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Evaluating electronics-grade gas-line purging requirements

OVERVIEW Appropriate pre-purging methods required to safely expose specialty gas delivery lines to atmosphere are not well defined. However, this consideration is particularly important when a process gas is toxic, flammable, or reactive with atmosphere.

An informal review of current industry practice shows that numerous permutations of vacuum and pressure cycling as well as extended inert gas flows are used for pre-purging gas lines, but no optimized set of conditions has been established. Therefore, controlled laboratory studies and tests on a CVD tool delivery line were conducted to obtain a more complete understanding. Repeated vacuum-pressure cycling was effective at removing the bulk of the residual gas, but these cycles eventually had a diminished effect. Subsequent flow purge could ensure the removal of residual gas along the main gas flow path. Even after this main flow path is thoroughly purged, significant quantities of gas may remain trapped and can diffuse over time into the main path. These observations might, for example, be used to formulate purging requirements that eliminate unnecessary downtime while still providing adequate protection from microcontamination.

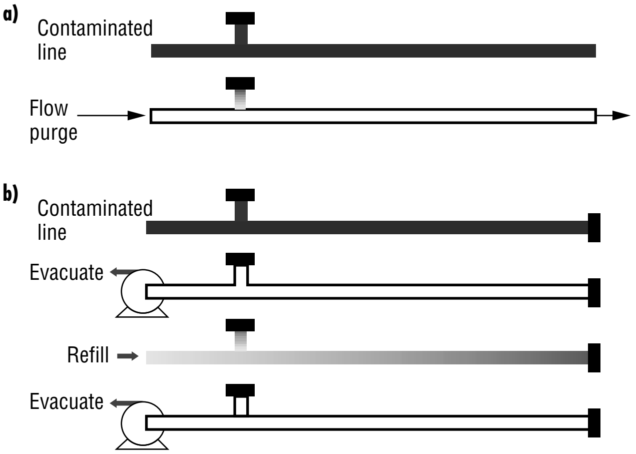

Figure 1. Schematic representation of the strengths and weaknesses of **a)** flow purge and **b)** vacuum-cycle purge for removing process gas from a delivery system. The simplified system depicted has one short unswept branch.

Process engineers know about the need to adequately purge atmospheric contaminants from process chambers [1] and gas delivery lines [2–5] prior to their use in semiconductor manufacturing; this has been the subject of numerous experimental and theoretical analyses. The related question of defining adequate "inerting" and purging of gas lines already charged with specialty gases just prior to opening them to atmosphere (e.g., for maintenance) is more complex and has been less thoroughly analyzed. Since many specialty gases are inhalation or reactivity hazards, the removal of a specialty gas from a system prior to maintenance is, of course, critical for worker safety. In addition, inadequate purging before or after exposing the system to atmosphere can also seriously compromise the purity of a gas delivery system.

This contamination concern is particularly relevant for air- or water-reactive gases, such as silane, tungsten hexafluoride, or boron trichloride, and for corrosive gases, such as hydrogen chloride or hydrogen bromide. Once residual quantities of such process gases are exposed to the atmosphere, an irreversible reaction that forms solids or other by-products can occur, permanently contaminating the delivery system. Similarly, moisture present in the atmosphere can potentially cause corrosion of metal surfaces when corrosive gases are present [6]. The deposition of reaction residue or corrosion products will contribute to particulate contamination of the process gas and may also make subsequent purging of the system less effective.

In recognition of the importance of proper process-gas purging, we see exhaustive purging regimens used in the industry. In the absence of definitive data to guide these procedures, however, valuable productivity of the process tool may be wasted by excessive or ineffectual purging practices. We have therefore undertaken an

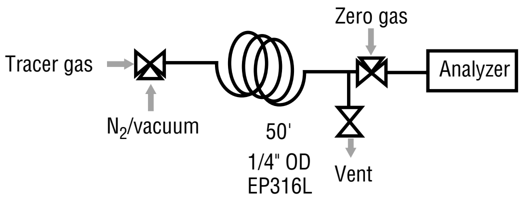

Figure 2. Experimental apparatus used for purging tests.

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experimental program to determine what parameters are most important when planning to purge a charged specialty gas system. Our work has resulted in general guidelines to aid in designing purging procedures.

Purging methods

In smaller gas distribution manifolds as found in gas cabinets, vacuum-cycle purging alone is effective, for instance, in removing contaminants before and after gas cylinder replacement. Anecdotal reports have suggested, however, that simple cycle purging is insufficient to thoroughly purge facility gas distribution lines before opening them for maintenance. Thus a common practice before opening a charged specialty gas line is to vent down the pressure and then perform some combination of flow purging and pressure-vacuum cycling using an inert gas such as nitrogen or argon. In theory, the two types of purging have complementary strengths and weaknesses (Fig. 1):

- In a flow purge, the inert gas is introduced into the system and is safely vented at a remote point. This technique is optimally effective at removing gas from the line directly between the gas inlet and outlet, but has little effect on side-legs and other unmixed areas of a gas distribution system (see Fig. 1a).
- In a cycle purge, the system is repeatedly pressurized with an inert gas and partially evacuated. This procedure is more effective in removing residual gas in the unmixed areas, but may require more time. Furthermore, vacuum cycling in relatively long, narrow, tubing runs is limited because of the poor mixing of the gas during refill, and thus contaminants may concentrate at the end of the tubing remote from the vacuum source (see Fig. 1b).

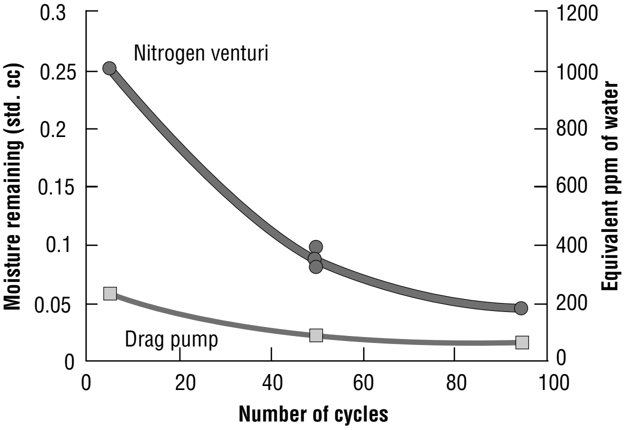

Figure 3. Comparison of the effectiveness of removing water vapor from 50 ft of EP316 tubing by cycle purging with a nitrogen venturi vacuum generator and with a molecular drag pump.

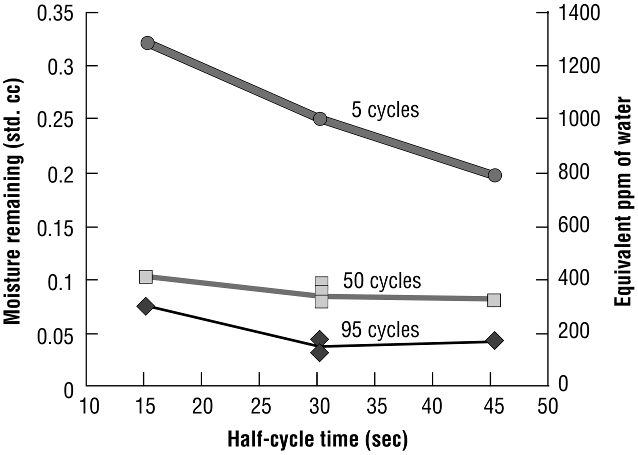

Figure 4. The effect of the number of venturi vacuum cycles and the cycle time on the removal of water from a 50 ft EP316L coil equilibrated at 50% relative humidity.

Laboratory tests

To better understand the fundamental variables that control the effectiveness of purging, we constructed a special test apparatus and procedure (Fig. 2): A 14-in.-dia. coil made from 50 ft of 1/4" EP316L tubing served as a model for a gas delivery line. To mimic both strongly adsorbing and weakly adsorbing impurities, we used water vapor (H_2O) and hexafluoroethane (C_2F_6) as tracers in our test. Before each experiment, we equilibrated the system with the desired tracer by establishing flow through the coil. We then sealed the outlet and performed the prescribed pressure-vacuum cycling at the inlet of the coil. At the end of cycle purging, we again established a flow through the coil with purified N_2 and recorded the concentration of the tracer as a function of the quantity of N_2 passed.

For water vapor tests, we investigated the role of cycle time, the number of cycles, and the vacuum pump on the amount of moisture left behind in the coil after cycling. As expected, there is a dramatic decrease in moisture remaining in the system as the number of pressurization-vacuum cycles is increased. This effect was most dramatic when the nitrogen venturi vacuum generator was used vis-à-vis a high-vacuum drag pump (Fig. 3). On the other hand, increasing the half-cycle time from 15 to 45 sec had a negligible effect except when the number of cycles was minimal (Fig. 4).

For removal of C_2F_6, we studied nitrogen venturi feed pressure (and vacuum level), purge gas pressure, the number of cycles, and the fraction of the one-minute cycles devoted to evacuation. We ran a screening experimental design with these four variables and evaluated their relative effect on the amount of C_2F_6 remaining in the lines. Figure 5 shows the normalized level average results; the size of the bars represents the amount of residual C_2F_6 in the line (i.e., smaller bars represent more effective purging). It is immediately evident that the supply pressure to the venturi (i.e., the better pumping speed and stronger vacuum) has the most pronounced effect. Higher refill pressure (e.g., 40 vs. 15psig) and longer evacuation time (50 vs. 30 sec) also improve purging. Surprisingly, the number of cycles did not show a strong correlation with the amount of C_2F_6 left behind in this experiment.

Field tests

To generalize our laboratory data into reliable guidelines, we needed to develop a benchmark with an actual specialty gas delivery system. Thus, we conducted an on-site test with a fully operational C_2F_6 delivery line to a chemical vapor deposition (CVD) tool in a semiconductor fabrication facility. The distance between the gas cabinet and the process tool was ~70 ft. These tests were done similarly to those in the laboratory: We charged the line to the process tool with C_2F_6 and then used the gas cabinet to conduct the programmed purge recipe. We established an inert gas flow from the gas cabinet and measured the concentration of C_2F_6 from a purge fitting just upstream of the tool's gas inlet, varying the number of cycles and the inert gas flow rate. In one experiment, we performed the evacuation using the tool's vacuum pump, which could achieve a lower pressure than the nitrogen venturi generator in the gas cabinet.

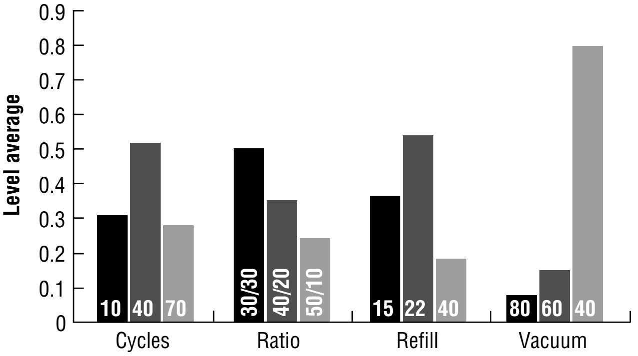

Figure 5. Normalized amount of C_2F_6 remaining as a function of the number of cycles (10, 40, or 70); evacuation time per cycle (30, 40, or 50 sec); refill pressure (15, 22, or 40psig); and vacuum generator feed pressure (80, 60, or 40psig).

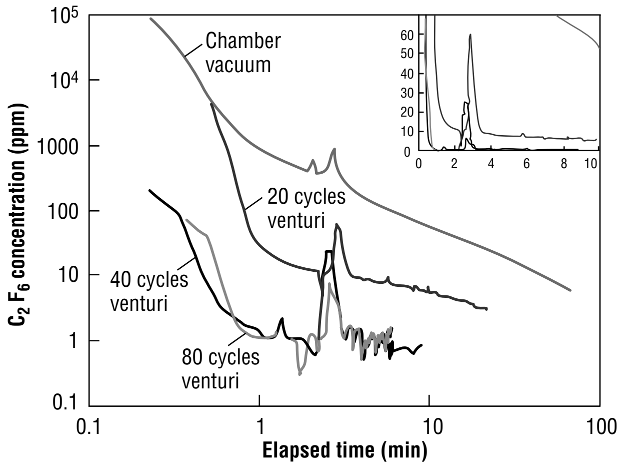

Figure 6. Results from purging C_2F_6 in a representative 70-ft delivery line to a CVD tool. The logarithmic scale in the main chart obscures the significant difference in the size of the concentration spike observed after 2 min, shown in the insert on a linear scale.

Figure 6 shows the results of several different purging conditions. From these data, several important results are immediately apparent: There was a significant reduction in the time to purge the system when the number of cycle purges performed at the gas cabinet was doubled from 20 to 40. Significantly, there was no further reduction in the time to purge down to a baseline concentration by doubling again to 80 cycles. This parameter, however, does not tell the entire story. As can be seen in the concentration profiles, there is a concentration spike that occurs ~3 min into the purge (at 1slpm, 45psig). This spike probably reflects gas remaining in a side branch, either in or near the gas cabinet, which has diffused into the purge gas flow path. As seen in the inset, increasing the number of vacuum cycle purges reduces the amount of gas released during this spike.

Interestingly, we also observed a concentration spike after the system was thoroughly purged when it had been left static for a period of time. To quantify this phenomenon, we stopped the inert gas purge flow once the effluent concentration of the tracer fell below 1ppm. After 30 min, we resumed flow and measured the maximum concentration of tracer. After flushing this concentration spike, we held the system static for 60 min and repeated the test, tabulating the results of these static accumulation experiments (see the table). The magnitude of these spikes may be high enough to be of concern, but does not seem to depend strongly on the cycle-purging procedure.

Results from C_2F_6 purge experiments

Run name	Cycles	Flow (sccm)	Total C_2F_6 (std. cm^3)	Max. concentration (% v/v)	30-min static (ppm-min)	30-min static (max. ppm)	60-min static (ppm-min)	60-min static (max. ppm)
20purge	20	230	0.58	0.41	105	250	132	280
20apurge	20	1000	0.84	0.41	—	—	—	—
40purge	40	1000	0.052	0.020	16.9	110.0	37	138
80purge	80	1000	0.021	0.0073	36	146	47	136
Vacpurge	1	1000	18.9	8.9	125	92	102	210
Vacpurg2	1	1000	20.0	7.0	106	290	94	240

Mechanical evacuation

In some situations, the vacuum source of the process tool is used to purge residual gases from the delivery lines. For comparison, we conducted a single, 10-min "extended" evacuation from the CVD tool's charged delivery line. As shown in Fig. 6, this purging was much less effective than the cycle purging that used a N_2-venturi vacuum generator. Although the base pressure of the process tool is far lower than that achievable with the venturi generator, evacuation from the process tool requires the gas to pass through a series of flow-restricting components (e.g., MFC, regulator, and filter) that hamper the tool's ability to evacuate the system effectively. As seen in the laboratory studies, better vacuum sources would be expected to dramatically enhance the effectiveness of purging, but this on-site experiment demonstrates that it is important to take potential flow restrictions into consideration.

Conclusion

It is important to properly design a purging procedure to ensure that residual process gas is thoroughly removed from a delivery system before attempting to open the system to the atmosphere for maintenance or component replacement. These procedures may entail a combination of vacuum-pressure cycling and flow purges. Even after flow purging brings the concentration of process gas at the outlet to a considerable level, there may still be an unacceptably high concentration of gas in dead-legs. In cases where there is a substantial amount of unswept volume in the system, adequate cycle purging is particularly important.

Also, after completion of purging to a safe level, it is advisable to maintain the purge flow, since reinstating flow after stopping can generate a relatively large spike of the process gas that propagates through the system. While cycle purging is significantly enhanced by using a stronger vacuum source, it is critical to take possible flow restrictions in the path to the vacuum source into consideration. By using the results of these laboratory and field experiments coupled with theoretical scaling factors to account for variations in delivery system design, it may be possible to more accurately design purging schemes for process delivery lines. Gas system integrity may thereby be maintained without wasting valuable time. ■

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References

1. N. Verma et al., "Kinetics and Mechanism of Gas Phase Purging in Process Tools and Components," in *Microcontamination 94 Conference Proceedings*, pp. 59–69, SEMATECH, San Jose, CA, October 4–6, 1994.
2. S. Dheandhanoo et al., *J. Inst. Env. Sci. Technol.*, 33, 1998.
3. S.M. Fine et al., "Optimizing the UHP Gas Distribution System for a Plasma Etch Tool," *Solid State Technology*, pp. 71–81, March 1996.
4. J. Sullivan et al., *J. Vac. Sci. Technol. A*, 10, 1869, 1992.
5. K.L. Siefering, W.H. Whitlock, *J. Vac. Sci. Technol. A*, 12, 1869, 1992.
6. S.M. Fine, R.M. Rynders, J.R. Stets, *J. Electrochem. Soc.*, 142, 1286, 1995.

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