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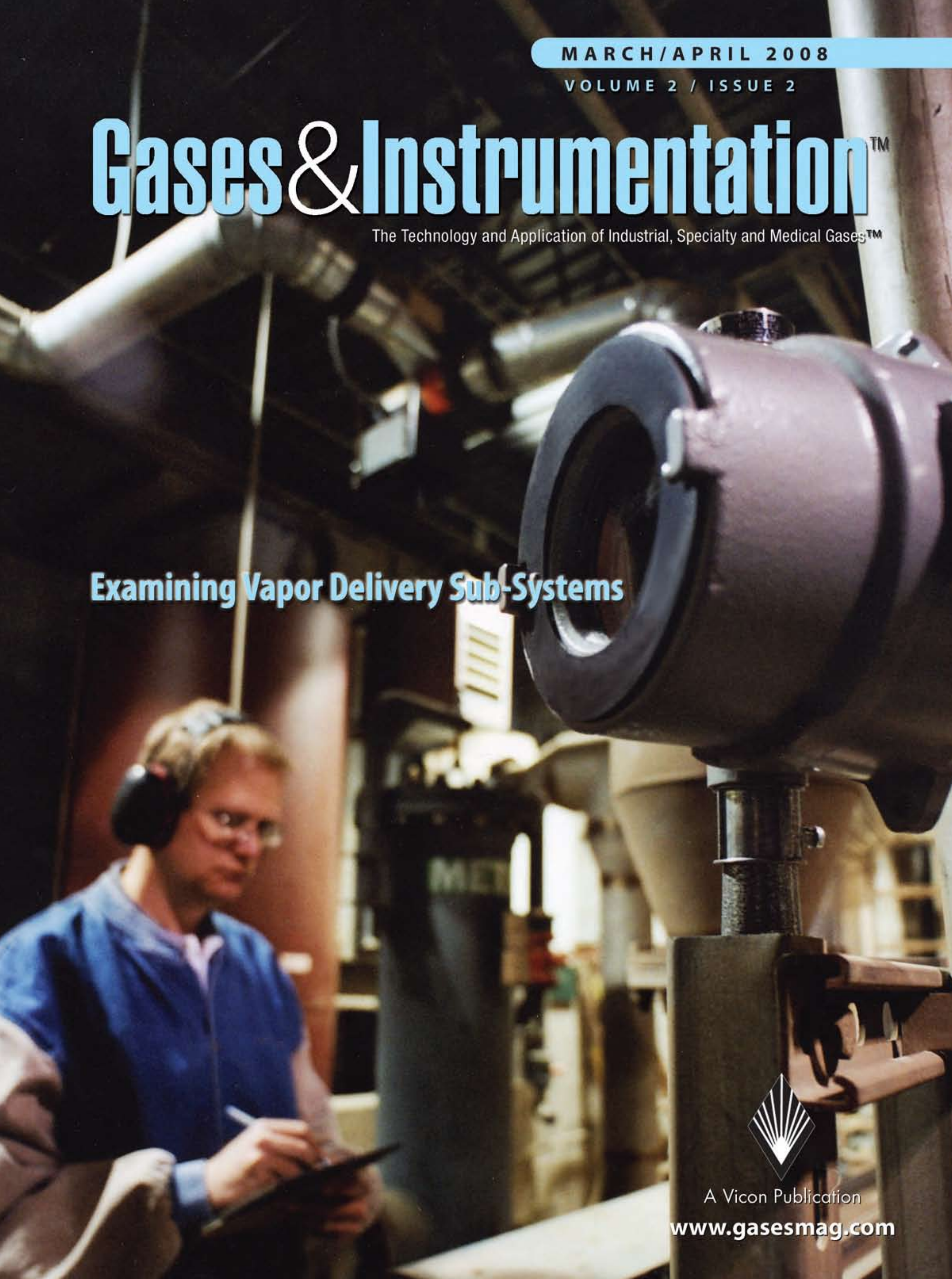

Examining Vapor Delivery Sub-Systems

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FEATURE

Considerations in the Selection of Vapor Delivery Sub-Systems

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This article examines the three most common types of vapor delivery sub-systems and explains their basic theory of operation and typical hardware configuration.

Introduction

For decades, process engineers have been developing deposition chemistries utilizing liquid phase precursors, converting them into a vapor phase prior to injection into a process chamber in order to deliver unique film characteristics. These liquid source materials are employed in a wide variety of industries ranging from high-tech applications such as semiconductors, flat panel displays and solar cell fabrication, to more industrial applications such as abrasion resistant coatings on cutting tools, optical lens/fiber and ceramics production. This review will examine the three most common types of vapor delivery sub-systems—bubblers, flash vaporizers and thermal vaporization systems—their basic theory of operation and typical hardware configuration. Close attention will be paid to each system's unique advantages and drawbacks, focusing upon an assessment of their relative mechanical size, integration considerations and vapor delivery performance, as well as their unique process considerations.

> There are a wide variety of factors that can influence the stability and reproducibility of bubbler systems.

General Considerations

Integration hardware that applies to all these systems would typically include heating of the delivery lines between the vaporizer and the process chamber, enclosure of the vaporizer within an enclosure that would provide spill containment, and exhausting of the space around the vaporizer in the case of a leak (Figure 1). The level of containment requirements are often dictated by the relative hazard of the precursor material and local and governmental regulations. Discussion of the relative size of the systems described herein does not include any accommodation for containment sub-systems, as these vary greatly by precursor material and governmental regulations. Some of the liquid precursors are extremely hazardous materials.

Examples of liquid precursors commonly used with vaporization systems include TEOS and POCl3. TEOS is used as a precursor for high quality SiO_2 films. It is a stable, but flammable liquid, and considered a health hazard. [1] Proper installation of a vaporizer for use with TEOS includes appropriate enclosures to contain the material in case of a leak, and alarms to alert upon leakage. POCl3 is a corrosive and reactive material used as a precursor for the phosphorous doping of silicon. It is incompatible with metals except nickel and lead, and is violently reactive with H_20. [2] Due to incompatibility with most metals, POCl3 can only be used with vaporization and refill systems incorporating compatible materials (such as quartz). Its high reactivity requires special containment and venting of all parts of the subsystem used in handling either the liquid or vapor state of the material.

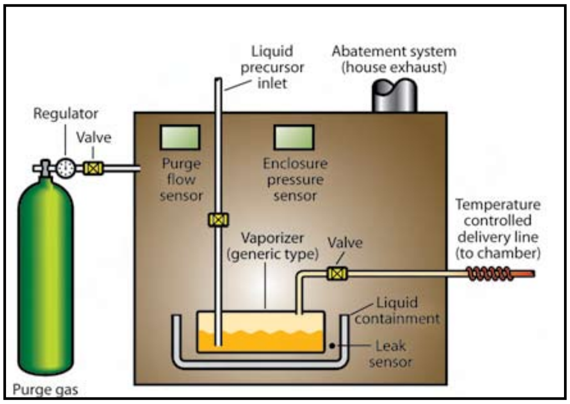

Figure 1. Typical integrated hardware system

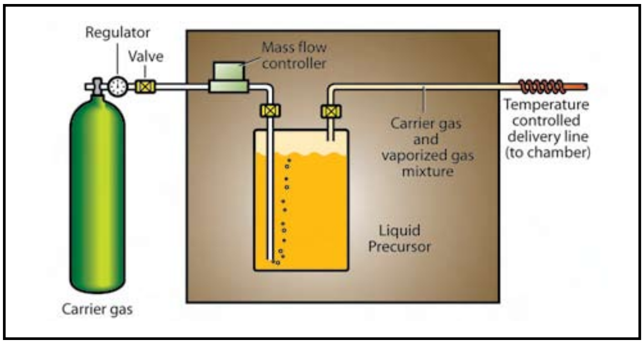

Figure 2. Bubbler basic concept

BUBBLERS

Theory of Operation

Bubbler-style systems (Figure 2) consist of a liquid reservoir, often referred to as an ampoule or flask that is held at a constant, often elevated temperature. The reservoir is wrapped with a heater and the associated temperature-management electronics. A carrier gas is flowed (bubbled) through the liquid. The rate of carrier gas flow through the reservoir is set with a flow control device that can range from a simple needle valve, a rotameter, or a mass flow controller (MFC) for more demanding applications. The bubbles of gas absorb some of the molecules of the liquid precursor and proceed through a heated line into the process chamber. These delivery lines are heated to ensure that none of the vapor condenses prior to arriving in the process chamber. Typical vapor flow rates are in the neighborhood of several hundred sccm to several slm. Process pressures range from vacuum to atmospheric applications.

Mechanical Considerations

Typical bubbler systems are less than one cubic foot, with the carrier gas inlet and vapor flow outlet on the top of the assembly. Since the temperature control electronics are an integral part of the module, ambient temperatures are of significant consideration. Depending upon the precursor employed, the reservoir may be made from SST (lined or electro-polished) or from a more fragile material, such as quartz. Obviously, routine handling of the reservoir can present a variety of hazards. Access to the reservoir is important since the vessel must be routinely replaced as the liquid level gets closer to the bottom. Depending upon the flow rates, the frequency of replacement can range from less than a day to many weeks. Some newer systems can be integrated with an automated refill system that can significantly reduce the need to replace the reservoir.

Relative Performance

Bubbler systems have been around for many years and their performance capabilities are well understood. There are a wide variety of factors that can influence the stability and reproducibility of this type of system. The amount of precursor vapor that is delivered to the processor chamber depends upon the flow rate and temperature of the carrier gas, the pressure of the vapor in the headspace above the liquid and, most significantly, the absorption rate of the precursor into the carrier gas. This absorption rate is dependent upon the relative size of the bubble, residence time of the bubble within the liquid, temperature of the liquid, and the stability of the carrier gas flow into the reservoir. With today's advanced mass flow controller technology, it will be assumed that the flow rate of the carrier gas is stable.

Most of today's carrier gas injection ports employ a delivery tube with multiple small orifices at the base of the tube. This is intended to provide a steady stream of bubbles, similar to what one might observe in a glass of champagne. Of course, as the bubble reaches the surface, there is a tendency for micro-droplets of liquid to form, and these droplets can be carried downstream to the process chamber. Over the course of the process(es), the residence time of the bubbles drops together with the liquid level in the flask. This results in decreasing the amount of vapor thereby impacting deposition reproducibility. Management of the temperature is also critical. As the precursor is vaporized, heat is removed from the reservoir. The ability of the temperature control system to deliver the heat energy required is dependent upon the level of the liquid within the reservoir. As this level drops over time, the surface area for contact between the liquid and the heated reservoir is reduced thereby limiting the ability of the system to maintain a stable temperature.

FLASH SYSTEMS

Theory of Operation

Flash systems (Figure 3) typically consist of a liquid MFC or metering pump and a heated vaporization apparatus. These vaporizer assemblies vary in configuration from a small heated chamber to a heated atomizer nozzle that employs a heated carrier gas to insure vaporization of the micro-droplets. Due to the temperatures required for nearly instant vaporization, the flash vaporizer apparatus tends to run at significantly higher temperatures than either bubblers or thermal vaporization systems. Typical vapor flow rates are in the neighborhood of approximately one slm to "tens" of slm. Pressures range from vacuum to atmospheric applications. Historically, flash systems have demonstrated difficulty with low flow applications .

Mechanical Considerations

Typical flash systems occupy less than one quarter of a cubic foot (depending upon the manufacturer) and lend themselves to locations much closer to the process chamber. The systems are mounted horizontally. Depending upon the chemistry employed, the vaporizer module can run at high temperatures thereby requiring thermal isolation from nearby electronics as well as providing for operator safety. One part of the system that requires special consideration is the chemical delivery system that provides a flow of precursor to the MFC. These liquids are usually kept in a bulk refill system that supports multiple flash systems. The refill systems deliver the liquid by means of a pressurized vessel or via a pump. Due to the constant pressure required for the source liquid material, flash systems require

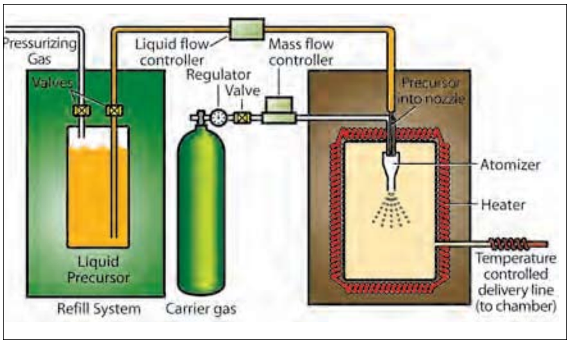

Figure 3. Flash vaporizer basic concept

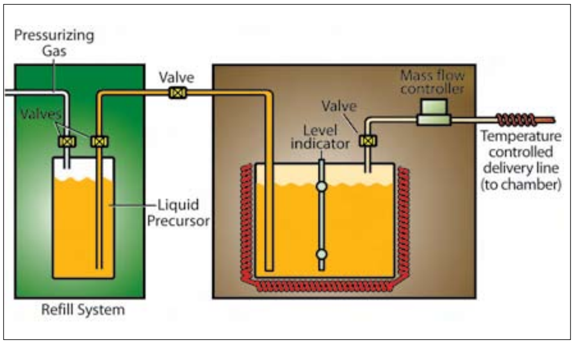

Figure 4. Thermal vaporizer basic concept

a very tight control of incoming liquid precursor material pressure. This may lead to a more complex refill system than would otherwise be necessary.

Relative Performance

Flash systems are capable of providing (under the correct conditions) stable vapor delivery with a wide variety of materials over a wide variety of temperature ranges. Additionally, flash systems have the smallest footprint of the three systems considered here. However, depending on model and the material to be vaporized flash systems may be prone to stability and maintenance issues. With vaporization taking place on a heated surface and at high temperatures, flash vaporizers are sometimes prone to the build up of materials on the vaporizing surface. This can lead to higher down time for maintenance as compared with thermal vaporizers. It can also cause particle generation. Depending on the material being vaporized, and the temperature required for instant vaporization, there is also a risk of materials being heated beyond their thermal decomposition temperature, causing premature decomposition.

As the control function of the flash vaporizer is based on the sensing of the liquid that is passing through the liquid MFC's sensor, the presence of outgassed pressurizing gas in the MFC's sensor can cause fluctuations in vapor delivery.

THERMAL VAPORIZER

Theory of Operation

Thermal vaporizers, sometimes called "baking systems," basically consist of an inlet, a heated tank, an MFC, and an outlet (Figure 4). Liquid precursor material is provided into the heated chamber from a refill system. Once the chamber is filled, the refill source is closed off, and the chamber is heated in order to raise the vapor pressure of the material. Once the material has achieved an appropriate temperature (and thus vapor pressure), the vapor is allowed to flow through the MFC and into the process chamber. The flow of material depends on a pressure drop between the heated chamber and the process chamber. Vapor flow is directly measured and controlled by the high temperature MFC. As a general rule, the temperatures used in thermal vaporizers are lower than those of flash vaporizers.

Mechanical Considerations

Thermal vaporizers are typically larger than 1 cubic foot. There is normally some length of delivery line required for the connection to the process chamber. It is very important that the temperature of this line be regulated to stay above the temperature of the vaporizer in order to avoid condensation of the precursor material. Depending on the model, some thermal vaporizers include the control circuitry and heat chamber in one box, separated by insulation. Other systems keep the circuitry in a separate control box, completely isolated from the heat of the vaporizing chamber. As the refill operation for a thermal vaporizer is periodic, refill may be accomplished with a less complicated system than would be required for flash vaporization systems.

Relative Performance

As the material flow is controlled in the vapor state by the MFC, thermal vaporizers tend to provide very stable flow. And, since thermal vaporizers do not rely on liquid MFCs for flow control, they are less subject to the flow fluctuations that can occur with the outgassing of pressuring gas from the liquid precursor.

Thermal vaporizers have the advantage of generally lower maintenance time than flash vaporizers, since they are not subject to the same materials-build-up issues at the point of vaporization. Also, they generally operate at lower temperatures than flash vaporizers, thus better avoiding premature thermal breakdown of the precursor material.

Thermal vaporizers are not subject to the high downtime incurred by bubblers due to flask changes. However, care should be taken in setting up the process to allow for small refills at appropriate steps in the process. If the heated chamber is allowed to deplete to a large degree before refill, time will be required to reheat the incoming material. This time can be shortened, or even eliminated with a preheated source.

For materials with relatively low vapor pressures, the thermal vaporizer may be limited to use with sub-atmospheric pressure processes. As previously described, the thermal vaporizer

	Bubbler	Flash	ThermalVaporizer
Maximum flow-rate	★	★★	★★★
Maintenance	★★★	★	★★★
Applicable Pressure range of the reactor	★★★	★★	★
Cost	★★	★★★	★
Foot print	★★★	★★★	★
Effects of bubbles in liquid precursor	★★★	★	★★★
Simplicity of refill system	★★★	★	★★★
Required temperature to vaporize	★★★	★	★★
Flow Accuracy	★	★★	★★★
Flow Reproducibility	★	★★	★★★
Flow stability	★	★★	★★★
Particulate source	★★	★	★★★

★ ★ ★ = "BEST PERFORMANCE"

Table 1. Performance table

relies on the pressure drop between the heated chamber and the process chamber. If the material to be vaporized has a relatively low vapor pressure, the system may not be able to raise the vapor pressure sufficiently to provide the requisite pressure drop for an atmospheric chamber.

Materials with a very low vapor pressure may not be appropriate for today's thermal or even some flash vaporizers. This limitation may be overcome in the future with the development of high temperature MFCs capable of operating in temperatures higher than current MFCs can tolerate.

Summary

Bubblers and flash vaporizers tend to compete in the same space in many regards, offering a wide variety of materials applicability. Thermal vaporizers may not be appropriate to some very low vapor pressure materials, but tend to offer better flow stability and reduced operator intervention.

The engineer should be careful to consider both process requirements and long term maintenance and performance goals when selecting a vapor delivery subsystem. A thoughtful review of the precursor material characteristics, process requirements, target maintenance periodicity, footprint, and total cost of ownership should yield a fairly clear picture of which subsystem is best for a given application. G&I

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