Inside A Vacuum Diffusion Pump

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High Vacuum Diffusion Pump

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Creating a usable vacuum anywhere within the earth's atmosphere means removing the molecules that make up the atmosphere (mostly nitrogen, oxygen, carbon dioxide, but also about 150 frequent contaminants). Removal of molecules continues until the desired base level of vacuum is obtained. The removal process typically continues after this point to maintain the desired process vacuum level while the work that requires the vacuum is performed. The work can involve an electron microscope, a GC mass spectrometer (GC-MS), surface analysis, molecular beam systems, welding of advanced materials, large-scale vacuum furnace, or even the simulation of a space environment.

The most common type of pump for use in high vacuum applications is the diffusion pump (or, more properly, vapor jet pump). Vacuum diffusion pumps are one of the oldest and most reliable ways of creating a vacuum down to 10^{-10} Torr, or even lower, at 25° C. The first designs of diffusion pumps go back to 1915, when the pump was invented by Irving Langmuir. The original diffusion pump fluid used was mercury, which could withstand elevated temperatures but had a disadvantage of being toxic. Pump and jet designs have evolved over the years and synthetic fluids have advanced, enabling higher levels of vacuum to be achieved. Numerous scales exist for the measurement of pressure in gasses, but Torr is the most frequently used.

The standard column of mercury on which the aneroid barometer is based measures 29.92 inches at normal atmospheric pressure at sea level; 29.92 inches equals 760 millimeters of mercury, or 760 Torr. For measurement of the very low pressures that exist in very high vacuums, microns (1,000 microns per millimeter) may be used. Thus a vacuum diffusion pump achieving a vacuum of 10^{-3} Torr may also be said to have reached a vacuum of 1 micron.

A vacuum of 10⁻³ Torr has removed a great many of the atmospheric molecules, but this is actually the level at which a vacuum diffusion pump begins to operate. A vacuum diffusion pump cannot begin its work with full atmospheric pressure inside the chamber. Instead, an ancillary mechanical roughing pump (or forepump), capable of a modest level of pumping, first brings the pressure inside the vacuum diffusion chamber down to about 10⁻³ Torr. At this point, the vacuum diffusion pump takes over to create a vacuum ranging from 10⁻³ to 10⁻¹⁰ Torr. Since the diffusion pump cannot exhaust directly to atmospheric pressure, the forepump is used to maintain proper discharge pressure conditions.

Although they have been replaced in some applications by more advanced designs such as cryopumps or ion pumps, vacuum diffusion pumps are still plentiful because they have several advantages: they are reliable, they are simple in design, they run without noise or vibration, and they are relatively inexpensive to operate and maintain. In fact, diffusion pumping is still the most economical means of creating high vacuum environments. These pumps also tolerate operating conditions such as excess particles and reactive gases that would destroy other types of high vacuum pumps. The

smallest are only slightly larger than a coffee cup; the largest are nearly one meter in diameter and two meters tall.

A vacuum diffusion pump is basically a stainless steel chamber containing vertically stacked cone-shaped jet assemblies. Typically there are three jet assemblies of diminishing sizes, with the largest at the bottom. At the base of the chamber is a pool of a specialized type of oil having a low vapor pressure. The oil is heated to boiling by an electric heater beneath the floor of the chamber. The vaporized oil moves upward and is expelled through the jets in the various assemblies. Water circulated through coils on the outside of the chamber cool the chamber to prevent thermal runaway and permit operation over long periods of time.

The only justification for calling them diffusion pumps is the observation that the molecules of the pumped gas penetrate some distance into the vapor jet in a manner resembling diffusion of one gas into another. (<u>"Source –</u> <u>M.H. Hablanian, page 207 High Vacuum Technology second addition"</u>). Exiting from the jets, which have fairly large diameters, the high-energy oil droplets travel downward in the space between the jet assemblies and the chamber wall at speeds up to 750 mph. The droplets may actually exceed the speed of sound, but there is no sonic boom, largely because the molecules in the partial vacuum are too far apart to transmit the sound energy. The capture efficiency of the vapor jet depends on its density, velocity and molecular weight. The high velocity jet collides with gas molecules that happen to enter it due to their thermal motion. This typically imparts a downward motion on the molecules and transports them towards the pump outlet, creating higher vacuum. At the base of the chamber, the

condensed molecules of atmospheric gases are removed by the forepump, while the condensed oil begins another cycle.

The effect of removing molecules is to create a high vacuum in the upper portion of the chamber. It is this part of the chamber that is connected with the application where a high vacuum is needed - in an electron microscope, for example. To prevent thermal runaway, water flows through cooling coils on the outside surface of the chamber.

The temperature at the base of the chamber, where the oil is being vaporized, ranges from about 190° C to about 280° C. There are several types of oil, based variously on silicones, hydrocarbons, esters, perfluorals, and polyphenyl ethers that can be used. The polyphenyl ether (Santovac® 5) fluid or oil has been a worldwide standard for more than 25 years and combines high molecular weight, low reactivity and exceptional vapor pressure. Criteria for selection of pump fluid includes low vapor pressure at room temperature, low toxicity, chemical inertness, heat of vaporization and cost. All of the oils would have a higher boiling point at full atmospheric pressure. Oils having lower molecular weights tend to boil at the lower end of this range; polyphenyl ethers, having higher molecular weight (446) boil between 230° and 270°, near the high end of the temperature range. The vacuum chamber may have a switch that automatically shuts the pump down when the temperature begins to rise. Thus a chamber that is using a polyphenyl ether, but whose trip switch is set for a lighter oil that boils at a lower temperature, may shut down if the switch's indicator temperature has not been reset.

The various oils also have different thermal breakdown temperatures - the temperature at which the oil molecules break down and combine with any available oxygen. For pump operation, the boiling point of the oil is not particularly important, but the thermal breakdown temperature may be. Oils with low boiling points also tend to have lower thermal breakdown temperatures.

Since the chamber itself has no moving parts aside from the oil droplets, a vacuum diffusion pump can operate with stability over long periods. In all diffusion pumps, a small amount of backstreaming occurs. By definition, backstreaming is the migration of minute levels of oil that moves in the opposite direction – toward the inlet of the pump and into the process stream, which may be the stage of an electron microscope or a welding chamber. In some applications minor backstreaming has no impact; in others, where the purity of materials is critical, backstreaming cannot be tolerated. For this reason, some systems add a liquid nitrogen cryotrap to remove oil particles before they can reach the process stream. In many applications the use of polyphenyl ethers makes a cryotrap unnecessary, since the high purity of polyphenyl ethers minimizes backstreaming. But some applications are so sensitive that even this solution does not give complete protection. This is why semiconductor manufacturers moved some years ago from vacuum diffusion pumps to more expensive cryopumps and turbomolecular pumps.

Since the purpose of the vacuum diffusion pump is to create a vacuum by removing molecules, the surfaces inside the chamber need to be very clean during operation. Technicians working on a pump are usually advised to

wear gloves, because even a single fingerprint can outgas water vapor and other molecules. When a vacuum diffusion pump is slower than normal in pumping down to the desired vacuum level, the reason is likely to be either outgassing of moisture on internal surfaces of plastics or other volatile substances, or a leak. All have the same effect - they add molecules to the atmosphere that the pump is trying to evacuate. When a pump is disassembled for routine maintenance and cleaning, one of the final steps is purging with dry nitrogen.

A vacuum diffusion pump chamber is not particularly difficult to disassemble, but for breakdowns that go beyond routine maintenance the work may be quite difficult. The worst breakdowns occur when there is a massive influx of oxygen and the heat rises to a level that will degrade or decompose the oil. Generally this occurs when the roughing pump fails or when a port breaks, or when the cooling system isn't functioning properly. The combination of high temperature and pressure tends to char the oil - a process that can create a gooey mess that is hard to clean up - but exactly what happens depends on the type of oil in the chamber, and the temperature at which it breaks down. Breakdowns with non-silicone hydrocarbon-based oils are hardest to clean up. The residue is much like tar, and is tenacious. After scraping out as much of the gunk as can be safely removed, maintenance personnel may use emery cloth followed by extensive, careful bead blasting (similar to sand blasting, but less damaging to surfaces, and particularly to the rather fragile aluminum jet assembly). Final cleaning stages may use alcohol, other solvents, soapy water, and de-ionized water.

Silicone-based oils leave a residue that is somewhat easier to remove. Oils containing perfluorals decompose to form fluorine compounds that can be extremely toxic and very damaging to the aluminum jet assemblies. The least messy, and least damaging, are the polyphenyl ethers, in part because their breakdown temperature is much higher (350° C vs 300° C), and in part because polyphenyl ethers tend to decompose into small non-toxic molecules such as water and carbon dioxide. But breakdowns involving polyphenyl ethers appear to be unusual. In one application, where diffusion pumps create a vacuum for the welding of highly specialized metals, the polyphenyl ether is so long-lasting that it is retained and re-used when a pump is replaced.

Pressure, Torr	Molecular Density	
760	2.36 x 10 ¹⁹	
1	3.25 x 10 ⁻¹⁶	
10 -3	3.25 x 10 ⁻¹³	
10 ⁻⁶	3.25 x 10 ⁻¹⁰	
10 ⁻⁹	3.25 x 10 ⁷	
10 ⁻¹²	3.25 x 10 ⁴	
10 ⁻¹⁵	3.25 x 10	

Figure 1 Molecules per cubic centimeter from atmospheric pressure (top) to extreme vacuum (bottom).



Figure 2 In a vacuum diffusion pump, specialized low vapor pressure oils capture molecules, which are continuously removed by the forepump to create a usable vacuum down to about 10^{-10} Torr. The bulbous shape of the diffusion pump body helps to create superior throughput – a measure of the quantity of gas per unit time that can flow through the pump.

Fluid Property	Polyphenyl Ether Santovac 5	Silicone Dow Corning	Hydrocarbon Oil Apiezon
Vapor Pressure, Torr @ 25° C	4x10 ⁻¹⁰	2x10 ⁻⁸	5x10 ⁻⁶
Molecular Weight	446	484	420
Density @ 25° C	1.20	1.07	0.87
Flash Point, °C	288	221	243
Boiling Point @ 1.3 mbar, °C	295	223	220
Viscosity, cSt @ 25° C @ 100° C	1,000 12.0	40 4.3	135 7.0
Surface Tension, dynes/cm	49.9	30.5	30.5
Refractive Index @ 25° C, 589 nm	1.67	1.56	1.48
Thermal Stability	Excellent	Good	Poor
Oxidation Resistance	Excellent	Excellent	Poor-Fair
Chemical Resistance	Excellent	Good	Poor
Radiation Resistance	Excellent	Good	Fair

Figure 3Typical properties for certain fluids used indiffusion pumps for ultra high vacuum UHV) applications. UHV is definedas the region of pressure below 10⁻⁸ Torr.



Figure 4Vacuum diffusion pumps in operation, here in avacuum coating operation.[Photo courtesy of Mill Lane Engineering]

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