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# Thermal-based mass flow control for SDS gas delivery systems

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**The economic advantage of Safe Delivery Source gas source feed materials depends largely on maximum use of cylinder contents. In early applications, more than 50% of the usable contents remained in the cylinder. Recent improvements in flow control technology have made it possible to utilize as much as 90% of the gas cylinder contents with bottle pressures below 8 torr. This article discusses the design and operating principles of thermal-based flow controllers and emphasizes design features for applications with inlet pressures below 8 torr. It will also show some additional advantages for thermal-based flow controllers designed for low input pressures over alternative mass flow control methods.**

Hazardous ion implant doping gases such as  $\text{AsH}_3$ ,  $\text{PH}_3$ ,  $\text{SiF}_4$  and  $\text{BF}_3$  can be stored safely in Safe Delivery Source (SDS) cylinders. The gases are stored by adsorption on a porous mass at subatmospheric pressure inside the cylinder, virtually eliminating the risk of catastrophic failure. The lower the operating pressure of the cylinder, the more gas can be extracted.

When the first subatmospheric SDS cylinder was successfully demonstrated in 1992, it was assumed that conventional thermal mass flow controllers (MFCs), which were designed for high-pressure applications, would not be suitable for SDS applications in ion implant because of the high pressure drop across the MFC. The 50–100 torr pressure drops produced by these thermal MFCs [1] resulted in more than 50% of the potentially usable contents remaining in the cylinder.

But when optimized for subatmospheric applications, thermal MFCs can control gas flow with inlet pressures below 8 torr,

and they offer other advantages over needle valve and pressure-based control methods, including greater flow stability and faster response times. With pressure-based flow controllers, response time at low pressures can be as much as 10× greater than that of thermal MFCs operating at low pressures.

The cost-effective use of SDS is primarily dependent on maximum cylinder utilization, and maximum desorption of the gas depends on the pressure drop across the flow control device. Reducing the pressure drop to below 8 torr can allow increased use of the SDS gas cylinder to as much as 90% (Fig. 1).

In needle valves, flow is proportionate to pressure. When used in SDS applications, the input pressure continuously drops and the needle valve requires constant adjustment in order to maintain the same flow into the chamber. Consequently, failure to make the necessary adjustments will result in insufficient flow of gas into the chamber or premature cylinder replacement.

## Theory

The thermal-based mass flow sensor uses the thermal properties of a gas to measure the mass flow rate directly:

$$\text{Sensor output} = m C_p \Delta T$$

where  $m$  is the mass flow rate,  $C_p$  is the specific heat at a constant pressure, and  $\Delta T$  is the net change in gas temperature.

Each gas molecule has a specific ability to pick up heat. This property, called specific heat ( $C_p$ ), directly relates to the

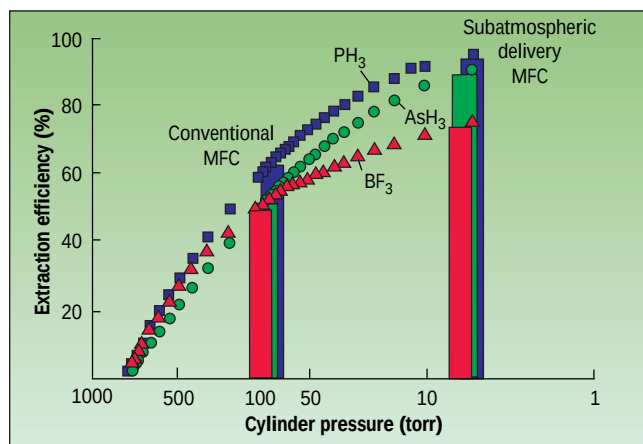


Figure 1. Gas use rate efficiency for an SDS cylinder.

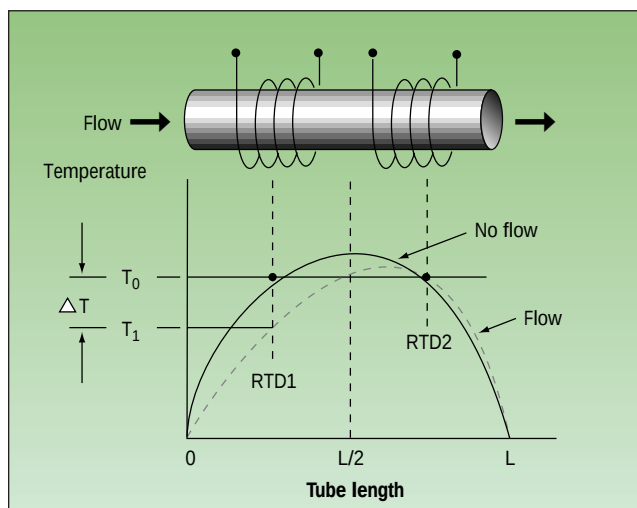


Figure 2. Gas is heated in a sensor tube and resistive temperature sensors measure the gradient along the tube.

mass and physical structure of the molecule. At reduced pressures, as are found in SDS applications, the gas behaves like a perfect gas and  $C_p$  becomes constant [2]. The heat capacity of a perfect gas is a function of temperature alone [3].

In a thermal mass flow sensor, gas is heated in a sensor tube and resistor temperature devices measure the temperature gradient along the tube (Fig. 2). The primary mechanism creating the temperature difference is the mass transport of the gas carrying the thermal energy gained in contact with the heated element by virtue of its heat capacity. For a given amount of heat applied to the gas, the temperature change of the gas is a function of mass flow as given in  $Q = m C_p \Delta T$ , where  $Q$  is the thermal flux,  $m$  is the mass flow,  $C_p$  is the thermal capacity, and  $\Delta T$  is the change in temperature.

The theory indicates that mass flow is measurable independently of gas pressure, which means that thermal sensors can inherently operate beyond the limits of laminar flow into the molecular flow region of fluid dynamics. This makes thermal MFCs ideal for reduced-pressure applications.

## Operation

**Pressure range.** High-pressure MFCs operate in the laminar flow region where gas flow characteristics are predictable. Understanding gas flow characteristics in the rarefied flow region is essential to increasing the operational pressure range of the thermal MFC. At subatmospheric pressures, gas velocities increase due to the reduced gas density for any given flow rate. When the gas velocity reaches Mach 1, a “choked flow” condition is produced, thus restricting further flow. To increase the flow under choked flow condition requires an increase in gas density or upstream pressure. This was one of the problems limiting low-pressure applications of early thermal MFCs, and it drove the need for a new approach to the thermal sensor and valve design. One such approach is used in a new thermal-based MFC, which features a sensor designed to address the special conditions presented in rarefied flow; it also includes flow improvements in the valve and reduced dead space volumes, allowing flow rates of 10 sccm below 8 torr (Fig. 3).

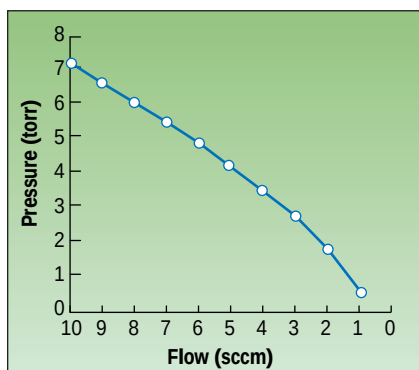


Figure 3. Minimum pressure drop.

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**Stability.** The changing pressure differential across the MFC in

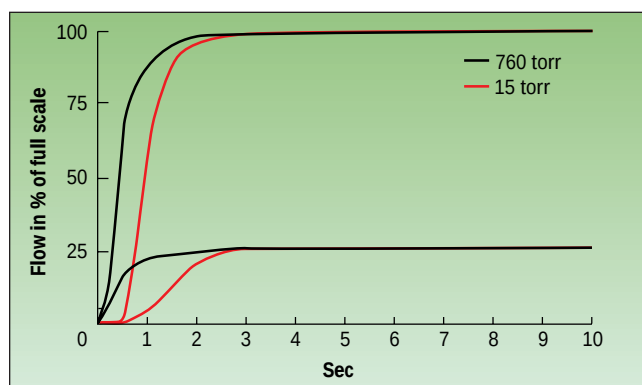


Figure 4. Typical response times at 760 and 15 torr.

SDS gas applications challenges the sensor and valve tuning to deliver a stable and linear flow across the operating pressure range. Consistent operation of the implanter depends on stable flow over the full pressure range of the SDS bottle. In thermal-based MFCs specially designed for subatmospheric pressures, MFC performance is the same at 15 torr as it is at 760 torr.

The thermal sensor coupled with a magnetic solenoid proportional control valve offers almost infinite resolution; gas flow remains stable at both extreme high and low pressures. The matched and tuned electronic circuitry compensates for the thermal lag time of the sensor to changes in flow. The reduction of internal flow volumes eliminates dead space and pneumatic lag. Response characteristics are stable and repeatable across the SDS bottle pressure range without the need to retune the MFC. Set point response times are relatively fast, achieving <3 sec at 10 torr. Typical response times at 760 torr and 15 torr are shown in Fig. 4.

**Calibration.** In the past, calibration accuracy was not an issue. Doping gases were diluted with hydrogen. The low doping concentration and higher flow rates reduced the requirement for flow control accuracy. The 100% concentration of the SDS source gas places greater requirements of accuracy and flow repeatability on the flow controller. In SDS applications, controllers must deliver accurate flows from as low as 0.3 sccm to 10 sccm with gas inlet pressures from 760 torr to below 8 torr. The use of primary calibration standards, where flow measurement is based on mass flow and time, provide a low flow accuracy of  $\pm 1\%$ .

The thermal mass flow sensor relies on the established thermal mass properties of gases. The  $C_p$  of implant gases and the flow relationship with respect to nitrogen are well defined. The primary sensor flow relationship between nitrogen and any other gas is the ratio of their respective molar  $C_p$  values. The temperature dependence of  $C_p$  is associated with the molecule’s rotational and vibrational degrees of freedom represented by  $N$ . Since a mole of any perfect gas occupies the same volume at standard conditions (22,414.00 cc / mol at 0°C and 1 atm pressure), it follows that molar specific heat is the specific heat (J / gm K) multiplied by the standard density (gm / mol):

$$C_p \times \rho_s = (\text{J / gm K}) \times (\text{gm / mol}) = \text{J / mol} \times K$$

where  $\rho_s$  is the standard density. The formula for the conversion factor then becomes:

$$CF = (N_{\text{gas}} / N_{\text{N}_2}) \times (C_p \times \rho_s)_{\text{N}_2} / (C_p \times \rho_s)_{\text{gas}}$$

Table 1. Conversion factors

| Gas type  | $N$  |
|-----------|------|
| Monatomic | 1.04 |
| Diatomic  | 1.00 |
| Triatomic | 0.94 |
| Polytomic | 0.88 |

with  $N$  shown in Table 1. For example, a sensor flow of 5-sccm nitrogen will correspond to a flow of 3.66-sccm phosphine or 3.36-sccm arsine (Table 2).

The molar-specific heat ratio allows the use of surrogate calibration gases that are nontoxic and noncorrosive. The surrogate gas thermodynamic and fluid dynamic characteristics are matched to the specific process gas. This is called gas-specific calibration.

Maximum bottle use is obtained through gas-specific calibration. The sensor, bypass, and valve are optimized to take advantage of the fluid properties of the specific gas,

Table 2. Gas flow correction factors

| Gas            | Thermal MFC |
|----------------|-------------|
| $\text{N}_2$   | 1.0         |
| $\text{AsH}_3$ | 0.742       |
| $\text{PH}_3$  | 0.673       |
| $\text{BF}_3$  | 0.528       |



resulting in a lower pressure drop across the thermal MFC. This is most important when flowing SDS  $\text{BF}_3$ , because of the higher cost / gm compared to high-pressure  $\text{BF}_3$ .

**Low pressure characterization.** The valve test point (VTP) voltage provides a simple and accurate means of characterizing an individual flow controller. Thermal MFCs used in subatmospheric applications should be individually tested and characterized for flow and pressure performance from 100 torr down to 5 torr prior to shipment (Fig. 5).

The VTP signal demonstrates stability of flow control at the lowest pressures. The VTP increases slowly and steadily as the operating pressure drops from 760 torr to the 10 torr range. When the device reaches the limits of the operating pressure range, the VTP increases rapidly. At any set flow rate, a VTP of 10 V may be used to indicate a warning of low pressure, while  $\geq 12$  V may be used to indicate the end of bottle. Flow control will continue until the VTP reaches 14 V, when the valve is fully open. Flow at that time will resemble flow through an orifice, limited by the total pressure drop across the gas path.

In conjunction with the bottle pressure transducer, the VTP voltage is useful in determining and troubleshooting MFC or system flow problems. A filter or line restriction will produce an area of high-pressure drop; the valve must open more, producing a higher valve voltage earlier in the bottle lifetime. The VTP voltage alone would lead to premature bottle replacement. Using the MFC VTP voltage and a bottle pressure transducer prevents premature bottle replacement.

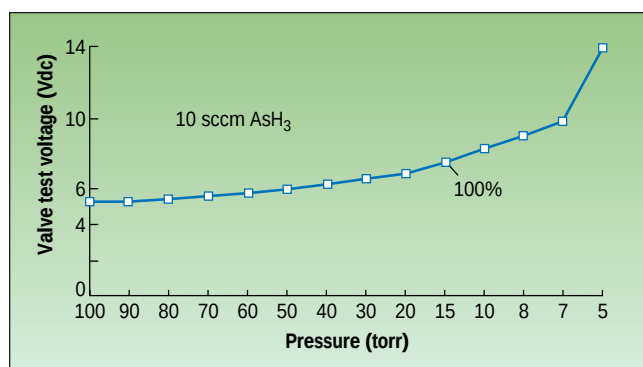


Figure 5. The VTP signal demonstrates the stability of flow control at the lowest pressures.

**Construction.** The construction of the sensor and valve in the latest thermal MFCs designed for SDS applications can operate up to 5 psig in a control mode and deliver a proof pressure of 2000 psig. Purge gas pressures of 150 psig can be safely used without fear of damage to the sensor or valve components.

Improvements to the MFC design extend beyond reduced pressure drop and flow characteristics. The all-metal valve construction reduces particle generation, while the elimination of all elastomers in the seals and valve makes gases compatible. Metal seat valve leakage is  $<1\%$  at 10 psig.

**Economics.** Safety was the primary reason for developing the SDS system, but it is also economical if maximum cylinder contents are used. High-pressure MFCs resulted in 50% of the product remaining in the SDS bottle, with ending pressures of 100 torr. This disproportionate relationship of pressure to extracted product drives the need for lower-pressure-drop MFCs. At 10 torr, 90% can be extracted and at 8 torr,  $>90\%$ . The ability to use SDS cylinders to below 8 torr offsets the higher cost of the special MFC [4].

Further savings are realized in additional tool uptime. Cost savings models place tool uptime from \$500–\$3500/hr [4, 5]. Bottle pressures  $<8$  torr translate into days of additional tool-processing time. Actual tool uptime is dependent upon multiple factors, including flow rate, specific gas, bottle size, process time, and final bottle pressure.

## Summary

Increased cylinder use and tool uptime are the first order of economic payback in a SDS gas source delivery system. Though safe delivery of highly toxic gases is its principle feature, the cost cannot be ignored. To make the SDS gas source delivery system economical for ion implantation requires that ending bottle pressure be between 8 and 20 torr. Economic advantages increase at  $<8$  torr.

Specifically developed sensors and improved flow through the valve provide both high and medium beam current implanters with a cost-effective delivery system for arsine, phosphine, silicon tetrafluoride, and boron trifluoride. Utilization of cylinder contents to 90% can be realized at flow rates  $\leq 10$  sccm. ■

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