Dry-down characterization of

diaphragm valves

OVERVIEW New polymer-seated valves dry two to three times slower than metal-seated valves depending on operating temperature. Brief exposure of thoroughly dried valves to ambient atmosphere, however, results in similar dry-down times for both metal- and polymer-seated springless diaphragm valves at room temperature, and even slightly faster dry-down of the polymer-seated valve at 70°C. No significant difference was found between the two valve types when the dried valves were briefly exposed to ~16ppb of moisture. Concentrations of <1ppb were reached within ~15 min at the low challenges.

Fast dry-down — removal of contaminating moisture — is an important performance characteristic for high-purity gas delivery systems. Delay during system maintenance or startup caused by long dry-down times can be a significant economic factor in semiconductor manufacturing. It is commonly assumed that polymer-containing components should be avoided because outgassing of moisture from the polymer matrix or from pores in the polymer can contribute ppb levels of moisture to the gas stream for extended time periods, and might even prevent the achievement of sub-ppb purity levels.

Thus, we set up to quantify the contribution of low-porosity polymer seats (specifically Kel-F seats) in state-of-the-art springless diaphragm valves and to compare the dry-down characteristics to metal-seated valves with otherwise similar design.

Experimentation

Our analytical tool was an ABB-Extrel atmospheric pressure ionization mass spectrometer (APIMS) equipped with a two-stage dilution manifold and heated line (Fig. 1). We maintained the inlet pressure of the APIMS at 950torr, an outlet flow rate of 1slpm, and a 2slpm flow rate through the test valve for all experiments. We measured a linear calibration curve up to ~20ppb in the counting mode with a permeation device that is an integral part of the first stage of the dilution manifold. Higher concentrations had to be extrapolated and so have a relatively large associated uncertainty. Detection limits for moisture in nitrogen were below 100ppt (3σ). We adjusted temperature on the valves with heating tapes and measured it with a thermocouple.

To maintain flow through the APIMS, we installed the valves to be tested while the dry gas stream was bypassing the sampling setup. Then, the dry gas stream was directed through the valve immediately after the fittings were tightened. Re-exposure to ambient atmosphere was accomplished by switching the gas stream to the bypass lines and physically disconnecting the fittings for 5 min. The starting conditions for the ambient air exposure experiments were not exactly defined, since relative humidity ranged from 10–30% in the laboratory at temperatures between 20–22°C during the experiments. The amount of moisture in ambient air, however, was sufficiently high to assume quick saturation of our study metal or polymer surfaces. We assumed that exposure of other system components like the heated bypass valves and connecting lines to ambient moisture during the assembling procedure contributed only slightly to the dry-down. Diffusion of moisture through the 1/8-in. line segments to the valve seats of the bypass valves was assumed to be slow enough to be neglected.

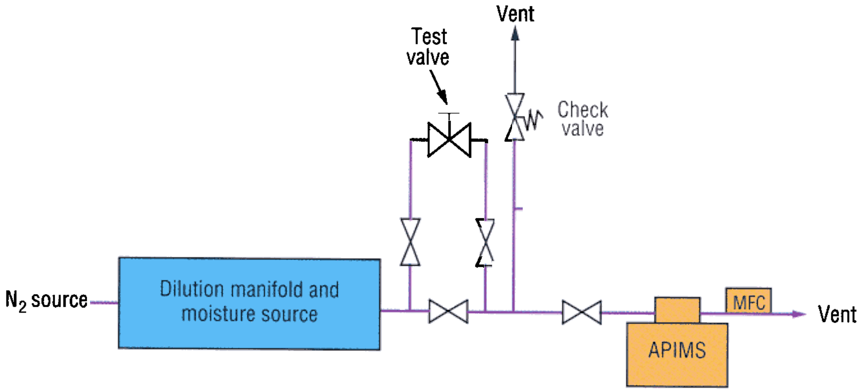

Figure 1. APIMS setup for valve dry-down tests.

The intrinsic characteristic of the moisture adsorbed on the internal valve surfaces started to dominate the dry-down behavior after a few seconds of purge because of very short residence times at 2slpm flow rates through 1/8-in. lines, and differences in the initial conditions became insignificant. The permeation device used for calibrations was applied for the low-concentration moisture challenges.

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COVER

The cover shows the inside of a TSMC fab cleanroom. TSMC enforces strict contamination controls to maintain the cleanliness of its fabs. The standard mechanical interface (SMIF) minienvironment the company uses is equipped for Class 0.1 cleanliness. *Photo courtesy of TSMC, North America*

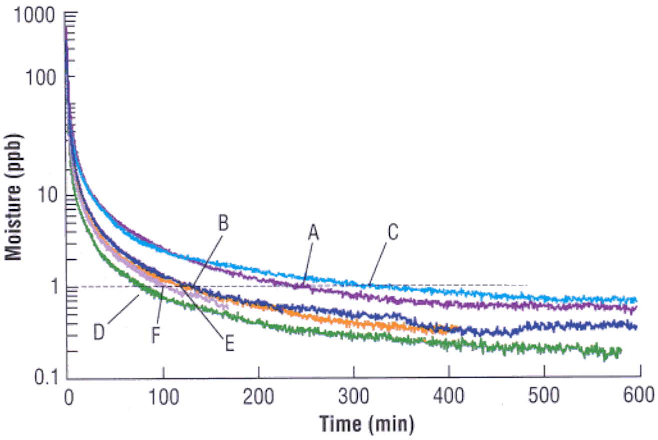

Figure 2. Comparison of polymer- and metal-seated valves at high moisture challenge. A) new Kel-F at room temperature (RT); B) Kel-F repeat at RT; C) new Kel-F at 70°C; D) dry Kel-F at 70°C (5 min open); E) metal at RT; and F) metal at 77°C.

Findings

In this study, we confirmed the valve dry-down times to ppt levels on the order of several hours, similar to those that have been reported [1, 2]. We obtained curve A in Fig. 2 with a new Kel-F-seated valve at room temperature; curve B is the result of a 5-min exposure of the dried valve to ambient air after the completion of curve A, also at room temperature. The dry valve reaches sub-ppb moisture levels significantly faster. Internal outgassing of the polymer seat that decreases after sufficient exposure to the dry gas stream during the measurement of curve A appears to cause the observed differences. Short-time exposure to ambient conditions did not result in significant moisture saturation of the bulk polymer matrix and the observed dry-down was mainly influenced by surface adsorption.

The valve was then exposed to ambient air for one day to recreate the behavior of a new valve at elevated temperatures. Curve C (see Fig. 2) shows the dry-down characteristics of the completely re-equilibrated valve at 70°C and curve D is the result of brief exposure of the valve to ambient air for 5 min after completion of curve D, also at 70°C, similar to curves A and B. The dry-down time in curve C is much longer than the initial dry-down of the new valve (curve A) at room temperature. Moisture absorbed in the core of the polymer seat diffuses faster to the surface at an elevated temperature and is released during the measurement. The contribution of the core moisture was apparently much smaller during the room temperature measurement and the initial dry-down was mainly affected by surface moisture. Curve D is significantly faster because the core moisture was removed during the previous test and surface moisture is less strongly adsorbed at elevated temperatures.

Metal-seated valves are generally expected to be superior to polymer-seated valves based on the lack of outgassing from the core polymer. At lower temperatures, however, thoroughly dried polymer-seated valves dry faster because the polymer surface is more hydrophobic than a metal surface and therefore adsorbs less moisture; Fig. 2 illustrates this behavior. Curves E and F were obtained with an all-metal valve at room temperature and at 70°C. The heated, metal-seated valve dried only slightly faster than the unheated valve. Both dry-down times were significantly shorter than the times obtained for the wet polymer valves (curves A and C), but longer than the dry-down of the well-dried polymer valve at elevated temperatures. All-metal valves will be more advantageous if the system can be heated above maximum operating temperature of the polymer valves (~70°C).

High-moisture intrusions, such as exposure to ambient air, are not usually encountered during APIMS measurements, since the system is mainly designed for low-ppb to ppt moisture levels. Figure 3 demonstrates that a 15ppb moisture challenge can be purged from the system within a few minutes to sub-ppb levels if the gas-handling system is sufficiently dry.

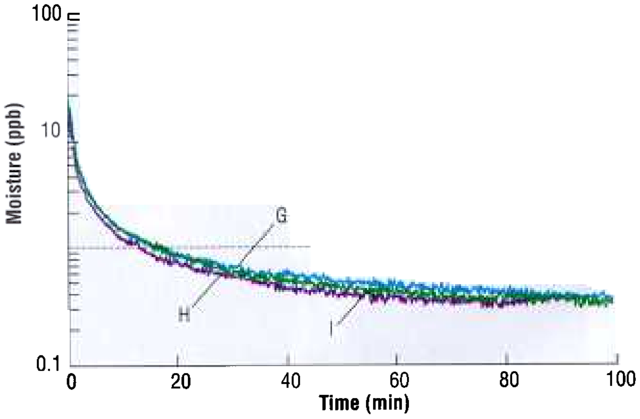

Figure 3. Dry-down of polymer- and metal-seated valves at low moisture challenges. G) Metal at 66°C; H) dry polymer at 65°C; and I) metal at 66°C.

Curves G, H, and I compare the characteristics of the dried polymer valve and the metal-seated valve at ~65°C. Curve I is a repeat measurement obtained with the metal-seated valve to demonstrate reproducibility. The metal valve dries slightly slower, possibly because of the more hydrophilic character of oxide-coated metal surfaces.

Dry-down times to 1ppb for all experiments

Seat material	Metal	Metal	Metal	Metal	Kel-F	Kel-F	Kel-F	Kel-F	Kel-F
Order of test, curve label in figures	1st, E (new valve)	2nd, F	3rd, H	4th, I	1st, A (new valve)	2nd, B	3rd, C	4th, D	5th, G
Temperature (°C)	Ambient	77	66	66	Ambient	Ambient	70	70	65
Challenge	Ambient air	Ambient air, 1 day	~16ppb	~18ppb	Ambient air	Ambient air, 5 min	Ambient air, 1 day	Ambient air, 5 min	~16ppb
Dry-down time (min)	119	103	15.4	15.6	233	129	318	77	13.9

APIMS used for valve experiments.

The results in Figs. 2 and 3 are summarized in the table, which lists the dry-down time to 1ppb for each of the experiments.

Conclusion

Metal-seated valves are preferred if fast initial startup is required and if the system can be heated above the maximum operation temperature of polymer-seated compounds. Polymer-seated valves compare well with all metal compounds after the system is well dried and moisture intrusions are brief to avoid diffusion into the core of the polymer seat. The more hydrophobic nature of polymer surfaces used as valve seats might even slightly improve dry-down after short system upsets, since the amount of moisture that is adsorbed on a polymer surface is smaller than on a similarly sized metal surface. ■

Acknowledgments

Kel-F is a registered trademark of 3M.

References

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