

SVC Topics

Donald M. Mattox Technical Director Society of Vacuum Coaters 440 Live Oak Loop Albuquerque, NM 87122 505/856-7188 • FAX: 505/856-6716 Internet: http://www.svc.org

PVD Processes: Safety Aspects of Vacuum Processing

he vacuum environment poses no direct safety hazard, unless a worker happens to be in the chamber when it is pumped down. The pressure differential that is established between the ambient and the vacuum can cause safety hazards. If a glass enclosure-such as that of a glass belljar chamber or the envelope of an ionization gauge-breaks, then the pressure differential will cause an implosion of the glass shards. In the case of the belljar, the flying glass can pose a safety hazard, and this is the reason that glass belljars are no longer commonly used. If they are used, they are surrounded by a wire enclosure to prevent the glass shards from escaping.

The glass envelope of an ionization gauge should also be surrounded by an enclosure—more to prevent it from being accidently broken than to ensure safety. When working around systems where implosion can occur, safety glasses should always be worn.

There have been several cases where large, vertical, top-opening vacuum chambers have been backfilled with a heavier-than-air gas, such as argon, and a person has died after entering the oxygen-deficient atmosphere. In one case, several would-be rescuers died, as well as the original victim. Vacuum chambers are not designed for pressurization, and if backfilling from a high-pressure source, such as tank nitrogen, causes the pressure in the chamber to exceed the ambient atmospheric pressure, a seal may release violently, causing injury or damage. This hazard can be avoided by capturing the seal with clamps or bolts or by having a pressure relief valve on the chamber.

The vacuum pumping system used to generate the vacuum poses the same safety hazards as those com-

monly encountered in electrical and mechanical equipment. Moving parts, such as belts and pulleys, should be shielded so that hands and clothing will not be caught in the moving part. Highvoltage leads should be shielded. Interlocks can be used to prevent the high voltage



A cabinet for containing toxic, flammable, explosive, carcinogenic or mutagenic high-pressure gases.

from being turned on, unless there is a vacuum in the chamber.

Liquid nitrogen is often used in vacuum technology to cool adsorption materials, traps and baffles. If the liquid nitrogen vaporizes in a poorly ventilated, enclosed space, it can displace enough air to form an oxygen-deficient atmosphere. This type of environment can cause workers in the area to pass out. One liter of liquid nitrogen will produce about 650 L (stp) of nitrogen gas. When using liquid nitrogen, care should be taken that the cold fluid or a cold surface does not contact and "burn" the skin. In particular, liquid nitrogen should not be allowed to drop into your shoes! The liquid nitrogen can also splatter, so safety glasses or a face shield should be used when transferring the fluid.

Oxygen is used for reactive plasma cleaning and the reactive deposition of oxide compounds. Compressing

pure oxygen in an oil-sealed mechanical pump, using hydrocarbon oil, can cause a diesel-type explosion that can blow the pump apart. This problem can be minimized by using an oxygen-nitrogen mixture, such as pure air, which is less explosive. More chemically stable fluids, such as silicone oil, can be used in the mechanical pump, but generally they are not good lubricants and maintenance can be a problem. Pumping pure oxygen in a cryopump, followed by pumping hydrogen formed by the decomposition of a hydrocarbon vapor in the reactive deposition of a carbide film, can cause an explosion in the cryopump on regeneration. Regeneration of sorption and cryopumps can generate high internal pressures. Such pumps should always be equipped with pressure-relief valves.

Plasmas, along with high voltages, can pose a safety problem if a metal vacuum chamber is not adequately

grounded. A plasma in contact with a surface at a high negative voltage can float to a high negative potential with respect to ground. If an electrically floating surface, such as a metal vacuum chamber isolated from ground by a rubber sealing gasket, is in contact with the plasma, it can attain a high voltage with respect to ground. This can present a shocking hazard. High voltages in contact with the plasma can come from such diverse sources as bias voltages on substrates or ionization gauges that are left on when the plasma is established. All metal surfaces in plasma systems that are not being used as electrodes should be well grounded to prevent such floating potentials.

High-pressure gases are often used in vacuum processing. High-pressure gas cylinders can pose a safety hazard if they fall and the tank-valve is knocked off. They then can become a jet-propelled "missile." Gas cylinders should be transported with the correct equipment, stored with a protective cap over the tank valve and tied down when not being transported, particularly when the pressure regulator is on the tank valve.

Plumbing between the tank and the point-of-use should have a flow restrictor and a pressure-relief valve to prevent over-pressurizing the gas line. When using toxic gases such as arsine, or flammable gases such as silane, the distribution system should be of double-walled tubing, which allows the outer jacket to carry escaping gases to a volume, such as the cylinder cabinet, where they can be detected (see figure). Gas plumbing should be helium leak-checked after installation. Detectors and alarms are available for toxic and flammable gases. Gas suppliers provide handling instructions and material safety data sheets (MSDSs) for gaseous materials.*

When changing gas cylinders or investigating a gas leak in a toxic gas distribution system, self-contained breathing apparatus (SCBA) equipment should be worn. Changing gas cylinders can introduce contamination into the gas lines. If this is a concern, a valve arrangement, such as shown in the figure, can be used to allow evacuation and purging of the gas distribution line prior to opening the cylinder valve. Gas cylinders should never be allowed to be emptied to ambient pressure because, when opened later, they can draw in air and water vapor if the new ambient pressure is higher than the pressure in the tank. Always leave 10–15 psig pressure in the tank. Regulator valves for use with oxidizing gases should not be lubricated with hydrocarbon lubricants.

Vacuum pumps are often used to pump flammable, corrosive or toxic gases. These gases can accumulate in the pump oils and present a maintenance hazard (e.g., pumping of chlorine-containing plasma). Corrosion can cause pump failure or sticking of pressure-relief valves. Corrosion can be minimized by using compatible metals or surfaces coated with a chemical-resistant material. Heavily anodized aluminum, for example, is used for containing chlorine-containing plasmas. Pressure-relief valves should be periodically calibrated and certified to make sure that corrosion has not affected their operation. Often flammable, corrosive or toxic gases are removed from the pump exhaust by burning and/or solution into water. For example: In the exhaust system, silane (SiH₄) can be burned to form nontoxic SiO₂. Chlorine-containing gases can be dissolved in water either by bubbling through water or in a water spray tower. The exhaust system of such systems should be monitored and alarmed for flammable or toxic gases.

Gas mass flow meters (MFM) generally are designed to only withstand several hundred psi inlet pressure. Higher pressures can result in the violent failure of the meter. Because the gas sources for PVD processing are often from highpressure gas cylinders, it is important that the full cylinder pressure never be applied to the flow meter. This can be avoided by using a pressure regulator on the gas cylinder and including an appropriated flow restrictor and pressure relief valve in the gas distribution line. In case the regulator fails and full cylinder pressure enters the line, the flow restrictor causes the line pressure to increase to the point that the pressure relief valve is actuated before pressure in the downstream line exceeds the design pressure of the mass flow meter. The MFM should be shielded from personnel, just in case.

Vacuum processing, such as plasma etching, low-pressure chemical vapor deposition (LPCVD), plasmaenhanced chemical vapor deposition (PECVD) or reactive deposition of carbide materials using hydrocarbon precursor gases, can produce solid particles ("soot"), which are swept through the pumping system. Etching aluminum with BCl₃ in the presence of water vapor will form B_2O_2 particles, for example, and the decomposition of acetylene in a plasma during carbide film deposition forms carbon soot. These particles can clog plumbing and reduce pumping speed, or can even stop the pumping action altogether. This produces a back-pressure that can cause components to fail. An oil-sealed mechanical pump system can be set up so that ballast gas is added to help sweep the particles through the pump, the oil is continuously filtered and a pressurerelief valve limits the back pressure if the exhaust plumbing becomes clogged. In some cases, oilless mechanical pumps, such as piston pumps or scroll pumps, should be used.

Concern has been expressed about forming toxic cyanide (CN) gas when combining nitrogen and a hydrocarbon vapor, such as acetylene (C_2H_2) , in a plasma when depositing a carbon-nitride film. To my knowledge, no harmful level of cyanide has ever been detected in the exhaust of such a plasma system. Cleaning vacuum systems generally involves innocuous chemicals, but the basic aspects of chemical safety should be observed. When using abrasive cleaning, such as grit blasting, appropriate eye protection should be worn. **PESF**

Bibliography

- Office of Safety and Health Administration (www.osha.gov)
- CRC Handbook of Laboratory Safety, A.K. Furr, 4th ed., CRC Press (1995).
- Fundamentals of Laboratory Safety, W. Mahn, Van Nostrand-Reinhold Publishing Co. (1991).
- Handbook of Compressed Gases, 2nd ed., The Compressed Gas Association, Inc., Van Nostrand-Reinhold Publishing Co. (1981).
- Sax's Dangerous Properties of Industrial Materials, 9th ed., Richard J. Lewis, Sr., John Wiley & Sons (1997).
- Safe Storage of Laboratory Chemicals, David A. Pipitone, Ed., John Wiley & Sons (1991).
- Safety in Academic Chemistry Laboratories, (1990) (Booklet available from American Chemistry Society).

^{*} Air Products (gas supplier) Safety-grams & MSDS (1-800/245-2746)