Kirk-Othmer Encyclopedia of Chemical Technology.

VACUUM TECHNOLOGY

Vacuum technology concerns the means to predict, effect, and control subatmospheric pressure environments (vacuum) (1). Increasingly, each vacuum environment must be not only safe and cost-, energy-, and materials-effective, but also tailored to serve each use profitably. However, as of this writing (1997), the likely interactions between a dynamic endeavor and a potentially profitably serving vacuum environment may still not be straightforward. A learning experience is likely. Adequate results can depend on luck in choosing and following recipes, cut-and-dry methods, guessing, and intuition, rather than understanding traceable to scientific first principles.

Vacuum production was essential in the early steam-actuated pumps used for pumping water (qv). In these engines, steam pressure raised a piston against atmospheric pressure. The work stroke in the engine was created by condensing the steam, which allows atmospheric pressure to perform the output of the engine. These vacuum-force-type engines were replaced by the steam engine of James Watt. Philosophical notions of void and vacuum developed over centuries turned out to be more realistic than the idea that subatmospheric pressure in a gas characterizes a vacuum.

The vacuum environment offers a great range and diversity of uses (Table 1). The host of parameters are set by the USE and may not include gas pressure except heuristically. Construction and control represent a challenge to the engineers and technicians. Vacuum environments can be grouped into the following operational levels: crude (CR), rough (R), controlled (C), highly controlled (HC), and ultracontrolled (UC) (2). In some instances, these correspond to the traditional categories where vacuum is referred to as low, medium, high, ultrahigh, and beyond ultrahigh. The traditional categorization focuses on the magnitude of pressure rather than on the parameters and their magnitudes that are essential to a given use. The degree of control of a vacuum environment demonstrated by the base pressure of the system is regarded as a balance between the rate at which molecules enter the gas phase and the rate at which molecules are pumped away. There is no explicit recognition of the types of molecules present, and the base pressure of a system at room temperature is not sufficient information for predicting the behavior of the system when a dynamic process or experiment is attempted in the chamber. Regarding vacuum as a molecular environment from the beginning can help to keep in view the importance of any possible consequence between the process and vacuum systems in part and/or as a whole.

Within vessels, vacuum environments comprise gaseous molecular phase(s) in contact but not necessarily in equilibrium with condensed molecular phases (2). The condensed phases consist of desirable, undesirable, and tolerable aggregations of molecules and structures such as grain boundaries, dislocations, vacancies, and defects. Typically, these are found in configurations that include undifferentiated bulk at interfacial, thin-film, and surface locations. The gaseous phase, especially under extreme dynamic conditions, may include neutral, excited, metastable, and electrically charged cluster, as well as particulate species that can consist of beneficial, tolerable, and deleterious molecular species, eg, contamination can be good or benign as well as bad.

Nonmolecular species, including radiant quanta, electrons, holes, and phonons, may interact with the molecular environment. In some cases, the electronic environment (3), in a film for example, may be improved by doping with impurities (4). Contamination by undesirable species must at the same time be limited. In general, depending primarily on temperature, molecular transport occurs in and between phases (5), but it is unlikely that the concentration ratios of molecular species is uniform from one phase to another or that, within one phase, all partial concentrations or their ratios are uniform. Molecular concentrations and species that are anathema in one application may be tolerable or even desirable in another. Toxic and other types of dangerous gases are handled or generated in vacuum systems. Safety procedures have been discussed (6, 7).

Through its committees, divisions, and chapters, the American Vacuum Society has produced a nearly complete bibliography (to 1996) (8), a dictionary of terms (9), a monograph series, and a number of other useful publications (10). Another source of information is the Association of Vacuum Equipment Manufacturers. A history of vacuum ideas and technology development from the Middle Ages to Newton has been given (11).

Table 1. Vacuum Applications

Vacuum environment category[a]	Gas pressure, ~Pa[b]	Pump[c]	Use
C, HC	$<10^3$	A, B, D	aneroid barometers
all	all	all	annealing
HC, UC	$<10^{-3}$	D	arc circuit breakers and switches
CR, R, C	<1	A, B, D	arc furnaces
HC	$<10^{-4}$	D	betatrons
C	3	B	blood-plasma dehydration
CR, R, C	10^3	A, D	capacitors
CR, R	all	A, D	casting
R, C	60	A	citrus-juice dehydration
CR	5×10^4	A	cleaning
C		A	coffee packing
CR	5×10^4	A	concrete casting
all	all	all	cooling
R, C	10^{-5}	cryo	cryogenic wind tunnels
R, C	3	B	dehydration of antibiotics
R, C	3	B	distillation of plasticizers
C, HC	$<10^{-2}$	D	electron- and ion-beam lithography
C	$<10^{-2}$	D	electron-beam furnaces
C	$<10^5$	A, B, D	electron-beam welding
HC, UC	$<10^{-3}$	D	electron-diffraction cameras
HC, UC	$<10^{-3}$	D	electron and ion linear accelerators
C, HC, UC	$<10^{-2}$	D	electron microscopes
C	50	A	essential-oil distillation
CR, R, C	10^3	A	filtration
C	$<2 \times 10^{-2}$	D	fluorescent lights
R, C	2	A, B, D	freeze-drying foods and pharmaceuticals
C, HC, UC	$<10^{-2}$	A, B, D	fusing analysis
HC, UC	$<10^{-1}$	D	fusion power research
HC	$<5 \times 10^{-3}$	D	geiger-counter tubes
C, HC, UC	10^{-2}	D	helium-leak detectors
CR, R	20	A	impregnation of cables
CR, R	20	A	impregnation of capacitors
CR, R	50	A	impregnation of castings
CR, R	10	A	impregnation of wood
C	<1	B, D	incandescent lamps
all	$<10^{-1}$	B, D	induction melting
R, C	$<10^{-1}$	A, D	infrared spectrometers
C, HC, UC	$<10^{-4}$	D	isotope separators
C, HC, UC	$<10^{-2}$	D	mass spectrometers
HC, UC	2	A, B, D	mercury switches
C, HC	2	A, B	mercury thermometers
CR, R	10^{-1}	A, B	metalizing capacitor paper
all	$<10^{-2}$	D	metal evaporation
C, HC	$<10^{-1}$	B, D	metal sputtering (triode)

Table 1. ***Continued***

Vacuum environment category[a]	Gas pressure, ~Pa[b]	Pump[c]	Use
R, C, HC, UC	10^{-1}	A, B, D	molecular distillation
C, HC, UC	$<10^{-2}$	A, D	molecular, ion, and electron beams
CR	5×10^4	A	milking machines
R, C	10^{-2}	D	neon signs
R, C	10^3	A	oil deodorizers
C	$<10^{-2}$	D	optics coating
CR	10^4	A	papermill equipment
CR, R	10^4	A	petroleum distillation
C, HC	10^{-4}	D	photoelectric cells
C, HC, UC	$<10^{-5}$	D	photomultiplier tubes
C, HC	2	B, D	radio-receiving tubes
C, HC, UC	$<10^{-3}$	D	ratio-transmitting tubes
C, HC	3	B, D	radiofrequency diode sputtering
R, C	3	A, B	refrigeration units
C, HC	1	B, D	relays
C, HC, UC	1	B, D	sealed resistors
C	5	B	serum ampules
all	$<10^{-1}$	D	sintering
R	10	A	solvent recovery
CR, R	5	B	smelting
all	all	all	space simulation
R, C	$<10^{-1}$	all	spectrophotometers
CR, R	2×10^3	A	steam-turbine exhaust
CR, R, C	50	A	steel degassing
HC, UC	$<10^{-8}$	D	storage-ring and colliding-beam machines
CR, R	10^4	A	sugar-evaporating pans
R, C	<50	A, cryo	supersonic wind tunnels
C, HC, UC	$<10^{-3}$	D	synchrotrons for electron, ion
R, C	2×10^{-3}	D	thermocouples
C	2×10^{-2}	D	thermos bottles
C	5×10^{-5}	D	television tubes
HC, UC	$<10^{-5}$	D	thin-film circuits
CR	50	A	transformer-oil drying
CR, R	$<10^{-2}$	D	ultracentrifuge
C, HC	$<10^{-2}$	D	ultraviolet instruments
C	2	B, D	vaccines
C, HC	<1	B, D	vapor lamps
CR	5×10^4	A	vehicular transportation, brakes, engines, etc
CR, R, C	$<10^4$	A, B, D	wind tunnels
HC, UC	$<10^{-4}$	D	x-ray tubes

[a] CR represents crude; R, rough; C, controlled; HC, highly controlled; UC, ultracontrolled.
[b] To convert Pa to torr, divide by 133.3.
[c] A represents mechanical pump or steam ejector; B, booster pump; D, cryo, turbomolecular, sorption, ion, or trapped diffusion pumps.

1. Vacuum Dynamics

1.1. Units and Concentration

In the gaseous as well as the condensed phases, molecular concentration by molecular species is of prime importance. By convention, total pressure in a Maxwellian gas is used as though it indicates the quality of the vacuum and as though Maxwellian gases were the rule rather than the exception (12). In general, in dynamic systems, gas pressure (or its partial pressure components) is neither isotropic nor an adequate indicator of molecular significance.

For a Maxwellian gas in steady state, one standard atmosphere is defined as being equal to 101,323.2 N/m^2; 1 N/m^2 = 1 pascal (Pa) $\longrightarrow$ 2.6×10^{20} molecules per cubic meter; 1 torr = 133.32 Pa $\cong$ 1 mm Hg = 1000 micrometers; 1 millibar = 100 Pa; 1 in. Hg = 3386.33 Pa; and 1 lb/in.^{2} = 6895.3 Pa (see Pressure measurement).

1.2. Condensed-Phase vs Surface-Phase Concentration

There are ca 4×10^{18} molecules/m^2 in a monolayer on the surface of a plane, depending on substrate and adsorbed species. At bulk impurities of 10^{-6} and a bulk thickness of 1 mm, ca 7×10^{19} molecules/m^2 can diffuse to the surface as a function of temperature and time. Thus, in a vessel of 1 m^3 at 100 μPa (7.5×10^{-7} mm Hg) and 300 K, the reservoir of molecules in the gas phase is 2.6×10^{16}, whereas surface and bulk are apt to hold ca 2×10^{19} and $>4 \times 10^{20}$ impurity molecules, respectively.

1.3. Interaction between Gaseous and Condensed Phases

In a closed vessel of volume V containing a nonionized, unexcited molecular gas having total number of molecules N, the change in the pressure P in the gas can often be predicted if the steady-state absolute temperature T is changed to another steady, constant level:

$$PV = NkT \qquad (1)$$

where k is the Boltzmann constant, relating the steady-state absolute temperature T and the equilibrium pressure P in the gas.

However, it is not practical to set the gas temperature in steady state without equally setting the temperature of the surface and bulk phases bounding the gas. Consideration of the response of the system as a vacuum environment can then provide a sufficiently precise prediction of the pressure P and the surface coverage θ at temperature T for molecules of a known species in a known state on a known surface. For example, an isotherm is established between the surface of the condensed and the gaseous phases, depending, eg, on the heat of desorption Q. For submonolayer coverage on a known surface, the pressure P is likely to be an exponential function of T. Among several isotherms, the Temkin isotherm (13) may be used to predict a specific pressure:

$$P = C \cdot \exp - (Q_\theta / RT) \qquad (2)$$

where C is a constant. Changing the temperature can follow or significantly depart from equation 1, the kinetic formula predicting the dependency of P on T.

An example would be a cubical chamber (1000 cm^3) at 298 K constructed of metal, ceramic, or glass that is evacuated (pumped down) and then filled with hydrogen to a pressure of ca 13 mPa (9.75×10^{-5} torr). On cooling to 77 K, the H_2 pressure drops to ca $77/298 \times 1.3 \times 10^{-2} = 3.4$ mPa (25.5×10^{-4} torr), in agreement with the kinetic theory. If a clean tungsten surface is deposited by evaporating tungsten, and then enough H_2 gas is admitted intermittently to give a nonvarying gas-phase pressure at 298 K of ca 13 mPa (9.75×10^{-5} torr), a concentration of $N_{H_2} = 3 \times 10^{15}$ is obtained. Then the tungsten surface (projected area only) would have absorbed ca 5.6×10^{18} hydrogen molecules/m^2, or $>3.4 \times 10^{17}$ molecules (100 times the total in the gas phase). If the chamber is again cooled to 77 K, the hydrogen partial pressure in the gas phase falls to <10 fPa ($<75 \times 10^{-18}$ torr), as predicted by the Temkin isotherm (13).

1.4. Kinetics Modified by Dynamic Interaction

The kinetic theory of gases is a valuable tool for vacuum technology. The unmodified kinetic theory must not be applied when the gas interacts significantly with itself or with the molecular phases that bound it. When interaction occurs, as it does for many molecular species in the systems considered here, the kinetic predictions must be modified by dynamic considerations. The condensed phase dominates the behavior of the gaseous phase in almost every respect under free molecular conditions. In general, measuring vacuum is not equivalent to measuring any single parameter (1).

1.5. Partial Concentration

The sum of the partial concentrations (pressures) in a free molecular gas is equal to the total concentration (pressure). However, all gaseous components, at the same partial pressure or absolute pressure or ratios thereof, are not likely to have the same significance to any or all vacuum applications. The significance of the condensed-phase concentrations must therefore be considered.

1.6. Essential Parameters

Traditionally, all vacuum environments are characterized in terms of one parameter, ie, pressure in the gaseous phase. However, when costs, energy, safety, hazardous wastes, and other requirements are taken into account, each system must be characterized by a host of parameters. Their magnitudes must be determined in order to judge system performance.

The role of a component as a function of position, use, history, and time may change. For example, a gas-pumping system is always a source of contamination, which may be negligible when the system is in good condition. The significance of a given component in a system at a given time must be taken into account. The following are examples of vacuum environments.

1.6.1. Electrical Breakdown

The electrical breakdown between parallel planar vacuum electrodes is seen to be a function of gas species and pressure (14, 15). Markedly higher a-c and d-c voltages can be held off at gas pressures of 100 μPa to 1 Pa (7.5×10^{-7} to 7.5×10^{-3} torr) (Fig. 1). The composition of the surface molecular phase is a key factor.

1.6.2. Zinc Coating of Capacitors

In the zinc coating of paper strip for capacitors, the paper strip is fed from air through locks into a vacuum environment. There, it is coated by thermally evaporated zinc. The rate of evaporation is so high that contamination of the zinc vapor is excluded. The paper is fed at the maximum rate permitted by its own strength.

1.6.3. Electron Phenomena

Deleterious electron (r-f) phenomena are erased in electron linacs by an improved vacuum environment. Before the development of the 3000-m-long Stanford linear accelerator (SLAC), electron linacs were plagued by so-called multipactoring because secondary electrons were trapped in the r-f cavities of the accelerator when voltage was first applied. No beam could be accelerated through the linac until these cavities were conditioned. The vacuum achieved was thought not to contribute to multipactoring. The vacuum thought to be satisfactory for the accelerator was based on the prevention of beam interaction with the residual gas. This mean-free-path consideration is species-dependent, but by custom the vacuum was specified in terms of total pressure in the gas phase only. When the SLAC was planned, each klystron r-f power tube was to be open to the accelerator vacuum because reliable r-f windows transmitting the power required had not yet been developed. Thus, SLAC was built to have a vacuum good enough for a klystron tube. However, r-f windows were developed but the

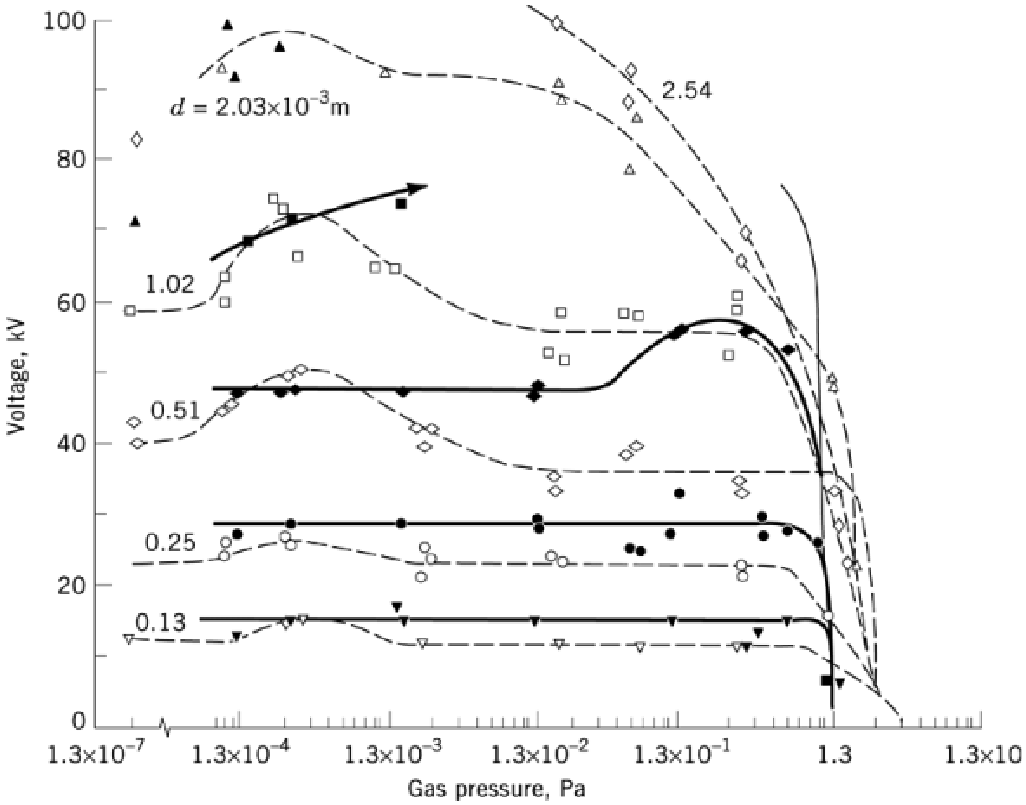


Fig. 1. Currents are d-c (dashed lines and empty symbols) and peak a-c (solid lines and solid symbols) breakdown voltage vs gas pressure in nickel for the various gaps, d, noted by values $\times 10^{-3}$ m on the curves (16). To convert Pa to torr, divide by 133.3.

accelerator vacuum environment was kept clean as a klystron. When a beam was first attempted at SLAC, no multipactoring occurred, and the beam passed through from electron source to target the first time r-f voltage was applied.

1.6.4. Film Contamination from Bulk Phase

The contamination of an epitaxial film of GaAs from an oven charge can be corrected by doping (4).

1.6.5. Oil Contamination of Helium Gas

For more than 20 years, helium gas has been used in a variety of nuclear experiments to collect, carry, and concentrate fission-recoil fragments and other nuclear reaction products. Reaction products, often isotropically distributed, come to rest in helium at atmospheric concentration by collisional energy exchange. The helium is then allowed to flow through a capillary and then through a pinhole into a much higher vacuum. The helium thus collects, carries, and concentrates products that are much heavier than itself, electrically charged or neutral, onto a detector that may be a photographic emulsion. If the helium is contaminated with pump oil, the efficiency of delivery to the detector is markedly increased. Oil contamination, anathema in some systems, is desirable for this purpose.

1.6.6. Field Emission of Electrons

Nonthermionic emission from Spindt-type arrays of cold cathode tips is affected by gas-phase concentration and species (Fig. 2) (17). The time response of emission at constant voltage to changes, and reverse, from negligible to significant concentrations of water, hydrogen, and oxygen indicates that this emission may depend on diffusion rates into and out of the bulk phase in addition to cathode surface adsorption–desorption. Emission from Spindt-type tips was also increased by other active gases tested, eg, NH_3, CH_4, and H_2S (not O_2), but was unchanged by corresponding concentrations of He, Ne, or Ar. The bulk and surface phases of the anode receiving the electron emission must be degassed at ca 1200 K in order to avoid electrical breakdown.

1.6.7. Action of Vacuum on Spacecraft Materials

For service beyond the atmosphere, the vacuum environment allows materials to evaporate or decompose under the action of various forces encountered (1, 18, 19). These forces include the photons from the sun, charged particles from solar wind, and dust. The action of space environment on materials and spacecraft can be simulated by a source–sink relationship in a vacuum environment. Thus, for example, the lifetime of a solar panel in space operation may be tested (see Photovoltaic cells).

A vacuum system can be constructed that includes a solar panel, ie, a leak-tight, instrumented vessel having a hole through which a gas vacuum pump operates. An approximate steady-state base pressure is established without test parts. It is assumed that the vessel with the test parts can be pumped down to the base pressure. The chamber is said to have an altitude potential corresponding to the height from the surface of the earth where the gas concentration is estimated to have the same approximate value as the base pressure of the clean, dry, and empty vacuum vessel.

In general, the test object cannot be heated above its operating temperature in space. As free molecular conditions are obtained around the object, it outgases and, if solar-spectrum photons impinge on the object, increases the release of gas. Because the object is in a vessel and the area of the hole leading to the gas pump is small compared with the projected interior area of the vessel, molecules originating from the test object can return to the test object provided that they do not interact in some manner with the vessel walls and the other components of the molecular environment. The object inside the vessel establishes an entirely different system than the clean, dry, and empty vacuum vessel. The new system no longer has the capability to reach the clean, dry, and empty base pressure within a reasonable time.

This simulation can be achieved in terms of a source–sink relationship. Rather than use the gas concentration around the test object as a target parameter, the test object can be surrounded by a sink of ca $2\text{-}\pi$ solid angle. The solar panel is then maintained at its maximum operating temperature and irradiated by appropriate fluxes, such as those of photons. Molecules leaving the solar panel strike the sink and are not likely to come back to the panel. If some molecules return to the panel, proper instrumentation can determine this return as well as their departure rates from the panel as a function of location. The system may be considered in terms of sets of probabilities associated with rates of change on surfaces and in bulk materials.

1.6.8. Electronic Vacuum Tube

In special electronic vacuum diode tubes, with spacing between the cathode and anode of 10 μm, high gas concentrations of some types are beneficial to the operation of the tube under proper control.

2. Pump Down

Many problems encountered in producing a highly controlled vacuum result from the system's design, history, contents, use, and maintenance.

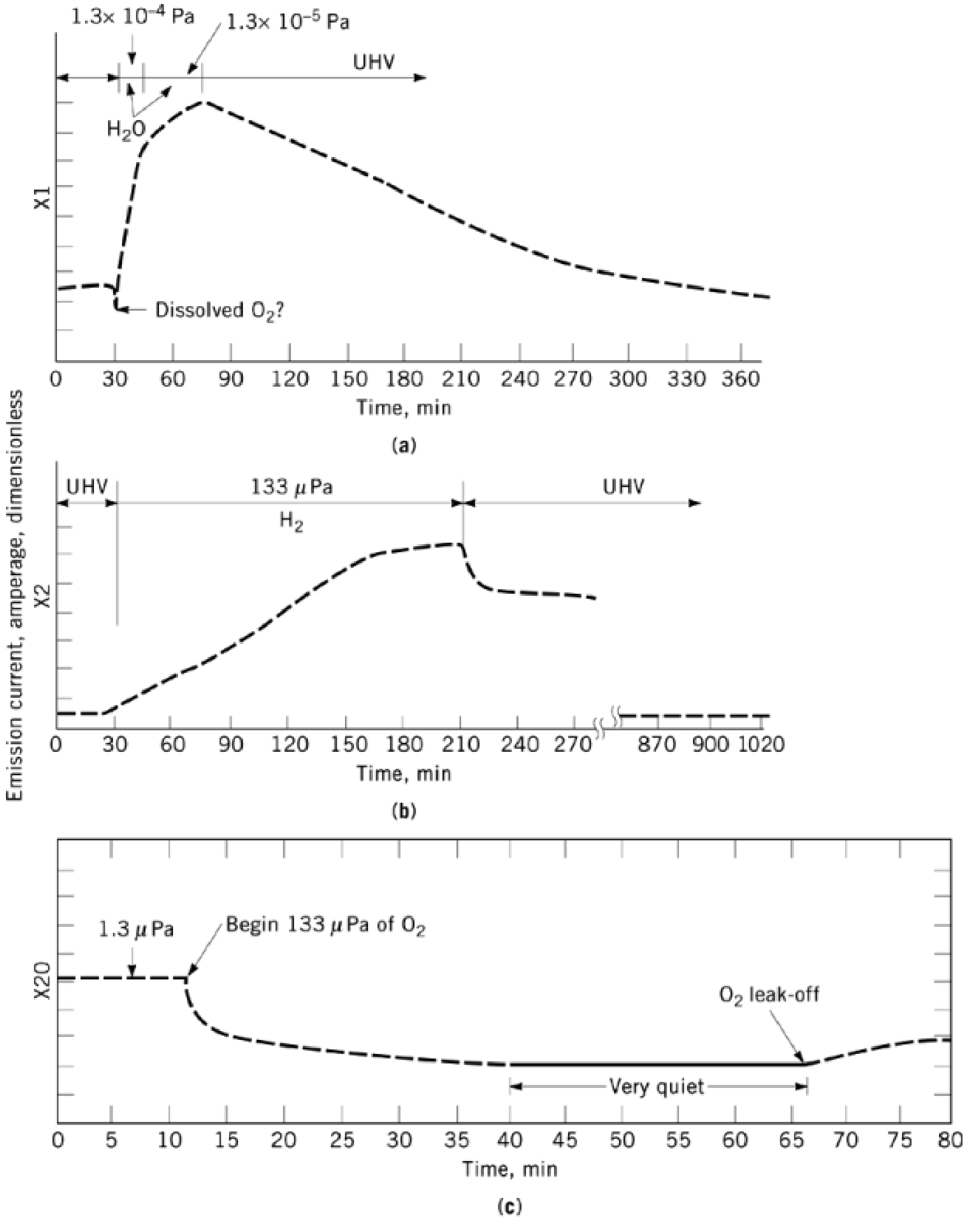


Fig. 2. Behavior of electron-field emission at room temperature from Spindt-type arrays of 5000 tips per mm^2, beginning and ending with ultrahigh vacuum (UHV), eg, ultracontrol (UC): (**a**) water; (**b**) hydrogen; and (**c**) oxygen, where the dashed line indicates noise. To convert Pa to torr, divide by 133.3.

Initially, the vessel is filled with ambient air. Any given macrosample of air may contain at least 1600 substances that have been identified (20). Among these are the gases and vapors listed in Table 2. Most important is usually water; others include viable and nonviable particulates (21), aerosols (21), and cluster species of molecules that can be in states of excitation or ionization. Aerosols originate from both anthropomorphic and

Table 2. Typical Average Diurnal Concentrations of Molecular Species in Nonpolluted Ambient Air[a]

Species	Concentration, molecules/m^3
More chemically reactive and surface adsorbing	
O_2	5×10^{24}
H_2O	ca 2.5×10^{23}
O_3	ca 1×10^{18}
NO	ca 1.5×10^{17}
HO_2	ca 6.5×10^{14}
HO	ca 4×10^{11}
Cl	ca 2×10^{10}
N_2O	ca 5×10^{19}
H_2	ca 5×10^{19}
Less chemically reactive and surface adsorbing	
N_2	2.1×10^{25}
CO_2	8.9×10^{21}
CH_4	ca 5×10^{19}
Chemically inert but slightly adsorbing	
Ar	2.5×10^{23}
Ne	4.9×10^{20}
He	1.4×10^{20}
Kr	3.0×10^{19}
Xe	2.3×10^{18}
Ra	2×10^{4}

[a] Ref. 20.

natural sources; in urban air, the former far outweigh the latter. Typical contaminants include cigarette smoke, lead (qv), and asbestos (qv). Gasoline and diesel fuel contribute significantly to contamination. Effluents and exudates are present, including skin particles, hair, and innumerable other substances from human sources (see Air pollution).

2.1. Microstructure on Surfaces

Gross cracks and voids are usually lined with microstructure, as indicated by Figure 3. As the depth–width, D/W, ratio of a crack is held constant but the dimensions approach molecular dimensions, the crack becomes more retentive. At room temperature, gaseous molecules can enter such a crack directly and by two-dimensional diffusion processes. The amount of work necessary to remove completely the water from the pores of an artificial zeolite can be as high as 400 kJ/mol (95.6 kcal/mol). The reason is that the water molecule can make up to six H-bond attachments to the walls of a pore when the pore size is only slightly larger. In comparison, the heat of vaporization of bulk water is 42 kJ/mol (10 kcal/mol), and the heat of desorption of submonolayer water molecules on a plane, solid substrate is up to 59 kJ/mol (14.1 kcal/mol). The heat of desorption appears as a exponential in the equation correlating desorption rate and temperature (see Molecular sieves).

2.2. Turbulent Gas Flow (Rough Pumping)

An oil-sealed mechanical pump in good condition, having vented or trapped exhaust, is gas purged by running for several hours. A liquid-nitrogen (LN) trap is between pump and vessel. It can consist of a U-shaped tube of thin-walled stainless steel clad externally with heavy-wall copper along its vertical legs (23). In each riser leg of the U-trap, a twisted piece of copper of width d, ie, the ID of tube, is inserted to ensure that under contaminated flow conditions, oil cannot pass without encountering a liquid-nitrogen-cooled surface. An in-line, hot (450 K), all-metal valve on the system side of the trap is connected in the line above the copper-clad leg by a 0.02-m-long

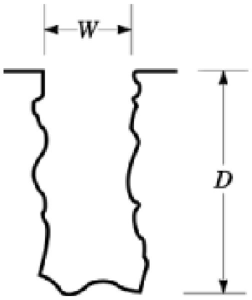


Fig. 3. Crack of width W and depth D; ratio $_{D/W=\text{constant}}$ (22).

stainless-steel neck (2.5×10^{-4}–m wall thickness). Thus, the rate of boil-off of the liquid nitrogen from the trap is kept reasonable. The trap can be filled automatically from a local reservoir, from a built-in LN supply line, or by hand. By keeping the all-metal valve always at 450 K, it is possible to close it, allow the trap to warm up when needed, refill the trap, and reopen the valve. This arrangement provides satisfactory control of contamination from an oil-sealed mechanical pump (23).

Using absorbent material is time-consuming and expensive, and can contribute minute, solid pieces of the sorbent into the system. Metallic bonded, high surface area materials can be used instead.

With valves open, the air in the vessel is exhausted through the U-trap by the oil-sealed pump. As the pressure falls, the composition of the gas in the vessel begins to change. At a pressure of ca 13 Pa (0.097 mm Hg), water is the dominant species in the gas phase. The surface phases then change appreciably, although initially water was the dominant species on the surfaces. The bulk phase is unlikely to contain any water molecules as such, except in voids and gross defects. Water is desorbed from glass as a result of OH radicals changing to H_2O at the surface.

2.3. Diffusion Pump System

After the pump line and trap have been shut off, a large valve is opened slowly enough that the mass flow of gas from the chamber through the valve into the oil-diffusion pump system does not disrupt the top jet of the diffusion pump (DP) (Fig. 4). When the liquid nitrogen is replenished after the trap has been operated for some time, release of previously trapped gas must be avoided. The so-called ionization-gauge response pips at the start of the liquid-nitrogen replenishment are an indication of trap ineffectiveness.

The trap fill line is separated from the high vacuum region by an overhanging copper skirt, ie, the creep barrier, which also serves to keep the interior surface exposed to the working environment at a temperature independent of the liquid level in the trap. Oil creepage in two dimensions along surfaces is effectively inhibited at <200 K. Contribution to creepage by the liquid-nitrogen trap is usually small compared with the contamination delivered during the filling of the trap and reduction of LN level.

2.3.1. DP Speed Factor

Pumping-speed efficiency depends on trap, valve, and system design. For gases having velocities close to the molecular velocity of the DP top jet, system-area utilization factors of 0.24 are the maximum that can be anticipated: eg, less than one quarter of the molecules entering the system can be pumped away where the entrance area is the same as the cross-sectional area above the top jet (see Fig. 4). The system speed factor can be quoted together with the rate of contamination from the pump set. Utilization factors of <0.1 for N_2 are common.

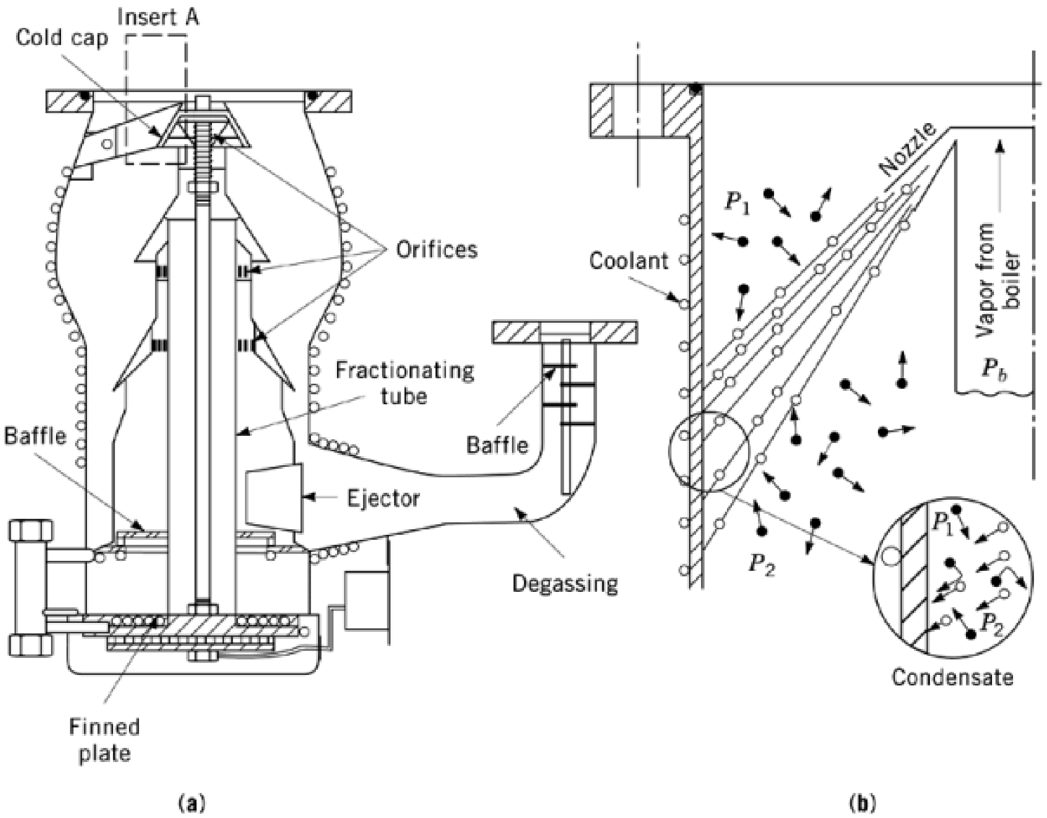


Fig. 4. (**a**) Multistage diffusion pump; (**b**) insert A, jet spray, where closed circles indicate gas molecules P_1 and open circles indicate vapor-jet molecules P_2. P_b is boiler pressure (23).

The rate of contamination from the pump set is $<10^9$ molecule/$(m^2{\cdot}s)$ for molecular weights >44 (23). This is the maximum contamination rate for routine service for a well-designed system that is used constantly and subject to automatic liquid-nitrogen filling and routine maintenance.

A fraction of gas, depending on species, is pumped by the diffusion pump or trapped on the cold surfaces of the LN trap; for example, 0.05 for H_2 and 0.9 for H_2O, respectively, have been measured. With enough flow, multilayers of gas can build up on the liquid-nitrogen-cooled surface, thereby evaporating at the bulk vapor pressure of the gas at LN temperature. To cope with such buildups, a well-designed valve is required immediately between the system and the LN-cooled surface. Designs incorporating this valve within the LN trap itself are available. The valve can be closed and the trap warmed sufficiently to purge accumulated gases such as carbon dioxide. Furthermore, the trap can be allowed to warm to room temperature without delivering contamination into crannies and surfaces of the valve exposed to the chamber when the valve is open.

3. Leaks

A vacuum system can be stalled by gas leaks (4, 6, 24). Traditionally, leaks are categorized as real or virtual. A real leak refers to permeation processes or cracks or holes that allow external gas (air) to seep into the vacuum environment. Atmospheric gases such as helium and hydrogen permeate glass equipment, especially

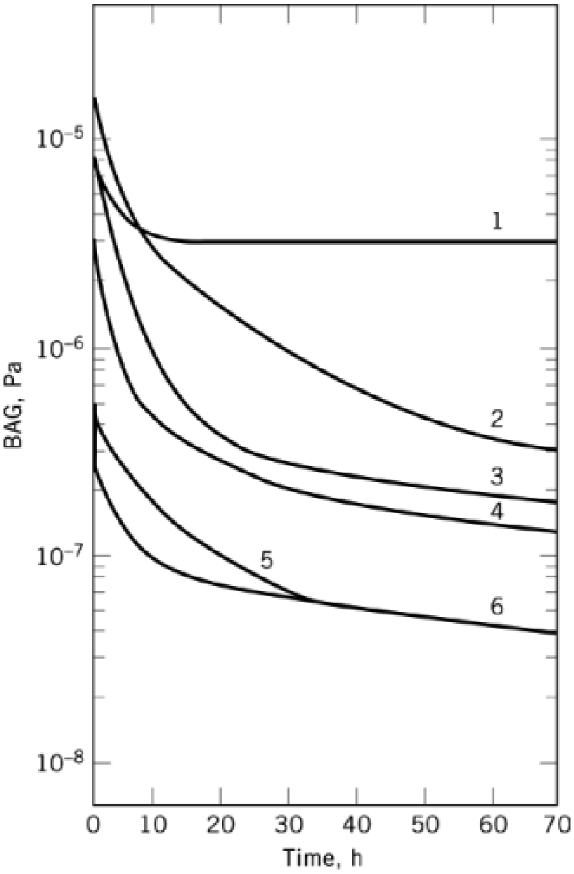


Fig. 5. Plots of pump-down performance for pumps operating on $0.1\text{–m–dia}\times 0.43\text{–m–long}$ stainless-steel tubing. Curves 1–4 are sputter-ion pumps of different makes; curve 5 is Orbitron type; and curve 6, LN-trapped oil DP. Pressure is measured using Bayard-Alpert gauge (BAG) (26). To convert Pa to torr, divide by 133.3.

at elevated temperature. The noble gases do not permeate metals, but hydrogen does. Virtual leaks refer to gases that originate from within, eg, from trapped volumes, the gauges, pumps and the bulk and surface-phase species. For example, carbon in bulk stainless steel may precipitate along grain boundaries and then combine with surface oxygen to give CO, which is then desorbed into the gas phase (25). Proper instruments readily distinguish real leaks from virtual leaks.

In practice, it is often necessary to take readings from hot-filament ionization gauges or other devices. Figure 5 gives pump-down curves for six different types of pumping equipment on the same vacuum chamber (23). The shape of curve 1 indicates that a real leak could be responsible for the zero slope demonstrated by the Bayard-Alpert gauge (BAG). The shape of the other curves could result from a combination of real and virtual leaks.

In fact, the leveling-off slope of curve 1 was entirely owing to gas issuing from the pumping equipment itself, and there were no other sources of leakage. Curves 2–6 all resulted from combinations of virtual and real leakage. Most of the leakage in curves 2–4 originated in the pumping equipment. This was also true for the early part of curve 5. The phenomenology of the early stage of curve 6 resulted from chamber-wall outgassing. The latter stages of curve 6 show a combination of wall outgassing and a small leakage from the pump itself.

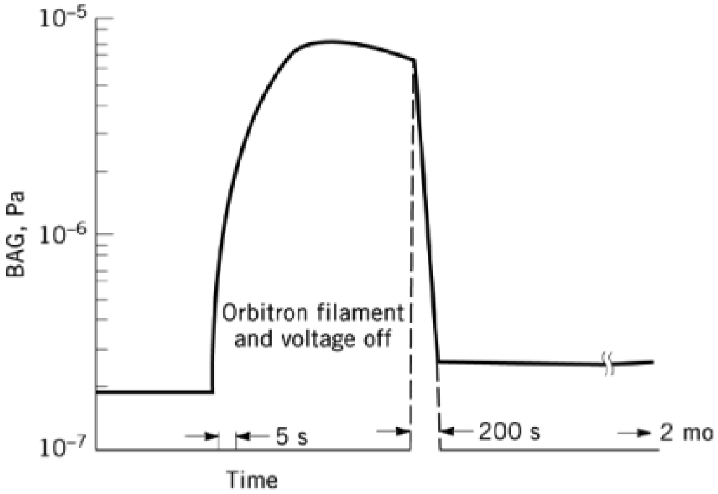


Fig. 6. Bayard-Alpert gauge response vs time upon cutting off power to Orbitron pump (27). To convert Pa to torr, divide by 133.3.

Figure 6 is instructive regarding the magnitude of leakage from pump 5, shown in Figure 5, after ca 5 h of pumping. Pump 5 was a commercial Orbitron made by the National Research Corporation. After ca 4 h from the start of the pump down, the electric power to the pump was turned off, whereas the power to the BAG remained on. The total pressure in the system rose very rapidly, and this response is recorded in Figure 6. Within a short time, however, this rise reached a maximum and assumed a small slope downward. After ca 200 s, the reading on the BAG was close to the reading achieved before the Orbitron power had been turned off. It remained essentially constant for two months after the initial pump down.

Figure 6 can be interpreted as follows. The Orbitron pump is delivering a virtual leak back into the vacuum environment. As with many other pumps, this quantity of gas is significant. The operational speed of the Orbitron just before the electric power was turned off was zero. Thus, an equilibrium was established between the source–sink properties of the vacuum environment. When the Orbitron power is turned off, a slug of electron-bombarded, evaporating titanium remains hot for some time and continues to outgas. As the slug cools down sufficiently, however, the BAG intrinsic speed is sufficient to balance the decreasing rate of gas evolved. The intrinsic steady speed for the BAG is known to be ca 100 cm^3/s. Thus, after ca 4 min from the time the electric power was switched off to the Orbitron, the operational speed of the system is provided by the ionization gauge and the sorption properties of the system. The ionization gauge is thus able to provide almost the same equilibrium base pressure provided by pump 5, which has a rated speed nearly 4000 times greater than that of the BAG. The significance of equilibrium base pressure is use-dependent. The category virtual leak must include evolution from the pumping system and the condensed phases, as well as from atmospheric air and gas trapped in cracks and voids. The question of virtual leak vs real leak may be important in every system.

4. Molecular Transport

Molecular transport concerns the mass motion of molecules in condensed and gaseous phases. The mass motions are driven primarily by temperature. As time progresses, the initial mass motion results in concentration gradients. In the condensed phase, flow along concentration gradients is described by Fick's law.

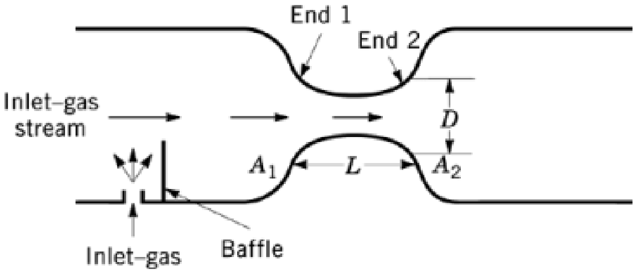


Fig. 7. Duct connecting two volumes of dimension $\gg D$, where A_1 is the area of end 1; A_2, that of end 2.

Standard texts may be consulted on the topic of diffusion in solids (6, 12, 13). Some generalizations, however, are possible. No noble gas permeates a metal. Metals are, however, permeated readily by hydrogen. Stainless steel, for example, can be permeated by hydrogen from concentrations likely in air. The least permeable material for hydrogen is carbon. Glasses are permeable, especially by the light noble gases at elevated temperatures.

After a bake-out of 600–700 K, the bulk phase is likely to far exceed the surface phase as a source of atomic (molecular) impurities that desorb into the gas phase (28). Bake-out at 1300 K greatly reduces bulk-phase impurities.

4.1. Gas Transport

Initially, in a vessel containing air at atmospheric pressure, mass motion takes place when temperature differences exist and especially when a valve is opened to a gas pump. Initial flow in practical systems has been discussed (29), as have Monte Carlo methods to treat shockwave, turbulent, and viscous flow phenomena under transient and steady-state conditions (5).

4.1.1. Viscous Transport

Low velocity viscous laminar flow in gas pipes is commonplace. Practical gas flow can be based on pressure drops of $<50\%$ for low velocity laminar flow in pipes whose length-to-diameter ratio may be as high as several thousand. Under laminar flow, bends and fittings add to the frictional loss, as do abrupt transitions.

4.1.2. Free Molecular Transport

The free molecular gas regime is illustrated by Figure 7. A duct of maximum transverse dimension D and length L connects two chambers, each of minimum interior dimension $\gg D$. Free molecular transport (Knudsen flow) is often sufficiently approximate when $\lambda \geq D$. For a right circular cylinder of length $L = D$, the diameter, the internal pressure drop in free molecular flow is ca 50% (8, 9, 10). In free molecular flow at steady state (Fig. 7), the temperature of the gas entering a duct determines the rate of passage through the duct, not the temperature (other than zero) of the duct itself. Volumetrically, Knudsen flow is proportional to gas entering velocity only; it is constant, independent of gas concentration or gradient. Free molecular flow can be described in terms of a statistically valid number of molecules interacting with the surfaces of a duct, provided that the entering spatial and ongoing reflection distributions from the walls by the molecules are known.

If a Maxwellian gas at steady state is entering end 1 in Figure 8, then the duct conductance, C, is

$$C = 1/4\ (\bar{v} A_1 W_{1\longrightarrow 2})$$

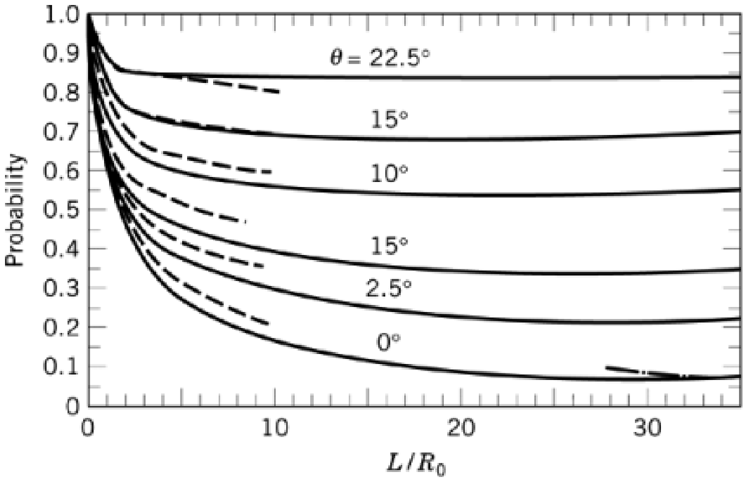


Fig. 8. Conical ducts having length L, small end of radius, R_0, and half angle θ (30). (———)represents Monte Carlo method (29, 31); (-·-·-), long-tube asymptote. The divergence of solid and dashed lines results from an error in maintaining the sample size during the Monte Carlo method (30, 31).

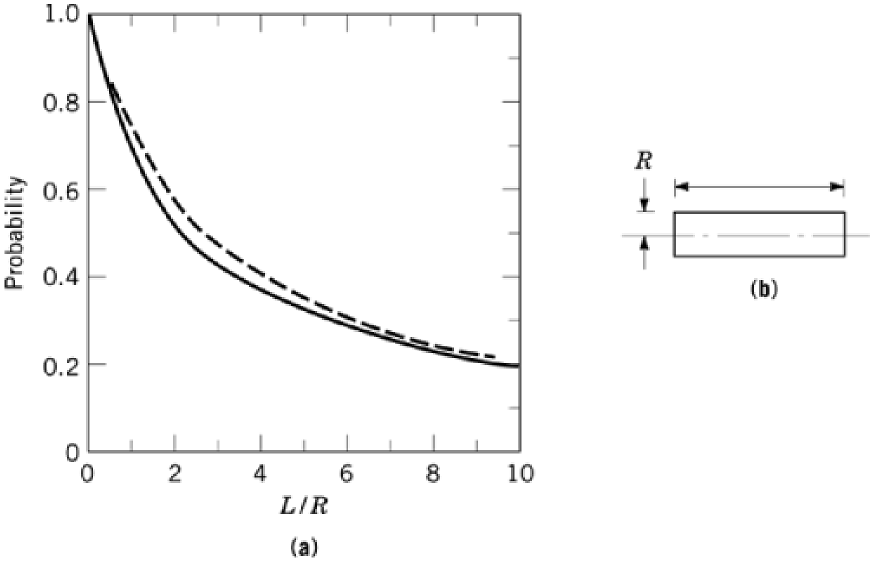


Fig. 9. Molecular-transmission probability: (**a**) for a cylindrical tube; and (**b**) as a function of the ratio of length to radius, L/R. The conductance is $C=1/4\ (\pi R^2 \bar{v} W_{1\longrightarrow 2})$. The solid curve is from Clausing's calculation; the dashed curve corresponds to the approximation $W=1/(1+(3L/8R))$ (32).

where v is the average velocity of the Knudsen Maxwellian gas entering end 1 (before wall encounter);A_1 is the area of end 1; and the transmission probability of a Maxwellian gas (typically reflecting from the walls by a cosine distribution) is $W_{1\longrightarrow 2}$ = number number in end 1 per number out through end 2.

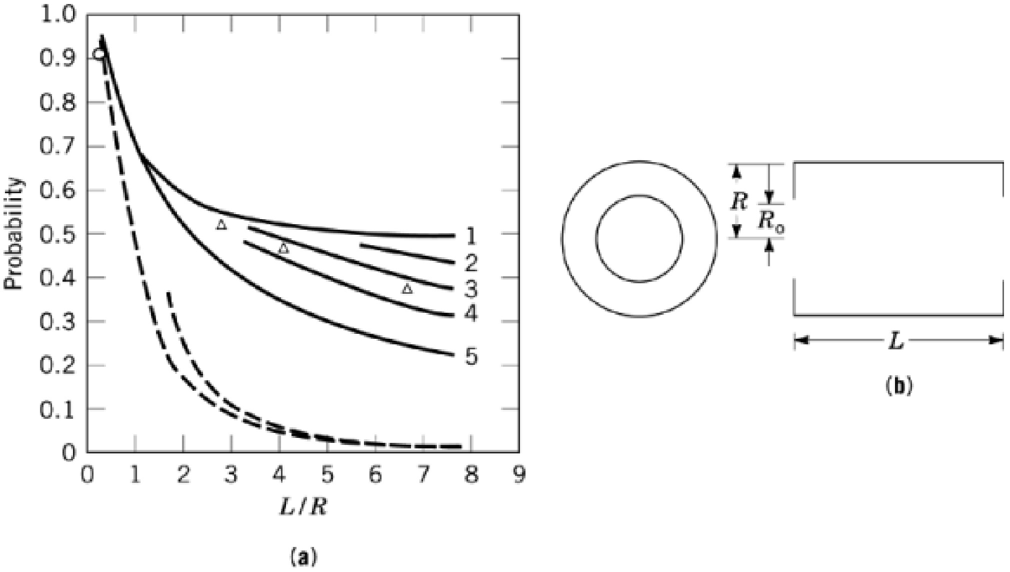


Fig. 10. Molecular-transmission probability (**a**) for circular cylinder (**b**) having two restricted ends where the solid line represents fraction transmitted without wall encounter; and the dashed line, prediction of this fraction by the formula R^2_c/L^2 vs L/R_0. $(R/R_0)^2$: 1, ∞; 2, 3; 3, 2; 4, 1.5; 5, 1.0. (○), Argon, $(R/R_0)^2=2$; and (△), nitrogen, $(R/R_0)^2=2$ (33).

Under isothermal conditions where energy is not added or removed from the system, the second law of thermodynamics obtains, and

$$A_1 W_{1 \longrightarrow 2} = A_2 W_{2 \longrightarrow 1}$$

Thus, the probabilityof transmission must be directional for $A_1 \neq A_2$, but the conductance cannot be directional (see Thermodynamics). If C is directional, energy must be supplied from external sources and C is a pumping speed. Thus, work is performed by pumping but not by conductance. Under free molecular flow, the volumetric rates of transport in the gas phase are independent of the pumps being on or off; bends in a duct little alter the probability of passage over a straight duct having the same axial length. The intuitive idea that the walls of transition sections must be fared in (smooth flow) does not apply in free molecular flow. For example, conical transitions (see Fig. 9) (30) and the area above the top jet in Figure 4 always have smaller transmission probabilities than right circular cylinders having one restricted end where diameters and length are the same as in a given conic frustum. A right circular cylinder without a restricted end has a free molecular conductance that is greater than that of the conic frustum. However, there can be other reasons than conductance for using conical transitions in a given instance.

4.2. Wall Geometries

Rougher-than-rough wall geometries can reduce transmission probabilities in Knudsen flow by as much as 25% compared to the so-called rough-wall cosine reflection (34, 35). For this and other reasons, conductance calculations that claim accuracy beyond a few percent may not be realistic.

In free molecular flow, if gaseous conductance were not independent of the flow direction, a perpetual-motion machine could be constructed by connecting two large volumes by a pair of identical ducts having a turbine in front of one of the ducts. A duct that has asymmetrically shaped grooves on its wall surface could alter the probability of molecular passage in such a way that for a tube of equal entrance and exit areas, the probability of passage would be made directional.

On purely kinetic grounds, however, the term random must be used carefully in describing a Maxwellian gas. The probability of a Maxwellian gas entering a duct is not a random function. This probability is proportional to the cosine of the angle between the molecular trajectory and the normal to the entrance plane of the duct. The latter assumption is consistent with the second law of thermodynamics, whereas assuming a random distribution entry is not.

The probability of passage is independent of the entrance velocity of free molecules and the subsequent velocity ($v \neq 0$) of these molecules within the tube, but depends on the entering angular and wall-reflection distributions of the molecules. It is difficult to predict the distribution functions of molecules reflecting from single-crystal surfaces (31, 34–36) and thus also from engineering surfaces. For engineering surfaces and gases at room temperature, reasonable results within ± 10 percent are obtained by assuming that a statistical number of molecules impinging on a surface exhibits a cosine distribution upon reflection from the surface. Some engineering surfaces may be classed as rougher than rough, however, and the distribution of reflected molecules from an element of surface shows a maximum near the normal to the wall of the tube (34) rather than the spherical shape predicted by the cosine law. Thus, cooling or heating a structure such as a trap does not alter the probability of molecular passage through the structure. This statement assumes that the spatial distribution of molecules reflecting from the walls of the trap is not a function of temperature and that a Maxwellian gas in steady state is entering the trap. In actuality, arranging constant temperature differences in steady-state flow can work on a free molecular gas and cause a pumping effect. This pumping effect is distinguished from gaseous conductance because net work is performed on the free molecular gas. In other words, in special cases, series arrays of ducts can be caused to pump when temperature differences are maintained in the arrays. These constant temperature differences are maintained because energy is supplied to the system from external sources to maintain the temperature differences. This energy from the external sources keeps the system operating at steady state at an end-to-end pressure (concentration) difference.

In free molecular flow, all tubes of a geometrically similar shape have the same free molecular probability of passage for the same entering gas distribution. When dealing with Maxwellian gases, therefore, the probability of passage through a duct can be plotted and this probability can be used to calculate the conductance for all pipes of that shape (Figs. 8, 9, 10, 11, 12). Under free molecular flow, bends in the tube of equal entrance and exit areas little alter the probability of molecular passage compared to a straight tube having the same axial length. In a few cases, the probability of passage through a duct can be obtained by inspection. For example, through a thin orifice, the probability is 1; through a large box that has a pinhole entrance and a pinhole exit, it is 0.5. If a small plate is placed in the box and obscures the line of sight between the pinholes, the probability of passage is still 0.5.

4.3. Combining Conductances

Combining short conductances may be difficult because, if a free molecular gas that is Maxwellian in steady state enters conductance 1 (length $\neq 0$), the gaseous distribution is no longer Maxwellian at exit 1. This corresponds to the so-called beaming effect. The overall conductance can be estimated if the probabilities of passage of the individual components are known and if the juxtaposed components do not vary more than about a factor of two in cross-sectional areas (6, 33). In general, exact methods of calculating conductance depend on knowing the spatial distribution of molecules encountering and reflecting from real surfaces.

4.4. Pumping Speed

If the standard formulas for gas flow in vacuum are applied, eg, pumping speed $S = Q/P$, where $Q = \text{mass}$ of gas transported and $P = \text{pressure}$, it is assumed that a Maxwellian free molecular gas is entering the pump.

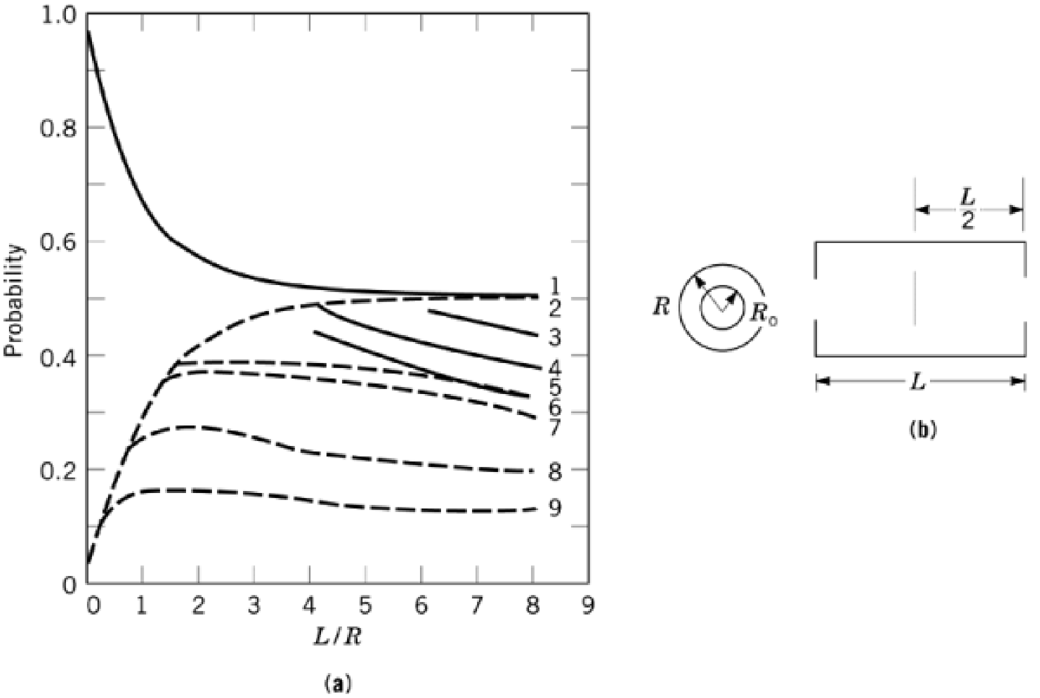


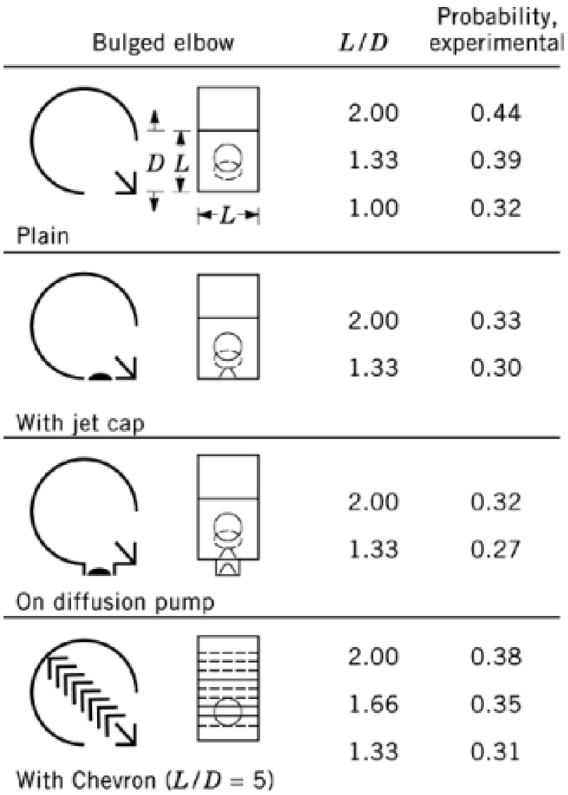
Fig. 11. Molecular-transmission probability (**a**) of a round pipe (**b**) with (—) and without (- - -) blocking plate and restricted entrance and exit apertures. $(R/R_0)^2$: 1, ∞; 2, ∞; 3, 3; 4, 2; 5, 2.25; 6, 1.5; 7, 2.0; 8, 1.5; and 9, 1.25 (37).

The pump must be used on a chamber having a cross-sectional area much larger than that of the cross-sectional entrance of the pump. The gas concentration is then sufficiently uniform in the chamber and across the entrance to the pump. As the chamber is made smaller, finally coinciding with the pumps' entrance area, the gaseous distribution entering the pump is increasingly non-Maxwellian. This does not present a problem, however, provided that this fact is taken into account when assessing the system. For example, the behavior of a free molecular gas from point to point can be predicted mathematically, whether it is Maxwellian or not (5). The calculations can be augmented by experimental measurement. When using only experimental measurements, the data are treated like those obtained with Maxwellian gases. Calculating the probability of passage and pumping speed for more complex shapes may require high speed computers, but once a code is obtained, only a limited amount of machine time may be needed (5). Similar codes have been worked out for neutron and radiation problems.

Instead of calculations, practical work can be done with scale models (33). In any case, calculations should be checked wherever possible by experimental methods. Using a Monte Carlo method, for example, on a shape that was not measured experimentally, the sample size in the computation was allowed to degrade in such a way that the results of the computation were inaccurate (see Fig. 8) (30, 31). Reversing the computation or augmenting the sample size as the calculation proceeds can reveal or eliminate this source of error.

4.5. System Pumping Speed

The operational speed of the pump is a systems effect. All current pumps perform more than one function in the system. Pumps are sinks and at the same time sources for molecules. Pumping speed relates to work on the gas phase. Each type of pumping system occupies a projected area on the vacuum vessel wall. This area may



Bulged elbow	L/D	Probability, experimental
Plain	2.00	0.44
	1.33	0.39
	1.00	0.32
With jet cap	2.00	0.33
	1.33	0.30
On diffusion pump	2.00	0.32
	1.33	0.27
With Chevron ($L/D = 5$)	2.00	0.38
	1.66	0.35
	1.33	0.31

Fig. 12. Transmission probability for bulged-elbow geometries (31).

be a hole in the wall, a surface that getters gas, or a cold surface that condenses gas. In the case of cryogenic or gettering surfaces, there can be some advantage in making the actual area of the surface larger than its projected area. If the primary interest is to increase the pumping speed of the system as distinct from the molecular handling capacity, the speed can be increased for molecular species having sticking coefficients of <0.3.

Increasing the specific area of a solid surface that retains the molecules that strike it impedes a free molecular gas in reaching more deeply into the extended surface area. This conductance effect is geometrical and thus nearly independent of the dimensions of the increased area. However, depending on temperatures and species, when molecular-size pores such as encountered in sorbent material are used, molecules that arrive at the pores initially can plug the pores and prevent subsequent molecules from entering. Furthermore, depending on the temperature of the surface and the molecular species, some two-dimensional adjustment can take place where molecules can penetrate into higher specific-area regions by two-dimensional flow along surfaces. The system pumping speed for holes cut in the side of a vacuum system can be evaluated in terms of efficiency and cost for this pumping. For Maxwellian gases in free molecular flow, the chance of molecules being pumped through a hole is independent of the concentration. This pumping probability has been incorrectly called the Ho coefficient for the system (6). The probability of a molecule exiting from a system through a hole and not returning is equivalent to the Clausing factor for Maxwellian free molecular gases. Pumping systems, whether based on turbomolecular, ionic, getter, cryogenic, or diffusion-pump principles, have yet been unable to utilize holes fully and be of reasonable size and cost. If the area of a hole in the vessel is equal to the cross-sectional entrance area of a pump operating on that hole for the light gases hydrogen and helium, the system pumping probability is ca 1:10. Thus, theoretically, another pump could be placed on the same hole and deliver 10 times the pumping speed available. This inability to utilize holes and still keep the cost of a pump within reasonable bounds stems from several difficulties.

Diffusion pumps, whether of oil or mercury, require a trap to prevent the working fluid of the pump from contaminating the vacuum environment. Liquid-nitrogen traps are usually employed. Their temperature, however, is not low enough to condense any hydrogen or helium gas even on sorbents of large specific area. In addition to the trap, a valve is often essential in the system. Analytical estimates of the maximum system probability realizable for a diffusion pump showed it to be less than 3:10 for molecular species having velocities near to or less than the molecular velocity of the top jet of the diffusion pump (Fig. 4). It thus appears impractical to increase the molecular velocity of the top jet. The pumping of light gases such as hydrogen and helium is fundamentally limited by the velocity of working fluid molecules in the top jet. From a user's point of view, there should be a sizeable market for pumping equipment that could utilize the potential of wall area better without increasing the system cost.

Turbomolecular pumps operating on holes are faced with the same fundamental problem as the diffusion pump. The tip speed of the turbine rotor in commercial pumps, because of the strength-to-density ratio of the turbine blade material, is limited to $<6 \times 10^2$ m/s. The average velocity for helium at room temperature is ca 1×10^3 m/s; for hydrogen, 2×10^3 m/s. Patents have been filed related to a novel vacuum pump (38). Magnetically suspended self-balancing rotating arrays of fibers permitting tip speeds of ca 3×10^3 m/s are self-cutting and of low mass (light weight).

5. Instrumental Measurement

Especially important under dynamic conditions, the role of a system and of each component can be disclosed by appropriate measurements. Thus, it can be established when the system environment is ready for a dynamic use, eg, if the pump is likely to perform as a molecular sink, a source, or some combination of these.

5.1. Gauges

Because there is no way to measure and/or distinguish molecular vacuum environment except in terms of its use, readings related to gas-phase concentration are provided by diaphragm, McCleod, thermocouple, Pirani gauges, and hot and cold cathode ionization gauges (manometers).

Ionization gauges (IG) do not give pressure in the gaseous phase but are set to provide readings proportional to the concentration of molecules in the gaseous and, to a lesser extent, the condensed phases. These concentrations are translated to units of gaseous pressure. The hot and cold cathode ionization gauges, such as those shown in Figures 13 and 14, provide information about vacuum environment as a host of parameters. Perhaps largely by trial and error, selection of a gauge readout indicates when to begin the process or experiment. Turning the gauge off and on provides the so-called flash-filament gauge response. When the filament is turned off, it cools, and molecules from the gaseous environment impinge and stick to the surface of the cooling filament. When the filament is turned on again and its temperature rises to incandescence, these molecules are desorbed. Sufficient electrons are provided by the filament to indicate the gas-phase concentration increase from this desorbed material. An abruptly rising pip having a longer decaying tail can be recorded from the output of the gauge. The area under this desorption peak is a measure of the integrated pumping effect of the cold bare filament. The filament is bombarded by the free molecular gas over a ca 2- π solid angle. The flux of particles striking the filament is given by $n{\cdot}\bar{v}$, where n is the molecular concentration in the gas phase and $\bar{v}$ is the average velocity of this Maxwellian gas. By changing the power supplied to the filament in its heating

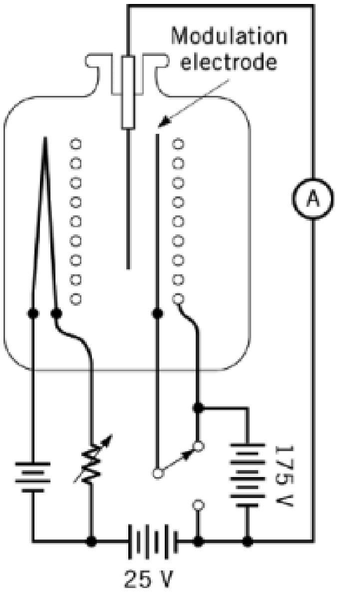


Fig. 13. The introduction of a modulator electrode in a Bayard-Alpert gauge permits the pressure range to be lowered by a factor of 10^{-1} to 133 pPa (10^{-12} torr) (39).

phase and varying the time interval when the filament is left cold, approximate but useful information can be obtained (13).

5.2. Residual Gas Analyzers

A gaseous molecular phase is analyzed using a mass spectrometer (41) (see Analytical methods). The surface and bulk phases of the vacuum environment can be probed for appropriate uses. Desorbed species, neutrals, and metastable, positive, and negative ions can all be read by the residual gas analyzer (RGA) through its ion source and/or by its electron multiplier (13). If heat is delivered to the condensed phase or if electrons are caused to strike its surface, molecular desorption provides a signal for the mass spectrometer analysis. Direct electron desorption does not involve heating. For contaminated surfaces, the probability of a molecule being desorbed per incident electron can be as high as 10^{-3}. For submonolayer coverage of some species, this probability may decrease to $<10^{-7}$. These probabilities are large enough to allow a significant signal from the mass spectrometer. The electron current delivered to the surface can be raised to deliver intense heat, providing information about the bulk phase near the surface. Photons desorb molecules directly from surfaces (except for water vapor).

Ultrasound frequencies can be introduced into the walls of the vacuum system. If a source of ultrasound is placed on the wall of an ultrahigh vacuum system, a large hydrogen peak is observed. Related phenomena, presumably from frictional effects, are observed if the side of a vacuum system is tapped with a hammer; a desorption peak can be seen. Mechanical scraping of one part on another also produces desorption.

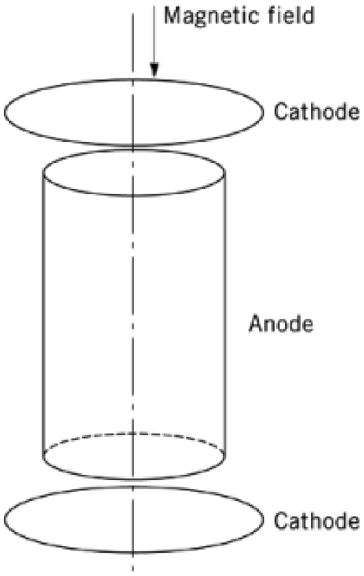


Fig. 14. Geometry of a single cell of a penning gauge. Space charge of the trapped, circulating electrons equalizes the axis potential with that of the cathode. Thus, the electric field is radial. Electron density is at a maximum a short distance from the anode. Electrons progress radially toward the anode only as they lose kinetic energy, mainly through inelastic (ionizing) collisions with molecules (40).

6. Vacuum Systems and Equipment

6.1. Wall Materials

Glass (qv) is often the material of choice for small laboratory systems and sealed systems in commercial practice. Glass has a wide range of useful properties, including high compressive strength but relatively low tensile strength, requiring careful selection of the glass and design. Evacuated glass tubes, such as photomultiplier tubes, are exposed to temperatures as high as 720 K. In some applications, glass has been displaced by high alumina ceramic. Borosilicate glasses, in the form of industrial glass pipe, have been used in large experimental systems that frequently require demountable joints.

Industrial glass pipe and other wall materials are connected to other materials that have demountable seals made of elastomers or soft metal wire, eg, lead, indium, tin and their alloys. The soft-wire seals, however, have led to difficulties. High temperature bake-out must be avoided, and glow-discharge cleaning must not reach the gasket. Furthermore, the gasket material can creep under load and admit a leak, except in the case of indium, which creeps but does not usually leak (see also Packing materials).

The choice of metals for vacuum walls is largely based on the ease of fabrication of the metal, machining, cleaning (26), welding, etc. Aluminum alloys are the material of choice for out-gassing at room temperature.

Demountable joints are commercially available in great variety in stainless steel, but less so in aluminum alloy or related materials. Experimental joints have been made in which aluminum flanges employ aluminum foil as gasket material.

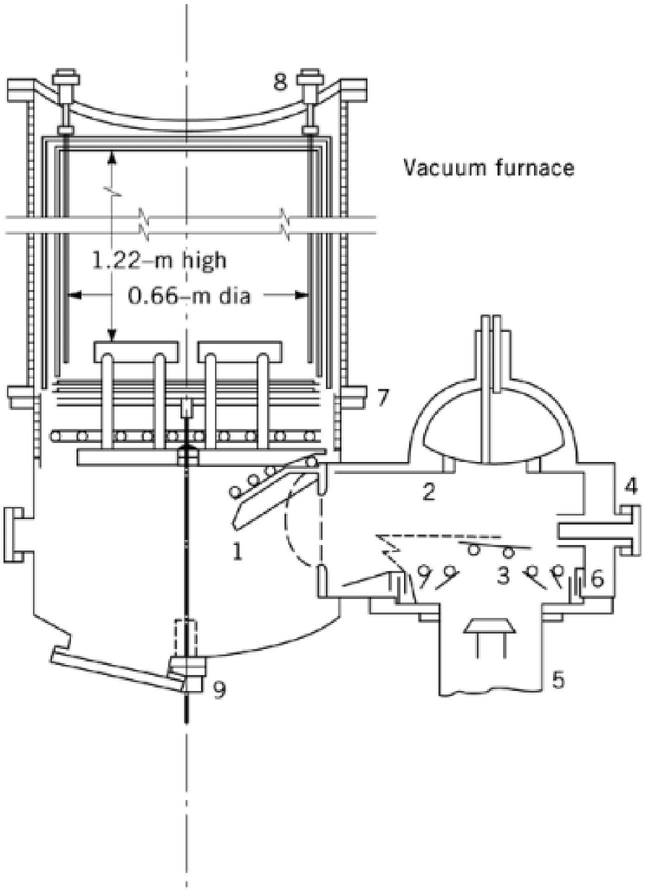


Fig. 15. A low contamination vacuum brazing furnace for brazing large metal-to-ceramic joints: 1, valve, open (—) and shut (___); 2, LN trap; 3, water-cooled baffle; 4, valve actuator feedthroughs for lowering the bottom heat shields; 5, diffusion pump oil, 1.25-cm dia; 6, creep barrier; 7, flange separation for opening the furnace; 8, electrical feedthroughs for lowering the bottom heat shields; 9, seal for raising and lowering of the furnace (42).

6.2. Vacuum Brazing Furnace

The cross sections of a vacuum brazing furnace of the bell-jar type are shown in Figures 15 and 16. Contamination is very low even when rapidly cycled from one work load to the next. The bell jar can serve on one hearth while another hearth is being loaded for the next operation.

Rapid cool-down by helium heat transfer is made possible at an interior of ca 100 Pa (0.1 atm). A convective fan transfers heat efficiently from the interior hot surfaces of the furnace to water-cooled base and wall parts.

During startup, the baseplate heat shields are lowered, thereby allowing pump-down through a large gap. The heat shields can then be raised to produce a narrow gap when maximum temperature is required.

Offset design gives ready access along the axis of the hot zone. This design permits routine operation and cycling of the furnace without sacrificing control of contamination, access, and speed for condensables or noncondensables.

An optimized relationship is obtained between the bell jar, 60° swing-leaf valve, LN trap, baffle for the oil, and the plane of action for the diffusion pump (DP) top jet. The valve open area equals 0.38 of the cross-sectional

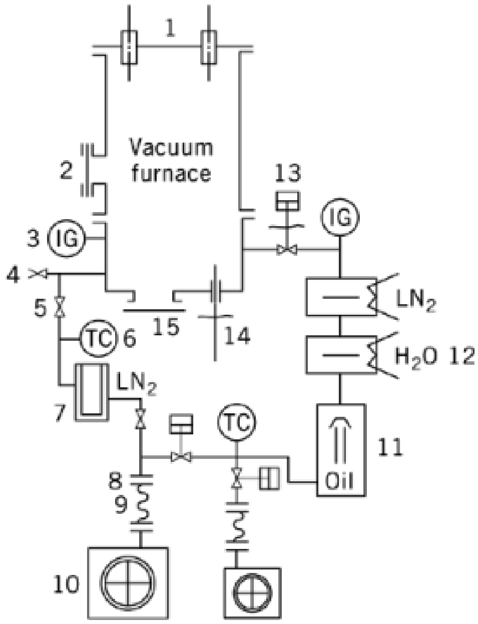


Fig. 16. Schematic representation of the vacuum furnace shown in Figure 15, where 1 is electrical feedthrough; 2, viewport; 3, vacuum-gauge hot-filament ionization; 4, air valve; 5, valve; 6, thermocouple vacuum gauge; 7, thimble trap; 8, demountabl coupeling; 9, flexible line; 10, two-stage liquid-sealed mechanical pump; 11, DP; 12, water-cooled baffle; 13, pneumatic valve having sealed bellows; 14, linear motion feedthrough having sealed bellows; and 15, blind-flange port (43). The graphic symbols used are among those contained in the American Vacuum Society Standard 7.1. $_{\mathrm{IG=ionization}}$ gauge.

area of the inside diameter of the furnace. The volumetric speed factor for water vapor is thus $0.38 \times 0.9 \simeq 0.34$, where 0.9 is the Clausing factor.

No gas pips occur during the filling of the LN trap, and the temperature of the trap surfaces does not vary more than 0.01 K with the liquid-nitrogen level variation in the reservoir. The high conductance water-cooled baffle provides a minimum restriction of flow to the DP, yet returns back-streamed pump oil along the walls of the pump attached at its room temperature edge only. A thin, stainless-steel, anticreep barrier, cooled by radiation to the LN-chilled annulus, prevents surface migration of pump oil to the furnace. The bottom heat shields may be lowered by a rod during the warming of the furnace to provide high conductance from the hot zone to the trap inlet. A hot-zone temperature of >1500 K has been achieved.

6.3. Liquid-Nitrogen Traps

The principal reason that cold traps are frequently ineffective in preventing the passage of oil or mercury is the warming of the trap and its internal filling lines when LN is added and/or as LN depletes. However, some designs have eliminated this problem (Fig. 15). A liquid-nitrogen trap need not have an active chemical surface, but in some cases, it is advantageous to cool a chemically active surface to liquid-nitrogen temperature in order to obtain effective trapping of methane and other low molecular weight species. This method, however, has a predictable finite life determined by the formation of monolayers on the chemically active surface. A plain metal surface cooled to liquid-nitrogen temperature in a trap has two primary functions, namely, to act as a cryopump for water vapor and to prevent contamination from DP working fluid from reaching a given vacuum environment. A well-designed LN trap can provide a pumping speed of at least 10^2 m^3/s per m^2 pf of system entrance area for water vapor (at room temperature, under free molecular conditions) and confine oil contamination to negligible levels. Measurements have shown that oil molecules and cracked-oil products having molecular weights of >60 pass through a particular design at rate of $<10^9$ molecules/$(\mathrm{m}^2{\cdot}\mathrm{s})$ (23). These rates correspond to a vapor pressure at room temperature of less than ca 0.1 pPa $(7.5 \times 10^{-16}\ \mathrm{mm\ Hg})$.

Typically, a baffle condenses a vapor flow to a liquid in such a way that liquid can drain off for recycle. A well-designed and useful trap, on the other hand, catches and retains condensables such as water and the higher vapor pressure fraction of the working fluid of a pump. Although the terms baffle and trap seem to describe the same type of function, namely, to slow down but not prevent the rate at which some species of molecules pass through, traps can completely stop some molecular species (23).

Molecules arrive at the surfaces of traps and baffles by volume flow and surface creep. Molecules are trapped in vacuum systems by binding with energies much greater than kT of the surface, where k is Boltzmann's constant and T the absolute temperature, or by lowering the temperature of the surface in such a way that kT is less than the heat of physisorption of a molecular species on a surface.

If a trap is placed directly over a DP top jet (Fig. 4) without baffling action, all of the working fluid is caught by the trap in the time predicted by the rate at which the fringe of the top jets feeds the working fluid to the trap. This rate may be many orders of magnitude greater than the rate predicted from the equilibrium vapor pressure of the working fluid at DP wall temperature.

Valve, trap, and baffle can be combined in such a manner that elastomers can be used in the valve and contamination is controlled from the valve-actuator mechanisms and from the gasket of the valve-plate seal (Fig. 15) (42).

The role, design, and maintenance of creepproof barriers in traps, especially those in oil DPs, remain to be fully explored. In general, uncracked oil from a DP is completely inhibited from creeping by a surface temperature of <223 K. On the other hand, a cold trap, to perform effectively in an ordinary vacuum system, must be <173 K because of the vapor pressure of water, and ≤ 78 K because of the vapor pressure of CO_2. For ultracontrolled vacuum environments, LN temperature or lower is required. CO_2 accumulation on the trap surface must be less than one monolayer. The effectiveness of a LN trap can be observed by the absence of pressure pips on an ionization gauge when LN is replenished in the reservoir.

DP systems can be shut down when not in use to conserve energy. If a liquid-nitrogen trap is incorporated, the manner in which this trap is warmed up and the DP is cooled down should be determined by the presence or absence of a valve between the chamber and the liquid-nitrogen trap. In critical systems, this head valve can be included in order to permit rapid shutdown and rapid return to operation. The assertion that dry nitrogen gas can be used to sweep contamination from traps and pumps in such manner that oil contamination is prevented from running counter to the nitrogen-sweeping flow direction is questionable. Proper placement of valves can eliminate the need of a sweep gas.

7. Process Equipment

A good survey of process equipment widely used in chemical engineering is available (44). The pressure in the gaseous phase characterizes the likely vacuum process environment. Rough vacuum ranges from 101 kPa to ca 100 Pa (760 to 0.75 torr); medium vacuum ranges from 100 Pa to ca 0.1 Pa (0.75 to 0.00075 torr). The chemical engineer is likely to work in the rough vacuum region in which distillation, evaporation, drying, and filtration are normally conducted. The medium vacuum range is employed in molten-metal degassing, molecular distillation, and freeze drying.

Vacuum equipment requires strength to withstand the pressure of the surrounding atmosphere. The full load is ca 101.3 kPa when the internal gas pressure in the system is sufficiently reduced (see Pumps).

7.1. Steam Ejectors

Ejectors are simple vacuum pumps. They have no moving parts but can accomplish compression through fluid-momentum transfer. A high pressure motive fluid enters the ejector and expands through the converging and diverging section of the nozzle. The initial pressure energy is converted to velocity. This increase in velocity entrains the load to be handled, which enters through the suction inlet. The motive and suction fluids are then mixed and recompressed through the defuser, which discharges this mixture into a higher intermediate pressure. By convention, an ejector represents a single-point design and is most efficient at a single set of conditions. Ejectors are either single-stage or multistage, depending on the suction pressures required. A practical compression ratio for single-stage ejectors is ca 6:1 when discharging to atmospheric pressure. This compression ratio can be as high as 10^6:1 for six-stage ejectors. Multistage steam ejectors are usually equipped with direct-contact or surface-type condensors between stages in order to prevent contamination of cooling fluid. When designing a multistage steam ejector, condensables and noncondensables in the intake must be distinguished. Booster stages followed by a condenser are used to handle large quantities of condensables. Intercondensers are sized for both the motive steam from the booster and the process vapors. Thus, the load to the ejector stages downstream is significantly reduced. Such an ejector system uses considerably less steam, and purchase and installation costs are lower than those of a system designed to handle process vapors as condensables.

7.2. Liquid-Ring Pumps

In a liquid-ring pump, the rotor is the only moving part. The liquid ring performs all the functions normally done by mechanical pistons or vanes. There are a number of variations of this design, but all operate on the principle that before startup the pump casing is partially filled with a sealant liquid. When the rotating impeller is turned on, the liquid is caused to contact the periphery of the casing centrifugally. Thus, a liquid ring is formed that seals off the cylindrical pump body. Because the rotor axis is offset from the body axis, a piston action is established as the liquid fills and then almost empties each of the chambers between the rotor blades. Compression ratios as high as 10:1 can be achieved when discharging to atmospheric pressure for a single-stage pump. Because of the apparent difficulty in rating a liquid-ring pump on the basis of swept volume, it is classed as an isothermal machine rather than a true positive displacement compressor. The liquid ring acts as a heat sink to maintain constant-temperature operation. Assuming a normal sealing-liquid flow, a temperature rise in compressing air from ca 26.6 to 101.3 kPa (ca 0.25 to 1 atm) is ca 3–6 K. For comparison, an uncooled adiabatic compressor can cause a temperature rise exceeding ca 90 K. In sizing the pump, both evaporative and condensing effects must be considered. Evaporative cooling takes place whenever dry gases are introduced at temperatures higher than those of the sealed liquid. Condensation occurs when pumping gas that is saturated with vapor, ie, when the pump behaves like a direct-contact condenser.

7.3. Rotary-Piston Pumps

Positive-displacement, oil-sealed machines that compress a specific volume of gas with each revolution, and compress and exhaust it to the atmosphere, are the rotary-piston pumps. The oil-sealed piston traps the aspirated gas ahead of it by closing the inlet port. The gas is compressed, the discharge valve opens, and the gas is exhausted to the atmosphere. Compression ratios can be as high as more than 10^6:1 for a single-stage pump. These pumps operate in an internal oil bath which lubricates the pump and seals against backstreaming from the exhaust into the intake. The rate of flow and the distribution of oil through the pump are the important features of the design. The piston must be sufficiently lubricated to prevent failure. Failures are often caused by breakdowns of the oil distribution systems.

Rotary-piston pumps are frequently stalled by condensation of process vapors in the lubricating fluid. In addition, condensate can accumulate in the pump oil, resulting in mechanical failure and permanent damage. Water vapor, a persistent source of oil contamination, is almost always present in vacuum-processing operations. Higher alcohols and other solvents normally encountered also have a tendency to condense in the lubricating oil during compression. A number of techniques have been developed to prevent condensation of process vapors in oil-sealed pumps. Gas ballast, the most common technique, involves drilling a hole in the head of the pump to admit air or other gas into the cylinder during the latter portion of the compression stroke. Ballasting takes effect while the gas being compressed is sealed off from the intake by the piston. The ballasting method reduces the partial pressure of the condensible vapor and thus reduces or eliminates condensation within the oil. The introduction of gas-ballast air into the pump increases the pressure differential across the seals between intake and exhaust. Ballasting results in increased leakage past the seals, which can significantly reduce the capacity of rotary-piston pumps operating below ca 100 Pa (ca 0.1 atm). However, the effect on pump capacity is negligible in most processing applications. Below the processing range, however, ballasting when handling the water in ordinary atmospheric air is not needed.

7.4. Rotary-Vane Pumps

Rotary-vane pumps are positive-displacement machines having spring-loaded vanes that contact the inside of the pump casing. Gas entering the pump is trapped between adjacent blades, compressed, and forced out to the atmosphere through the discharge point. Maintenance requirements have severely limited the use of these pumps in process applications. Rotary-vane pumps are still found in laboratory applications and can achieve compression ratios well above 10^6:1 for discharge directly into the atmosphere. Oil contamination by process fluids causes a deterioration in the pump performance and can necessitate frequent cleaning and reassembly of the pump.

7.5. Rotary-Blower Pumps

This type of pump employs two interlocking rotors to trap and compress gases. The rotors are prevented from touching one another, and there is no sealing liquid in the pump. The gears and rotor bearings are lubricated with oil, but are external to the rotors. Clearance between the rotors is generally 25–100 μm. Typically, these pumps operate at high speeds of 3000–4000 rpm. Because there is no positive seal between the rotors, the rotary blower is limited to small compression ratios, though it can also be designed for higher throughput than any other mechanical pump. In process and most other applications, the rotary blower is limited to operation in conjunction with other mechanical pumps. Inherently, rotary blowers are potentially subject to overheating because of the lack of a discharge valve separating the heated gas. As a result, the compression ratio of single-stage blowers is limited to ca 2.3:1. However, if the rotary blower discharges into a rotary-piston, oil-sealed pump, the combination can exceed 10^6:1. When operating below 133 Pa (1 torr), overheating need not be a consideration because the work done in compressing the process load is small.

8. Economic Aspects

Vacuum systems, largely for the semiconductor industry, are the main source of sales (see Semiconductors). The sales of all vacuum equipment, pumps (qv), valves, sensors (qv), etc, in the United States, including applications not in vacuum systems, generally exceed $\$500 \times 10^6/\mathrm{yr}$. A reasonably comprehensive list of high vacuum manufacturers is supplied by the American Vacuum Society's exhibitor's list. In Europe, a special issue of the journal *Vacuum* serves similarly.

Capital investment, capital costs, operating costs, return on investment, and energy conservation have all been discussed (6). In the economic analysis, the speed of each type of pump considered is normalized to 1 m^3/s as a common basis.

With regard to fixed amortized investment (45), utilization of the wall area of the chamber to be evacuated using a given pumping method must be considered. Depending on the process, it may or may not be possible to expose gettering or cryogenic pumping surfaces directly. Many uses would contribute dynamically to a gettering or cryogenic surface, making it uneconomic to handle the energy flux, sputtering processes, etc, which direct exposure might entail (see also Cryogenics).

Energy costs are not directly related to the energy efficiency of the process (6, 42). Even if the thermal efficiency of a steam ejector, for example, is less than that of mechanical equipment run by an electric motor, the overall cost of the energy to run the steam ejector may still be less.

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