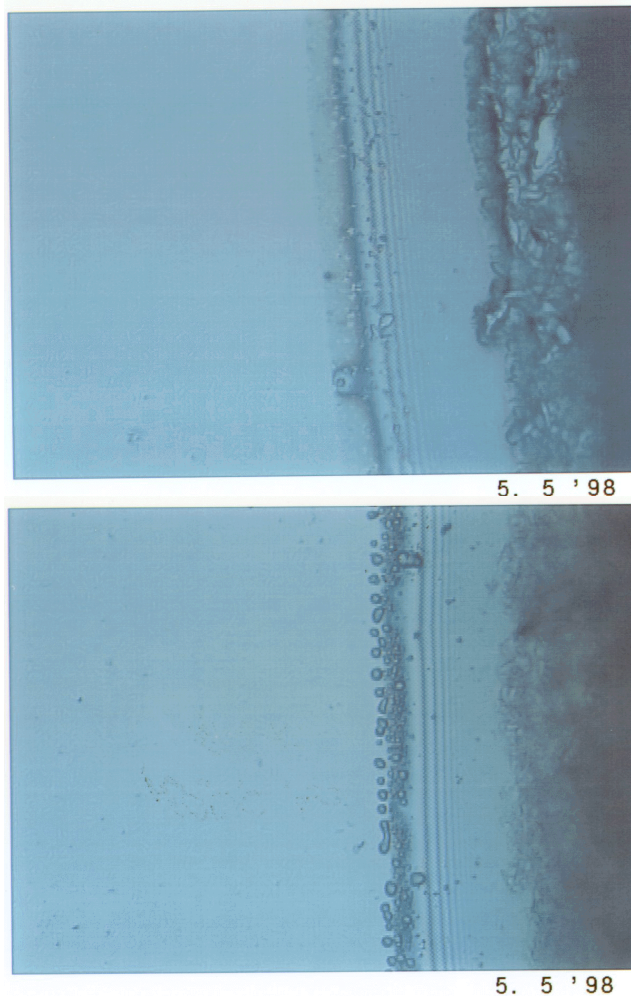


Figure 9: Nomarski photomicrographs of silicon surfaces coated with die attach material. Magnification is 400x. Bare silicon is on the left of each picture, while the die attach material is coated on the right side. Upper: prior to exposure to concentrated HBr. Lower: after exposure to concentrated HBr.

The die attach material was also measured quantitatively for changes in its mechanical properties following similar exposure to HBr. The results are shown in Table IV. The measurements were obtained using an Instron Series IX Automated Materials Testing System.

	Tensile Strength (psi)	Elongation at Break (%)	Young's Modulus (psi)	Load @ Max Load (lbf)
HBr Mean	1550	8.8	57800	6
HBr Sigma	100	0.7	6550	0.85
Control Mean	1310	9.2	34500	3.9
Control Sigma	40	0.6	5200	0.8

Table IV: Results of mechanical behavior tests of PMFC die attach material, following exposure to HBr.

Results: Cycling Studies

A variety of factors lead to the estimate of mean time to fail (MTTF) for PMFCs. As with TMFCs, the essential moving part is the valve. In PMFCs, then, the long-term reliability of this microvalve must be

considered. *A priori*, silicon as a single-crystal material is expected to demonstrate no significant wear-out mechanisms, such as mechanical deformation or creep. Long-term studies of Redwood's microvalves have been carried out, both internally and in Japan. For microvalves identical to those used in PMFCs, with silicon diaphragm diameters of 4 mm and thicknesses of 50 μm , dozens of microvalves have been tested for periods in excess of a year, for over ten million cycles, with no observed breakage or degradation in performance.

Results: Particle Generation and Dry-Down Time

Measurement studies are currently underway at Air Products, and at another semiconductor equipment supplier, to determine the particle generation and dry-down time characteristics of PMFCs.

Conclusions

PMFCs created using MEMS-based components offer a number of demonstrated and anticipated benefits in terms of contamination reduction for IC processing. These include:

Materials compatibility:

- Silicon is superior for most gases and liquids of interest, including water vapor in conjunction with HF/HBr/HCl. In the event that additional corrosion resistance is required, Si₃N₄ or SiC films may be deposited on the silicon, as appropriate;
- PTFE-based die attach materials have been demonstrated to be mechanically robust in harsh environments, specifically HBr;
- Available package substrate materials (either alumina or nickel) are compatible with all but a very few of gases and liquids encountered in IC manufacture;
- Stainless steel manifolds provide a compatible interface with the remaining portions of gas delivery systems of choice.

Particle generation:

- A ten-fold reduction in dead volume yields a commensurate reduction in particles generated via reaction of reactions in these volumes under stagnant conditions;
- The simultaneous reduction in contact surface area further reduces particles generated via reaction between reactants and sidewall materials;
- Additional tests related to particle generation are underway.

Dry-down:

- As with particle generation, the reduction in contact surface area should result in a reduction in

overall system dry-down times. Again, tests are underway to demonstrate this reduction.

Erosion:

- MEMS components have been demonstrated to have substantial erosion resistance, even under high flows in harsh environments. Given the relatively low Reynolds numbers encountered in MEMS-based PMFCs, erosion is expected to play little role in performance degradation or IC process contamination.

Reliability:

- Single-crystal silicon has been demonstrated to have outstanding MTTF characteristics (tens of millions of cycles, with no degradation), when used as the mechanical component in a PMFC microvalve, and compared to the electromagnetically-actuated valves found in standard TMFCs.

Acknowledgments

The authors acknowledge gratefully the efforts of Mark Eckert, and use of the facilities of Air Products, Inc. in performing some of the HBr corrosion resistance evaluations of the die attach material. At Redwood, Errol Arkilic and Ruben Leni provided much of the MEMS-based PMFC performance data, while Bob Kozen and Les Lilly contributed to the contamination studies and evaluation. Some of the work at Redwood Microsystems was supported by the Defense Advanced Research Projects Agency through Contract DAAL01-94-C-3401. Mark George of Deposition Sciences (formerly of Air Products) led the early contamination collaboration efforts. John Fitch of Xerox Corp. (formerly of Redwood) made major contributions to the MFC development and packaging efforts.

References

- [1] G. Chizinsky, "Recent advances in mass flow control." *Solid State Technology*, p. 85 (September, 1994)
- [2] generic reference on MKS PMFC applications, esp. to ion implantation
- [3] P. W. Barth, "Silicon microvalves for gas flow control." In Proceedings, *Transducers '95 (1995 Int'l. Conf. Sol. State Sens. and Act.)*, pp. 276-279 (IEEE, Piscataway, NJ, 1995); and references therein.
- [4] J. Robertson, "An electrostatically-actuated integrated microflow controller." Ph.D. dissertation, U. Michigan, 1996
- [5] M. Esashi, S. Eoh, T. Matsuo, and S. Choi, "The fabrication of integrated mass flow controllers." In Proceedings, *Transducers '87*, pp. 830-833 (Inst. Elec. Eng. Japan, 1987); and, S. Shoji, B. Van der Schoot, N. de Rooij, and M. Esashi, "Smallest dead volume microvalves for integrated chemical analyzing systems." In Proceedings,

Transducers '91 (1991 Int'l. Conf. Sol. State Sens. and Act.), pp. 1052-5 (IEEE, Piscataway, NJ, 1991)

[6] M. J. Zdeblick and J. B. Angell, In Proceedings, *Transducers '86 (1986 Int'l. Conf. Sol. State Sens. and Act.)*, pp. 827-829 (IEEE, Piscataway, NJ, 1986); M. Zdeblick, "A planar process for an electric-to-fluidic valve." Ph.D. dissertation, Stanford University, June, 1988; and, M. Zdeblick, "Integrated, microminiature electric-to-fluidic valve and pressure/flow regulator." U.S. Patent 4,821,997 (1989).

[7] O. N. Tufte, P. W. Chapman, and D. Long, "Silicon diffused-element piezoresistive diaphragms." *J. Appl. Phys.* **33**, p. 3322 (1962)

[8] A. K. Henning, "Microfluidic MEMS for semiconductor processing." In Proceedings, *Intl. Conf. on Innovative Systems in Silicon*, pp. 340-349 (IEEE, Piscataway, NJ, 1997). Accepted for publication in *IEEE Trans. Components, Packaging, and Manufacturing Technology*

[9] A. K. Henning, "Microfluidic MEMS." In Proceedings, *IEEE Aerospace Conference*, Paper 4.906 (IEEE Press, Piscataway, NJ, 1998)

[10] J. S. Fitch, A. K. Henning, E. B. Arkilic, J. M. Harris, "Pressure-based mass-flow control using thermopneumatically-actuated microvalves." In Proceedings, *Sensors and Actuators Workshop*, pp. 162-165 (Transducers Research Foundation, Cleveland, OH, 1998)

[11] Frank M. White, Fluid Mechanics. (2nd edition, McGraw-Hill, New York, 1986)

[12] G. Kasper and H. Y. Wen, "Particles in ultrapure process gases," p. 301; B. J. Tullis, "Particle contamination by process equipment," p. 410; M. Naggar, "Contamination control in microelectronic chemicals," p. 370. In Handbook of Contamination Control in Microelectronics, D. L. Tolliver, ed., (Noyes Publications, Park Ridge, NJ, 1988)

[13] E. B. Arkilic, M. A. Schmidt, and K. S. Breuer, "A technique for high resolution mass flow measurements at atmospheric pressures." *Experiments in Fluids* (1997)

[14] S. Lau, *et al.*, "Performance of diaphragm valves in chlorine." *Solid State Technology*, p. S21 (June 1997)

[15] K. Williams and R. Muller; "Etch rates for micromachining processing," *IEEE J. Microelectromech. Syst.* **5**, pp.256-269 (1996)

[16] H. Jansen, *et al.*, "A survey on the reactive ion etching of silicon in microtechnology." *J. Micromech. Microeng.* **6**, pp.14-28 (1996)

[17] G. Eriksen and K. Dyrbye, "Protective coatings in harsh environments." *J. Micromech. Microeng.* **6**, pp. 55-57 (1996)

[18] H. B. Pogge (ed.), Electronic Materials Chemistry (Marcel Dekker, NY, 1996)

[19] P. Walker, W. Tarn (eds.), CRC Handbook of Metal Etchants (CRC, Boca Raton, FL, 1991)

[20] E. Flaherty, *et al.*, "Reducing the effects of moisture in semiconductor gas systems." *Solid State Technology*, p. 69 (July 1987)

[21] S. M. Fine, *et al.*, "The role of moisture in the corrosion of HBr gas distribution systems." *J. Electrochem. Soc.* **142**, p. 1269 (1995)

[22] P. M. Bhadha, *et al.*, "Joule-Thomson expansion and corrosion in HCl systems." *Solid State Technology*, p. S3 (July 1992)

A. K. Henning, J. M. Harris, R. Pearlstein and B. Hertzler, "Contamination reduction using MEMS-based, high-precision mass flow controllers." In *Proceedings, SEMICON West Symposium on Contamination Free Manufacturing for Semiconductor Processing* (SEMI, Mountain View, CA, 1998)

[23] T. Ohmi, "Corrosion-free Cr₂O₃ passivated gas tubing system for specialty gases." *Solid State Technology*, p. S18 (October 1995)

[24] P. B. Henderson, *et al.*, "Cylinder package effects on the purity of electronic specialty gases." *Solid State Technology*, p. S5 (June 1997)

[25] A. F. Flannery, *et al.*, "PECVD silicon carbide for micromachined transducers." In Proceedings, *Transducers '97 (1997 Int'l. Conf. Sol. State Sens. and Act.)*, pp. 217-221 (IEEE, Piscataway, NJ, 1991)

[26] Patent application from Lawrence Livermore National Laboratories and Redwood Microsystems, in progress.

[27] A. Klumpp, U. Schaber, H. L. Offereins, K. Kuhl, and H. Sandmair, "Amorphous silicon carbide and its application in silicon micromachining." *Sensors and Actuators A* **41-42**, pp.310-316 (1994)

[28] N. Rajan, C. A. Zorman, M. Mehregany, R. DeAnna, and R. J. Harvey, "Effect of MEMS-compatible thin-film hard coatings on the erosion resistance of silicon micromachined fuel atomizers." In Proceedings, *International Conference on Metallurgical Coatings and Thin Films* (San Diego, 1998); R. J. Harvey, "Progress on comparison study of the erosion of conventionally machined and microlaminated pressure-swirl atomizers." In Proceedings, *International Gas Turbine and Aeroengine Congress and Exhibition* (1997).

[29] A. K. Henning, J. S. Fitch, D. J. Hopkins, Jr., L. Lilly, R. Faeth, E. Falsken, and M. J. Zdeblick, "A thermopneumatically actuated microvalve for liquid expansion and proportional control." In Proceedings, *Transducers '97 (1997 Int'l. Conf. Sol. State Sens. and Act.)*, pp. 825-828 (IEEE, Piscataway, NJ, 1997)

[30] J. Cestari, D. Laureta, and H. Itafuji, "The next step in process gas delivery: a fully integrated system." *Semiconductor International*, pp. 79-87 (January 1997)

[31] G. Bitko, D. J. Monk, T. Maudie, D. Stanerson, J. Wertz, J. Matkin, and S. Petrovic, "Analytical techniques for examining reliability and failure mechanisms of barrier coating encapsulated silicon pressure sensors exposed to harsh media." In Proceedings, *Micromachined Devices and Components II* (International Society for Optical Engineering, Bellingham, WA, 1995; R. Roop and K. Chau, eds.), volume 2882, pp. 248-258 (1996)

[32] S. M. Fine, *et al.*, "Optimizing the UHP gas distribution system for a plasma etch tool." *Solid State Technology*, p. 69 (March 1996)

[33] H. Kobayashi, "How gas panels affect contamination." *Semiconductor International* **17**, p. 81 (September 1994)

[34] M. George, D. Bohling, W. Baley, T. Del Prato, K. Harlan, and C. Magnella, "Minimizing system contamination potential from gas handling." *Semiconductor International* **16**, pp. 98-104 (July 1993)

[35] O. Hallberg, *et al.*, "Recent humidity accelerations, a base for testing standards." In Proceedings, *Quality and Reliability International Conf.* **7**, pp. 169-180 (1991)

[36] M. A. George, *et al.*, "Compatibility of 316L stainless steels and tungsten hexafluoride." Presented at: Tungsten and Other Refractory Metals for ULSI Applications, Dallas, TX, October 1990.

