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Technical Note

Analysis of moisture purge in high purity gas distribution systems

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Abstract

Moisture can easily adsorb on the inner surface of high purity gas distribution systems for semiconductor manufacturing processes, when the inner surface is exposed to the ambient air during regular or troubleshooting services. Before restarting the equipment, the adsorbed moisture has to be removed, typically by purging the gas distribution system with a high purity gas. An important system design issue therefore is to minimize the required moisture drydown time, so as to increase the productivity of the equipment. Here, the moisture purge/drydown process is analyzed using a simple phenomenological model, in which the moisture desorption kinetics is extracted from experimental data for moisture drydown in a single straight pipe. Based on that model, we also propose to minimize the overall moisture drydown time of a gas distribution system by properly allocating the purging gas flowrates in all branches of the system. It is demonstrated by a case study that, without altering the piping network design and total flowrate, our flowrate allocation scheme substantially reduces the overall moisture drydown time of the gas distribution system.

Keywords: Gas distribution systems; Moisture adsorption; Purge; Drydown time minimization

1. Introduction

The minimum feature length in modern integrated circuits (ICs) has been reduced to the submicrometer range [1]. The decrease in semiconductor device size, however, also brings about more stringent requirements on process gas purity. As a matter of fact, for ultralarge-scale integration (ULSI), it is necessary to decrease gaseous impurities down to low parts per billion (ppb) levels [2]. In particular, moisture is a critical impurity in semiconductor manufacturing processes, and its generation, transportation, and reduction have been studied extensively (see [3–7], for example, and the references cited therein).

Generally speaking, a high purity process gas is delivered to the actual points of use through a piping network with numerous valves, sensors, particle filters, and other components. Each of the components is a potential source of contamination. Moreover, in a newly installed or serviced gas distribution system, moisture almost inevitably is adsorbed on the inner surface of the system. Before (re)starting the equipment, the adsorbed moisture has to be removed, typically by purging the system with a high purity gas, and this purging process may take days or even weeks for larger systems [3]. During the purging stage, the adsorbed moisture continues to contaminate the gas flowing to the end points of the distributing system, so that the manufacturing processes there have to be halted. It is therefore highly desirable to minimize the required drydown time of a gas distribution system, so as to increase the productivity of the equipment, and here we shall address this important system design issue.

First, in Section 2 we shall discuss a simple model for the moisture purge/drydown process in a gas distribution system. The model builds on a mathematical model used previously by Haider and Shadman [3] to analyze their experimental data of moisture desorption from stainless steel tubes and alumina filter tubes. However, unlike the simple nth order moisture desorption kinetics assumed by

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Haider and Shadman [3] ($n \approx 1$ for their data covering exit moisture concentrations of several hundred ppb), we shall relate the moisture desorption rate to the surface concentration of adsorbed moisture using a more general phenomenological relationship, which can be determined from experimental data covering the entire moisture concentration range of interest. In particular, we shall determine the moisture desorption kinetics from the experimental data of Dheandhanoo et al. [7] for an electropolished stainless steel pipe (of 1/2-in. OD and 10 Ra surface finish). In their experiments, the adsorbed moisture was purged at room temperature and 6 atm (gauge pressure) by ultrahigh purity (UHP) nitrogen having a moisture content of less than 0.2 ppb, and the initial moisture condition in the pipe was about 150 ppb.

Now, as moisture desorption is a micro-scale phenomenon, intuitively its kinetics should depend on the surface finish and temperature, and the purging gas pressure, but not on the macro-scale geometry of a gas distribution system. So, one would expect that the moisture desorption kinetics determined from a single pipe should remain valid for a piping network consisting of pipe segments of the same material and surface finish as that of the single pipe, and operating at the same temperature and pressure. By the above reasoning, in Section 3 we shall use our phenomenological approach to analyze a gas distribution network previously studied by Dheandhanoo et al. [7]. It will also be shown that the drydown time predictions of our model are consistent with the experimental data of Dheandhanoo et al. [7] for the gas distribution network.

More importantly, on the basis of our phenomenological model, it is readily identified the possibility of minimizing the overall drydown time of the gas distribution system by properly allocating the flowrates in all branches of the network. We shall show that, without altering the piping network design, and keeping the total flowrate fixed, our minimizing scheme can substantially reduce the overall moisture drydown time of the system (by about 30% for the case studied). Finally, in Section 4, a number of general comments on system design from the viewpoint of moisture drydown time minimization will be given to conclude this paper.

2. Moisture drydown model

2.1. Mass balance

Here, we discuss a simple moisture purge/drydown model that forms the basis of this work. However, in order to justify some of the assumptions to be made in the model, let us first describe the experiments of Dheandhanoo et al. [7] in more detail. As mentioned in the previous section, the outer diameter of the tube used in Dheandhanoo et al. [7] is 1/2 in. Moreover, the length of the tube is 7.1 m and the flowrate of UHP nitrogen is 2 slm. Taking the tube wall thickness to be 0.065 in. (a standard product dimension; see, for example, www.swagelok.com), the average flow velocity is calculated to be 0.48 m/s and, using properties of nitrogen at 20 °C [8], the Reynolds number is about 300. The purging gas flow therefore is expected to be laminar. Also, the convection time scale is estimated by dividing the pipe length by the average flow velocity to be on the order of 10 s.

Now, Fig. 1 shows the experimental data of Dheandhanoo et al. [7] for the temporal variation of moisture concentration at the exit of the pipe described above. It is seen that the moisture concentration varies on a time scale of 100 min—much longer than the convection time scale (estimated above to be on the order of 10 s). So, clearly, the moisture concentration variation primarily is controlled by the mechanism of desorption from the inner surface of the pipe. Furthermore, in view of the disparity between the convection and desorption timescales, and the extremely low moisture concentration level (<150 ppb), it is appropriate to assume steady purging gas flow and quasi-steady concentration variation for the moisture purge/drydown process. An additional assumption here is that, for isothermal pipe wall and extremely low moisture content of the purging gas, the spatial variations of both the surface concentration and desorption rate of adsorbed moisture are negligible.

Accordingly, we deduce by mass balance that the moisture concentration at the pipe exit (C_{out}) exceeds that at the pipe entrance (C_{in}) by an amount proportional to the moisture desorption rate from the pipe surface:

$$C_{\mathrm{out}} - C_{\mathrm{in}} = AR_{\mathrm{d}}/Q, \tag{1}$$

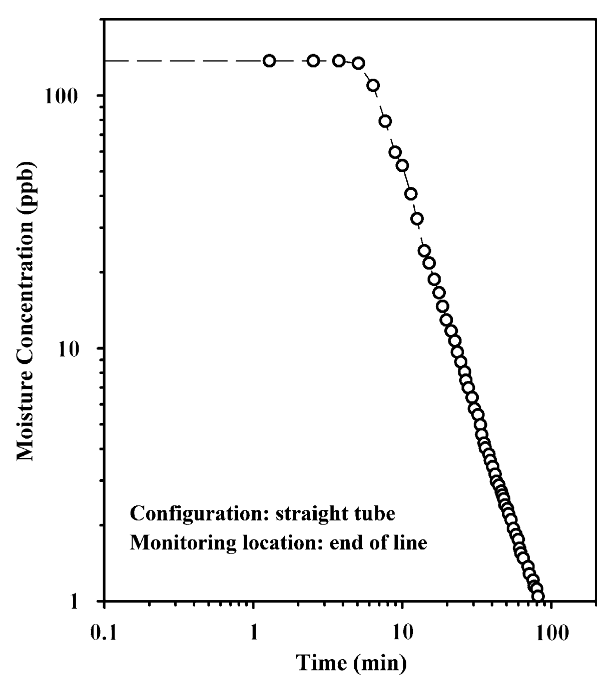

Fig. 1. Experimental data of moisture drydown in a straight pipe (adapted from Dheandhanoo et al. [7]). The system parameters are detailed in the text.

where A is the total pipe surface area, R_d is the moisture desorption rate per unit surface area, and Q is the volumetric flowrate of the purging gas (UHP nitrogen). Of course, moisture desorption from the pipe surface decreases the surface concentration of adsorbed moisture (denoted by γ below), so that we have again by mass conservation that

$$R_d = -d\gamma/dt, \tag{2}$$

where t denotes time.

So far we have only used mass conservation to construct a mathematical model for the moisture drydown process, and Eqs. (1) and (2) actually also appear in the model of Haider and Shadman [3]. To complete the modeling, however, one still needs a relationship between the desorption rate and surface concentration of adsorbed moisture. In particular, Haider and Shadman [3] assumed an nth order kinetics model for moisture desorption, i.e.,

$$R_d = k\gamma^n,$$

where k is a desorption rate constant. Note also that their experimental data cover exit moisture concentrations of several hundred ppb, and can be reasonably accurately fitted with $n = 1$.

However, as pointed out by Dheandhanoo et al. [7], for extremely low surface concentrations, the moisture is chemically adsorbed (as opposed to being physically adsorbed for higher surface concentrations), so that more complicated physicochemical models are needed to describe the moisture desorption kinetics (see, for example, the introductory treatise by Hudson [9]). In fact, such models have been constructed by Dheandhanoo et al. [7], and the resulting differential equations can be integrated numerically to accurately reproduce the experimental results shown in Fig. 1.

The main purpose of this paper, however, is to address the issue of moisture drydown time minimization for gas distribution systems, so we shall not discuss the details of the theoretical model of Dheandhanoo et al. [7]. Instead, we shall determine a phenomenological relationship between the desorption rate and surface concentration of adsorbed moisture from the data shown in Fig. 1. As pointed out above, such an approach is based on the intuitive understanding that since moisture desorption is a micro-scale phenomenon, its kinetics should not depend on the macro-scale geometry of a gas distribution system. So, the moisture desorption kinetics determined from experimental data for a single straight pipe should also be applicable to the analysis of moisture drydown in gas distribution networks consisting of pipe segments of the same material and surface finish, and operating at the same temperature and pressure. The general procedures for the determination of moisture desorption kinetics from experimental data will be demonstrated below using the data of Dheandhanoo et al. [7] as an example. As it turns out, our treatment of the desorption kinetics also allows us to analyze moisture drydown of gas distribution systems without the need for numerical solution.

2.2. Determination of moisture desorption kinetics

Let us now deduce the moisture desorption kinetics from the experimental data of Dheandhanoo et al. [7]. First, recall that the UHP nitrogen entering the pipe has a moisture content of less than 0.2 ppb, whereas the initial moisture condition in the pipe is about 150 ppb. It is therefore a good approximation to neglect the moisture concentration of the purging gas at the pipe entrance, and hence we shall take $C_{in} \approx 0$ below. Accordingly, for the gas flowrate $Q = 2$ slm and pipe surface area $A = 0.210\ m^2$ (as can be calculated from the pipe dimensions given above), the instantaneous moisture desorption rate R_d is determined from the data shown in Fig. 1 by use of Eq. (1). The results are shown in Fig. 2; note in particular that as $t \to \infty$ the data points are fitted with good accuracy by

$$R_d \sim 48.7/t^2, \tag{3}$$

where R_d is in ppb m/min and t is in minutes. The asymptotic expression (3) will be useful later for estimating the drydown time.

Now, Eq. (2) can be integrated to calculate the temporal variation of the surface concentration of adsorbed moisture, yielding

$$\gamma(t) = \gamma_0 - \int_0^t R_d(\tau)\,d\tau. \tag{4}$$

Furthermore, assuming that the adsorbed moisture eventually is completely desorbed by purging, i.e., $\gamma(\infty) = 0$, the initial surface concentration therefore is found to be

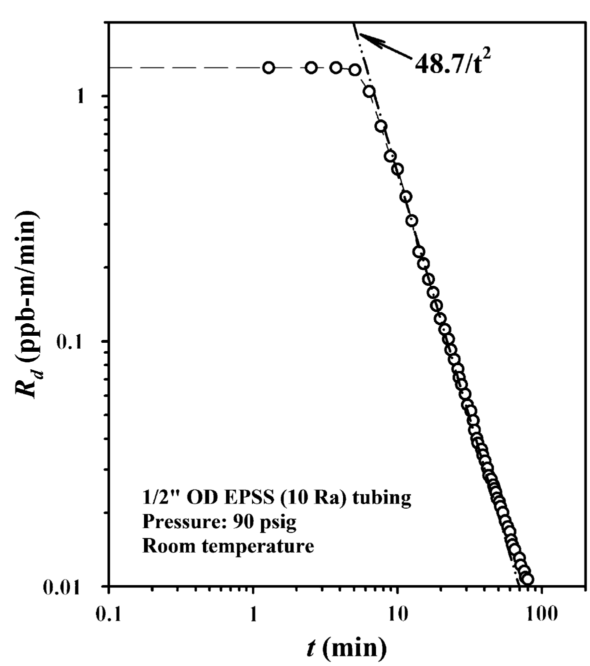

Fig. 2. Moisture desorption rate as a function of time, determined from the data shown in Fig. 1. The system parameters are detailed in the text.

$$\gamma_0 = \gamma(0) = \int_0^\infty R_d(t)\,dt. \tag{5}$$

Using Eq. (5), it is then calculated from the data shown in Fig. 2 that $\gamma_0 \approx 15.7$ ppb m. Also, using Eq. (4), the temporal variation of surface moisture concentration is calculated and plotted in Fig. 3. Note in particular that, consistent with Eq. (3),

$$\gamma \sim 48.7/t \quad (t \to \infty), \tag{6}$$

where γ is in ppb m and t is in minutes.

Then, we combine the data shown in Figs. 2 and 3, and obtain the relationship between the desorption rate and surface concentration of adsorbed moisture; the result is plotted in Fig. 4. As noted above, this relationship is expected to be applicable as well to the analysis of moisture drydown in gas distribution networks consisting of pipe segments of the same material and surface finish, and operating at the same pressure and temperature. (In the next section, we shall justify this argument by comparing the drydown time predictions of our model with the experimental data of Dheandhanoo et al. [7] for a particular gas distribution network.) Fig. 4 also shows that for the surface concentration range between 1 and 10 ppb m, the desorption kinetics approximately is of second order, whereas approximately first-order kinetics is observed for higher surface concentrations and more complex models (see, for example [7]) are needed to describe the kinetics for lower surface concentrations. However, for our present purpose of minimizing the drydown time of a gas distribution system, it suffices to use the results obtained above.

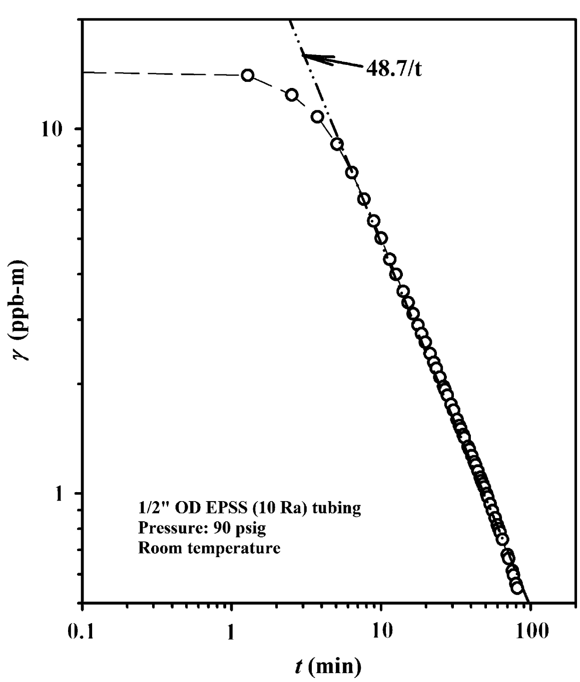

Fig. 3. Surface moisture concentration as a function of time, determined from the data shown in Fig. 2. The system parameters are detailed in the text.

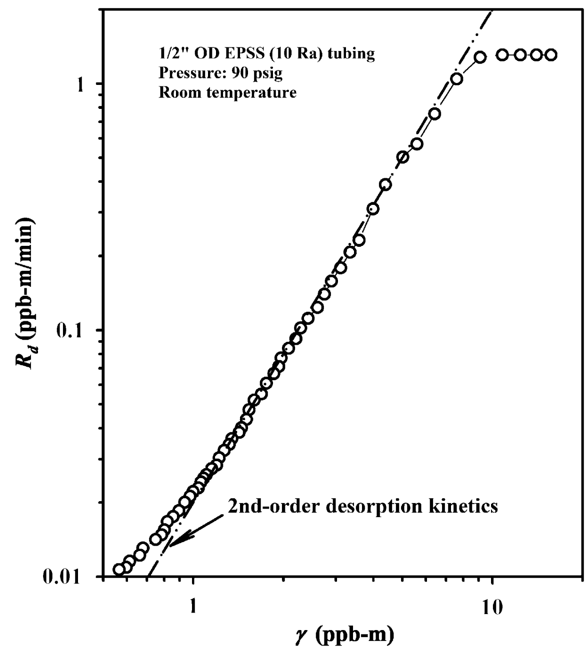

Fig. 4. Dependence of the moisture desorption rate on the surface concentration of adsorbed moisture, determined from the data shown in Figs. 2 and 3. The system parameters are detailed in the text.

3. Network drydown time minimization

Here, we shall demonstrate by a specific case study how the overall moisture drydown time of a gas distribution system may be minimized. It will become clear that the methodology may also be readily generalized to analyze general piping networks.

Fig. 5 shows the schematic of a gas distribution network studied by Dheandhanoo et al. [7], which will also serve the purpose of a practical case study here. Like in the single pipe experiments discussed in the previous section, here the total purging gas flowrate will be fixed at $Q = 2$ slm. The lengths of the main line segments of the network are $L_{M1} = 1.5$ m, $L_{M2} = 1.8$ m, and $L_{M3} = 3.6$ m, while the four branch line segments have the same length $L_{B11} = L_{B12} = L_{B21} = L_{B22} = 1.3$ m. The same labeling rules apply to the flowrate in each segment of the piping network as well (see Fig. 5).

Suppose now that the junctions of the network have negligible size and do not cause significant concentration jump across the junctions. It can then be assumed that

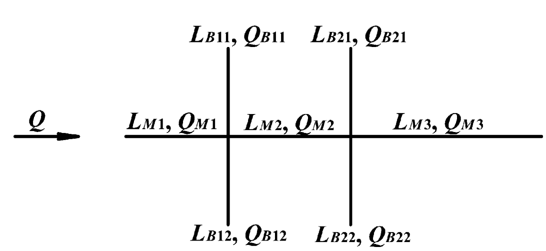

Fig. 5. Schematic of a gas distribution system studied here as a particular example.

the moisture concentration has a definitive value at each junction. Furthermore, the end of main line segment M1 can be considered as the common starting point of the M2, B11, and B12 segments. Similarly, the end of segment M2 simply is the common starting point of the M3, B21, and B22 segments. The simplification of continuous moisture concentration at each junction then allows us to determine the moisture concentration variation at each exit of the network in a particularly simple manner. Specifically, taking the exit of branch line segment B11 for example, as the exiting purging gas has traveled along the M1 and B11 segments, the moisture concentration there can be calculated from Eq. (1) to be

$$C_{\mathrm{out,B11}} = R_{\mathrm{d}}(A_{\mathrm{M1}}/Q_{\mathrm{M1}} + A_{\mathrm{B11}}/Q_{\mathrm{B11}}),$$

where A_{M1} and A_{B11} are the surface areas of the M1 and B11 segments. It is assumed that $C_{\mathrm{in}} \approx 0$, and R_{d} is the same for all segments.

The moisture concentration at other exits of the network can be calculated by the same token. In general, we may write for each exit

$$C_{\mathrm{out}} = R_{\mathrm{d}}A', \tag{7}$$

where the coefficient A' is the sum of the constituent A/Q ratios of all relevant segments. Table 1 lists the expressions for A' corresponding to the exits of the network shown in Fig. 5. Clearly, for given segment lengths the surface areas can be readily calculated. The values of the coefficients A', however, also depend on how the flowrates in all the segments are allocated. Therefore, in view of Eq. (7), the specific flowrate allocation scheme clearly would affect the temporal variation of moisture concentration at each exit of the network. In order to calculate the drydown time for each exit of the piping network, we need to be more specific about the flowrate allocation. So, below we shall consider two ways of flowrate allocation, and show how the overall drydown time of the system may be minimized.

3.1. Equipartitioned flowrate

Consider first the scenario that all the exits of the system have the same flowrate, i.e., $Q_{\mathrm{B11}} = Q_{\mathrm{B12}} = Q_{\mathrm{B21}} = Q_{\mathrm{B22}} = Q_{\mathrm{M3}} = 0.4$ slm. The value of the coefficient A' for each exit therefore can be readily calculated using the corresponding expression listed in Table 1. The results listed in Table 2 show that the exit of the M3 segment has the largest value of A' among all exits of the network. Therefore, for the exit

Table 1
Expressions of A' appearing in Eq. (7) for each exit of the network shown in Fig. 5

Exit	A'
B11	$A_{\mathrm{M1}}/Q_{\mathrm{M1}} + A_{\mathrm{B11}}/Q_{\mathrm{B11}}$
B12	$A_{\mathrm{M1}}/Q_{\mathrm{M1}} + A_{\mathrm{B12}}/Q_{\mathrm{B12}}$
B21	$A_{\mathrm{M1}}/Q_{\mathrm{M1}} + A_{\mathrm{M2}}/Q_{\mathrm{M2}} + A_{\mathrm{B21}}/Q_{\mathrm{B21}}$
B22	$A_{\mathrm{M1}}/Q_{\mathrm{M1}} + A_{\mathrm{M2}}/Q_{\mathrm{M2}} + A_{\mathrm{B22}}/Q_{\mathrm{B22}}$
M3	$A_{\mathrm{M1}}/Q_{\mathrm{M1}} + A_{\mathrm{M2}}/Q_{\mathrm{M2}} + A_{\mathrm{M3}}/Q_{\mathrm{M3}}$

Table 2
Results of drydown time calculations for the case of equipartitioned flowrate

Exit	A' (min/m)	$R_{\mathrm{d@3\,ppb}}$ (ppb m/min)	T_{dry} (min)
B11 and B12	119.2	0.0252	44
B21 and B22	164.2	0.0183	52
M3	337.5	0.0089	74
Optimized[a]	181.0	0.0166	54

[a] The issue of system optimization is discussed in Section 3.2.

moisture concentration to reduce below a prescribed level (here taken to be 3 ppb), it would take a smaller value of the desorption rate R_{d} (see Table 2), which can be readily calculated from Eq. (7). In other words, since the desorption rate decreases with time (see Fig. 2), the overall drydown time of the system thus is controlled by the M3 segment.

In fact, using the asymptotic expression for the desorption rate, Eq. (3), the drydown times for all exits can be estimated, and the results are also tabulated in Table 2. It is seen that while the gas exiting from the B11 and B12 segments would have satisfied the humidity requirement of 3 ppb moisture concentration after 44 min of purging, the M3 segment would take 74 min to satisfy the requirement. The above drydown time estimates compare favorably with the experimental data of Dheandhanoo et al. [7] (see Fig. 6), and hence provide some support for the intuitive reasoning that the supposedly micro-scale moisture desorption kinetics is not sensitive to the macro-scale geometry of the piping network. (As a matter of fact, the particular flowrate allocation producing the data shown in Fig. 6

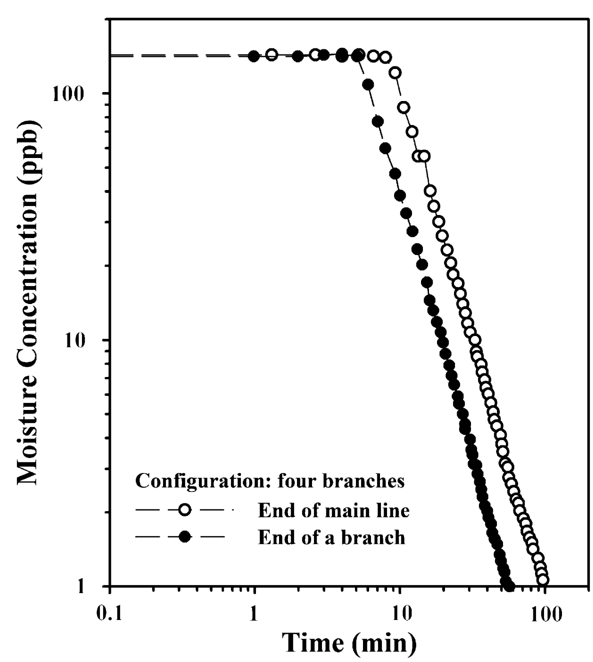

Fig. 6. Experimental data of moisture drydown in a gas distribution network consisting of a straight main line and four branches (adapted from Dheandhanoo et al. [7]). The system parameters are detailed in the text.

was not specifically described in Dheandhanoo et al. [7]. Based upon the above calculations, however, we speculate that in the actual experiment the flowrates at all the exits of the network are nearly the same.)

From the above example, it is clear that the drydown time for each exit of the network is controlled by the corresponding value of the coefficient A'. In particular, unequal values of the coefficients A' for the exits would render the drydown times different. In particular, while the moisture contents of the purging gas at some exits have been satisfactorily low, the system as a whole still has to wait for the slowest exit to dry down, and this is a waste of time and gas usage from a practical point of view. The key to minimizing the overall drydown time of a gas distribution system therefore is to allocate the flowrates in all segments of the piping network in such a way that the values of the coefficients A' for all the exits are equalized. It is interesting that in a wide variety of engineering and natural systems, the system performance typically is optimized when all parts of the system work equally hard (see the examples discussed in the very interesting book of Bejan [10]).

3.2. Optimized flowrate allocation

Now we present the computational technicalities leading to drydown time minimization. Since the B11 and B12 branches here have the same length (and hence the same surface area), and share the same main line segment M1, their flowrates should also be equal in order to have the same value of A' for both branches. The same argument also applies for the B21 and B22 branches. Therefore, suppose that the optimizing flowrates in both the B11 and B12 branches are

$$Q_{\mathrm{B11}} = Q_{\mathrm{B12}} = \alpha Q$$

and the flowrates in both the B21 and B22 branches are

$$Q_{\mathrm{B21}} = Q_{\mathrm{B22}} = \beta Q.$$

Then, by mass conservation, the flowrates in the main line segments are calculated to be

$$Q_{\mathrm{M1}} = Q, \quad Q_{\mathrm{M2}} = (1-2\alpha)Q, \quad Q_{\mathrm{M3}} = (1-2\alpha-2\beta)Q.$$

The task then is to choose the ratios α and β to equalize the values of the coefficients A' (whose expressions are tabulated in Table 1) for all exits.

With some straightforward algebra, the optimizing flowrate ratios are calculated to be

$$\alpha = \frac{A_{\mathrm{B11}}}{A_{\mathrm{M2}} + A_{\mathrm{M3}} + 2(A_{\mathrm{B11}} + A_{\mathrm{B21}})} \tag{8}$$

and

$$\beta = \frac{\alpha A_{\mathrm{B21}}(A_{\mathrm{M2}} + A_{\mathrm{M3}} + 2A_{\mathrm{B21}})}{A_{\mathrm{B11}}(A_{\mathrm{M3}} + 2A_{\mathrm{B21}})}. \tag{9}$$

For the piping network under consideration, we find from Eqs. (8) and (9) that the optimizing flowrate ratios are $\alpha = 0.122$ and $\beta = 0.158$, respectively. Also, at the optimized flowrates, all exits of the network share the same value of the coefficient A':

$$A'_{\mathrm{B11}} = A'_{\mathrm{B12}} = A'_{\mathrm{B21}} = A'_{\mathrm{B22}} = A'_{\mathrm{M3}} = A_T/Q, \tag{10}$$

where $A_{\mathrm{T}} = A_{\mathrm{M1}} + A_{\mathrm{M2}} + A_{\mathrm{M3}} + 2(A_{\mathrm{B11}} + A_{\mathrm{B21}})$ is the total surface area of the system. (Recall that here $A_{\mathrm{B12}} = A_{\mathrm{B11}}$ and $A_{\mathrm{B22}} = A_{\mathrm{B21}}$.) It can be readily deduced from Eq. (10) that, in general, reducing the surface area of the system and increasing the purging flowrate would further decrease the value of A', and hence reduce the overall moisture drydown time of a gas distribution system.

For the present piping network, however, taking the total flowrate to be $Q = 2$ slm as before, the optimized value of A' (same for all exits) is calculated to be 181.0 min/m. Accordingly, by Eq. (7), the exit moisture concentration becomes 3 ppb when the desorption rate $R_{\mathrm{d}} = 0.0166$ ppb m/min. Furthermore, using Eq. (3), the drydown time (same for all exits) is estimated to be 54 min. Compared with the case of equipartitioned flowrate (results summarized in Table 2), we have thus saved the drydown time (and hence gas usage) by about 30%, simply by properly allocating the flowrate in each branch of the piping network.

Finally, it is worth emphasizing that despite this is just a particular case study, the strategy of minimizing the moisture drydown time of a gas distribution system by flowrate allocation is applicable for general piping networks. Also, the calculations carried out above can be readily generalized to other more complex settings.

4. Concluding remarks

Here, we have discussed a simple model for the moisture purge/drydown process in a gas distribution system. While the more fundamental model of Dheandhanoo et al. [7] has to be integrated numerically to predict the evolution of moisture concentration in a gas distribution system, our model allows us to calculate the moisture concentration evolution analytically, at the expense of having to determine the moisture desorption kinetics from experimental data. We therefore do not claim to have constructed a more accurate or powerful theoretical model for moisture desorption.

The simplicity of our approach, however, points out the possibility of minimizing the overall drydown time of a piping network by properly allocating the flowrate in each segment of the network. As the practical case studied here in Section 3 indicates, the saving in drydown time and gas usage can be significant (as much as 30% in the case study). The minimizing strategy and the calculation methodology therefore are the main contributions of this work.

We also emphasize in closing that, although we have only presented a particular case study, the calculations can be readily generalized to other more complex settings. Furthermore, from the viewpoint of minimizing the drydown time of a gas distribution system, it is advisable to reduce the surface area of the system and increase the purging flowrate, if such options are at one's disposal.

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