Practical Aspects of Micromachined Gas Distribution Systems for Semiconductor Processing

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ABSTRACT

Previously, we have reported the science and technology of micromachined valves and orifices, and their integration to form high-performance mass-flow controllers (MFCs), and vacuum leak rate shut-off devices (SOVs). In this work, we expand the science and technology base of these devices, to include not only performance, but more practical aspects of their behavior. Specifically, for MFCs we have studied long-term drift, mean time to fail, particle generation, dry down after moisture contamination, gas replacement, effects of gravitational orientation, and sensitivity to inlet pressure and ambient temperature. For SOVs, we have studied mean time to fail, particle generation, dry down after moisture contamination, vacuum leak rate, and sensitivity to inlet pressure and ambient temperature.

Keywords: MEMS, microvalve, microfluidics, mass flow control, reliability, mean time to fail

1.0 INTRODUCTION

Silicon-based microvalves, appropriate for the control of gas flow, have been studied for some time.[1,2] Commercial microvalves have been available from several manufacturers for some time, including Redwood Microsystems, IC Sensors, TiNi Alloy, and Hewlett-Packard.[3] The availability of such valves carries a presumption of rigorous reliability and quality testing prior to release, though little such information has been presented in research literature.

Integration of these valves into gas distribution systems, such as mass flow controllers and gas panels, has been approached from several directions. The earliest research attempts encompassed a near-monolithically integrated controller,[4] with only one or two components added on a device-by-device basis, rather than at the wafer level. More recent research efforts have focused on monolithic integration,[5] including encapsulation.[6] In each of these cases, the devices so constructed have been academic in nature, and no commercial versions have been reported to date. Measurements of long-term reliability, drift in flow sensors, generation of particles, and other important data have either not been made, or reported.

Surface mount MFCs, and vacuum leak-rate shut-off valves, where silicon microfabricated components are attached to a metal substrate, have also been reported.[7] Some performance data has been presented, but rigorous measurements related to such parameters as long-term reliability, mean time to fail (MTTF), and particle generation (which is especially important for devices intending for semiconductor manufacture) have been absent. In this work, we report data related to these practical aspects of MFCs and SOVs, laying the foundation for their full use in commercial applications.

2.0 DEVICE CONSTRUCTION

The MFCs and SOVs have been reported elsewhere,[7] and only a sketch of the construction will be provided here.

The SOVs consist of a double or triple layer of silicon and Pyrex wafers, etched using bulk micromachining techniques, and bonded using both fusion and anodic bonding. A cantilever structure is attached on a valve-by-valve basis, using a PTFE-based die attach material having low chemical reactivity with virtually all of the gases encountered in semiconductor manufacture. The cantilever structure provides the vacuum leak-rate character of this normally-closed device. Incorporated in its structure, again on a valve-by-valve basis, is a Chemraz™ valve seat material. The micromachined valve components are attached to a 316L stainless steel manifold, again using a PTFE-based die attach material.

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The MFCs have five silicon components, which are attached to a 316L stainless steel base using a PTFE-based die attach material. The MFC valve is a monolithically integrated, normally-open valve, comprised of two silicon layers which are patterned and fusion bonded, and a Pyrex layer which is bonded anodically. The flow sensor is comprised of: a silicon temperature sensing integrated circuit; a micromachined silicon orifice; and two micromachined silicon piezoresistive pressure sensors. The pressure sensors monitor the pressure upstream and downstream of the orifice. The voltage outputs of the sensors are related to the flow through the orifice, measured independently. The flow calibration information is stored in an EEPROM on the MFC.

2.0 PERFORMANCE DATA

2.1 Mass flow controller performance

MFC flow measurements are made using a DHI Molbloc as a primary standard. Verification is made using a rate-of-rise flow measurement system. Both systems are traceable to NIST primary standards.

Data from four MFCs are presented. Each MFC is calibrated for each of four gases: nitrogen, helium, CF_4, and SF_6. Measurements are performed according to accepted standards.[8,9] Accuracy data is shown for nitrogen, helium, and SF_6, in Figures 1 through 3. These three gases represent the norm, low, and high value of molecular weight of gases typically used in semiconductor manufacture, and so exercise the extremes of the flow model used in these MFCs (see Paper 4175-06 of this conference, for full reference of the flow model.)

Linearity and reproducibility are shown for nitrogen only, in Figures 4 and 5. The linearity and reproducibility results for the other three gases are comparable to nitrogen.

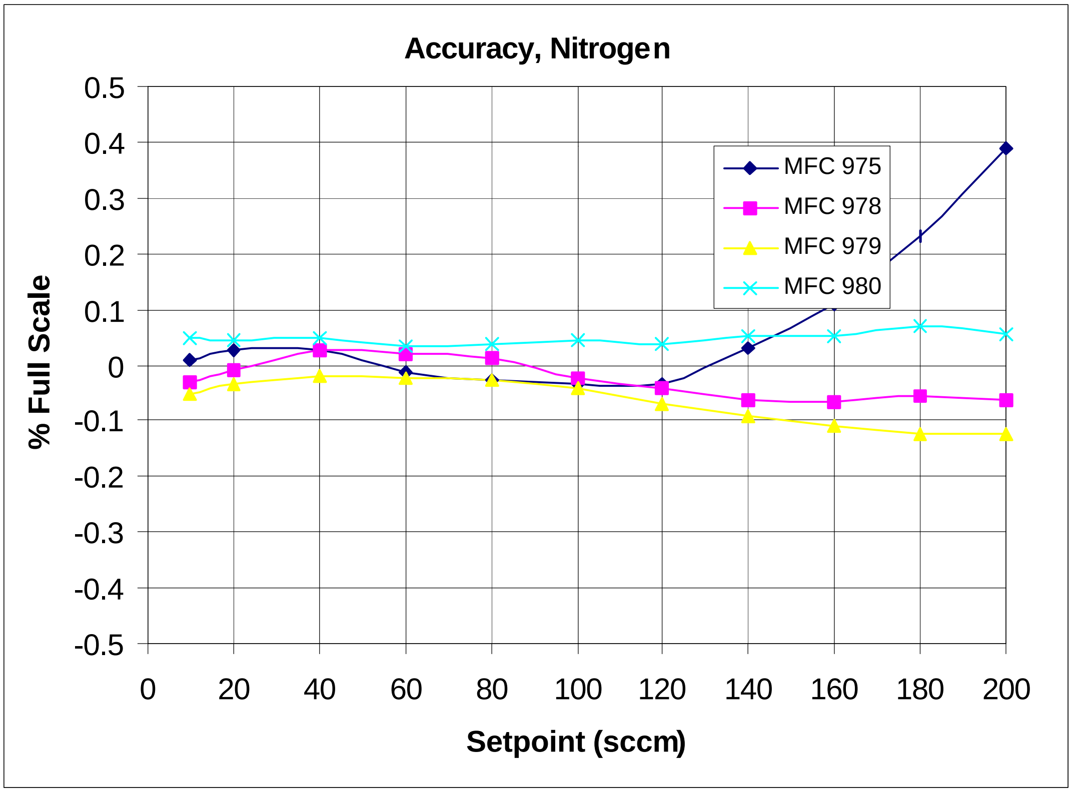

Figure 1: Accuracy of four different MFCs, for nitrogen. The maximum flow rate of each MFC is 200 sccm.

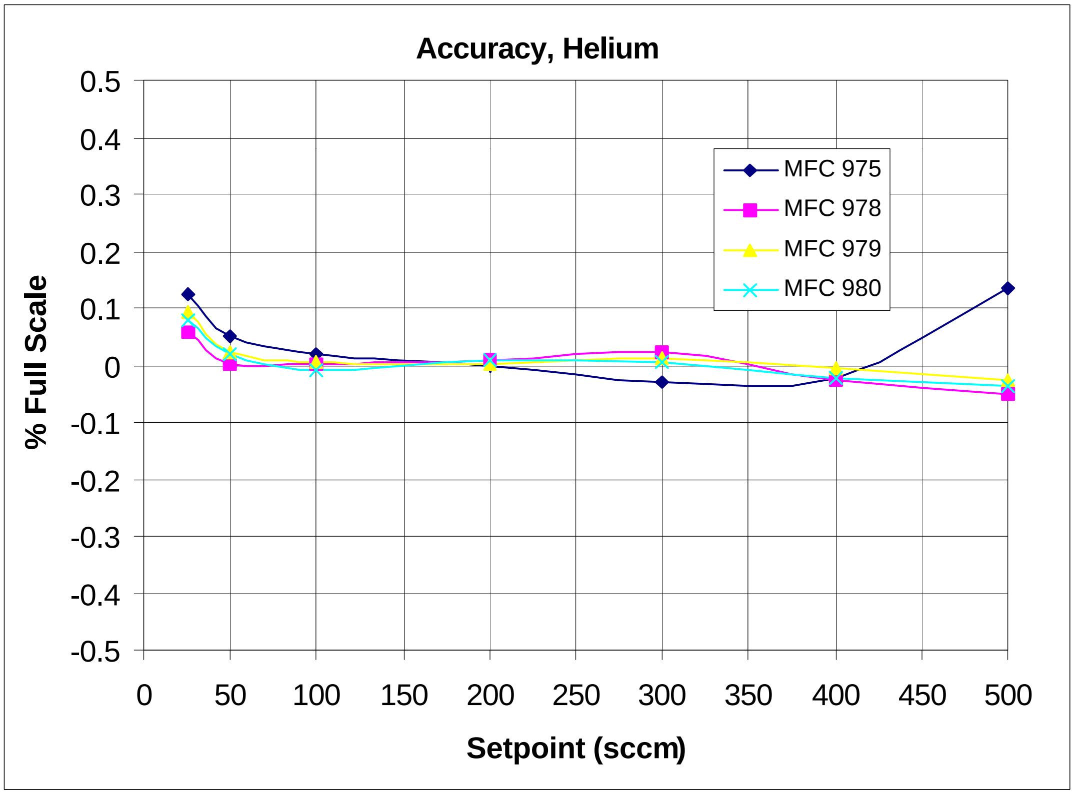

Figure 2: Accuracy of four different MFCs, for helium. The maximum flow rate of each MFC is 200 sccm.

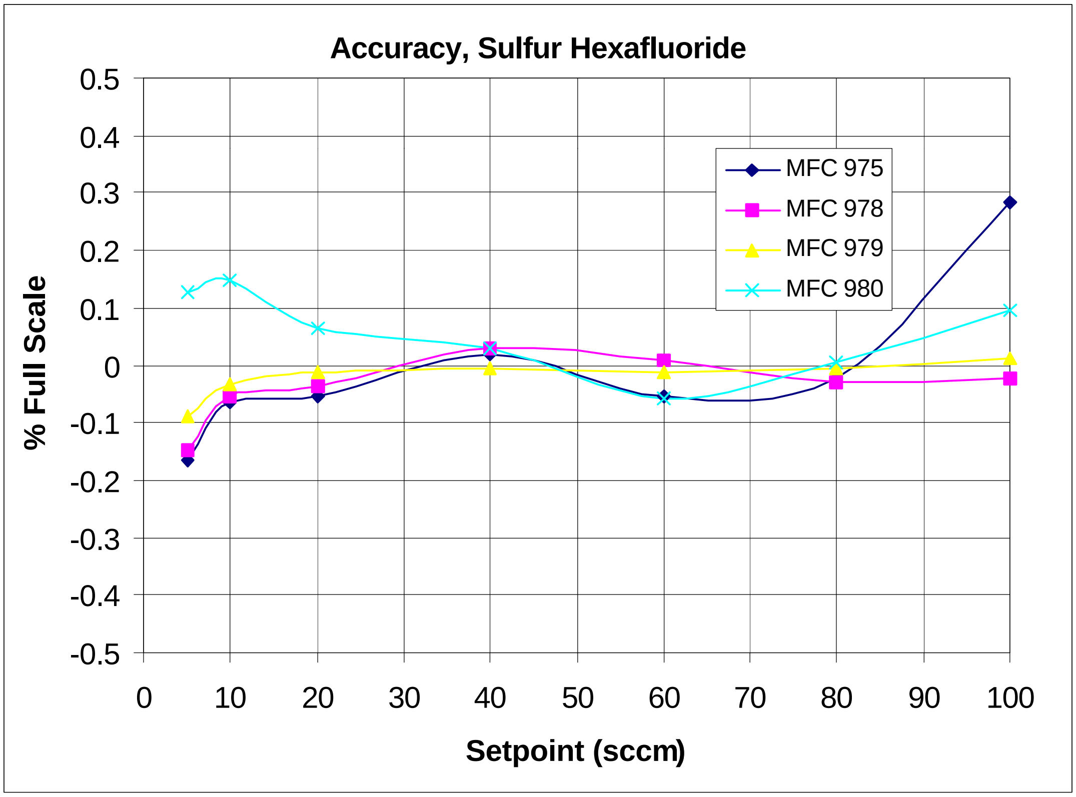

Figure 3: Accuracy of four different MFCs, for SF_6. The maximum flow rate of each MFC is 200 sccm.

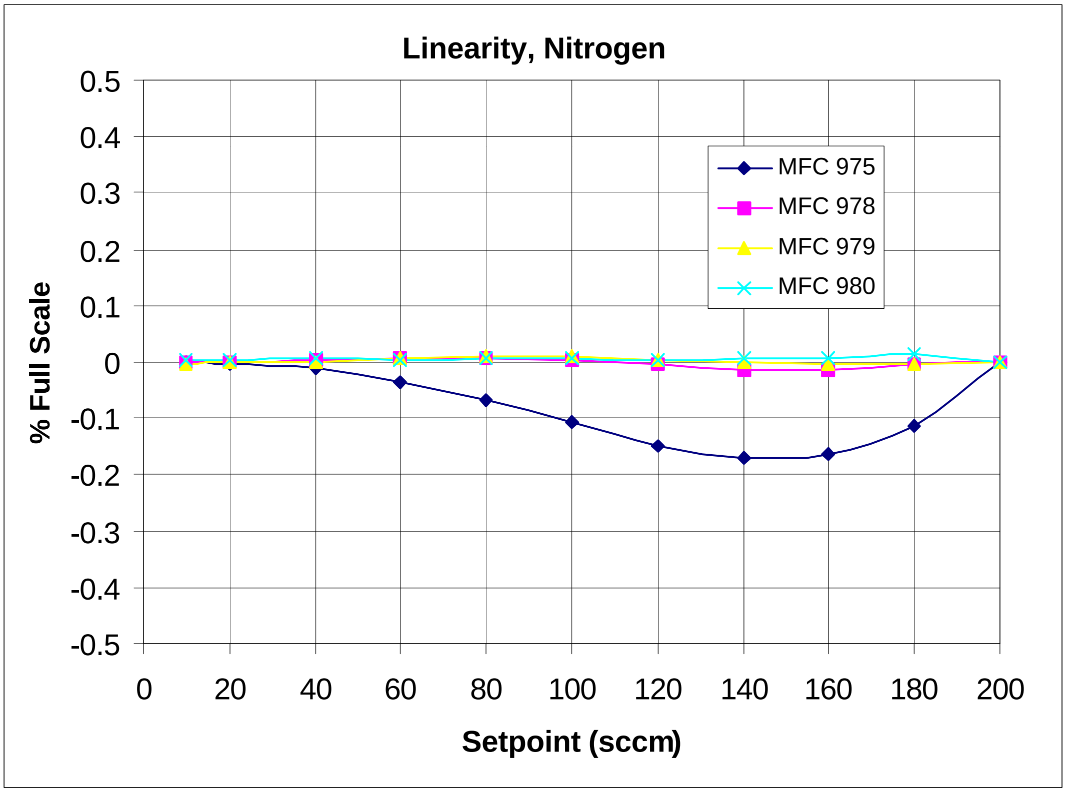

Figure 4: Linearity of four different MFCs, for nitrogen. The maximum flow rate of each MFC is 200 sccm.

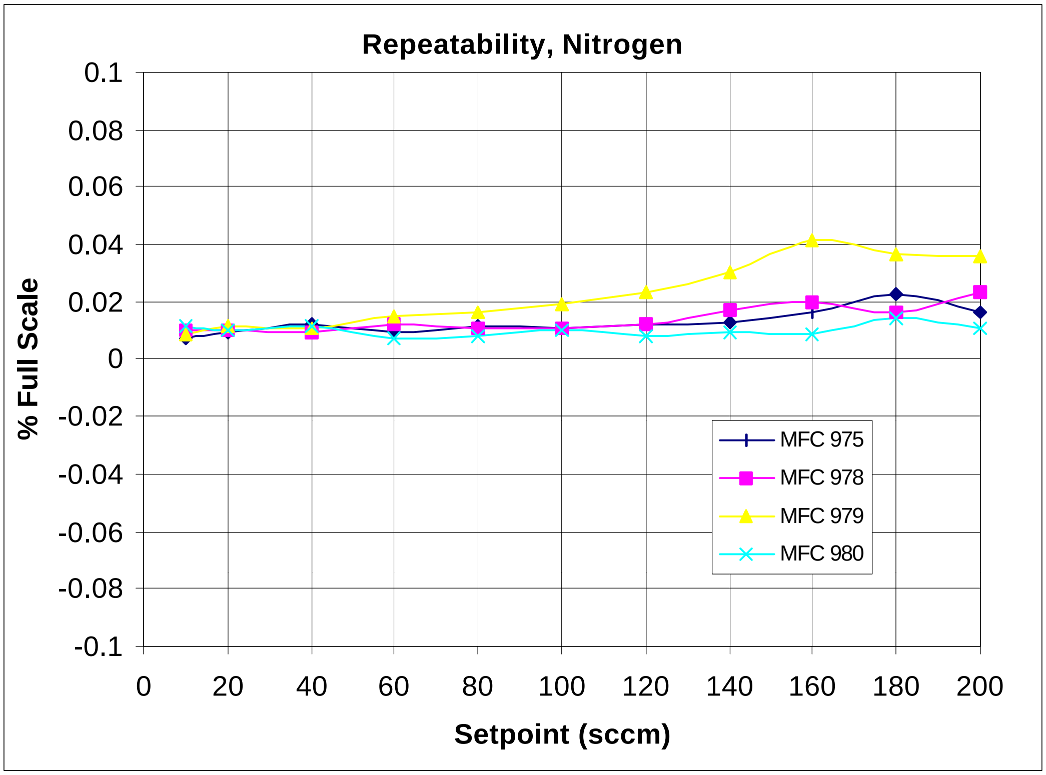

Figure 5: Repeatability of four different MFCs, for nitrogen. The maximum flow rate of each MFC is 200 sccm.

2.2 Shut-off valve performance

The flow coefficient C_v was measured according to accepted standards[10] on five units, with results shown below. These values are small relative to conventional shut-off valves used in semiconductor manufacture, which reflects the 'micro' nature of the valve structures. However, higher values of C_v appear to be feasible, by extending the valve actuation mechanism of the present valve to control larger cross-sectional flow areas.

SOV Serial Number	# 931	# 932	# 934	# 935	# 936
C_v	0.0067	0.0069	0.0065	0.0071	0.0072

Figure 6 shows the transient (closed-to-open) response for a series of shut-off valves. The response for Unit #712 shows the open-to-closed response of the valve.

Figure 7 shows the flow versus inlet pressure for a typical SOV. Because of the nature of the valve cantilever, the application of reverse pressure serves to open the valve without electrical actuation, for reverse pressures above 20 psig.

Figure 8 shows the vacuum leak rate for a typical SOV, measured according to specified procedures.[11] Because of the nature of the valve cantilever, the application of reverse pressure serves to open the valve without electrical actuation, for reverse pressures above 20 psig.

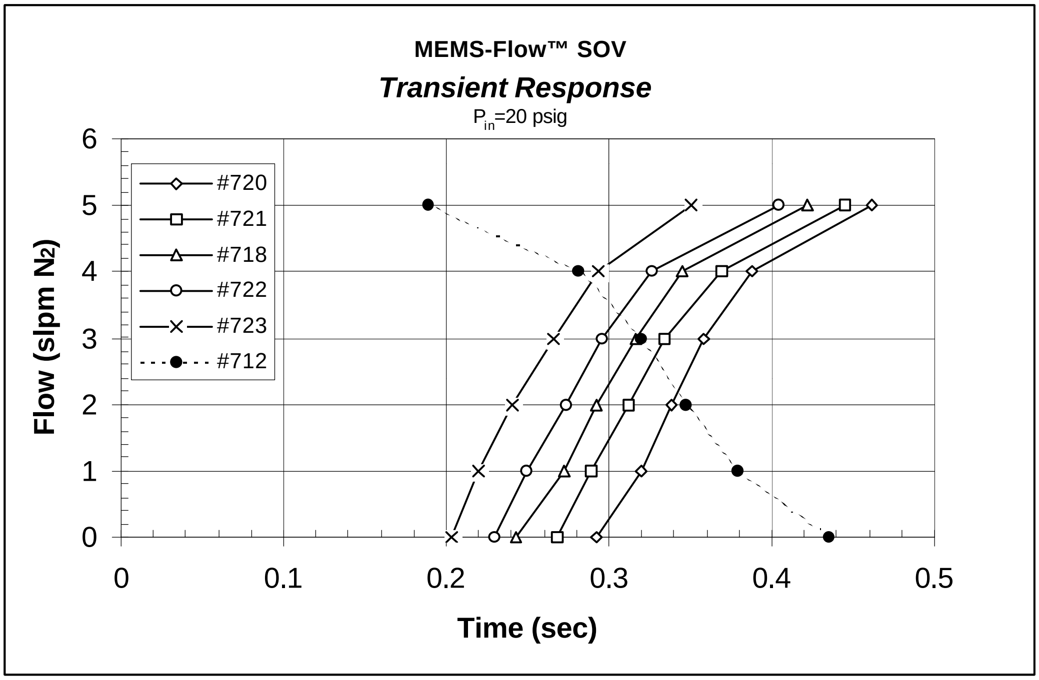

Figure 6: Transient response time of shut-off valves. In these measurements, the outlet pressure is 14.7 psia.

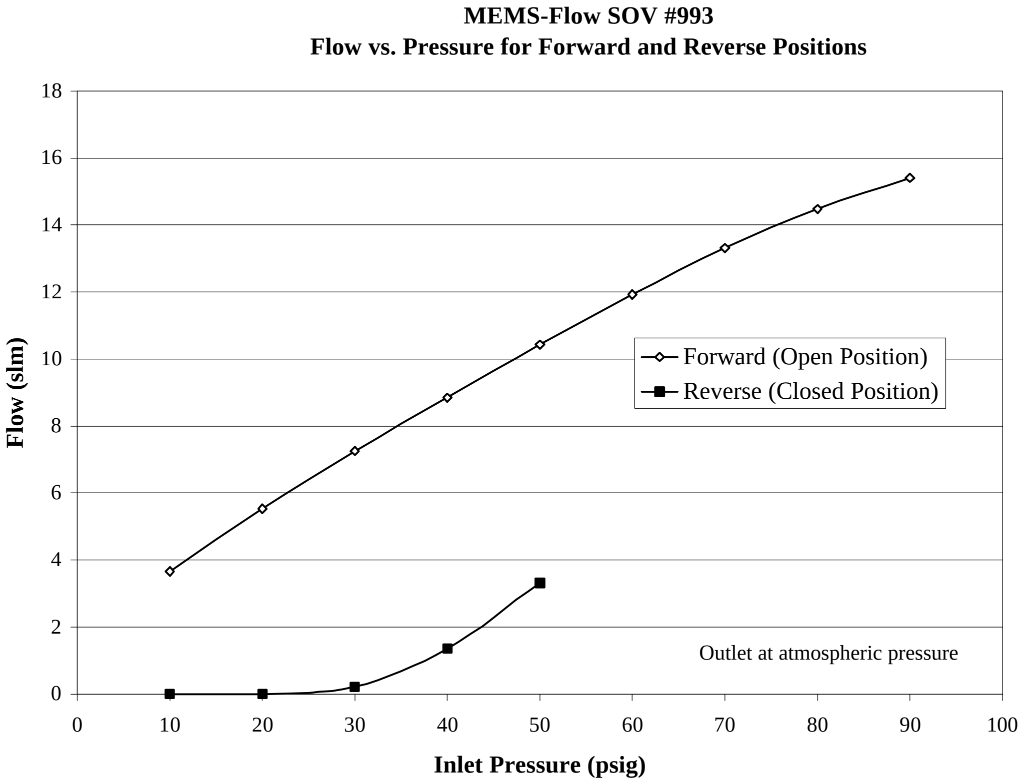

Figure 7: Shut-off valve flow rates versus inlet pressure, for both forward and reverse pressure drops across the valve seat.

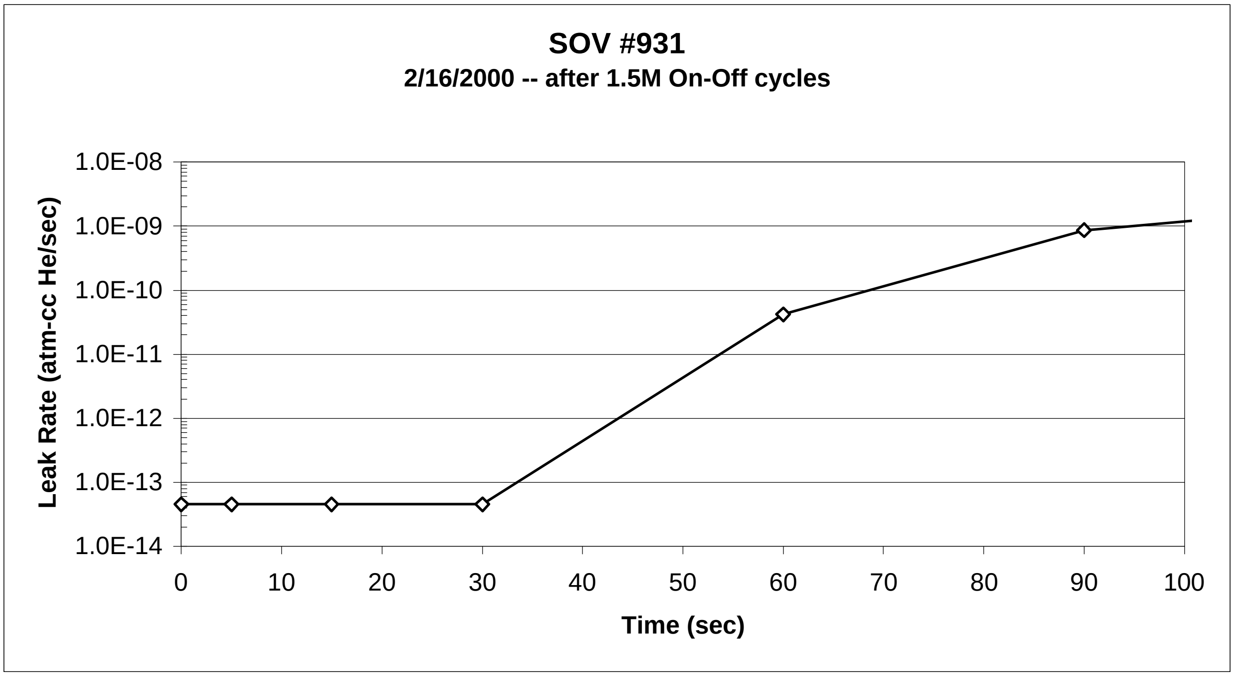

Figure 8: Vacuum shut-off leak rates through the SOV in forward mode. Note that the results shown are for a valve which has been actuated through 1.5 million cycles. The reverse leak rate characteristics are comparable, for reverse inlet pressures of 20 psig or less.

3.0 CYCLING DATA

3.1 Mass flow controller cycling

The MFCs described in Section 2.0 were cycled for one million (1M) cycles, according to specified procedures.[12] The activation process involves an initial setpoint of 0%, followed by successive setpoints of 100, 25, 75, and 0%. This process represents two cycles. Each setpoint is separated in time by 1.25 sec, so that the cycle time is 2.5 sec.

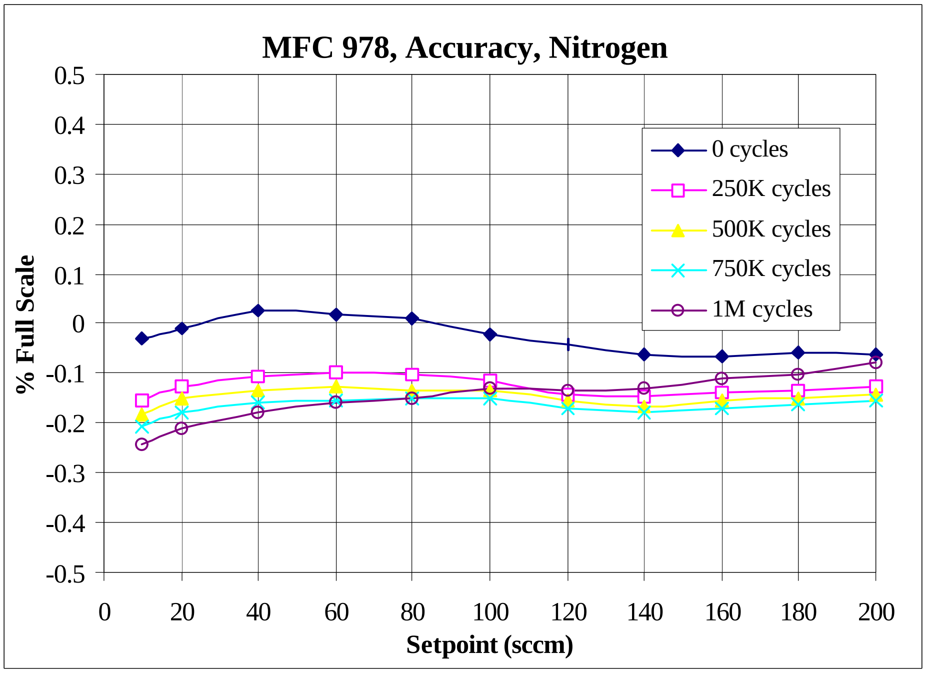

Figure 9: Accuracy vs. cycling data for one MFC, for nitrogen. The maximum flow rate of the MFC is 200 sccm.

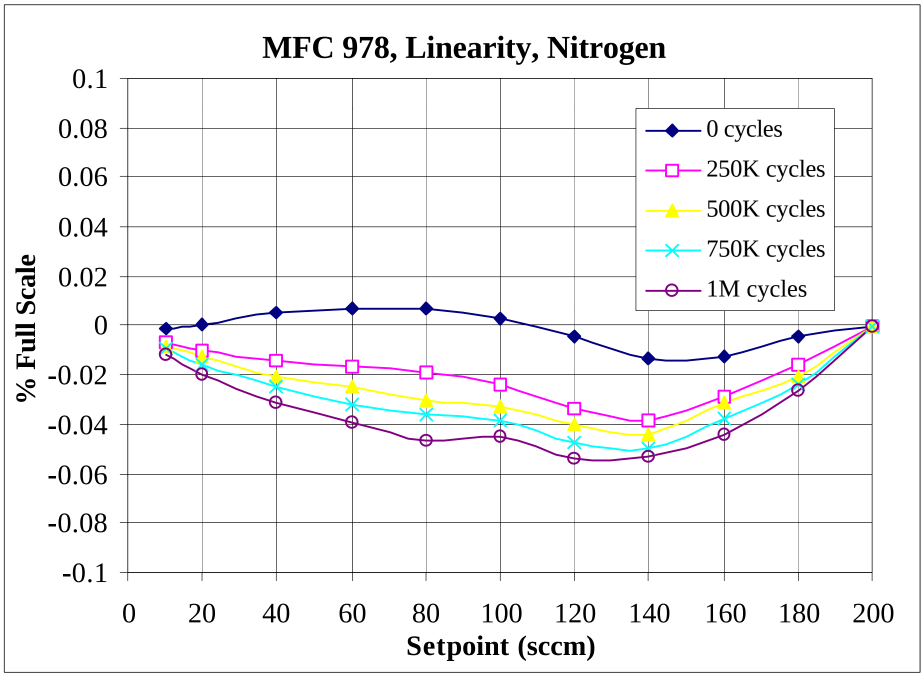

Figure 10: Linearity vs. cycling data for one MFC, for nitrogen. The maximum flow rate of the MFC is 200 sccm.

3.2 Shut-off valve cycling

Five shut-off valves were cycled from 'off' to 'on' and back, with a cycle time of 2 sec, for up to 3 million cycles, according to specified procedures.[13] Response time, flow rate, C_v, and particle generation were monitored at regular intervals during this process. No significant changes in response time, flow rate, or C_v were observed. Particle generation results from these measurements will be discussed separately, below.

4.0 OTHER RELIABILITY AND QUALITY MEASUREMENTS

4.1 Recovery from exposure to moisture

Both MFCs and SOVs were subjected to moisture recovery testing, according to specified procedures.[14] The test consists of exposing the device under test to a specified concentration of water. The moisture concentration in a gas stream moving through the device under test is then monitored as a function of time, with the device heated to an elevated temperature, in order to determine the desorption of water vapor from the wetted internal surfaces of the device under test. Figure 11 shows the results from a shut-off valve; similar results have been obtained for the MFCs described earlier.

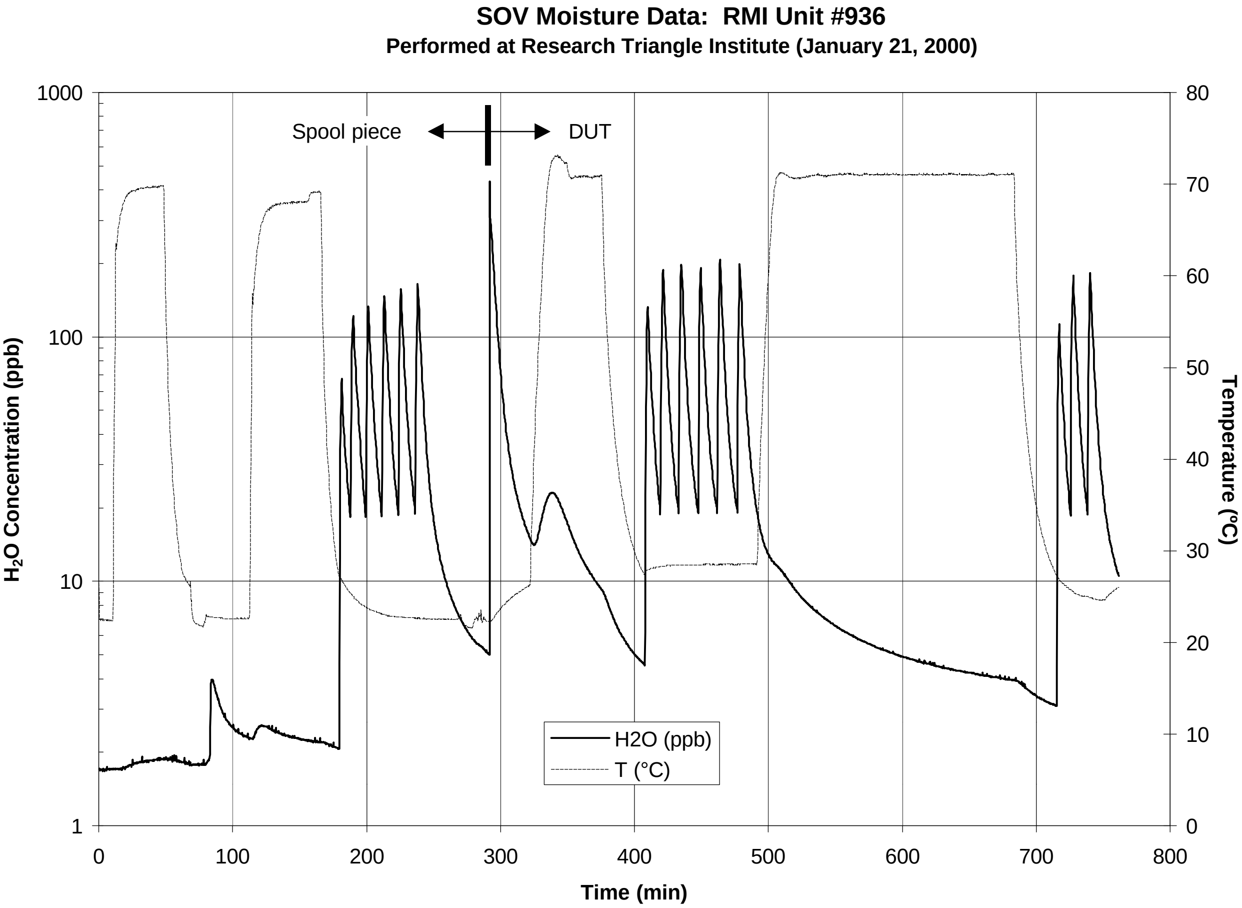

Figure 11: Moisture recovery of a shut-off valve.

4.3 Ionic contamination

The total ionic contamination from a shut-off valve and its stainless steel manifold was determined according to specified procedures.[15] Measurements on a spool piece were also made. The results are shown in the table immediately below. The stainless steel manifold contributed about 66% of the total volume in the “Test Component”. The total ionic contribution from the SOV+Manifold is 0.91 µg/component, or 334 µg/l water; for the spool piece, 1.47 µg/component, or 172 µg/l water.

Ion	**SOV 934 + Manifold (mg/l Water)**	**SOV 934 + Manifold (mg/Test Comp.)**	**SOV 936 + Manifold (mg/l Water)**	**SOV 936 + Manifold (mg/Test Comp.)**	**Spool (mg/l Water)**	**Spool (mg/Test Component)**
Fluoride, F$^-$			< 3	< 0.007	23	0.20
Bromide, Br$^-$			< 10	< 0.023	<5	<0.04
Chloride, Cl$^-$			62	0.141	61	0.52
Nitrite, NO_2^-			< 5	< 0.011	<5	<0.04
Nitrate, NO_3^-			19	0.043	34	0.29
Phosphate, PO_4^-			< 10	< 0.023	<10	<0.09
Sulfate, SO_4^-			39	0.088	33	0.28
Sodium, Na$^+$	108	0.323			<20	<0.17
Potassium, K$^+$	106	0.316			21	0.18
Lithium, Li$^+$	< 20	< 0.06			<20	<0.17
Ammonium, NH_4^+	< 20	< 0.06			<20	<0.17

The total ionic contamination from a shut-off valve and its stainless steel manifold was determined according to specified procedures.[15] Measurements on a spool piece were also made. The results are shown in the table immediately below. The stainless steel manifold contributed about 66% of the total volume in the “Test Component”. The total ionic contribution from the SOV+Manifold is 0.91 µg/component, or 334 µg/l water; for the spool piece, 1.47 µg/component, or 172 µg/l water.

4.4 Total hydrocarbon contribution

The contribution of hydrocarbons by the SOV and its manifold was measured according to specified procedures.[16] The SOV results did not differ from the spool piece; emissions did not exceed 10 ppb. The results are shown in the Figure 12.

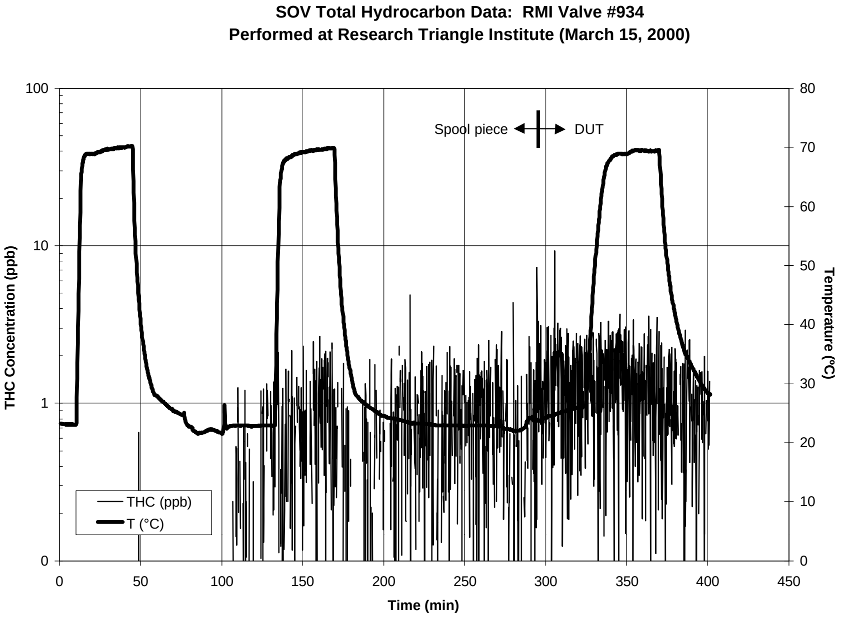

Figure 12: Total hydrocarbon contribution of a shut-off valve.

4.5 Total oxygen contribution

The contribution of oxygen by an SOV and its manifold was measured according to specified procedures.[17] The SOV did not contribute more than 10 ppb oxygen at 22.5 minutes after installation. The results are shown in the graph below. The results are shown in the Figure 13.

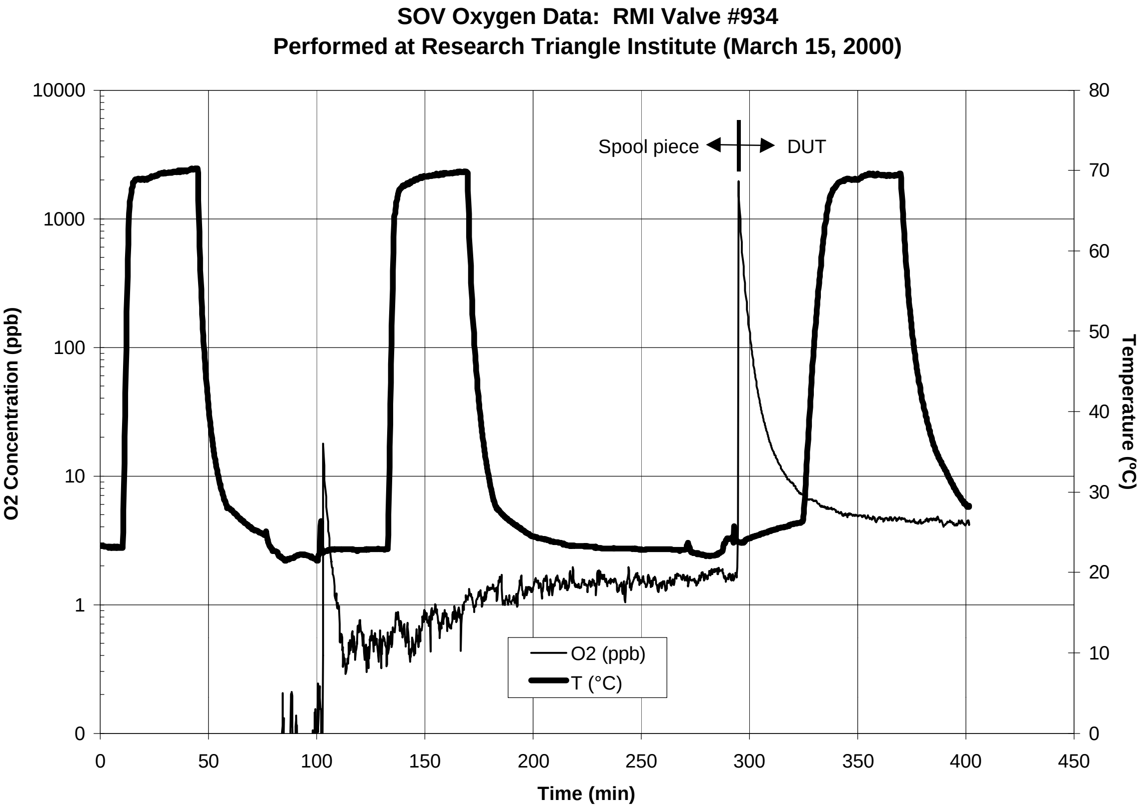

Figure 13: Total oxygen contribution of a shut-off valve.

4.6 Particle generation

The generation of particles by an SOV and its manifold was measured according to specified procedures.[18] The following table shows particles of size greater than or equal to 0.1 µm, measured using a laser particle counter. Results for smaller particle sizes, as a function of cycle life, are shown in the next section.

Unit Number (Num. of cycles)	Dynamic Particles (Particles/cuft)	Static Particles (Particles/cuft)
#931 (2.51 M)	≤ 3	≤ 3
#932 (2.74 M)	5	≤ 3
#934 (2.83 M)	≤ 3	≤ 3
#935 (750 K)	≤ 3, after 20 min clean-up period	≤ 3
#936 (2.14 M)	≤ 3	≤ 3

4.7 Accelerated life test/Mean-Time-To-Fail (MTTF)

Shut-off valves were subjected to cycle stress, according to specified procedures.[13] At regular intervals, particle generation, flow coefficient, and transient response were measured. Departures from specified limits resulted in the counting of a 'failure'. The number of failures was accumulated throughout the cycle procedure, and a mean-time-to-fail was calculated.[19] The particle generation results, in particular, are shown in the table below. Since there were two failures for five valves over 10.97M cycles, a MTTF of 2.06M cycles, with 90% confidence, is calculated.

MTTF and accelerated life tests were also performed on mass flow controllers, with results superior to those reported here for the SOVs. The four MFCs presented in Section 3.0 were employed. The criteria used for the MFCs differed from the SOVs. Accuracy, short-term reproducibility, linearity, hysteresis, transient response, and particle generation were monitored at specified intervals. The calculated MTTF exceeded 3M cycles for the MFCs.

Unit Number (Num. of cycles)	Dynamic Particles (Particles/cuft)	Static Particles (Particles/cuft)	Inboard Leak (Atm scc He/s)	Leak Across the Seat (Atm scc He/s)
#931 (1.51 M)	25	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#931 (1.76 M)	36	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#931 (2.01 M)	50	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#931 (2.26 M)	31	13	$< 10^{-10}$	$< 10^{-10}$
#931 (2.51 M)	12	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#932 (1.74 M)	51	33	$< 10^{-10}$	$< 10^{-10}$
#932 (1.99 M)	17	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#932 (2.24 M)	10	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#932 (2.49 M) fuse fail, repaired	9	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#932 (2.74 M)	13	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#934 (2.08 M)	≤ 3	4	$< 10^{-10}$	$< 10^{-10}$
#934 (2.33 M)	8	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#934 (2.58 M)	5	4	$< 10^{-10}$	$< 10^{-10}$
#934 (2.83 M)	25	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#934 (3.08 M)	4	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#935 (0)	≤ 3	20	$< 10^{-10}$	$< 10^{-10}$
#935 (250 K)	64	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#935 (500 K)	≥ 75	≥ 75	$< 10^{-10}$	$< 10^{-10}$
#935 (750 K)	6	8	$< 10^{-10}$	$< 10^{-10}$
#936 (1.39 M)	18	≤ 3	$< 10^{-10}$	$< 10^{-10}$
#936 (1.64 M)	≤ 3	7	$< 10^{-10}$	$< 10^{-10}$
#936 (1.89 M)	≤ 3	4	$< 10^{-10}$	$< 10^{-10}$
#936 (2.14 M)	≤ 3	57	$< 10^{-10}$	$< 10^{-10}$
#936 (2.39 M)	21	≤ 3	$< 10^{-10}$	$< 10^{-10}$

5.0 CONCLUSIONS

We have developed commercial mass flow controllers and vacuum leak-rate shut-off valves, and gas distribution systems based on these devices. We have demonstrated the performance, reliability, and quality of these devices, based on commonly accepted standards and specifications, especially in the context of their utility in controlling gas flow for semiconductor manufacture. The devices show superior performance, equal to or better than comparable devices based on conventional technology. Because many of the reliability and quality attributes, such as moisture contribution, are dependent on the total wetted surface area, and on the dead volumes in the devices, it is expected that further reduction in the size of these devices, using micromachining techniques, will result in even better performance, relative to that reported here.

ACKNOWLEDGEMENTS

The efforts of John Hill and Dean Hopkins have been essential to the development of the gas flow control devices described in this work.

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