

Thermopneumatically actuated microvalves and integrated electro-fluidic circuits

Mark J. Zdeblick, Rolfe Anderson, Joe Jankowski, Bob Kline-Schoder, Lee Christel, Robin Miles, Walter Weber
Redwood Microsystems, Inc.
1030 Hamilton Court
Menlo Park, California

Abstract

An electro-fluidic multi-chip module (E/F MCM) technology incorporating microfabricated sensors and actuators has been developed for diverse research and commercial applications. The first E/F MCM incorporates a thermopneumatically actuated valve, a piezoresistive pressure sensor, and feedback electronics to create a complete electronically programmable pressure regulator. Both the operation of the valve and the pressure regulator are described. By routing chemicals between various reaction vessels and detectors, future E/F MCM's may be used to process chemical information in a manner analogous to the way computers manipulate electronic information.

Introduction

The idea of using silicon as an electro-fluidic substrate for chemical analysis evolved in the laboratory of Professor James B. Angell at Stanford University in the late 1970's and early 1980's¹. In that same laboratory, other microfluidic devices were built^{2,3,4} and the first complete integrated microvalve was produced^{5,6}. Later, the concept of using silicon as a substrate for multiple valves, electronics, and detectors to perform chemical operations as complex as DNA analysis evolved^{7,8,9}. Others have built micropumps^{10,11,12}, microvalves with a variety of actuation mechanisms^{13,14,15}, flow sensors¹⁶, and electrophoretic separators¹⁷. A nice review paper documenting the surge of interest in microfluidics has recently been published¹⁸. This paper focusses on thermopneumatically-actuated microvalves and their role in integrated circuits that have fluidic as well as electronic elements, enabling chemical instrumentation of increasing complexity but decreasing size.

Principles of Operation

An Electro-Fluidic Multi-Chip Module (E/F MCM) may be comprised of five functional groups: 1) a fluid management system including flow channels, valves, mixers, and reaction chambers; 2) one or more sensors for measuring any of a number of parameters, including pressure, flow, pH, optical properties, and electrical properties; 3) separation systems for refining chemical products from reagents; 4) interface and control electronics; and 5) a multi-chip module manufacturing technique that integrates these electronic and fluidic functions. In the example of an E/F MCM described herein, the fluid management system is comprised of a thermopneumatic-microfabricated valve, an orifice, input and output ports, and a flow channel connecting them; the sensor is a pressure sensor; the separation system is omitted; the electronics are an analog feedback control circuit that provide pressure regulation; the MCM manufacturing technique is a direct extension of standard PC board manufacturing methods used in the IC industry.

Operation of the microfabricated valves

The key component of a microfabricated thermopneumatically actuated valve is a silicon diaphragm made by etching a precisely controlled recess in a silicon substrate. A Pyrex wafer with a resistor fabricated onto its surface covers the liquid-filled recess, creating a hermetically sealed control cavity. Dissipating energy in the resistor heats the liquid, increasing the pressure inside of the sealed vessel, increasing the volume of the vessel. This pressure is used to modulate the flow of either gas or liquid in the adjacent channel. In a Normally Open (NO) configuration (Figure 1), the edges of the diaphragm are fixed in space. Increasing control cavity volume pushes the silicon membrane towards the valve seat. In a Normally Closed (NC) configuration (Figure 2), a point on the diaphragm is fixed in space. Increasing control cavity volume causes the edges of the diaphragm to move away from the valve seat. The devices shown in Figures 1 & 2 are both designed for gaseous operation. Similar devices optimized for liquid operation prevent the liquid from contacting the electrical connections.

Since the NO and NC valves are made using a similar process, only the Normally Closed process is illustrated in Figure 3.

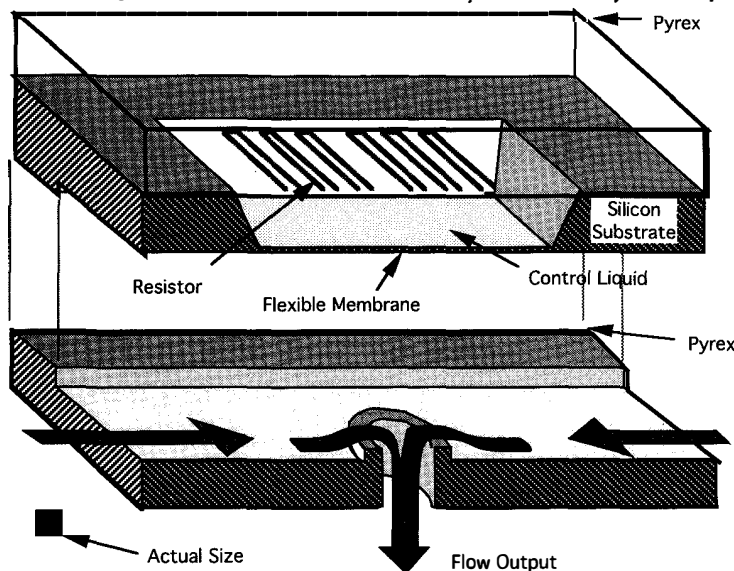


Figure 1 : The Normally Open thermopneumatically actuated Fluistor™ Microvalve features a liquid filled cavity which when heated, forces a silicon diaphragm outward over valve seat.

These drawings represent an illustrative cross-section through a single device; typically nearly one hundred devices are made simultaneously on a single wafer. Figure 3a) shows the top pyrex wafer after one side has been coated with 1000Å Pt and patterned into a resistor. It also shows an electric / fluidic via through the pyrex. Figure 3b) shows the first step in the processing of the silicon wafer. Here a thick (1.2 µm) layer of SiO₂ is grown in a humid environment at 1100°C and photolithographically patterned as shown. Figure 3c) shows the silicon wafer after it has been etched in a hot (80°C) solution (33% w/w) of KOH, defining the control cavity and membrane. A second lithography and etch is performed, leaving a 40 µm thick silicon membrane and sealing ring, as shown in Figure 3d). The bottom wafer, shown in Figure 3e) is made of pyrex, which is ultrasonically drilled and polished. Figure 3e) shows the three wafers after they are bonded together. After being filled with the control liquid, Figure 2) illustrates how the cap seals the liquid in the control cavity. The complete valve, pictured in Figure 4, measures 6.3 x 6.6 x 2.0 mm.

One of the advantages of thermopneumatic actuation over other micro-actuated mechanisms — such as thermal bimorph, electrostatic or piezoelectric — is the independence of the actuation and the object being actuated. In this case, the object being moved is a diaphragm, whose flexibility, chemical inertness, and other properties can be completely optimized independently of the actuator. The actuator, i.e., the cavity full of gas and or liquid, can also be optimized independently from the membrane for a particular application by adjusting cavity shape, boiling point of the control liquid, quantity of both gas and liquid molecules, and other properties. The dynamic range of the device can thus be quite broad: vacuum pressures to hundreds of atmospheres; gas, liquid, and corrosive fluids; ultra-low (µl/min of gas) to industrial (10's slpm) flow rates. Valves of this type have been built that control liquids at 204 bar (3000 psig). Flow rates of similar valves have been built that exceed 15 slpm of N₂ at 7 bar (103 psid).

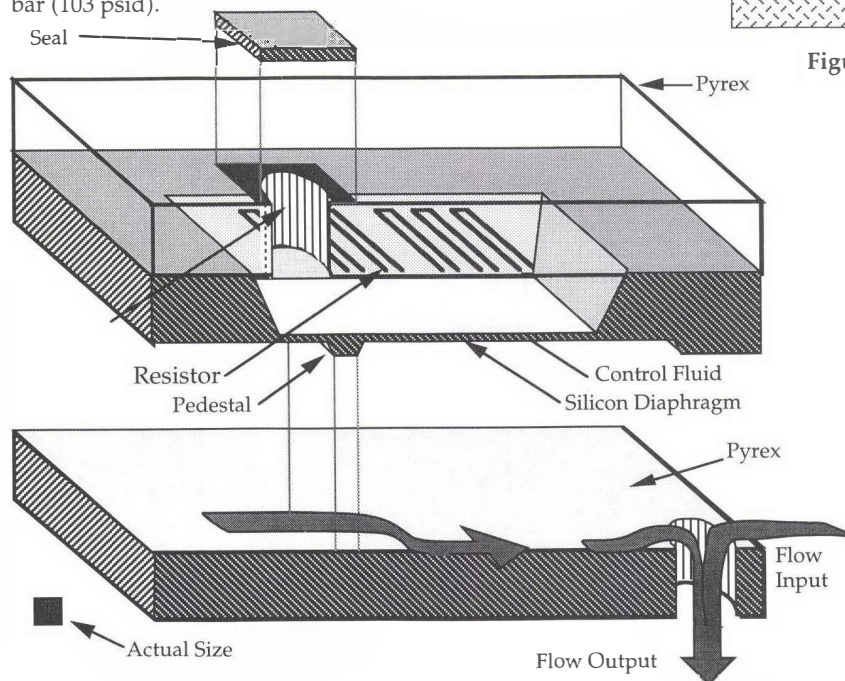


Figure 2: Normally Closed thermopneumatically actuated Fluistor™ Microvalve features a liquid filled cavity which when heated, flexes a silicon diaphragm forcing the valve cover to lift off the valve seat.

Compared to other microvalve technologies, thermopneumatic actuation exerts tremendous force through a long stroke. The maximum displacement of a NO valve is about 50 µm. Since the upper portion of a NC valve rotates, its maximum vertical displacement is about 150 µm. A thermopneumatic actuator is capable of providing significant force throughout this displacement — over 20 N (200 bar x 6900 N/m²/bar x 1.6 x 10⁻⁵ m²) through a 50 µm stroke has been demonstrated in the case of the NO valve.

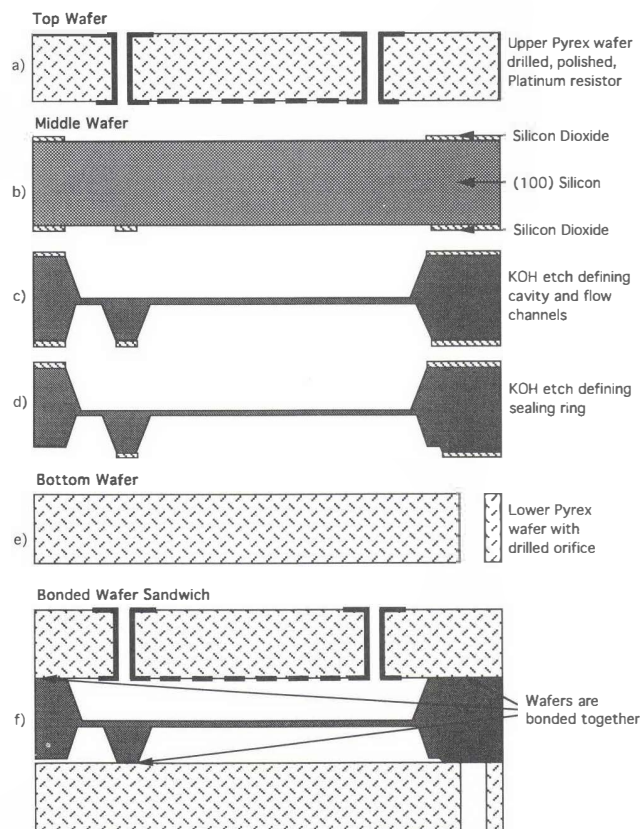


Figure 3: Process flow of a normally closed valve

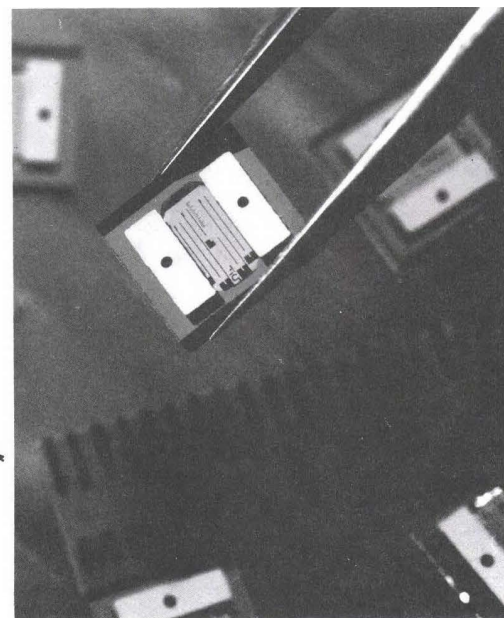


Figure 4: Photograph of a Fluistor™ microvalve.

This long stroke allows a thermopneumatically-actuated microvalve to control high flow rates and pressures relative to other microvalve technologies. The high operating pressure capability not only extends its application to high pressure applications, it allows the use of significant force for preventing leaks across the valve seat. Long stroke and smooth actuation allows stable control of flow rate over a wide dynamic range: single valves are capable of controlling stable flow rates continuously from 0.001 sccm to 10 slpm. Flow v. pressure at a variety of ambient temperatures and flow v. applied power at a variety of supply pressures are shown in Figure 5a & 5b (Normally Open) and Figure 6a & 6b (Normally Closed).

Pressure Sensor

The sensor used in this E/F MCM is a piezoresistive, silicon membrane device (SenSym, Milpitas CA). The silicon sensor is encapsulated in a plastic "button" package that facilitates PC-board mounting. The sensor has a linearity and accuracy of about 1%. Linearity and precision may be extended to about 0.1% through the use of well-established digital compensation techniques that correct for nonlinearities in the sensor caused by temperature and pressure variations.

Feedback electronics

The analog control circuit provides the necessary power signal for the pressure sensor and conditions the sensor output appropriately for the implementation of the pressure control system. The analog pressure control system calculates a valve control signal as the sum of three terms; one term proportional to the pressure error, one term proportional to the integral of the pressure error, and one term proportional to the filtered derivative of the pressure error. Therefore, the controller is a classical PID (proportional-integral-derivative) controller. Since the system is non-linear, however, the gains have been set to maximize the time of response without unduly sacrificing transient overshoot or steady-state performance. The selected gains result in a controller which closely resembles a modified-bang-bang controller: bang-bang control until the operating point is within range of linear behavior at which point the PID control behavior dominates. The output of the control electronics is a 0 - 15 Volt signal which serves as the command to the valve chip drive electronics. As the valve membrane deflects due to changes in the voltage applied to the heater, the flow through the valve will vary, changing the sensed pressure, which is used by the controller to continually adjust the valve heater voltage until the sensed pressure matches the desired pressure.

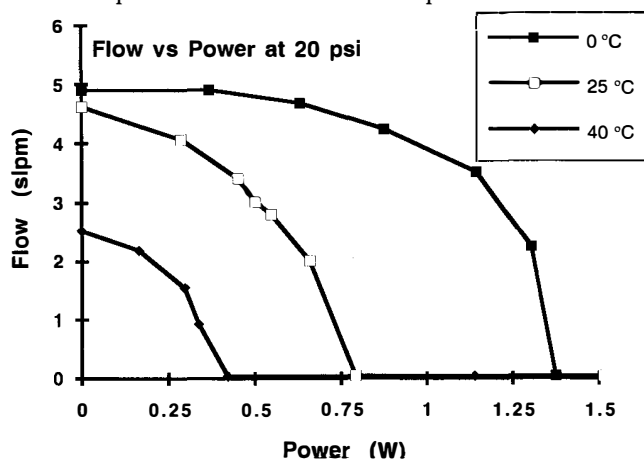


Figure 5a: Flow vs. Power for a Normally Open valve at 20 psig supply pressure and a variety of ambient temperatures.

E/F MCM Manufacturing technique

Multi-Chip Module is a widely known manufacturing technique in the electronics industry for integrating electronic chips together at the die-level to reduce the volume of the package and improve the performance of the system. Non-packaged silicon die are bonded to a substrate, which provides electrical connections. In this way, a system designer has the flexibility of purchasing functionally specialized components or systems from different vendors and integrating them in an optimal way. Lower system cost, smaller size, greater reliability, higher frequency response, and electro-optic transduction are all attributes that make MCMs higher value-added products.

Here, we demonstrate an MCM for electro-fluidic circuits, i.e., consists of fluidic and electronic elements. The construction and choice of materials of the fluidic element has an influence on the performance of the valve in the module. Since currently available micromachined valves are thermal devices, the thermal mass and heat conduction of the manifold materials plays an important role in the response time and stability of the system. Ideally, the package should be a perfect heat sink, i.e. the chip interface stays near ambient temperature while dissipating heat from the chip.

Since fluidic interfaces (input/output ports) for E/F MCMs must conform to industry standard fluidic fittings and tubing, these interfaces prevent shrinking the modules even further. For high density packaging requirements, a manifold with flange seal type O-rings is often the best choice. This format can eliminate fittings and tubing since the module can be pressed directly against larger manifolds or instrument bulkheads. In some applications, in-line connections with tube fittings are required — a solution that allows easy interface but sacrifices size efficiency.

The MCM substrate contains milli-flow channels that are used to interconnect various fluidic and electro-fluidic components. This E/F MCM uses three such components: the microvalve, the pressure sensor, and an optional microfabricated capillary or orifice for relieving pressure applications. These three devices are interconnected with a channel buried in the substrate. Electronic traces are patterned on the surface of the substrate using standard PC-Board manufacturing techniques. The first example of an E/F MCM to use both microactuators and microsensors is shown in Figure 7 — the Mini Pressure Regulator.

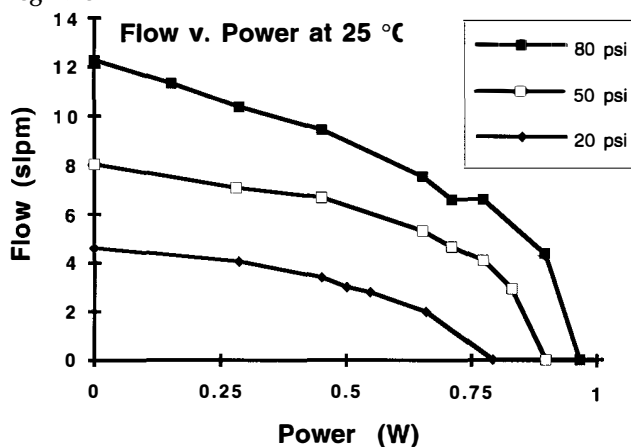


Figure 5b: Flow vs. Power for a Normally Open valve at a variety of supply pressures and 20 °C ambient temperature.

Pressure regulators built using E/F MCM technology have excellent performance characteristics including electronic pressure programmability, broad dynamic range of flow (100,000:1), excellent supply pressure rejection (>100dB) and flow rate rejection (<0.1% full scale pressure/full scale flow), temperature insensitivity (<0.1% full scale pressure/°C), minimal ripple (0.01% full scale pressure), and long term stability (variations <0.1% full scale pressure/year). When compared with manual mechanical regulators, these performance characteristics are striking. Mechanical devices typically have small dynamic range (100:1), less supply pressure rejection (<40 dB), lower flow rate rejection (0.5% full scale pressure/full scale flow rate), reduced temperature insensitivity (1.0% full scale pressure/°C), and poor long term stability (variations greater than 1.0% full scale pressure/year). Although small in size — measuring 6.3 x 8.6 x 1.2 cm — it is capable of regulating continuously over a range of 0 to 100 psig with 0.5% accuracy. The full performance specifications are summarized in Table 1.

Virtually all of the closed-loop performance limitations of the E/F MCM pressure regulator arise due to the sensor calibration limitations. Higher performance E/F MCM pressure regulators will be based on digital control electronics and will incorporate accurately calibrated pressure sensors. Off-the-shelf pressure sensors with minimal compensation yield pressure signals which are accurate to no better than 1.0% full scale pressure. Advanced digital calibration techniques will result in a pressure sensor with accuracies of at least $\pm 0.1\%$ full scale pressure over a wide range of temperatures. Incorporation of a microprocessor for control opens up the possibilities of using intelligent control algorithms which can more easily implement nonlinear functions making fuzzy logic and neural network-based controllers attractive. Further, the use of high accuracy pressure sensors combined with a microprocessor makes the development of a flow regulator a simple extension of the pressure regulator.

One of the key advantages of the E/F MCM technology is the efficient use of space. Since these modules can be made compactly, individual units can be combined to form more complex systems without sacrificing cost or size over specifically dedicated designs. For example, a manifold of pressure regulators can be efficiently constructed by mounting numerous E/F MCM pressure regulators to a common input gas line. This type of configuration is required by numerous industries including analytical instrumentation,

gas chromatography, and other mixing applications. More complex modules for regulating pressure and flow of gases and liquids will be developed offering greatly reduced size, lower cost and higher performance than systems utilizing mechanical valves.

Application of E/F MCM's in Medical Instrumentation

The trend in the Medical marketplace is constantly towards reducing size and costs. Medical applications more commonly involve carrying liquids not gases and require on-off switching rather than proportional control. For these applications, E/F MCM materials require a higher degree of corrosion resistance and the feedback circuitry must close the control loop very quickly.

There are numerous medical applications where current E/F MCMs can perform proportional control of gas pressure. Anesthesia machines can be upgraded to perform automatic gas mixing and flow control — functions which today are still performed with manual valves and regulators. Insufflators, instruments which inflate the abdominal cavity during surgery, can be equipped with Micro Pressure Regulators to provide dynamic closed-loop control of pressure during surgery, replacing larger, more expensive and lower performance electro-mechanical valve-based systems. For Non-Invasive Blood Pressure monitoring (NIBP), E/F MCMs can be used to rapidly inflate and deflate pressure cuffs in a stable and predictable manner to facilitate rapid and accurate blood pressure measurement — the size and affordability of the E/F MCMs will enable the NIBP industry to utilize closed-loop control of pressure to replace unstable, open-loop solenoid valve systems.

Future E/F MCM's designed for liquid operation could provide a new platform on which a variety of clinical chemistry operations could be performed. A concept sketch of such a "Universal chemical processor" is shown in Figure 8. Such a processor would have a number of different elements: an array of input ports to connect to a supply of reagents and samples; an array of valves to route selected chemicals to and from various nodes; a number of temperature controlled reaction chambers where the chemical reactions would take place; an array of separation devices for separating certain chemicals from each other; an array of detectors that sense the presence of chemical products; an array of output ports for disposing of waste materials as well as any chemicals that have been synthesized. A number of these microfabricated elements have been developed by various researchers around

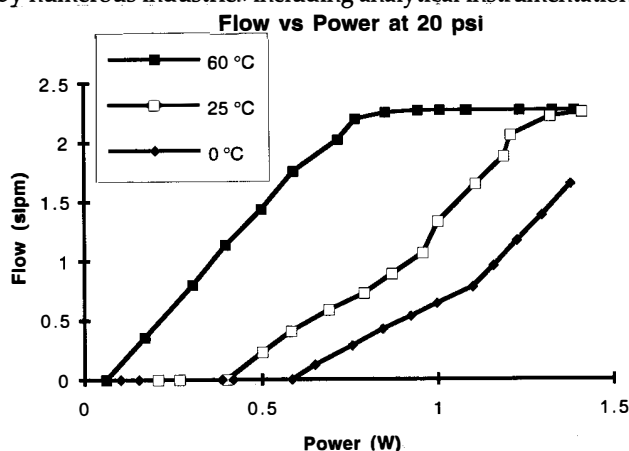


Figure 6a: Flow vs. Power for a Normally Closed valve at 20 psig supply pressure and a variety of ambient temperatures.

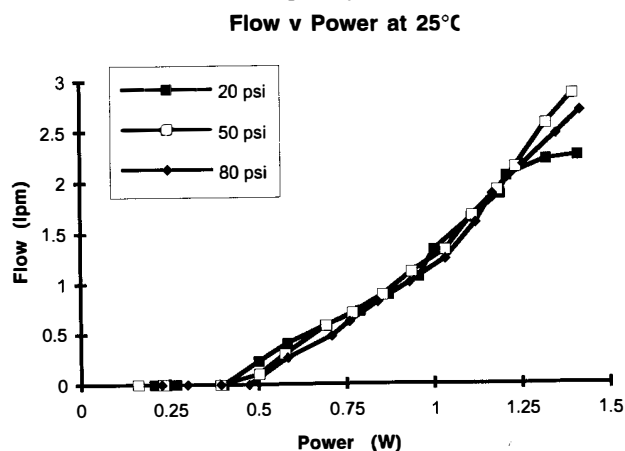


Figure 6b: Flow vs. Power for a Normally Closed valve at a variety of supply pressures and 20 °C ambient temperature.

the world, including electrophoresis separation, optical detectors, pH sensors, valves, and temperature controlled reaction chambers. Integrating them together will provide a compact chemical analysis system with tremendous flexibility and sensitivity.

Conclusions

A microfabricated valve has been developed for operation on both gases and liquids. This device has enabled the development of Electro/Fluidic Multi-Chip-Modules. Already, this level of integration provides an architecture capable of perform complex functions in a small space. The next challenge will be to provide these chips in a monolithic construct, with multiple valves or sensors manufactured simultaneously on a common substrate — the first Electro-Fluidic Integrated Circuits. Mirroring the semiconductor industry, these chips will rapidly increase in capabilities, as their size and costs are reduced. The revolutionary impact on the instrumentation marketplace will be analogous to the impact of semiconductors on the computer marketplace. Today's instruments — the equivalent of yesterday's mainframe computers — will be replaced by the instrumentation equivalents of workstations, lap-tops, and field portable units.

-TABLE 1-
Performance Benefits of E/F MCM Pressure Regulators

Performance Criteria	E/F MCM	Mechanical
Dynamic Range:	100,000:1	100:1
Supply Rejection:	> 100 dB	< 40 dB
Flow Rate Rejection: (percent of full scale pressure)	< 0.1%	0.5%
Temperature Insensitivity: (percent of full scale pressure/°C)	< 0.1%	1.0%
Long Term Stability: (percent pressure variation/year)	< 0.1%	1.0%
Programmability:	yes	no

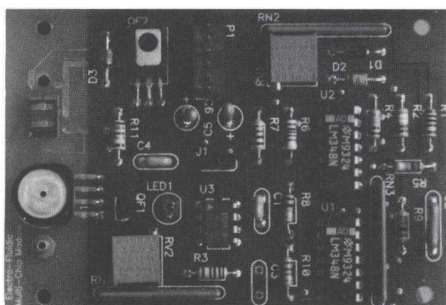


Figure 7: The size and cost benefits of E/F MCM products increase as product complexity increases. Custom manifolds and multiplexors — such as this pressure regulator for a protein synthesizing instrument — will replace larger, more expensive electro-mechanical systems.

- ¹ Terry S C, Jerman J H and Angell J B, 1979, "A gas chromatographic air analyzer fabricated on a silicon wafer," *IEEE Trans. Electron Devices*, **ED-26** 1880-6.
- ² Tuckerman D B and Pease R F W, 1981, "High performance heat sinking for VLSI," *IEEE Electron Device Letters*, **ED-2** 126-9.
- ³ Zdeblick M J, Barth J B and Angell J B, 1986, "Microminiature fluidic amplifier," *Tech. Digest IEEE Solid-State Sensor and Actuator Workshop (Hilton Head Island, SC, 1986)* (New York: IEEE)
- ⁴ Zdeblick M J, Barth J B and Angell J B, 1986, "A microminiature fluidic amplifier," *Sensors & Actuators*, **15** (1988) 427-433 (Elsevier Sequia/ The Netherlands)
- ⁵ Zdeblick M J and Angell J B, 1987, "A microminiature electric-to-fluidic valve," *Tech. digest IEEE Transducers 87 (Tokyo, 1987)* (New York: IEEE) pp 827-9
- ⁶ Zdeblick M J "A planar process for an electric-to-fluidic valve," PhD Thesis, Stanford University (Menlo Park, CA: Redwood Microsystems)
- ⁷ Zdeblick, M.J., "Electro-Fluidic Amplifiers and Actuators on Silicon," Seminar at the University of California at Berkeley, Berkeley, CA, 22 February 1987.
- ⁸ Shoji S, Esashi M and Matsuo T, 1988, "Prototype miniature blood gas analyser fabricated on a silicon wafer," *Sensors and Actuators* **14** 101-7.
- ⁹ Nakagawa S, Shoji S and Esashi m, 1990, "A microchemical analyzing system integrated on a silicon wafer," *proc. IEEE Mems 90 (Napa Valley, CA, 1990)* (New York: IEEE) pp 89-94
- ¹⁰ Petersen K E, 1979, "Fabrication of an integrated planar silicon ink-jet structure," *IEEE Trans. Electron Devices*, **ED-26** 1918-20.
- ¹¹ Smits J G, 1990, "A piezoelectric micropump with three valves working peristaltically," *Sensors and Actuators* **15** 153-67
- ¹² van Lintel H T C, van der Pol F C M and Bouwstra S, "A micro pump based on micromachining of silicon," *Proc. Eurosensor I (Cambridge, UK, 1987)* (New York: elsevier) pp 20-1.
- ¹³ Jerman J H, 1990, "Electrically-activated micromachined diaphragm valves," *Proc. Micro System Technologies 90 (Berlin 1990)* (Berlin: Springer) pp 806-11
- ¹⁴ TiNi paper
- ¹⁵ Huff M A and Schmidt m M A, 1992, "Fabrication, packaging and testing of a wafer-bonded microvalve," *Tech. Digest IEEE Solid-State Sensor and Actuator Workshop (Hilton Head Island, SC, 1992)* pp 194-7.
- ¹⁶ Petersen K E, Brown J and Renken W, 1985, "High-precision, high-performance mass flow sensor with integral laminar flow micro-channels," *Tech. digest IEEE Transducers 85 (Philadelphia, PA, 1985)* (New York: IEEE) pp 830-3
- ¹⁷ Mans A, Fettingier J C, Verpoorten E, Harrison D J, Ludi H, and Widmer H M, 1991, "Integrated electroosmotic pumps and flow manifolds for total chemical analysis," *Tech. Digest IEEE Transducers 91 (San Francisco, CA, 1991)* (New York: IEEE) pp 939-41.
- ¹⁸ Gravesen P, Branebjerg J, and Jensen O S, "Microfluidics — a review," *J. Micromech. Microeng.* **3** (1993) 168-182.

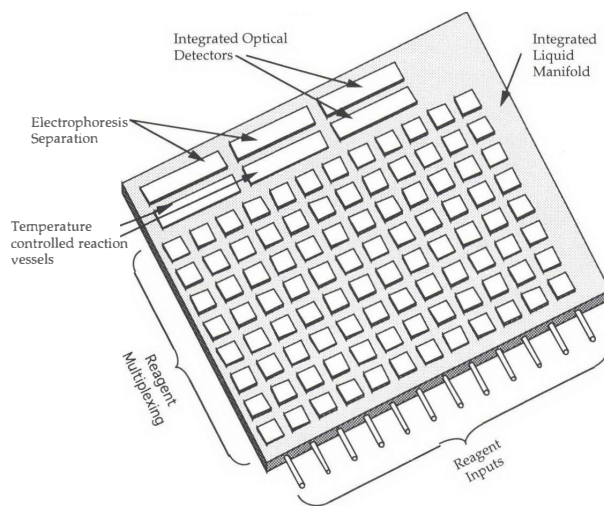


Figure 8: Universal chemical analysis manifold