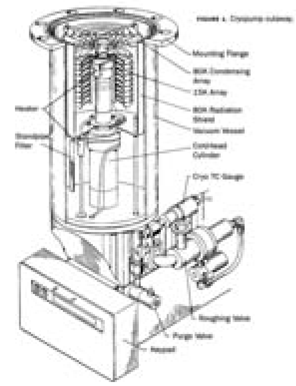
Cryogenic High Vacuum Pumps

by Gary Ash



High vacuum processing has wide application in the manufacture of semiconductor devices, computer hard disk systems, thin films for optical and electronic equipment, and basic research. In most vacuum process applications, water vapor is a primary concern. Unlike other gases, water vapor binds to chamber walls and other surfaces in relatively large quantities. High-speed cryogenic pumping of water vapor can allow the system to achieve acceptable base pressure in a minimum timeframe. Cryogenic high vacuum pumps provide high pumping speeds for all gases, in addition to their high water pumping speed. Cryopumps capture and store gases as frozen solids or ice. Because gases at atmospheric pressure occupy nearly 1,000 times more volume than cold solids, large quantities of gas can be stored in a pump. Eventually, the pump needs to be defrosted or regenerated. For most gases, the capacity of the pump is determined only by the available space within the pump to hold the ice. This article details the components of a cryogenic system and gives an overview of applications that use such pumps.

Applications

While the term "vacuum" may cover the general condition of reduced pressure in a process chamber, there are many pressure levels and types of process gas used. Today's cryopumps are designed to match not only specific processes, but also the size and shape of pump required. Pumps with vertical orientation have a minimum footprint, while "flat" pump designs can fit underneath equipment with waist-high loading ports.

***Sputtering**.* Sputter deposition of metals is the largest single application of cryopumps. Argon or nitrogen gas, with flow in the range of *50* to *250* sccm, is used to produce pressures of *2* to *5* mTorr for process conditions. With the ability to capture and store *500* to 1,000 standard liters of gas, cryopumps can operate continuously for a week or two before regeneration.

Ion implant. With gas loads primarily composed of hydrogen, hydrocarbons, CO2 and water vapor, ion implanters use cryopumps on beam lines and wafer end stations to obtain pressures in the 10^{-8} to 10^{-4} torr range.

Evaporation. Evaporation of metals and dielectrics for thin-film production typically requires vacuum of 10^{-5} to 10^{-7} torr. Reduction of water vapor during the process improves control and repeatability of film properties. Cryopumps have been used successfully with all types of thin-film optical coatings, including zinc sulfide and other high vapor pressure materials. Film adhesion is improved by the elimination of oil diffusion pumps and by reduced backstreaming from oil-sealed rough pumps as a consequence of higher crossover pressures.

***Loadlocks**.* Primary evacuation of loadlocks on multi-chamber systems has been a strong application of cryopumps. With the highest pumping speed for a given inlet diameter, rapid pumpdowns are obtained. Because cryopumps can operate in any position, they may be mounted anywhere there is a port available.

Static vacuum. Vacuum test chambers and a wide range of research equipment use cryopumps to achieve base pressures in the 10^{-7} 10^{-11} torr range. High pumping speeds are maintained to low levels of pressure. With low gas loads, pumps can operate many months before requiring regeneration.

The Pumps

Cryogenic pumps condense gases on cold surfaces to produce vacuum. In fact, a cryopump is really three pumps in one. The inlet array of the pump operates at 60 to 100 K to condense water vapor and heavy hydrocarbons on metal surfaces. Behind the inlet array, a condensing array operates at 10 to *20* K to capture argon, nitrogen, oxygen and most other gases. At these temperatures, nearly all gases form dense, ice-like solids with low vapor pressures. Hydrogen, neon and helium do not form solids at these temperatures and must be held by adsorption into activated carbon at 10 to *12* K. A third pumping mechanism is achieved by adhering carbon granules or pellets to the inner, shielded surfaces of the 10 K condensing array. Condensable gases deposit on the outer metal surfaces on their first collision, keeping the carbon only available for light gases.

A cryogenic pump consists of a mechanical refrigerator, a high temperature array for water vapor, a low temperature array for all other gases, and a vacuum vessel to house the components. Additionally, there are accessories to enhance the operation and regeneration of the pump. These include a rough valve to allow creation of an initial insulating vacuum during regeneration, a purge valve for nitrogen flow to flush regenerated gases and water vapor from the pump, and a pressure relief valve to prevent build-up of high pressure in the vacuum vessel, as condensed gases are re-vaporized during regeneration. The pumps also have a thermocouple-type vacuum gauge to measure pressure; temperature sensors to measure the temperature of the two stages; electrical heaters to control stage temperatures during pumping and to add heat in large quantities to speed the regeneration process; and a microprocessor controller to monitor operating conditions, control accessories, and communicate between the pump and external control systems.

Refrigeration

Two low-temperature stages required to use a cryopump are produced by mechanical refrigeration. A remote compressor supplies helium gas at room temperature (300 K) with a pressure of about 300 psig. Gas passes through a heatexchanger material in a piston-like displacer within the cylinder of the refrigerator. The gas cools to the first-stage operating temperature near 65 K as it passes through the displacer. The displacer is moved by a motor and crank or by pneumatic pressure to transfer gas from the space at the warm end to the cold end. As the displacer reaches the point where the warm volume is minimum and the cold volume is maximized, the exhaust valve is opened. The refrigerator exhaust line is maintained at about 100 psig by the compressor. Cold gas in the cylinder expands to produce even lower temperatures and flows back through the heat exchange material in the displacer to the compressor. As it leaves the refrigerator, the gas is at room temperature. Helium is transported to and from the compressor by flexible metal lines.

A second stage of refrigeration is necessary to produce the 10 to 20 K temperature. Part of the gas from the 65 K first stage passes through another heatexchanging displacer. After precooling to about 10 K, the helium gas expands and cools some more when the exhaust valve opens. Heat is conducted from the second stage array parts through the wall of the cylinder to the cold gas. The helium then flows back through the second and first stage displacer heat exchangers and exits the refrigerator slightly above room temperature. The first and second stage displacers are linked together and are moved by a motor-driven crank mechanism or by pneumatic action.

Helium is used in the refrigeration cycle because it does not become liquid or solid at very low temperatures, even well below 10 K. The compressor continuously recirculates the helium and maintains the pressure difference; compressing helium makes the gas hot, so the compressor has air or water cooling to bring the gas back to room temperature. Because the compressor must be lubricated and internally cooled by oil, the compressor also includes components to separate oil from the helium stream. Contaminants in the helium stream can reduce the performance of the refrigerator and cause noise or mechanical damaged, so the absorber must be replaced from time to time.

Regeneration

Regeneration is the process of warming the interior of the cryopump to revaporize the frozen gases and vent them to atmosphere. A complete regeneration is accomplished by stopping the refrigerator, warming the arrays to above room temperature, and purging residual gases and water from the pump vessel. Evacuation of the vessel with a roughing pump then removes gases adsorbed into the charcoal during purging, and it creates an insulating vacuum. Starting the refrigerator cools the parts to operating temperature.

During a full regeneration, all of the interior parts of the pump can be brought to 400 to 60° C to ensure that all of the water vapor is eliminated. In addition, it requires a minimum of 50 to 100 std cubic feet of nitrogen purge gas to evaporate 1 oz (28 g of liquid water at room temperature. Sufficient purge gas must be provided to remove all of this water.

A partial or fast regeneration removes condensable gases and hydrogen from the second stage arrays of the pump but leaves the water on the first stage (inlet) array. Partial regeneration can be completed in about one hour. If completed properly, all of the argon, nitrogen and hydrogen will be removed, restoring full speed and capacity for all gases. Fast regenerations are particularly effective in reducing downtime for loadlocked sputtering systems. For systems with water partial pressures below i0 torr, even a year's worth of pumping will collect less than three teaspoons (15 cm3) of water. In ion implanters, fast regeneration is also effective in restoring hydrogen capacity, although water accumulation may limit the number of times this can be done before a full regeneration is required.

Waterpumps

A waterpump is a single-stage cryogenic vacuum pump. It operates at a temperature of 100 to 130 K to provide high speed pumping of only water vapor. In some cases, a copper or aluminum plate is placed directly in the vacuum chamber as a pumping surface. Each square centimeter of area provides about 141/sec of pumping. Plates large enough for more than 10,000 1/sec of water pumping speed are common. The refrigerator is mounted externally to the chamber, and only a small port is needed to connect the panel to the refrigerator. Alternatively, a small cold panel or ring may be placed in front of a turbomolecular pump to raise the water pumping speed by a factor of two to four with little loss of speed for other gases.

Pump Characteristics and Sizing

Speed. Speed is a measure of the volume of gas removed per unit time, usually expressed in liters per second. Water vapor speed is strictly a function of the area of the pump inlet, because all cryopumps are essentially 100 percent efficient for pumping water at 14.4 1/sec per cm2. Speeds for other gases depend on array geometry. Except for three light gases (H2, He and Ne), the speeds for all gases are nearly independent of the amount of gas pumped. In fact, speeds may increase slightly because of the increased area of surfaces at 10 K. Heavier gases have lower pumping speeds in proportion to (1/mass)"'. For example, a typical pump of nominal 8" inside diameter has a nitrogen speed (mass = 28) of about 1,500 1/sec and an argon speed (mass = 40) of 1,200 1/sec. Speeds are constant at pressures below 10 torr. As molecular flow changes to transitional flow at higher pressures used in sputtering, speeds actually increase by 20 to 40 percent in the 2 to 5 mTorr range.

Because geometry of the pump parts determines pumping speed, each pump of a particular model has exactly the same pumping speed, and this speed is maintained over a wide range of pressures. Moreover, the speeds for all gases are set in this way. As long as the pump is at operating temperature, it must pump all condensable gases at rated speed. Even if the power fails, the pump will continue at full rated speed for five minutes or more.

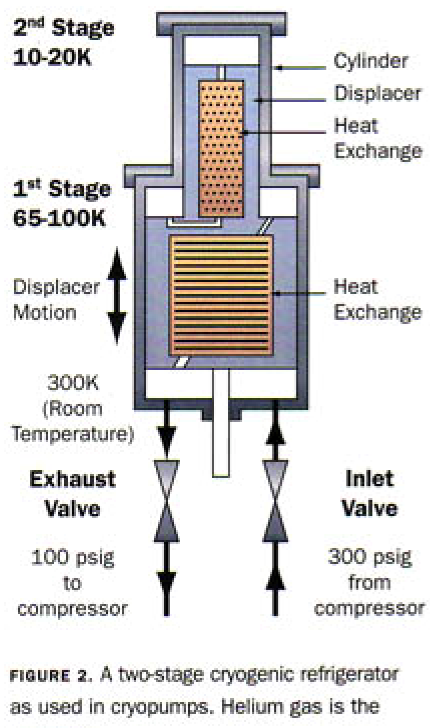


FIGURE 2. A two-stage cryogenic refrigerator as used in cryopumps. Helium gas is the refrigerant.

Crossover. When the high-vacuum valve is opened between the pump and chamber that has been rough pumped t 0.1 to 1 torr, the gas remaining in the chamber rapidly flows into the pump The thermal load from condensation typically much more than the stead state output of the refrigerator, so the pump parts warm up slightly. Crossover ratings are set to insure that no part the second stage array exceeds about 20 K. If the activated carbon on the second stage reaches temperatures much above 20 K, hydrogen can be release into the vacuum vessel and the pressure can increase. Extended operation at high temperatures and pressures can cause a unintended regeneration of the pump.

The crossover rating is the maximum product of chamber volume and pre sure that the pump can accommodate With typical ratings of 150 torr-liters fc 8" pumps and up to 300 torr-liters fc larger pumps, crossover from rough pumping can be done at pressures up t 1 torr. Because the pumping speeds of cryopumps are higher than roughing pumps, total pumpdown time is minimized by crossover at the highest possible pressure. Most pumps have sufficient capacity to do accomplish 3,000 to 5,00 crossover cycles at maximum pressure volume product before reaching capacity for condensed gases.

Throughput. Energy is required t cool gases as they condense, so higher gas flows place more of a load on the refrigerator. Part of the energy of the g is removed by an initial collision with a inlet array or radiation shield surface 65 K, with the remainder of the energy; removed when gas molecules hit the second stage array. In general, an argon g flow of 100 std cm3/min produces a load of about 1 W on the second stage of the refrigerator. This limits practical gas flow in cryopumped vacuum systems to 30 to 1,000 std cm3/min, depending on the pump size and configuration. For mo applications, throughput rating is the only aspect of vacuum pumping affected by the cooling ability of the refrigerator.

Capacity. Capacity describes the amount of gas that can be condensed or adsorbed in the pump before pumping speed or other properties are adversely affected. This is typically of concern where significant gas flows are introduced into the vacuum chamber during processing. For condensable gases, such as nitrogen or argon, the reduction in volume from gas at atmospheric pressure to frozen solid is about 700 to 900 to one. That is, *1,000 1(1,000,000* std cm3) of argon reduces to less than 1,200 cm3 of solid. Spread out on the second stage array surfaces like a mushroom cap, it produces a layer about 3/4 to I" thick.

At a continuous flow rate of 100 std cm3/min, it takes about one week to reach raw pump capacity for argon or nitrogen. For a comparison, pumping on a static vacuum chamber with an air leak at 10' torr requires 1.6 years to fill the pump.

Water vapor capacity is very high for cryopumps; even an 8" pump can collect up to a liter of ice with little loss of speed for other gases. In general, however, only a few grams or less of water collect in a cryopump or on a waterpump between regenerations.

Hydrogen capacity. Because hydrogen is pumped by a different mechanism, capacity is more limited. Also, H2 speeds drops as capacity is reached. Activated carbon at 10 to 20 K can hold about 200 std cm3 of hydrogen per gram at an equilibrium pressure of 5 x 10-6 torr. As this quantity is exceeded, the ability of the carbon to adsorb hydrogen decreases, and the equilibrium pressure rises. Hydrogen speed testing of cryopumps is performed by introducing sufficient flow into a vacuum test dome to produce a pressure of 2 x 10-1 torr. The flow is then kept constant and the pressure is noted as flow continues. As the carbon reaches saturation, the hydrogen speed drops and the pressure in the dome rises as a consequence. When the pressure reaches twice the initial value, 4 x 10 torr, the pump is considered full.

The total amount of hydrogen required to double the pressure defines the hydrogen capacity. For instance, an 8" pump that holds 1,0001 of argon can hold 10 to 20 1 of hydrogen, depending on pump configuration. Because of the flammability of hydrogen and potential for ignition by sparks or arcs in the process chamber, the pump manufacturer purposely limits hydrogen capacity.

Pressure Recovery. Some recently developed processes require the process chamber to cycle between process chambers at 2 to 4 x i0 torr at high flow rates and then recover to base pressures in the low 10' range within 30 seconds of cessation of argon flow. If the chamber has been thoroughly degassed of water and is free from virtual leaks, this performance is readily achievable. It does, however, test a different pump characteristic than raw capacity at constant pressure. Rapid recovery requires that the condensed gases are in contact only with surfaces well below 20 K, and that the solids are well-shielded from thermal radiation that might cause temperature gradients within the solids. In addition, if significant amounts of water are pumped, adsorption of argon in water ice at 65 K may slow recovery. For most applications, however, pressure cycling is not an issue.

Controls and Integrated Systems

As requirements have increased for control of vacuum processes, it is necessary to monitor and control operating characteristics of cryopumps. Measuring the temperatures of **the two** stages is the first step, so an operator knows when the pump has reached operating temperature after regeneration. However, to ensure that high-vacuum conditions are obtained reliably, the temperature of the first stage must be kept in the 65 to 90 K span; this prevents argon or other gases from partially condensing on the first stage and limiting the base pressure. Electrical heaters on the first and second stages can be used to control the temperatures at the desired operating point. Only a few watts of heat are necessary for this control function. These same heaters deliver 100 to 200 W to the arrays and refrigerator cylinder to melt the frozen gas and water for regeneration and warm the pump itself.

A microprocessor controller integrated on the pump can perform the temperature monitoring and control function. A microprocessor also opens and closes the rough and purge valves at appropriate times, turns on and off the refrigerator, and operates a thermocouple vacuum gauge. Recipes for regeneration for a variety of operating conditions are stored. Complete and thorough regeneration is obtained by coordinating the heating, purging and rough pumping of the cryopump to meet established parameters. The controller can also detect and compensate for external system failures, such as a loss of purge gas. Microprocessors on cryopumps also provide diagnostic and communication functions. Higher-level system software needs only to send basic codes to start, stop or regenerate the pumps. Pumps can report their operating or regeneration status, array temperatures, and detected faults back to the host computer. Networking of cryopumps through the host can allow for remote reporting of operating data from many pumps.

Advanced application issues

High heat loads. The refrigerator that cools the cryopump parts has a limited ability to remove heat. The inlet arrays of pumps in the 4 to 20" diameter range may have heat loads of 5 to 100 W from room temperature infrared emissions from the chamber surfaces. When elevated temperatures occur on components within the vacuum chamber, like heated wafer chucks, heaters and lamps, or evaporation sources, then attention must be paid to the thermal loads imposed on the pump. In the longer wavelength infrared- 5 to 25 jim - stainless steel and aluminum chamber walls have more than 90 percent reflectivity. Even if surfaces are rough enough to scatter radiation, heat eventually enters the pump inlet. A thin layer of water frozen on the inlet array is an efficient absorber of heat. Shields and baffles may be needed in the chamber to keep the loads within the range of the pump.

Safety. Cryogenic vacuum pumps have good safety records in a myriad of applications. Several points should be considered in their application. First, cryopumps are storage devices. They should not be used to pump gases that are pyrophoric or flammable, other than the amount of hydrogen for which the pump is rated. Similarly, they should not be used for toxic or highly corrosive gases, such as used in etch processes. Throughput-type pumps may be more appropriate for these applications so that only a small quantity of gas is in the system any point in time.

Care must be taken when pumping oxygen containing gases. Sputtering in oxygen rich conditions in the mTorr range with high RF power can create ozone within the pump.

This condition must be avoided, as explosive reactions with liquid ozone can occur within the pump during regeneration. While ozone formation is minimized under other vacuum processing conditions, like evaporation in partial pressures of oxygen in the 10 4 torr range, caution should always be exercised when ion ionization of oxygen can occur during pumping. Normal pumping of air during system evacuation does not create problems.