Safety Aspects of Vacuum Processing

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The vacuum environment poses no direct safety hazard unless you happen 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. This is the reason that glass belljars are not commonly used nowadays. Glass belljars can easily break if heated non-uniformly such as having an electron beam hitting one area. 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 accidentally broken than it being a safety hazard. When working around systems where implosion can occur, safety glasses should be worn.

There have been several cases of death where large, vertical, top-opening vacuum chambers have been backfilled with a heavier-than-air gas, such as argon, and a person has entered the oxygen-deficient atmosphere. In one case, several would-be rescuers died as well as the original victim. When situations like this are possible the worker should have a safety-line that allows them to be pulled out of the chamber.

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 and having a pressure relief valve on the chamber. Doors should have stops that prevent them from opening more than a centimeter or so without removing the stop, this prevents them from flying open unexpectedly.

The vacuum pumping system used to generate the vacuum poses the same safety hazards as those commonly encountered in electrical and mechanical equipment. Moving parts, such as belts and pulleys, should be shielded so that hands and clothing will not get caught. Ties can be a concern when working on mechanical equipment. High voltage leads should be shielded. Interlocks should be used to prevent the high voltage from being turned on unless there is a vacuum in the chamber. If an interlock is overridden for maintenance reasons there should be a flashing red light for everyone to see.

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 oxygen-deficient environment can cause workers in the area to pass out. One liter of liquid nitrogen will produce about 650 liters (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 in your shoes! The liquid nitrogen can 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, that is less explosive. More chemically stable fluids, such as silicone oil or perfluorinated polyethers (PFPE) such as Fomblin™, 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, such as is 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 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, particularly in the degas mode, 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.

Plasmas generate ultraviolet radiation that can be transmitted through glass windows, particularly if the window is quartz. Ultraviolet radiation can harm eyes and skin on excessive exposure. The UV can be adsorbed by filters on the windows or by eye glasses worn by the operator. These are the same types of glasses used when working with lasers or with glass blowing.

In thermal evaporation the material being evaporated is at a high temperature and the thermal radiation can harm skin and eyes, particularly those wearing contact lenses. An optically clear "heat mirror", which transmits the visible and reflects the infrared, can be used to prevent the radiation from reaching the observer. This is the type of mirror that is used in projection and lighting systems to prevent heating of objects being illuminated. Such heat mirrors should be cooled by air blowers.

High-pressure gases are often used in vacuum processing. High-pressure gas cylinders can pose a major 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. This allows the outer jacket to carry escaping gases to a volume, such as the cylinder cabinet, where they can be detected as shown in Figure 1. Gas plumbing should be helium leak-checked after installation. Detectors and alarms are available for toxic and flammable gases. The exhaust system for the storage cabinet should not exhaust near the intake for another area. Gas suppliers provide handling instructions and MSDSs for gaseous materials (see general references).

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 since, 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 to 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. For example, pumping of chlo-
rine-containing gases with a hydrocarbon-oil-containing vacuum pump in the presence of oxygen or water vapor can produce phosgene (COCl₂), a toxic gas. Pumping fluorine-containing gases with pumps containing hydrocarbon oil can lead to the formation of hydrofluoric acid (HF) which can accumulate in the oil.

Etching using chlorine-containing vapors, such as BC13, or fluorine-containing vapors, such as CF4, can cause corrosion in the pumping system. In particular, stainless steel is rapidly corroded by a 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. For example, heavily anodized aluminum 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 by solution in water. For example: In the exhaust system, silane (SiH₄) can be burned to form non-toxic 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. Since the gas sources for PVD processing are often from high pressure 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), plasma enhanced 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. For example, etching aluminum with BC13 in the presence of water vapor will form B₂O₃ particles, 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 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 should be continuously filtered and a pressure relief valve should be used to limit the backpressure if the exhaust plumbing becomes clogged. In some cases oil-less mechanical pumps such as screw pumps, scroll pumps or piston pumps should be used.

In high rate vaporization of oxygen-active materials such as titanium and zirconium in an inert gas environment vapor phase nucleated particles can form “soot” that deposit on the walls of the chamber. These fine particle form a very thin passivating layer when exposed to air. When disturbed the layer can catch fire and the fire can spread over the whole film. Such deposits should be wet-cleaned in order to prevent a fire.

Concern has been expressed about forming toxic cyanide (CN) gas when combining nitrogen and a hydrocarbon vapor, such as acetylene (C₂H₂), in a plasma when depositing a carbide film. To my knowledge, no harmful level of cyanide has ever been detected in the exhaust of such a plasma system.

Cleaning vacuum systems, fixtures and substrate holders generally involves chemicals and the basic aspects of chemical safety (eye protection, skin protection, etc) should be observed. Removing particulates of film deposit should be done by wet chemical methods to avoid forming “dust” of the material. When using dry abrasive cleaning, such as grit blasting, appropriate eye and respiratory protection should be worn and the work performed in a well-ventilated area. Silica grit (silica sand) should not be used for grit blasting because of respiratory concerns (silicosis) - use alumina.

References

General References
Air Products (gas supplier) Safetygrams and MSDSs - 800/245-2746
Office of Safety and Health Administration (USA) - www.osha.gov (internet web site)
Fundamentals of Laboratory Safety, W. Mahn, Van Nostrand-Reinhold 1991
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Note: Comments and suggestions for material for inclusion in this Educational Guide would be greatly appreciated. Actual case histories would also be appreciated.