TRAPPING – WHY, HOW, AND WHERE

INTRODUCTION

The gaseous effluent stream from deposition and etch processes contains excess precursors and process byproduct materials with characteristics of the process chemistry. These precursors and byproducts can condense or react to produce solid and/or crystalline structures (particles) that adhere to walls, components, and even transform under certain conditions. The buildup of these deposits throughout the exhaust system are of concern because they can adversely affect the process parameters, increase tool maintenance, and affect film parameters. Even the in-situ cleans can be affected by changing line conductance. The solution for these problems is to catch the materials before they deposit in a trap.

It is critically important to manage the effluent gas stream in such a way that material deposition on downstream system components is minimized. A necessary first step is a clear understanding of the chemistry and the chemical behavior in various areas of the exhaust stream in order to conclude which species is causing issues with downstream components along with the affects on component lifetime and performance.

Thorough chemical analysis of the process utilizes both theoretical and experimental methods. Knowing the physical characteristics of the downstream equipment allows for proper treatment of the effluent while minimizing the cost and complexity of the support equipment. Managing the chemistries by reducing buildup on system components optimizes tool performance, minimizes tool downtime, and provides for safer handling by reducing the amount of chemically hazardous materials. This can only be done through effective design of both the process and the downstream gas handling system so that deposits on the equipment surfaces are minimized.

It is important to note that there is no one-size-fits-all solution for optimal handling of process effluent gas. Each application must be analyzed before process-specific applications for addressing the conditions that exist in that process and exhaust stream can be applied. Some of the fundamental process and equipment characteristics that produce buildup and the physical and chemical trapping methods that can be used to reduce or eliminate buildup problems are discussed in this paper.

CHARACTERISTICS THAT INFLUENCE BUILDUP

Vacuum Conductance

Vacuum conductance is defined as the reciprocal of the resistance to gas flow. Flow resistance is due to the friction between gas molecules and the walls of equipment containing the gas and from the friction between gas molecules themselves as well as pressure differences. Conductance is usually expressed in terms of pumping speed, typically liters per unit time.

We can show how the conductance of a line coated with buildup differs from that of an uncoated line using a simple model that compares a clean line vs. one with a small amount of coating. Using air to simplify the calculation, assuming the pressure drop is negligible and assuming laminar flow, we can use Poiseuille’s law to determine the maximum flow. Then the maximum flow/conductance of the vacuum line (pipe), which is the same as flow Q, is proportional to the pressure difference between the inlet and outlet according to the relationship:

$$Q = \frac{(P_2 \ - P_1)\, \pi r^2}{8\eta l}$$

Where *r* is the radius of the pipe, *l* is the length of the pipe, and P_2 - P_1 is the change in pressure between the inlet and the outlet. In the model, we assume a 2-inch (5.08 cm) diameter pipe (*r* = 1 inch (2.54 cm) that is 10 feet (304.8 cm) long. For the pressure change we will assume it is neglible and assume a pressure of 1 Torr (133.32 Pascals).

To simplify the model, it is assumed the walls are smooth and that valves and other line components produce no reduction in conductance. Under these conditions, the conductance is:

$$Q = \frac{(P_2 \ - P_1)\, \pi r^2}{8\eta l} = 395 \text{ L/s}$$

If the walls of the pipe have a smooth 2 mm coating, the radius, *r*, is reduced by 2 mm to 2.34 cm. Using Equation (1), the maximum flow of the coated pipe is:

$$Q = \frac{(P_2 \ - P_1)\, \pi r^2}{8\eta l} = 284.5 \text{ L/s}$$

This is a decrease of almost 30% of the pumping capacity compared with the uncoated pipe. This is a significant change. Of course, vacuum lines are never uniformly coated and this causes additional issues such as:

- Variable conductance over the length of the line
- Increased buildup rates as coating thickness grows owing to the creation of a more chemically reactive surface
- Localized turbulent flow that further increases the rate of buildup

Mean Free Path and Gas Flow Regimes

Mean free path is the distance that a molecule or particle can travel without hitting another molecule/particle. In terms of temperature and pressure, the mean free path λ, is defined as:

$$\lambda = \frac{RT}{\sqrt{2}\pi d^2 N_A P}$$

where *R* is the universal gas constant, *T* is the temperature, *d* is the effective molecular diameter, N_A is Avogadro's number and *P* is the pressure. Equation (2) shows that the mean free path of a gas molecule is directly proportional to the temperature and inversely proportional to the molecular diameter and the pressure of the gas.

Mean free path is relevant to buildup considerations in vacuum systems owing to the fact that, as the mean free path is reduced, more collisions occur between molecules and nanoscale particles, and the walls of the vacuum line. The increased collision rate directly correlates with increased rates of particle formation and buildup on the wetted surfaces of lines and components. Figure 1 shows a schematic of molecular motion under different flow regimes. Under laminar flow, molecules all move in the same direction, but at varying speeds they move slower near the pipe wall and faster at the center line of the pipe. In the turbulent flow regime, molecular collisions dominate, resulting in a directed, but randomly varying molecular path. Molecular flow occurs when

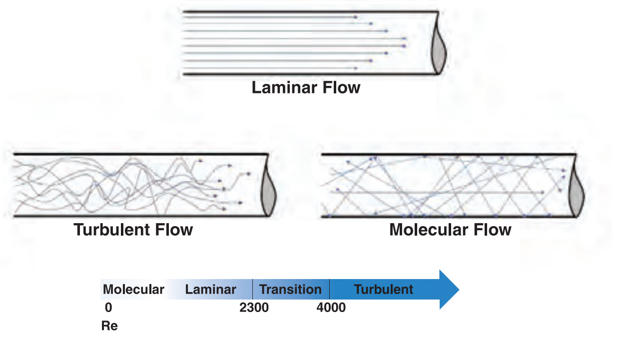

Figure 1 - Molecular vs. laminar vs. turbulent flow

the mean free path of the molecules is greater than the diameter of the confining pipe and molecular trajectories are effectively linear, changing only through molecule-wall collisions.

The Knudsen number, *Kn*, is a dimensionless characteristic number that is used to predict the gas flow regime in vacuum systems such as those used for semiconductor device fabrication. It is calculated according to the equation:

$$Kn = \frac{\lambda}{d}$$

where λ is the mean free path and *d* is the pipe diameter. Viscous flow regimes (pressures in the ambient to low vacuum range) in which either laminar or turbulent are present have Knudsen numbers of < 0.01 while Knudsen numbers of greater than 0.5 indicate a molecular flow regime. Medium vacuum environments such as those employed in semiconductor processes have Knudsen numbers between 0.01 and 0.5. Gas flow in this environment is often described as “Knudsen flow” and both viscous and molecular flow is possible. Figure 2 shows a plot of conductance vs. pressure in which the Knudsen numbers and different flow regimes are identified.

Another dimensionless number that is used to characterize fluid flow is the Reynolds number, *Re*. It is calculated as:

$$Re = \frac{\rho \cdot v \cdot l}{\eta}$$

where ρ is the gas density, ν is the average flow velocity of the gas, *l* is the characteristic length of the pipe, and η is the dynamic viscosity of the gas. A high value for the Reynolds number (> 4000) indicates a system in which the gas flow is turbulent while a low number (< 2300) indicates laminar flow. Systems with Reynolds numbers between 2300 and 4000 exhibit predominantly turbulent flows; however, laminar flow is possible, albeit unstable. Very low Reynolds numbers are indicative of a molecular flow regime.

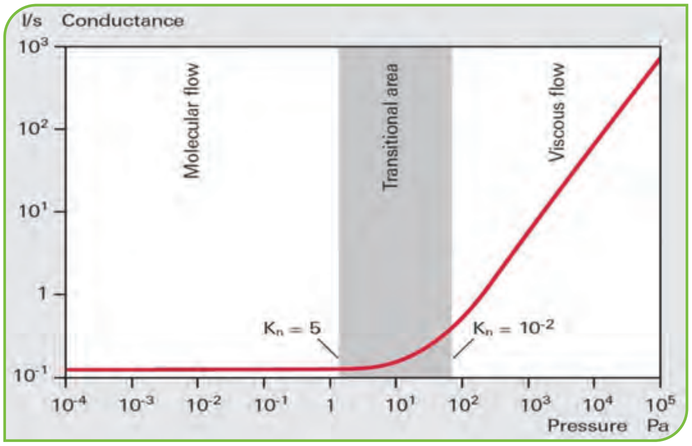

Figure 2 - Conductance vs. pressure showing characteristic Knudsen numbers for different flow regimes [2]

Understanding the characteristics of gas flow within the exhaust lines of a deposition or etch tool is important. Buildup occurs much more readily under gas flow regimes that exhibit high collision frequencies of exhaust components and the exhaust line walls. Viscous flow regimes typically exhibit high collision frequencies, with turbulent flow being the worst flow regime for where buildup is concerned.

Gas Flow Patterns in Exhaust Lines

Many published scientific studies on fluid dynamics examine the characteristics of gas flow in a pipe at different pressures and temperatures. Reference [1] is representative. This study used ANSYS fluent fluid dynamics software to study the gas velocity profile in 90° bends in vacuum piping. The results reported the following conclusions:

- At high bend curvatures (i.e., 90°), the velocity profiles at the bend inlet are shifted towards the inner pipe wall, whereas at low curvature the velocity profiles remain symmetric.
- At low Reynolds numbers, the flow passes through the bend without any change in the velocity independent of the bend curvature.
- When the curvature is high, at the bend exit, the velocity profiles are shifted towards the outer wall at high Reynolds numbers and towards the inner wall at low Reynolds numbers. When the curvature is low, all velocity profiles are shifted towards the outer wall at the bend exit.

- When the curvature is high, the velocity profiles along the exit pipe downstream of the bend show maxima and minima, but this behavior does not exist in the cases of low Reynolds numbers and low curvatures. In all cases, the velocity profiles become parabolic downstream.

A representative velocity profile for various Reynolds numbers published in this study is shown in Figure 3. The symbol “R” in the Figure represents the bend axis’ curvature radius, “r” signifies the radius of the pipe bend, δ is the curvature of the pipe’s cross-sectional profile and u is the gas velocity.

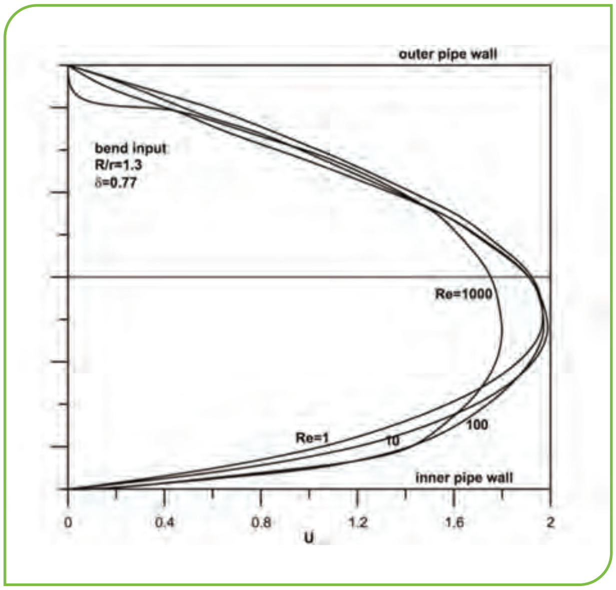

Figure 3 - Velocity profiles at different Reynolds numbers

Consideration of the gas flow pattern within the piping of an exhaust system is important for understanding the likelihood of buildup on different surfaces.

Adhesion

Deposition on the exhaust line walls typically occurs via one of four adhesion mechanisms. The first of these mechanisms relates to mechanical interference between the nanoparticles entrained in the gas stream and rough wall surfaces, the next two relate to chemical bonding, and the fourth to molecular diffusion.

Mechanical adhesion is the major contributor to exhaust line buildup. It occurs on surfaces that protrude into the gas stream and provide points of collision for adhesive particles. As well, uneven surfaces and protuberances provide a source of turbulence that produces localized high pressures that promote the adhesion of particles through methods like condensation or other adhesion mechanisms discussed here. At the atomic scale, surface roughness provides an initiation site for adhesion and growth of nanoscale deposits that also increase roughness contributing to buildup.

The best measure of surface roughness is the arithmetic average roughness, R_a, measured in micrometers, µm. A surface with a R_a value of 0.05 µm or 500 Ångstroms (2 µin) provides significant obstructions for molecular and nanoparticle collision and adhesion when molecule size is only a fraction of the roughness size. Typical roughness is more on the order of 0.4 µm (16 µin) to 0.8 µm (32 µin). Electro-polishing can remediate this problem, at least to some extent, since it removes the smaller, sharper protrusions. However, it does not affect large, broad undulations in vacuum line surfaces, unless the electro-polishing step is very long. As a consequence, even with electro-polishing, the surface roughness reflects the grinding step that is used prior to the electro-polish step. As well, many exhaust lines have connections, welds, and elbows that contribute to turbulence and particle collisions. In most systems, these factors lead to dominance of mechanical adhesion as the primary factor causing buildup.

It should also be noted that, as line pressure increases or conductance decreases, the mean free path of particles in the gas stream is reduced and molecule and particle collisions increase. Particle mass differences result in diversion of the paths of especially lighter particles and this increases the probability of their collision with the walls of the vacuum line. This in turn increases the deposition rate on the exhaust line walls and other surfaces along the exhaust path.

Both physisorption and chemisorption play a role in the buildup that occurs due to molecular adhesion on the surfaces of a vacuum line. Physisorption is due to the Coulombic attraction between a molecule and the surface that is strong enough to retain the molecule at the surface. Coulomb attraction between a gas molecule and the vacuum line surface occurs when regions of opposing charge are present in the molecule and the surface. A temporary dipole in a gas molecule is created when London dispersion forces between the molecule and the surface of the vacuum line induce an instantaneous dipole in the molecule; physisorption to the line surface is due to the Coulomb attraction between the induced dipole in the molecule and the region of opposite polarity induced on the metal surface.

On a clean vacuum line surface, the molecular dipole will induce an opposite polarity at the metal surface since electrons in the metal are highly mobile. Debye force, the Coulomb force between a permanent and an induced dipole, can also play a role in physisorption and buildup on vacuum line surfaces. A molecule with a permanent dipole will induce surface polarity in the vacuum line surface, leading to physisorption similar, albeit stronger, to that observed due to London dispersion forces. Whether or not Debye forces play a significant role in vacuum line buildup depends on the process chemistry. When the vacuum line already has buildup present, Coulomb forces between gas molecules and permanent areas of electrical polarity on the growing buildup surface will produce physisorption and buildup.

Chemisorption occurs via the formation of conventional, shared-electron bonds between the gas molecule and the surface of existing buildup on the walls of an exhaust line. Chemisorption can occur with the formation of fully covalent bonds or through hydrogen bonding. Hydrogen bonding has similarities to dispersive adhesion; however, hydrogen bonds are much stronger than conventional Coulomb interactions. Deposition and etch processes frequently employ highly electronegative chemicals from group 7A in the Periodic Table along with oxygen and water and it is difficult to prevent chemisorption from occurring in when these chemicals are present in the process system.

Diffusive adhesion, the last and least frequent mechanism of adhesion, occurs via the absorption of atoms, molecules, or nanoparticles into a material rather than adsorption on the surface. In most deposition and etch applications, the exhaust lines are steel or metal blends that do not absorb atoms or molecules from the gas phase, and buildup in these processes is not usually absorbant, making this mechanism one that is rarely, if ever, encountered.

TRAPPING

Traps capture material that causes coating and buildup on the exhaust lines before it can deposit on line surfaces. Traps use different methods and can be deployed in various locations to achieve reductions in buildup. Traps may use surface roughness, changes in conductance, phase change, or increased surface area to enhance adhesion within a controlled environment and to produce trapping of those materials that lead to buildup.

Some “traps” contain materials that react with process byproducts. While this can be considered trapping, it is more accurately termed abatement since there are multiple chemical reactions rather than a simple collection of the material as it arrives at the trap. This paper will discuss only trapping methods.

The physical properties (i.e., boiling, sublimation, melting, and condensation points) of an effluent material can be exploited to reduce or eliminate buildup. By adjusting the conditions in vacuum lines and traps, it is possible to either maintain the effluent material in the gas phase, prevent condensation and buildup, or to effect a phase change that retains the material as a condensed phase within a trap. Metal nitride and oxide compounds often have a boiling point well over 1000°C at atmosphere and these compounds tend to condense on the first cold surface they encounter. When a condensable material has a boiling point below about 250°C, at the line pressure, it is possible to heat the vacuum lines to keep the material in gas form thus preventing buildup, although higher temperature heating does exist.

Without heating, the lower boiling point materials will condense on the first cold surface that they encounter and, if a vacuum pump is used, will condense in the high pressure side unless heat is applied there too. Some pumps can use hot nitrogen to reduce condensation but heating must still be applied between the pump and the exhaust. Note that heating long, large diameter vacuum lines can represent a significant operational expense. In certain systems, cold traps can be employed that maintain a temperature well below the melting point of condensable materials in the effluent stream.

It is important to consider the placement of traps in the system exhaust line. Ideally, a trap should be placed as close as possible to the process chamber, before any pumps or valves. However, placing a trap that collects loose, solid material near the process chamber introduces a significant source of particles at a sensitive point in the process. As well, outgassing of the trapped materials may cause process issues with thin film composition. Therefore, traps are normally placed between the chamber exhaust and the pressure control valve, if there is one, with the trap in a higher pressure region significantly downstream of the process chamber exit. Placement of a trap before the pressure control valve provides the added advantage of keeping the pressure control valve free of deposits. Note that, for maintenance purposes, the pressure control valve position can be used to monitor the change in conductance of the trap as it gets full. The same tracking can be done when the trap is downstream of the pressure control valve.

Since traps should be positioned at some distance from the process chamber exit, this usually means that some length of the exhaust line must be heated, depending on the process.

For a tetraethyl orthosilicate (TEOS) process, line heaters are normally used between the chamber exit and the trap. Many furnace applications have high (> 300°C) near the chamber exit that are not compatible with heater materials; in these applications line heating must be deployed further downstream. MKS provides a unique product, the Virtual Wall™ [3] that was originally conceived for TEOS applications that can be used in areas where the temperature is too high to safely use line heaters. It produces a gas boundary layer along the exhaust line walls that prevents coating of the lines between the chamber and the trap.

Trap Size

The optimal size of a trap is a consideration in any process since this affects the conductance in the system. In determining the optimal trap size, the primary considerations are the removal efficiency and the volume of material that a trap can collect before requiring maintenance. A trap with too small capacity will determine tool uptime simply by being the most frequently maintained part in the exhaust system. Process effluent flows can range from a few hundred standard cubic centimeters (sccm) to thousands of liters per minute and this also influences the trap size in a process.

It is also important to know the expectations for the optimal volume or weight of material collection or for some other process metric compatible with existing cleaning protocols in other parts of the system (i.e., the number of substrates, deposition minutes, etc). Note that traps typically have an “effective” volume that is less than the total volume of the trap enclosure. The effective volume is the volume of the enclosure minus the volume of any mechanical components inside of the trap. Thus, for a deposition or etch system processing 500 substrates and generating condensable effluent at a rate of 0.01 Kg per substrate, the “effective” volume of the trap should be sufficient to collect 5 Kg of material. TEOS traps, for example, typically require a volume of 5.4 liters for service in line with typical maintenance requirements.

Traps are typically cylindrical, with a diameter of 6 inches and a length of about 12 inches. These dimensions are compatible with most exhaust lines and commensurate with maintenance cycle times. It should be noted that if room for the correctly sized trap is not available, smaller traps can be configured in parallel, using appropriate valve arrangements that allow switching of the exhaust path from a full trap to a clean trap. This method also eliminates any downtime for trap maintenance since one trap can be cleaned while the other trap is running.

Some process effluents contain materials that condense at temperatures close to 0°C or below. In such a case, a trap may require some sort of cooling, either internal or external or both, to cool the trap surfaces to a temperature low enough to condense the material. When a cooled trap is used, it is important to consider the residence time of the material in the trap since material condensation is directly proportional to the residence time. Normally, this is not very long and efficient trapping of the material can only be accomplished when the combined consideration is given to the incoming gas temperature, the residence time in the trap, the cooling method, the proximity of cooled surfaces to the material flow, and the temperature of the coolant.

Residence time within a trap can be increased by making the effluent path in the trap longer. One approach is to add baffles that cause the gas flow to change directions, increasing the flow path length. However, for every change in direction there is an accompanying decrease in the conductance. Adding filter materials is another way to increase residence time; similar to the situation with baffles, filter materials produce a decrease in conductance. These changes in conductance must be taken into account in any trap design and conductance decreases must be related to the pumping capacity of the system. In practical terms, a system's base pressure should be at least two orders of magnitude below the expected process pressure. This rule provides enough pumping capacity for changes in process flow and any conductance decreases produced by exhaust line changes.

Powder Filter and Traps

Many traps, especially the powder traps used in processes such as silicon dioxide (SiO_2) deposition and etch, use filter media as the trapping principle. This introduces several factors that may not be compatible with a system's pumping capacity, process, and/or maintenance requirements. The primary concern with filter traps is the pore size of the filter media. If the filter pores are too small, the conductance will be reduced to a point where severe and negative impacts on the process occur. If the filter pores are too large, the efficiency of the trap is diminished.

Pressure and the type of process play a large role in determining the best pore size in filter media. This is due to the fact that trapping effectiveness is diminished at lower pressures. As noted earlier, the formation of particles is dependent on the frequency of molecule-molecule and molecule-particle collisions. Since the collision frequency is determined by the mean free path, when there are fewer collisions the particles do not have a chance to grow to a size big enough to be effectively trapped in filter media (i.e. longer mean free paths at lower pressures yield fewer collisions). As a consequence, traps in low pressure processes should use filter media with as small a pore size as possible. Even so, effective trapping using filter media is very hard to achieve at very low process pressures. This causes additional problems further down the exhaust system since, in higher pressure regions of the pumping system, the particles will grow in size due to increased collision frequencies. This problem typically manifests itself as a large volume of powder at the outlet of the pump. Often, low pressure processes that exhibit no coating or deposition problems in the exhaust lines have issues with the production of large quantities of powder in the pump outlet.

As an example, consider a system in which there is a trap at a location in the exhaust that normally operates at 10 mTorr pressure. If the molecule size in the process is 0.2 nanometers (nm), then the mean free path is about 16mm. If, say, 1000 collisions are needed to build up a particle with an effective diameter of 1 micron then the chance of a particle being 1 micron in diameter before it reaches the trap media is very low. With some exhaust lines having lengths shorter than 3 meters and with lower pressures, the number of larger particles developing is very low. Since the particles do not grow large, to be effective, the filter media pore size would need to be less than 1 micron. Inserting filter media with such a very small pore size into a vacuum line where high conductance is needed is obviously problematic. More bends in the exhaust path can help to increase the number of collisions, but at the expense of conductance.

SUMMARY OF TRAPPING METHODS

Traps can be designed with a variety of internal systems, but they rely on one of four mechanisms to trap the material that can deposit on the surfaces of vacuum lines and/or damage downstream components such as vacuum pumps. The first and simplest mechanism is the use of a cold trap to condense the materials within the trap. In most cases, it is best to have the capability to lower the temperature of the trap at least 20°C lower than the condensation temperature of the incoming effluent. For example, the boiling point of tetraethyl orthosilicate, TEOS, is 169°C at atmosphere. Therefore, a trap at 25°C (room temperature) is at a sufficiently low temperature to condense this material.

Other processes may require colder temperatures to trap lower boiling condensable materials in the effluent. Cooled trap surfaces can be produced by simply using the available fab water to cool the internal surfaces. This can typically take the internal surfaces of a cold trap down to about 10°C. For colder water temperatures, a chiller can be used to lower the temperature of the water circulating in the trap closer to 0°C.

When a process requires even colder temperatures to trap volatile effluent compounds, cryogenic traps that employ liquid nitrogen (boiling point - 196°C) can be used. The capture efficiency of a cold trap is proportional to the volume of gas flowing through the trap, the size of the trap (these combine to determine the residence time in the trap), and the temperature of the gas entering the trap. If the residence time of the gas in the trap is not sufficiently long, then efficiency will be reduced. Likewise, if the temperature of the incoming gas is high and the residence time is short then efficient trapping will not occur.

The second mechanism of trapping uses mechanical adhesion to trap particles. In these traps, filter media are employed or the flow direction is changed multiple times by baffles. There is a balance between trapping efficiency and pore size that yields acceptable conductance and between the number of times the flow is diverted in a baffled trap, how it is diverted, and the effect on conductance. Especially in systems where the pumping speed is low and the pump is easily affected by changes in conductance, the reduction in conductance in these traps can affect the process characteristics. In general, it is good practice to have a pump that can reach a pressure that is at least two orders of magnitude below the typical process pressure with a flow of gas roughly equal to the normal process maximum total gas flow.

A third mechanism employs chemical decomposition for trapping. For example, the excess organometallic precursors in the effluent stream from atomic layer deposition (ALD) metal deposition processes can be pyrolyzed in a hot trap. Pyrolysis of the organometallic compound in the trap produces organic byproducts such as methane and ethane that are pumped away. Other energy sources such as plasma devices, lasers, lamps or arcing can be used to induce the decomposition process in these traps.

The fourth mechanism of trapping is to introduce a co-reactant into the effluent stream that produces a solid product that can be easily trapped. Oxidation works well because the resulting product is typically a powder that can be captured using a simple filter trap. With such reactive approaches to trapping, pumping speed, cooling, and safety need to be considered since most reactions generate significant amounts of heat. Trap cooling may be required. In some cases, reactive chemistries can increase the volume of effluent so the trap and pump, if any, must be properly sized for the increased gas volume.

CONCLUSION

As process exhaust lines become coated, they reduce system conductance with concomitant reductions in system pumping capacity and increases in process control problems. Deposits can foul line monitoring devices such as pressure gauges, pressure control valves switches, and other directly connected monitoring equipment. Trapping can be used to control deposition on exhaust lines as well as deposits that cause instrument failures.

A variety of trapping methods have been developed that efficiently remove any unwanted byproducts from process effluent streams that can foul downstream components. When selecting a trap for a particular process, it is important to understand trapping mechanisms and how they influence the selection of the best type of trap for the application.

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