Novel Safe Approach to Process Gas Delivery

INTRODUCTION

Gases are typically available in a compressed (high pressure) format, or in some cases, set to deliver at lower pressures — typically subatmospherically. Collectively Subatmospheric pressure Gas Systems (SAGS), have been differentiated[1] in the fire,[2] insurance,[3] and transportation[4,5] codes as uniquely separate from compressed gases.

The adoption of SAGS gas storage and delivery technologies has occurred steadily over the last 25 years, mainly in the semiconductor industry. Used initially to supply highly toxic dopants for ion implantation, the number of tool types and applications employing SAGS continues to increase every year. In fact, offerings based on SAGS technology have now expanded to 50-liter and larger cylinders, support a wide variety of gases and gas mixtures, and can even be calibrated to deliver at low positive pressures for processes that require positive pressure.

This article examines how SAGS function, where they've found utility, and suggests future uses in semiconductor manufacturing, various other industry applications, as well as laboratory settings.

DESCRIPTION

The first reference to SAGS in NFPA's standard 318 ("NFPA318") occurred in 2008. The definitions and specifics, included below, have been refined as the technology evolved and implementation methods and benefits were better understood. Currently, SAGS are defined by both the storage pressure inside the cylinder and the delivery pressure leaving the cylinder.

> 3.3.35.5.1 *Subatmospheric Gas Storage and Delivery Source (Type 1 SAGS)*. A gas source package that stores and delivers gas at subatmospheric pressure and includes a container (e.g., gas cylinder and outlet valve) that stores and delivers gas at a pressure of less than 14.7 psi at NTP.

> 3.3.35.5.2 *Subatmospheric Gas Delivery Source (Type 2 SAGS)*. A gas source package that stores compressed gas and delivers gas at subatmospheric pressure and includes a container (e.g., gas cylinder and outlet valve) that stores gas at a pressure greater than 14.7 psi at NTP and delivers gas at a pressure of less than absolute pressure of 14.7 psi at NTP.

It is the intent here to focus on the Type 2 SAGS, referred hereafter as a Vacuum Actuated Cylinder (VAC®). Type 1 SAGS technology has been described in detail elsewhere[6,7] and has found utility in a wide range of applications where intrinsically-safe subatmospheric gas supply is desired.

The VAC operates as its name implies, allowing flow only when a demand pressure below a subatmospheric threshold pressure has been achieved. Its basis is the incorporation of set pressure regulators (SPR) embedded within the cylinder body and located upstream of the primary cylinder valve. Thus, while the pressure inside the storage cylinder can exceed 1500 psi, the gas leaving the VAC cylinder is 500 torr, nominal.

Figure 1. Gas stick assembly.

The orientation of the regulators relative to the cylinder valve is shown in Figure 1. The gas stick assembly, which includes a particle filter, is welded to the base of the cylinder valve and resides completely within the cylinder body. Gas is introduced into the cylinder using a separate dedicated fill port.

The heart of the SPR is an internal pressure sensing assembly (PSA) used to both actuate and attenuate gas flow and control discharge pressure, as illustrated in Figure 2. The PSA is a sealed, edge welded bellows unit, which is calibrated by back filling with an inert gas to a preset pressure. When pressure less than the PSA set point is applied downstream, the welded bellows expands unseating a poppet and allowing gas to flow through the regulator and around the PSA. With flow initiated, the bellows expands and contracts accordingly to limit gas flow and control the downstream pressure. An added benefit to this design is that the contents of the cylinder cannot be contaminated if the delivery port is exposed to other materials.

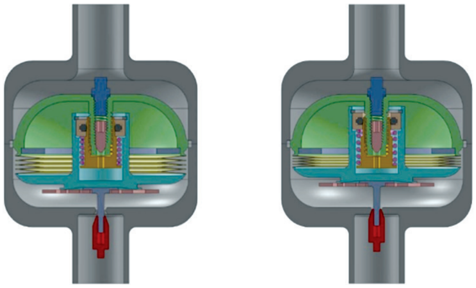

Figure 2. Section view of the SPR. Left image flowing, right image no flow.

As the poppet is normally closed, its design will leave it closed in the unlikely event of a failure. Should the PSA lose its calibrated charge pressure, the system will require a stronger applied vacuum (i.e., lower absolute pressure) to operate or it will ultimately fail in the closed position.

The complete subatmospheric delivery pressure cylinder configuration is shown in Figure 3. The cylinder shell is fully DOT compliant and utilizes either a manual or pneumatic cylinder valve.

Figure 3. Section view of the VAC cylinder.

While initially designed to operate at subatmospheric pressure, the VAC SPR can also be calibrated to operate at a nominally low super-atmospheric pressure, e.g., 50 – 100 psi, to meet application requirements. This implementation of the VAC technology has enabled users that require super-atmospheric gas delivery pressure for their application.

Users and gas component suppliers recognize the benefit of a low positive pressure for safety as well as equipment reliability purposes. Higher pressure within the gas delivery system typically reduces component lifetimes. One valve supplier has conducted a study and reported that cylinder valve lifetimes are significantly increased when operating pressure is capped at 100 psi.

SAFETY IMPLICATIONS AND LOW PRESSURE GAS DELIVERY

All gas cylinders, high pressure or SAGS, can have connection problems if proper procedures are not followed or other failures occur. The difference will be in the impact to the operators, equipment, and environment.

Worst case release rates for gases are predicated on gas pressure and flow through an orifice. In the 1980's the use of restrictive flow orifices (RFO) became commonplace and allowed users to develop more comprehensive safety practices, e.g., setting minimum ventilation rates for gas enclosures as well as sizing scrubbers to abate releases. The RFO effectively limited gas releases to more manageable levels but didn't eliminate them.[8]

Type 1 SAGS

In the case of type 1 subatmospheric pressure gas delivery, a prospective release will be very small both in rate of release, as well as the total mass lost. As the gas is adsorbed and without a pressure driver, releases are diffusion controlled. In contrast to a high pressure gas source, the SAGS 1 effectively eliminates gas releases as a concern.

Type 2 SAGS

A VAC gas source also effectively removes pressure as a risk element. This occurs as the embedded regulator will not permit flow if the 500 torr threshold set pressure is not achieved. In an integrated gas delivery system, detection of an upward deviation from the set pressure is configured to trigger the cylinder pneumatic isolation valve to close isolating gas flow.

Low Pressure Delivery Systems

The PDS®+100 is a new class of gas cylinder based on the VAC technology, set for low super-atmospheric delivery pressure. These cylinders are used when a process requires a positive pressure to operate. The advantages of operating at 50 to 100 psi vs 500 to 1500 psi are discussed in the following section. Cylinders in this category are no longer classified as a SAGS package but offer additional safety and reliability benefits over a typical high pressure cylinder.

Release Rate from Cylinders

Increasing the deliverables from a compressed gas cylinder and beneficially decreasing cylinder replacement frequency is highly desirable, but not when it comes with proportionately increased risk. In response, manufacturing facilities have increasingly moved to bulk delivery for pyrophoric materials like silane.

To illustrate this point, consider the case for delivery of silane at various pressures in a standard 49 L cylinder with a 0.010" RFO[8] as shown in examples A, B, and C in Table 1 below. Most users and jurisdictions eschewed the 15 kg package because of the high worst case release (WCR), and risk that came with it.

Consider the same situation using a low pressure delivery system [PDS +100] but where the outlet pressure is pre-regulated to 100 psi. The examples D and E employ a 0.010" and 0.014" RFO, respectively and reduce the WCR from the 15 kg package by 10 – 20 fold.

Safer gas packaging enables higher productivity. In the silane example, expected savings arise from reduced labor/better logistics (8%), better cylinder utilization (heels) (4 – 7%) and reduced qualifications, system reliability, and exhaust reduction (3 – 6%).[9]

CASE STUDY – ENABLING SAFETY, EFFICIENCY, AND PROCESS PERFORMANCE

The following case demonstrates the advantages of reducing the worst case release (WCR) potential. In a recent experience, a user was being restricted by local authorities to using a ~20% phosphine gas source (1.5 Kg PH_3 with cylinder pressure >1500 psi) instead of neat high pressure gas. When the user decided to use a VAC-based cylinder, they were permitted to use 100% phosphine (neat gas, 20 Kg PH_3), a greater-than tenfold on-site quantity increase. In addition to the favorable logistics of fewer cylinder changes, there was an improvement in the tool and process performance.

EXAMPLE OF SAGS IMPROVING PERFORMANCE AND EFFICIENCY

Prior to the introduction of SAGS technology gas, process gases were delivered as a very dilute mixture. Phosphine and arsine, for example, were typically limited to very small cylinders of 15% gas in balance hydrogen. This was due to the risk of releasing these highly toxic gases from the high pressure cylinders. With the introduction of SAGS, suddenly users were able to use 100% phosphine, arsine and other toxic gases in an inherently safe package/gas cylinder. This provided huge benefits to the users. Ion implanter throughput, tool performance and time between

Table 1. Worst case release rates for different cylinder configurations

Configuration	RFO size	Cylinder pressure	Outlet pressure	WCR	Mass
A	0.010"	800 psi	800 psi	30.5 slpm	5 kg
B	0.010"	1200 psi	1200 psi	55.7 slpm	12 kg
C	0.010"	1600 psi	1600 psi	70.8 slpm	15 kg
D	0.010"	1600 psi	100 psi	3.5 slpm	15 kg
E	0.014"	1600 psi	100 psi	6.9 slpm	15 kg

maintenance events were all drastically improved. The impact of SAGS technology to the world-wide ion implanter base allowed improved fab design and reduced the number of implanters required due to significantly higher tool availability and higher tool throughput.

Ventilation rates for gas enclosures are set based on the expected worst case release rate. Texas Instruments has taken advantage of the low release rates expected from Type 1 and Type 2 SAGS to achieve energy savings[10] with their (ion implant) process tools. They have implemented the following changes:

- Returning all of the terminal enclosure exhaust air back to the factory for direct reuse, reducing the overall tool make-up air requirement by 75%
- Reducing the gas box ventilation rates by about 50% and reclassifying it as general exhaust

Returning the ion implanter process tool shell exhaust to the factory rather than expelling it to the environment avoids the energy costs of moving and conditioning make-up air to replace it and saves an estimated $8000 per tool per year. Additional capital savings were achieved throughout the make-up air system simply by reducing its size proportionately. Reducing the individual gas box exhaust rate resulted in a savings of $800/tool.

GAS MIXTURES ENHANCING PRODUCT RESULTS AND ENABLING NEW PROCESS

Unlike the very dilute inefficient gas mixtures that had been used in Ion Implant prior to the introduction of SAGS, a new class of highly specialized gas mixtures are being widely adopted. VAC-based SAGS cylinders have been adopted to supply specialized gas mixtures which can enhance process performance, improve product results and even enable new processes. Because of the unique regulator-based design, gas mixtures incorporating various-sized molecules will flow at a consistent mixture ratio at varying gas flow rates and also over the cylinder deliverable-gas lifetime. This has been shown to provide great benefit over the co-flow of multiple gases from separate gas cylinders, where variables such as mass flow controller (MFC) calibration and variation – either within-tool or tool-to-tool, can cause process results and product performance to vary. There is a trend of users adopting gas mixtures from VAC-based SAGS cylinders to limit risk while achieving the benefits of the precision mixture.

SUMMARY

The risk of using hazardous materials arises from potential release scenarios that can be postulated to occur during transport, storage, use and cylinder change outs. The likelihood of “release incidents” is affected by cylinder delivery pressure, replacement frequency, and safety training/experience of the technical staff.

As an added benefit, operating gas delivery systems at reduced pressure will extend the lifetime and performance of valves and regulators, as well as other components in a gas manifold.

Supplying toxic or pyrophoric gases at low pressure (as in SAGS cylinders) improves the safety and efficiency of operations. The theme throughout this paper is that “pressure is the problem”. Largely on this basis, NFPA 318 recommends that SAGS be used whenever process compatibility allows.[11]

The use of SAGS-based cylinders is increasing world-wide, with larger cylinders, gas mixtures and low-positive-pressure offerings to expand the opportunity for new applications and industries, and even lab environments, to realize operational, process and product benefits.

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