HIGH VACUUM TECHNOLOGY

George A. Sofer and Harold C. Weingartner

Nuclear Development Corp. of America, White Plains, N.Y.
and Arthur D. Little, Inc., Cambridge, Mass.

I. Introduction

High vacuum is a term generally referred to pressures below 0.1 mm. of mercury. It is understood to be the pressure region where the laws of fluid flow in the turbulent and laminar ranges do not apply, and where molecular flow either partially or totally characterizes the pattern of fluid flow.

The transition from laminar to molecular flow is gradual. This transitional region is called the "slip flow" region. Flow values in this region lie between values predicted by the laws of laminar flow and those predicted by the laws of molecular flow.

A criterion for determining whether high vacuum conditions exist is that the mean free path of gases in a system must be of the same order of magnitude as the dimensions of the system, or larger.

High vacuum is not and should not be regarded as a unit operation in itself. Rather, it should be viewed as a very low pressure range in which conventional unit operations such as heat transfer, fluid flow, distillation, and extraction are put to use in the light of physical and chemical equilibria, reaction rates, and transfer rates characteristic of this pressure range.

The beneficial influences of high vacuum are entirely analogous to those obtained in the use of high pressure for certain processes.

The influence of high vacuum on diffusional unit operations, such as distillation, drying, impregnation, evaporation and desorption, is to reduce the partial pressure of the inert component (usually air) and thereby increase the rate of mass transfer of the diffusing fluids. In addition to affecting the rate of diffusional operations, high vacuum also influences equilibrium conditions. These two factors work together in bringing such operations nearer to completion. Its effect on heat transfer and fluid flow stems from the drastic reduction of intermolecular collisions and the greater significance assumed by gas-to-wall collisions.

Chemical reactions involving gaseous reactants or products can be influenced by high vacuum in the direction of greater molar volume, as in the case of the dissociation of certain metallic oxides into oxygen and free metals, and the reduction of such oxides by carbon with the formation of CO or CO_2.

Quite aside from physical or chemical reactions, an important function served by high vacuum is the provision of collision-free space, such as required in radio and television tubes, and particle accelerators. In these applications, charged particles must travel relatively long distances before reaching their target. Obviously, their path will be unimpeded only when the probability of collision with residual gas molecules is very low. A similar function is served in vacuum coating, where metal vapor is condensed on a suitable substrate some distance from an evaporation source.

The requirement for high vacuum often stems from the need for chemically inert surroundings necessary for handling reactive metals at high temperatures. For metals such as titanium and zirconium, the heat treating atmosphere must be free of oxygen, nitrogen, and hydrogen, as all of these gases tend to enter into reaction and result in a highly embrittled product. Such operations are either conducted under high vacuum, or in an inert gas atmosphere. The inert gas is usually introduced to the heat-treating equipment following the reduction of air pressure to the high vacuum range.

Research is currently being conducted at extremely low pressure, in the range of 10^{-10} to 10^{-14} mm. Hg abs. In commercial processes today the lower range of pressures employed is of the order of 10^{-6} and 10^{-7} mm. Hg abs., and pressures below this range are frequently referred to as ultrahigh vacuums. It should be readily apparent that the only sound basis for designating high vacuums is in terms of absolute pressure.

In the material which follows, the industrial use of high vacuum is discussed. It will be noted that recently gained knowledge has not yet been reduced to systematically applicable data that are useful and available for engineering design purposes. Furthermore, since useful applications of high vacuum are under investigation and development in many different industries, there exist differences in concept and nomenclature which hinder the unification of information.

A commendable action aimed at overcoming this condition is the organization of the Committee on Vacuum Techniques. This organization is devoting a considerable effort to the development of nomenclature and standards in this field.

II. Historical Development of High Vacuum Technology

The attainment of vacuum dates back to the seventeenth century. Torricelli, in 1643, demonstrated that atmospheric pressure can support the weight of a column of mercury 76 centimeters high. The pressure he attained in the evacuated space above the mercury column, must have been well below 10 mm. Hg absolute. Boyle recognized the influence of vacuum by his statement that "Nature abhors vacuum." Otto von Guericke, in 1654, developed a mechanical pump which he used to evacuate the famous Magdeburg spheres.

Not until the latter part of the nineteenth century, however, did vacuum become a very useful tool. In the hands of J. J. Thomson, it played an important part in the discovery of the electron, and in the hands of Thomas Edison, it made possible the invention of the first electric lamp. In later years, vacuum was useful in studying the internal structure of the atom, and in creating an entirely new industry, the electronics industry.

During World War I, the German firm Heraeus Vakuumschmelze found it advantageous to melt various metals, including steel, under pressures in the range from 1 to 50 mm.

The years of World War II witnessed an extensive industrial application of high vacuum in a number of major war efforts. This was made possible by development of the following vacuum pumps: (a) mechanical vacuum pumps with pumping speeds as high as 200 cu. ft./min. and capable of reaching pressures as low as 0.01 mm. Hg; (b) multi-stage, high capacity steam ejectors capable of producing an ultimate pressure of 0.1–0.05 mm. Hg; (c) vapor diffusion pumps with volumetric speeds as high as 25,000 cu. ft./min. and ultimate pressure of the order of 10^{-6} mm. Hg.

The following projects are good examples of wartime high vacuum application.

A. Freeze Drying

Blood plasma was dehydrated from the frozen state under pressures of the order of 0.3 mm. Hg, and temperatures near −30°C. The need for dehydration arose because plasma in aqueous solution spoiled, whereas storing as a dry powder drastically reduced deterioration. Dehydration from the solid state permitted easy re-solution in water or saline solution, by virtue of the high surface-to-volume ratio characteristic of the lyophilic structure produced by this technique. The rigidity of the solid state does not permit substantial shrinkage of the drying material. Dehydration from a liquid solution, on the other hand, is accompanied by excessive volumetric shrinkage, and the result is an amorphous coagulum of low surface-to-volume ratio, and hence poor resolution. Penicillin was dehydrated in very much the same manner, then streptomycin and many other pharmaceutical products were similarly processed.

B. Reduction of Magnesium

The Pidgeon process for reduction of magnesium from dolomite contributed a large portion of total output of this critical metal during World War II. This process (B1) consists of heating calcined dolomite and ferrosilicon in horizontal retorts, and collecting the distilled metal on removable condenser linings. Reaction temperature is held at approximately 1150°C., and a pressure of 0.100 mm. Hg absolute is maintained, which serves to protect the magnesium vapor from reoxidation. The reaction can be represented as follows:

$$\underset{\text{Dolomite}}{MgCO_3 \cdot CaCO_3} \xrightarrow{\text{heat}} \underset{\substack{\text{Calcined}\\ \text{dolomite}}}{MgO \cdot CaO} + 2\ CO_2$$

$$2\ (MgO \cdot CaO) + \tfrac{1}{6}\ \underset{\substack{\text{Ferro-}\\ \text{silicon}}}{FeSi_6} \xrightarrow[1150°C.]{} 2\ Mg\uparrow + \tfrac{1}{6}\ Fe + (CaO)_2 \cdot SiO_2$$

High vacuum serves further, by reducing the reaction temperature a few hundred degrees below that necessary under atmospheric pressure, for the same reaction yield.

Reduction of calcium from lime by a similar reaction was carried out in retorts similar to those used for magnesium. Here aluminum, rather than silicon, was used as the reducing agent, in order to maintain a sufficiently low reaction temperature.

$$6\ CaO + 2\ Al \xrightarrow[0.100\ \text{mm. Hg}]{1150°C.} 3\ Ca\uparrow + (CaO)_3 \cdot Al_2O_3$$

Other low boiling metals, such as lithium, barium, and columbium, have been reduced by similar reactions, but not on the large scale of magnesium or calcium.

C. Vacuum Melting

In order to achieve the maximum degree of homogeneity and purity in fissionable metals, melting under pressures below 1 mm. was utilized in the war years to reduce the content of dissolved and entrapped gases.

Although vacuum melting in this country was limited, Heraeus Vakuumschmelze was producing a large percentage of the nickel-chromium alloys used in Europe by a vacuum melting technique.

D. Isotope Separation

Separation of fissionable uranium-235 from nonfissionable uranium-238, was carried out by diffusion under high vacuum of suitable gaseous compounds of these metals. This application demanded gigantic installations of high vacuum equipment (B3).

E. Vacuum-Tube Manufacture

Although all vacuum tubes require evacuation, the expanded demand for cathode ray tubes imposed the need for pumps with ultimate pressures of the order of 10^{-6} mm. Hg. The distance of travel of electrons in cathode ray tubes is considerably greater than in receiving tubes; the residual gas pressure must therefore be sufficiently lower to prevent collisions with the electron beam.

Following World War II, a number of new applications opened up for high vacuum, and many wartime uses were extended to peacetime needs. Among the post-war applications, which attained significant commercial importance, are the following.

F. Vacuum Coating

This process, which was developed just prior and during World War II, found a nation-wide market in the metallizing of plastics and other materials. During the war, high vacuum was employed in the evaporation of magnesium fluoride onto optical lenses for the purpose of reducing reflection at the air-to-glass interface, thereby improving the light transmission efficiency of multi-lens systems. Since the war, the same technique was found commercially applicable for evaporating aluminum onto plastic novelty articles, and inexpensive aluminum or zinc die castings for use in imitation jewelry.

It was found necessary to pre-coat the substrate with a smooth, glossy, synthetic organic finish prior to vacuum metallizing. This permits the attainment of a highly specular finish for only a fraction of the cost required for polishing. In those applications where the coated article must remain serviceable after appreciable handling, the evaporated aluminum film is protected by a synthetic finish topcoat. At present this finish is still inferior to electroplating in respect to abrasion resistance. Rapid improvements in vacuum coating, however, are likely to make these processes directly competitive.

A similar use of the technique of vacuum evaporation, is put to work in the manufacture of aluminized TV picture tubes. In this case, the picture tube is used as the vacuum chamber and aluminum is flashed from a heated tungsten filament projecting into the neck of the tube. The aluminum deposits on the inside surface of the tube and covers the phosphor screen. When in use, the aluminum film serves to reflect the light emitted from the phosphor screen which would otherwise be lost inside the tube. In this respect, the aluminum film fulfills the same function as the mirror in back of a kerosene lamp.

Although the majority of vacuum coating installations now in use are batch-type units, a few have been made for semicontinuous coating of paper and plastic films such as cellophane, cellulose acetate, and Mylar. Mylar exhibits unusual adhesion characteristics for the vacuum-deposited aluminum film, and is potentially the source of a host of decorative and functional products.

G. Vacuum Metallurgy

Since the end of the war, the use of vacuum melting was extended to include steels, copper, chromium-nickel alloys, titanium, etc. The actual melting process may or may not take place under high vacuum, but adequate time is allowed subsequently for desorption of dissolved gases under high vacuum.

Vacuum-melted steels have been proven to have longer fatigue life, and their freedom from inclusions has spurred their use in the manufacture of miniature ball bearings. Vacuum-melted copper has found applications in large power vacuum tubes, and vacuum-melted chromium-nickel alloys are mainly used for high temperature turbine blades. Because of their high melting point and high chemical reactivity, titanium, zirconium, and molybdenum are now melted under vacuum as a standard technique.

Heat treating, sintering, and brazing of these metals under vacuum has also become an accepted practice. The increasing use of titanium and related titanium alloys, is accompanied by an expansion of the use of these techniques.

H. Vacuum Drying

The use of freeze-drying for pharmaceuticals did not grow appreciably after the end of the war, because it was no longer used for penicillin, and today plasma is no longer being stockpiled. Other vacuum drying processes, however, have come to the fore. In the food industry, the concentration of orange juice under vacuum has revolutionized the marketing of citrus products. Annual sales of vacuum-concentrated citrus juices now exceed $100 million. Concentration of orange juice is carried out at a pressure of 5–25 mm. Hg, maintained by multistage steam ejectors. A four to one concentration is achieved.

Recently, vacuum drying has been extended to hermetically sealed refrigeration and air conditioning compressors. Since the motor windings in such compressors communicate with the passages of the refrigerant, it is of prime importance to reduce the moisture content of these units to an absolute minimum. Failure to reduce this moisture results in ice clogging of the expansion capillary tube. The development of the gas ballast mechanical vacuum pump is a large factor in making this process commercially feasible. In these pumps, fresh air is introduced toward the end of the compression cycle to dilute the water vapor and prevent it from condensing, by maintaining its partial pressure lower than the vapor pressure at the temperature of the pump. Thus by preventing condensation in the vacuum pump, it is possible to pump larger quantities of water vapor without deterioration of the pumping speed and ultimate pressure. In the absence of gas ballast, the condensing water vapor emulsifies with the lubricating oil, finds its way back to the vacuum side of the pump, and prevents the attainment of low ultimate pressure.

I. Future Development

In the future, vacuum metallurgy, perhaps, will be the fastest growing application of vacuum technology. The increasing use of refractory metals will be a major factor in this growth. Vacuum brazing for high temperature application will assume more significance. Compressor dehydration and continuous coating are also likely to expand. The increasing production of color television will see a second round of expansion in exhaust and aluminizing equipment for the electronics industry. More automatically operated equipment assemblies will be put into use. It is likely that further growth of vacuum applications in the chemical process industries will take place, but further advances of chemical engineering technology in this field will depend upon the commercial incentives which exist in the near future.

III. Chemical Engineering and High Vacuum Technology

In this section the laws that govern high vacuum processes are briefly outlined. Although in many instances development of these laws has not progressed to a point where application to practical situations can be achieved by simple and straightforward calculations, it is useful to recognize the limitations and potentialities revealed by these laws.

A. Mass Balance

Mass balance considerations of high vacuum processes might appear strange to the chemical engineer who is accustomed to thinking in terms of pounds or pound-moles. The units of mass commonly used in high vacuum literature are the micron-liter and the micron-cubic-foot, in the United States, and the torr-liter or the micron-cubic meter in Europe. A micron-liter is a unit of mass and not a unit of volume, as it represents the mass of one liter of the gas in question at a pressure of one micron (0.001 mm. Hg abs.), and room temperature (usually meant to be 26°C.). A torr is a pressure of 1 mm. Hg, thus a torr-liter is a thousand times greater than a micron-liter. The units of throughput take the form, micron-liter per second, micron-cubic-foot per minute, or micron-cubic-meter per hour.

The use of such units as measure of throughput permits a simple relationship between throughput, pressure, and the volumetric rate of displacement. Thus if the volumetric rate of displacement is measured in liters per second and the pressure in microns, the mass throughput, measured in micron-liters per second, is simply the product of the volumetric rate of displacement and the pressure. The volumetric rate of displacement is termed "speed," whence the common formula

$$Q = SP \tag{1}$$

where Q is throughput, S is speed, and P is pressure.

When studying the steady state gas flow through a pipe or a series of pipes and fittings, the mass balance principle implies a constant throughput, Q. If the pressure at the mouth of the vacuum pump is known and the speed of the pump at this pressure is given, the product of these two factors gives the constant throughput which must hold at each point upstream of the pump (in the absence of any outgassing from the walls of the flow lines). Since pressure must be higher upstream in order for the gas to flow, it follows that the "speed" will vary along the length of the flow lines inversely as the pressure. "Speed" corresponds to the product VA in the familiar equation

$$W = \rho VA \tag{2}$$

where W = rate of mass flow, ρ = density, V = velocity, and A = area.

The units of speed and throughput in use in the field of high vacuum, although offering a simple short-cut for relating these variables to pressure, lead to great confusion when attempting to write force balance and energy balance equations. The chemical engineer has solved the difficulties arising from simultaneous use of the mass-pound and the force-pound. He has an even greater task in reconciling mass units expressed in terms of pressure and volume to those written in terms of force and true mass.

B. FLUID FLOW

Comparatively little experimental work has been done on the flow of gases under vacuum. Although theoretical relationships can be derived for molecular flow (K1) and for viscous flow (P1), few data other than those reported by Cheng (C1) and Brown *et al.* (B2), throw any light on the behavior of gases in the region of transition from viscous to molecular flow.

Theoretical flow equations were derived for the molecular flow region by Knudsen (K1) as far back as 1909. These equations for molecular flow and Poiseuille's Law for laminar flow, were the basis for vacuum flow computation until the later years of World War II. Normand (N1) was the first to translate these equations into practical forms for engineering applications. In this reference Normand gives useful empirical rules for applying Knudsen's equations to ducts of rectangular cross-section, nonuniform cross-section, baffles, elbows, etc.

Cheng (C1) in collaboration with National Research Corporation was the first to carry out exhaustive experimental tests for correlating pressure drop under high vacuum with such parameters as pipe length, pipe diameter, mean gas pressure, and gas throughput. The range of pressures investigated covered the regions of laminar flow, molecular flow and the region of transition between laminar to molecular flow, labeled as the region of slip flow. Cheng's data were correlated by an equation essentially in the form of Poiseuille's Law, with a correction factor which assumes increasingly greater importance at lower pressures. This equation applies throughout the range of laminar flow, i.e., Reynolds Number < 2100, as well as in the molecular flow region where the mean free path is so large that intermolecular collisions in the gas phase are very infrequent. This equation is

$$Q = \frac{\pi g_c R^4}{16\mu l}(p_i^2 - p_e^2)\left[1 + 4\left(\frac{\pi}{2}\right)^{1/2}\left(\frac{2}{f} - 1\right)\frac{\mu}{p_m R(\rho_1 g_c)^{1/2}}\right] \qquad (3)$$

where Q is gas throughput, micron-cm.^{3}/sec.

g_c is a constant numerically equal to the acceleration of gravity and having for dimensions

$$\frac{\text{mass-length}}{\text{force-(time)}^2} = 1.33 \frac{\text{g. mass}}{\text{micron Hg} - \text{sec.}^2\text{-cm.}}$$

f is a molecular reflection factor whose theoretical significance lies in the fact that it is the fraction of all incident molecules which are "diffusely" reflected off a wall (the remaining molecules are reflected "specularly," i.e., with angle of incidence equal to angle of reflection)

R is pipe radius, cm.

l is pipe length, cm.

ρ_1 is $\dfrac{\text{mass density}}{\text{pressure}}, \dfrac{\text{g.}}{\text{cm.}^3 - \text{micron}}$

p_i is inlet pressure, microns

p_e is exit pressure, microns

p_m is mean pressure, microns

μ is viscosity at atmospheric pressure, poises

Equation (3) reverts to Poiseuille's Law at sufficiently high pressures, where the second term in the large parenthetical factor becomes negligible compared to unity. As the pressure is decreased and intermolecular collisions become less frequent, a flow velocity profile is established where the forward velocity component near the wall becomes a finite value which increases with lower pressures. The reason for this condition is that at these pressures many molecules can stream from the bulk of flow, where the forward velocity is relatively high, to the wall, without suffering collisions with molecules having low forward velocities. Gas flow under such conditions is termed slip flow. Pressures corresponding to this type of flow are such that the second term of the "correction factor" in equation (3) is finite compared to unity. At lower pressures, where the mean free path is considerably larger than the radius of the pipe, the second term in the "correction factor" becomes dominant resulting in the equation for molecular flow

$$Q = \left(\frac{\pi}{2}\right)^{\frac{1}{2}} \left(\frac{2}{f} - 1\right) \frac{R^3 g_c}{l(\rho_1 g_c)^{\frac{1}{2}}} (p_i - p_e) \tag{4}$$

Thus we have one universal equation which applies throughout the entire range of high vacuum. In fitting these equations to experimental data, it was discovered that a molecular reflection factor f, equal to 0.84, best represents the data for glass and copper pipe. A factor f equal to 0.9, gives better agreement with data obtained on iron pipe. The above equations do not apply as such to short pipes or orifices, but are sufficiently accurate for pipes having a length to diameter ratio greater than 10.

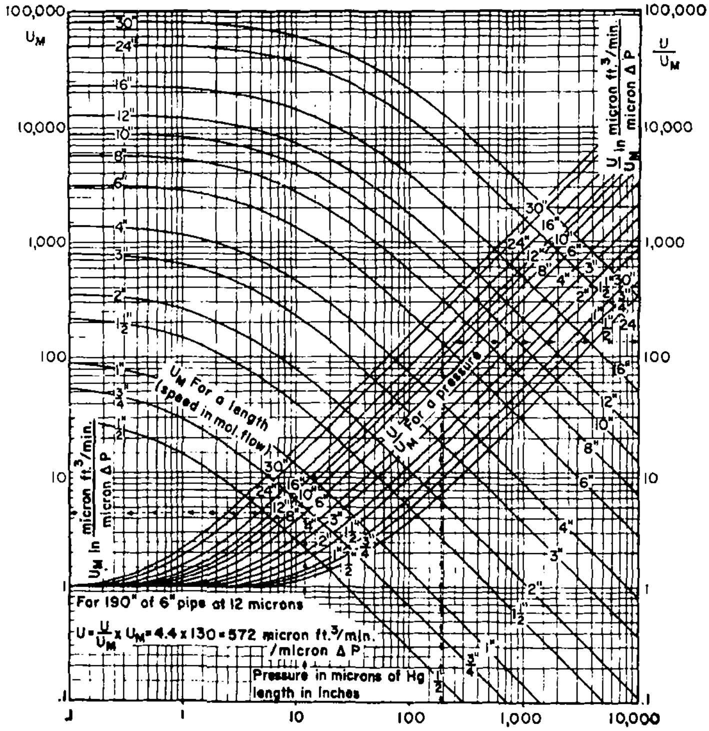

FIG. 1. Brown-DiNardo, and Sherwood curves for pressure drops through pipe.

A set of curves based on the above equations is given by Brown *et al.* (B2), which is useful for calculating throughput pipe size, pipe length, and inlet and exit pressures are given. These curves are given in Fig. 1. The molecular conductance U_m is read from the series of curves " U_m for length." The ratio of actual conductance to molecular conductance, U/U_m, is read off the curves "U/U_m for a pressure." The pressure used in this reading should be p_m, the arithmetical mean of the inlet and exit pressures. U can now be calculated from the equation

$$U = \left(\frac{U}{U_m}\right) \cdot U_m \tag{5}$$

and Q is determined from

$$Q = (p_i - p_e)U \tag{6}$$

To solve the common problem where determination of the inlet pressure and the pressure drop is required, given throughput, pipe size, and exit pressure, tedious trial and error solution is necessary when using these curves. The importance of solving such a problem can be appreciated when one considers the condition where a mechanical or diffusion pump of a known size, and hence known throughput, is connected at the end of a pipe of a known size and length. The exit pressure of the pipe is the inlet pressure of the pump, and hence defines a throughput value. In this instance we know the throughput, the exit pressure, and the pipe diameter and we are usually interested in pressure drop across the pipe and the inlet pressure. It is important to have a direct solution for this sort of problem, particularly where more than one pipe size is interposed between a given pump and a vacuum chamber. Lawrance (L2) has rewritten Eq. (3) so that a direct solution can be obtained in each of the following cases:

(1) Given exit pressure, throughput, pipe diameter. Required: pressure drop and inlet pressure

(2) Given exit pressure, inlet pressure, and throughput. Required: pipe diameter

(3) Given exit pressure, inlet pressure, and pipe diameter. Required: throughput.

For the problem where inlet pressure, throughput, and pipe diameter are given, the Lawrance equation can lead to a negative or imaginary value for the exit pressure, if the known variables are not physically compatible. The Lawrance equation is

$$p_i - p_e = \sqrt{p_{ea}^2 + W} - p_{ea} \tag{7}$$

where

$$p_{ea} = p_e + A\left(\frac{2-f}{f}\right)\frac{l}{D} \tag{8}$$

and

$$W = \frac{BK_L lQ}{D^4} \tag{9}$$

Table I gives values for the constants A and B for air and water vapor for a number of commonly used units.

For length-to-diameter ratios greater than 10, K_L can be taken equal to 1. For short pipes of length-to-diameter ratio less than 10, the proper value for K_L can be obtained from Fig. 7 of Lawrance's paper (L2). D in the above equations is the pipe diameter, K_L is a constant. All other symbols have the definitions given in conjunction with Eq. (3).

TABLE I

Units			Air at 26°C.			Water vapor at 26°C.		
p	Q	l and D	A	B	C	A	B	C
micron Hg	micron-liter/sec.	cm.	40.9	11.4	0.033	26.8	5.82	0.0257
micron Hg	micron-liter/sec.	in.	16.1	0.694	0.0051	10.54	0.354	0.00397
micron Hg	micron-ft.^{3}/min.	in.	16.1	0.327	0.0024	10.54	0.167	0.00187

For short pipes, in addition to pressure drop thus calculated, allowance must be made for inlet losses given by the semi-empirical equation

$$\delta p_i = \frac{CQ}{D^2} \tag{10}$$

where C is a constant given by Table I for air and water vapor.

It should be pointed out that the Lawrance equation and the Brown-DiNardo curves are true only at room temperature. Data such as those obtained by Cheng should be extended to gases other than air and water vapor, to non-circular conduits, and to temperatures encountered in vacuum metallurgy, and in freeze drying. Whether or not such work might be undertaken will, of course, depend on its commercial and technological significance.

C. HEAT TRANSFER

The effect of high vacuum on heat transfer is best understood in the light of low molecular density and a low frequency of intermolecular collisions.

Transfer of heat by radiation is not influenced by pressure except in rare cases where absorption of heat in the gas is important. Under reduced pressure, the effect of such absorption would be reduced or become negligible, thus simplifying radiant heat calculations. The relative importance of heat transfer by radiation is increased under vacuum.

Transfer of heat by convection assumes a lower degree of importance in vacua. Since convection is defined as the transfer of heat by the movement of a large number of molecules en masse, this type of heat transfer is non-existent under conditions of molecular flow, where collision between individual gas molecules is a rare occurrence, and where for the large part, gas molecules travel about unaffected by the presence of other molecules.

In the region of slip flow, heat transfer by convection is significant only at a distance from the heat source greater than the mean free path. Within a distance of one mean free path, just as in molecular flow, heat transfer occurs by molecular conduction alone. Kyte *et al.*, describe a method of calculation for heat transfer in slip flow (K2). The technique employed is essentially a trial and error solution whereby a temperature, t_a, is determined at a distance of one mean free path away from the heat source. The rate of heat flow from the hot surface is calculated on the basis of molecular conduction over the distance of one mean free path. The rate of heat flow beyond the distance of one mean free path is calculated on the basis of convection. The correct value of the temperature t_a, is that which results in equal rates of heat flow by conduction and by convection.

Under conditions of laminar flow, the usual natural convection equations can be used. Reference (K2) gives a table of heat transfer equations for spheres and cylinders recommended for use when molecular conduction is a factor, and a second table applicable to natural convection under laminar flow conditions.

The equations for spheres, applicable when the product of the Grashof number and the Prandtl number lies between $10^{1.5}$ and 10^9 are as follows:

(1) Effect of free-molecular conduction negligible:

$$q = 2\pi D k_{w,g} \frac{t_w - t_g}{1 - \dfrac{1}{1 + 5.01/(GrPr)^{0.26}}} \tag{11}$$

(2) Effect of free-molecular conduction important: (a) for heat transfer by convection,

$$q = 2\pi D' k_{a,g} \frac{t_a - t_g}{1 - \dfrac{1}{1 + 5.01/(GrPr)^{0.26}}} \tag{12}$$

(b) for heat transfer by molecular conduction:

$$q = \pi D^2 \Lambda_0 P \alpha \sqrt{\frac{273.2}{T_a}} \cdot (t_w - t_a) \tag{13}$$

For a correct solution, a temperature t_a must be chosen so as to give equal values for q when calculated by Eqs. (12) and (13).

It will be noted that Eqs. (11) and (12) are identical except that

Eq. (12) is written for the equivalent diameter $D' = D + 2\lambda_a$ at which point the temperature is t_a, rather than the actual diameter D.

q = heat flow, heat units per unit time, Btu/hr.
D = diameter, ft.
$k_{a,g}$ = conductivity of the gas at a temperature $(t_a + t_g)/2$, Btu/(hr.)(ft.)(°F.)
$k_{w,g}$ = conductivity of the gas at a temperature $(t_w + t_g)/2$
t_a = temperature at one mean free path from the wall, °F.
t_g = temperature of the gas at a large distance from the wall, °F.
t_w = temperature at the wall, °F.
Gr = Grashof number $(D^3\rho^2 g_c/\mu^2)(\beta\,\Delta t)$
Pr = Prandtl number $(C\mu/k)$
D' = equivalent diameter at one mean free path from the wall, ft.
T_a = absolute temperature, °K.
Λ_0 = free-molecular conductivity of the gas at 0°C., Btu/(hr.)(ft.^{2})(°F.) (micron Hg)
P = pressure of the gas, microns
α = accommodation coefficient, equal to 0.90 for air, 0.50 for helium, and 0.95 for argon
λ_a = mean free path, ft.

Although very useful, the above solution to heat transfer under slip flow and molecular flow conditions, is rather tedious leaving a definite incentive for simplification. Extension of this treatment to other geometrical shapes and elimination of the trial-and-error solution might be the next objective.

D. MASS TRANSFER

With reduction of pressure the mean free path grows larger, and with it the resistance to mass transfer due to intermolecular collision is progressively diminished. At pressures corresponding to mean free paths larger than the dimensions of the vessel in question, the gas phase resistance to mass transfer is negligible and the only limiting factor on the rate of material movement is the rate of emission from the interface.

Examples of mass transfer under high vacuum are distillation of thermally unstable organic compounds, high-vacuum freeze drying, vacuum concentration of fruit juices, vacuum drying of coffee concentrate, vacuum purification of molten metals, etc. The mechanism of mass transfer in the concentration of fruit juices can be adequately described by the Gilliland-Sherwood equations (G1), since the mean free path is negligible relative to the dimensions of the vessel at the pressures used. These equations show that the mass transfer coefficient is inversely proportional to the pressure. By extrapolation to very low pressures, it should be possible to reach an ever-increasing mass transfer rate. Of course, these equations do not take into account the surface limitation to mass transfer, which at low pressures can be the controlling factor. Under molecular flow conditions the emission from the surface is controlling, and the rate of mass transfer can be fully described by the Knudsen (K1) or Langmuir (L1) equation for surface emission derived from the kinetic theory. Here the rate is independent of the ambient pressure and is proportional to the vapor pressure of the material in question.

$$\frac{dW}{d\theta} = k_1 A \tag{14}$$

$$k_1 = P_v \sqrt{\frac{Mg_c}{2RT}} \tag{15}$$

where $dW/d\theta$ = rate of mass transfer, g./sec.
k_1 = mass transfer coefficient, g./(sec.)(cm.^{2})
A = area of surface in question, cm.^{2}
P_v = vapor pressure of solid or liquid in question, microns Hg
M = molecular weight, g./g. mole, of emitted substance
g_c = constant, numerically equal to the acceleration of gravity whose dimensions are

$$\frac{\text{mass-length}}{\text{force-(time)}^2} = 1.33 \frac{\text{g. mass}}{\text{(micron Hg)} - (\text{sec.}^2)(\text{cm.})}$$

R = gas constant $= 62.37 + 10^6 \dfrac{\text{(microns Hg)} - (\text{cm.}^3)}{\text{(g. mole)} - (^\circ\text{K.})}$

T = absolute temperature, °K.

The above equations give the maximum rate of evaporation. Although validated by many observations, these equations at times give rates of evaporation as much as 100 times higher than the measured values. This deviation from the Langmuir equation is not limited to a few singular solids or liquids. The same liquid which is noted to obey the Langmuir equation in one test, can be found to deviate widely from it in another. Hickman (H1) has proved that the Langmuir equation will hold true for all liquids he tested, including water, if the interfacial surface is constantly renewed, thereby removing surface impurities which retard the traffic across the interface. A surface once highly emissive becomes "torpid" or inactive upon standing in a glass flask. Upon removal of the impurities collected at the surface by overflowing the liquid into a second flask, the surface loses its torpidity and becomes active again.

The Langmuir equation can be written

$$\frac{dW}{d\theta} = \alpha k_1 A \tag{16}$$

where α is the emission coefficient (also termed the accommodation coefficient). The emission coefficient is an illusive variable lying between zero and unity depending upon the purity of the exposed surface.

Mass transfer data in the region of transition between molecular flow and laminar flow have been recently gathered by Cooke (C2). In a study of sublimation of naphthalene spheres, under vacuum, he discovered that the data collected can be correlated by a concept of "over-all" mass-transfer coefficient equal to the reciprocal of the sum of the resistance in the gas phase and a resistance corresponding to the Langmuir equation for the interface. An emission or accommodation coefficient of 0.10 was found to fit the data.

To illustrate this work, the mass transfer equation for sublimation from spheres at pressures near atmospheric, according to Ranz and Marshall (R1), is

$$\frac{dW}{d\theta} = \frac{2\,D_0 M\,A\,P_v}{R\,T\,P d_s}\,[1 + 0.30(Re_s)^{1/2}(Sc)^{1/3}] \tag{17}$$

This can be written as

$$\frac{dW}{d\theta} = \frac{k_2 A}{P} \tag{18}$$

and the principle of additive resistances leads to an equation applicable over the entire range of pressure

$$\frac{dW}{d\theta} = \frac{\alpha k_1 A}{1 + \dfrac{\alpha k_1 P}{k_2}} \tag{19}$$

where

$$k_2 = \frac{2\,D_0 M\,P_v}{R\,T\,d_s}\,[1 + 0.30(Re_s)^{1/2}(Sc)^{1/3}] \tag{20}$$

where D_0 = diffusivity of vapor at 0°C. and 1 atmosphere × atmospheric pressure, $\dfrac{(\text{microns Hg})(\text{cm.}^2)}{\text{sec.}}$

M = molecular weight, g./g. mole

A = area, cm.^{2}

P_v = vapor pressure, microns Hg

R = gas constant = 62.37 × 10^6 (microns Hg)(cm.^{3})/(g. mole)(°K.)

T = absolute temperature, °K.

d_s = diameter of sphere, cm.

P = system total pressure, microns Hg

Re_s = Reynolds number, $\dfrac{d_s V \rho}{\mu}$

Sc = Schmidt's number $\frac{\mu}{\rho D}$

k_1 = maximum coefficient of mass transfer for molecular flow, g./sec.-cm.^{2}

k_2 = coefficient of mass transfer, micron Hg-g/cm.^{2}-sec.

α = emission (or accommodation) coefficient

ρ = density, g./cm.^{3}

V = velocity, cm./sec.

μ = viscosity, g./sec.-cm.

D = diffusivity, cm.^{2}/sec.

Inspection of Eq. (19) reveals that at low pressure the second term in the denominator vanishes, and Eq. (19) reduces to Eq. (16). At high pressure the second term in the denominator dominates, and the equation is reduced to Eq. (18).

The biggest obstacle to progress made in establishing mass transfer rates under vacuum will continue to be the variable emission coefficient. Whether or not the accommodation coefficient can become predictable will not be known before a wealth of data is accumulated, and such data will not be accumulated in the near future until there arise commercial incentives sufficient to justify the expense.

E. Reaction Equilibrium

By the same mechanism that high pressure can be used to drive a certain reaction in the direction of smaller molar volume, as for example, in the synthesis of ammonia from hydrogen and nitrogen, high vacuum is employed to shift reaction equilibria in the direction of greater molar volume. Examples of such reactions beneficially carried out under vacuum are the reduction of copper or nickel oxide impurities in copper or nickel melts. Reduction of iron, titanium or aluminum oxides by dissociation under vacuum is impractical because of the extremely low oxygen partial pressures required. For instance, the equilibrium partial pressure of oxygen over titanium oxide at its melting point (1750°C.) is of the order of 10^{-13} mm. Hg, and for aluminum at its melting point, 10^{-50} mm. Hg. The equilibrium partial pressure of carbon monoxide over carbon and oxide mixtures is considerably higher, however. Reduction of metallic oxide impurities in the presence of carbon is, therefore, a common vacuum melting practice for metals like steel where a relatively small concentration of carbon is permissible. The solubility of carbon in molten titanium is very high, so that vacuum melting of titanium in the presence of carbon is generally avoided.

The reaction of titanium with carbon is utilized, however, in the vacuum fusion technique for micro determination of oxygen content in titanium. The conversion of titanium into titanium carbide in the presence of an excess of graphite is permissible so long as all oxygen in the melt can be collected as carbon monoxide.

Vacuum reduction of oxides of the alkali and alkaline earth metals, has proved a very useful technique, of which the Pidgeon ferrosilicon process for reduction of magnesium (B1) has been an outstanding example. Although this process is not currently competitive with electrolytic reduction, the cost differential is not so great that improved vacuum equipment and vacuum techniques could not swing the balance in favor of this process.

TABLE II

VACUUM THERMAL REDUCTIONS WHICH HAVE BEEN INVESTIGATED RECENTLY

Metal	Reaction
Magnesium	$2\,(MgO \cdot CaO) + \frac{1}{6}\,FeSi_6 \xrightarrow[\text{(0.005 to 0.5 mm. Hg)}]{\text{(1100 to 1175°)}} 2\,Mg\uparrow + \frac{1}{6}\,Fe + 2\,CaO \cdot SiO_2$
	$3\,(MgO \cdot CaO) + 2\,Al \xrightarrow[\text{(0.005 to 0.5 mm. Hg)}]{\text{(1050 to 1175°)}} 3\,Mg\uparrow + 3\,CaO \cdot Al_2O_3$
Calcium	$6\,CaO + 2\,Al \xrightarrow[\text{(0.005 to 0.1 mm. Hg)}]{\text{(1100 to 1175°)}} 3\,Ca\uparrow + 3\,CaO \cdot Al_2O_3$
Lithium	$6\,LiOH + 4\,Al + 6\,CaO \xrightarrow[\text{(0.005 to 0.1 mm. Hg)}]{\text{(1100 to 1175°)}} 6\,Li\uparrow + 2(3\,CaO \cdot Al_2O_3) + 3H_2$
	Spodumene $[LiAl(SiO_3)_2] + Al \xrightarrow[\text{(0.005 to 0.1 mm. Hg)}]{\text{(1050 to 1150°)}} Li\uparrow +$ complex silicate
Barium	$4\,BaO + 2\,Al \xrightarrow[\text{(0.1 mm. Hg)}]{\text{(1050 to 1200°)}} 3\,Ba\uparrow + BaO \cdot Al_2O_3$
Columbium	$Cb_2O_5 + 5\,CbC \rightarrow 7\,Cb + 5\,CO$

In this process, the free metal, which is more volatile than the other reaction components, is distilled under vacuum and collected in a suitable condenser. A pressure of less than 0.100 mm. Hg is necessary to prevent excessive reoxidation of the metal vapor. The use of vacuum has the advantage of lowering the temperature level of the reaction by several hundred degrees below that needed at atmospheric pressure. Several improvements are foreseen which might enhance the economic feasibility of the Pidgeon vacuum reduction process, e.g., it might be changed from a batch to a semi-continuous process. This might be achieved by introduction of the feed and removal of waste products through air locks. The vapors might be condensed on the walls of a rotary condenser similar in construction to the ice condenser developed for freeze-drying installations. The scraper blades might be rotated slowly enough to permit a substantial build-up of metal and flaking off due to shrinkage away from the wall. The metal flakes might be withdrawn from the condenser through an air lock. Since the operating pressure is about 0.1 mm. Hg, low maintenance mechanical booster pumps might be used. These pumps are much cheaper than ordinary mechanical pumps of the same capacity at this pressure, and are entirely free from the problems associated with the operating fluid of steam jets, oil ejectors, and diffusion pumps. Further description of these pumps is given in Section IV.

Examples of reduction reactions which can be carried out by the above approach are given in Table II (S1).

IV. Vacuum Pumps and Gages

For proper selection of a suitable pumping system, throughput requirements at various pressures should be known. A wide variety of vacuum pumps are available commercially, suitable for widely different ranges of operation. The pump with the lowest ultimate pressure is not universally desirable. A pump which can reduce the pressure in a particle accelerator down to 10^{-7} mm. is usually inadequate for maintaining a vacuum melting furnace at a pressure of 10^{-1} mm. An oil ejector pump capable of handling a high throughput between 1 and 10^{-2} mm. generally will not yield a pressure lower than 10^{-3} mm. Throughput *vs.* pressure requirements can be established on the basis of: (1) pumpdown time to a certain pressure; (2) extrapolation from small-scale operation; and (3) stoichiometric calculation of gas volumes to be handled in a given length of time.

The pumpdown time from atmospheric pressure to 10^{-1} mm. can be used to set the pumping capacity required in that range, if only bulk evacuation is significant. The following equation can be used to determine the pumping speed required, assuming a constant speed between atmospheric pressure and 10^{-1} mm.

$$S = \frac{V}{\theta} \ln \frac{p_1}{p_2} \tag{21}$$

where, S is the speed of the pump at the chamber to be evacuated (volume/unit time), θ is the time, V is volume, and p is pressure.

Extrapolation from a smaller scale unit is, in many applications, the most satisfactory approach. Scaling upwards may be done in proportion to the weight of the charge, the surface area exposed to vacuum, or the volume of the pumping chamber, depending on whichever of these variables is the controlling factor.

Stoichiometric calculations may be used as a basis for establishing pumping requirements for desorption of gases from metals, or in vacuum freeze-drying, under conditions where the rate of removal of gas is the controlling variable.

A well recommended procedure for determining the pumping requirements is to plot throughput *vs.* pressure over the entire pressure range, preferably on log-log paper, and in units such as micron-cubic feet/min. or micron-liters/sec. The advantage of these units is that a quick mental conversion from throughput to speed is then possible. The throughput curves of selected pumps can then be drawn up on the same plot for comparison with the required curve. Allowance for pressure drop in the lines should be made by using the Lawrance equations or the Brown-DiNardo curves, Section III, A.

A. VACUUM PUMPS

A great variety of pumps are available for producing and maintaining high vacuum. They differ widely in their principle of operation, and the range of pressures for which they are best suited. A brief description of the pumps most commonly used is given below.

1. *The Mechanical Vacuum Pump*

The mechanical vacuum pump consists of an eccentrically mounted rotor driven inside a cylindrical housing. Two types of mechanical pumps are constructed: (a) the rotary piston type, where the rotor comes in close contact with the housing and thus makes the seal between intake and exhaust compartments, and (b) the vane type pump where two vanes, spring-mounted in the rotor, make contact with the walls of the housing and thereby divide the space between the rotor and the housing. Typical construction is shown in Figs. 2 and 3.

The rotors in these pumps are driven at speeds in the neighborhood of 400–500 RPM. Rotational speeds higher than this range result in setting up excessive vibrational stresses. The volumetric displacement or speed of these pumps is, therefore, somewhat limited, though they are capable of handling large gas throughputs in the range approaching atmospheric pressure. Single-stage mechanical pumps are capable of reaching pressures of the order of 0.01 mm. Hg, and two-stage pumps can go down to 0.001 mm. Hg. They can discharge directly to atmosphere and hence can be used as forepumps to back diffusion pumps or oil ejectors. They exhibit a fairly constant volumetric speed from a suction pressure near one atmosphere to about 0.5 mm. Below this pressure their pumping speed decreases, diminishing to zero in the range 0.01 to 0.001 mm. Condensation of water vapor in these pumps materially increases the ultimate pressure that they can produce.

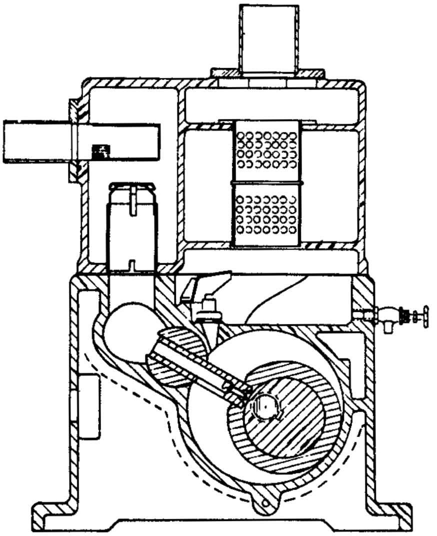

FIG. 2. Rotary—piston mechanical vacuum pump.

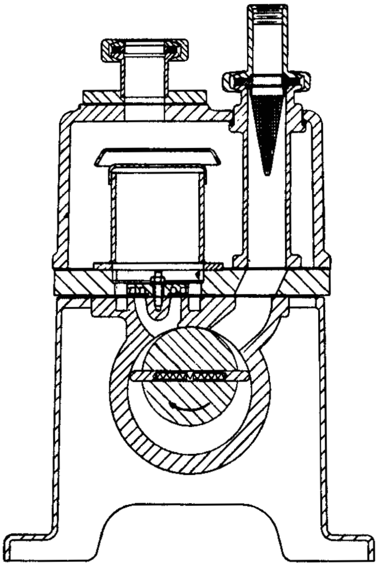

FIG. 3. Vane—type mechanical vacuum pump.

In recent years, the application of gas ballast to these pumps has reduced oil contamination when pumping water vapor.

2. *The Mechanical Booster*

This type of pump generally has two "figure-eight" impellers meshed together as in a gear pump. The impellers are symmetrical relative to their axes of rotation, therefore permitting high rotational speeds, and hence high volumetric displacement. This type of pump has been used both as blower and vacuum pump for several decades; however, its use for producing pressures below 10 mm. Hg dates back only two to three years and is the result of development work aimed at minimizing leakage through the shaft seals. One model of this pump (Kinney KMB-1200) operates at a rotational speed of 1800 RPM and is capable of handling 1000 cu. ft./min. in the range between 10 and 0.001 mm. Hg. These pumps do not discharge directly into the atmosphere; they are backed by forepumps of suitable size. No lubricating or sealing fluids are used, hence there is little chance of contaminating the vacuum system with vapors originating from them. The mechanical booster is not designed for operation with a suction pressure higher than 10–30 mm. Hg. Protective means, such as a pressure switch, are used to open a by-pass line to the forepump, or slow down the rotation of lobe pump as this suction pressure is exceeded. A wide usage is foreseen for these pumps, particularly in vacuum metallurgy, since they require little maintenance and retain their maximum pumping speed over a wide pressure range.

3. *Multi-Stage Steam Ejectors*

When more than three stages of steam jets are used in series, pressures as low as 0.050 or 0.025 mm. Hg can be obtained. A steam ejector system requires no backing pump, the final stage being capable of compression to atmospheric pressure. For many applications, steam ejectors offer great advantages over mechanical vacuum pumps, particularly in processes such as orange juice concentration where large quantities of water vapor are evolved. Steam jets require relatively small initial investment, but consume large quantities of high pressure steam. In general, steam ejectors are not economical below a pressure of 1 mm. Hg. For many applications, steam jets must be avoided as "flash-back" of steam into the vacuum system may cause contamination. If wet steam were to flash back into a titanium melting furnace, the result might be a violent explosion. Steam ejectors are used commonly in installations requiring a continuous demand for pumping large quantities of water vapor between 1 to 50 mm.

4. *The Oil Ejector*

Pumps of this type are exemplified by Consolidated Electrodynamics Corporation's KB-300 and KJ-5000. They consist of a diverging vapor nozzle and a diffusor section similar in construction to a steam ejector. The difference lies in using an organic vapor as the pumping fluid, generated at a boiler pressure of the order of 30–40 mm. Hg. The vapor is condensed and returned to the boiler for re-evaporation. This type of pump can produce pressures as low as 0.001 mm., but has a maximum speed between 0.1 and 1 mm. Hg. Most of the oil ejectors in use are single-stage units. A two-stage oil ejector was developed and is marketed under the commercial designation KJ-5000. Single–stage oil ejectors have highly peaked speed curves, because the diverging nozzle achieves maximum efficiency at a fixed ratio of upstream to downstream pressures. Oil ejectors are more economical than steam jets in their operating pressure range which is between 3 mm. and 0.010 mm. They must be backed by a forepump which can handle their maximum throughput while maintaining a forepressure less than 3 mm. Hg. Accidental exposure of these pumps to atmospheric pressure for more than a few minutes will result in oxidation and thermal breakdown of the oil, and may lead to contamination of the vacuum system. This situation is aggravated if the boiler heaters continue to operate under atmospheric pressure and the temperature of the oil rises above the normal operating level. A typical use of these pumps is for vacuum melting of steel, where large quantities of gas are evolved in the range between 0.30 to 0.05 mm. Hg.

5. *The Diffusion Pump*

A cross-section of a typical diffusion pump is shown in Fig. 4. It consists of an electrically heated boiler where a high boiling organic liquid is vaporized into a vertical stack which feeds two or more annular nozzles. The vapor travels through the nozzles at sonic or higher velocities and discharges into the space between the stack and the wall. The walls are water- or air-cooled and hence act to condense the vapor and return the liquid to the boiler. Pumping takes place as gas molecules, wandering into the vapor jets by their random thermal motion, collide with organic vapor molecules and are given a velocity component in the direction of the vapor stream. Because the pressure in the gas is very low, and hence the mean free path very high, the pumping action is somewhat different from that of an ejector. The difference is that the pressure in the vapor stream can be several orders of magnitude higher than pressure in the gas just above or below the jet. Gas molecules wandering into the vapor stream are trapped through the action of intermolecular collisions inside the jet. As the vapor condenses on the wall, the entrapped gas is diverted downstream of the vapor jet where it can be pumped by another vapor jet.

The pumping action of a given jet is maintained so long as the forepressure, i.e., the downstream pressure, is lower than that required to break through the curtain of vapor. This maximum forepressure is greater the smaller the space between the jet nozzle and the wall of the casing, and the more nearly the direction of vapor flow approaches the axis of the pump. On the other hand, the maximum rate of gas entrainment, since it depends on the rate of collision of gas molecules with the vapor stream, is proportional to the admittance area of the jet and the pressure of the gas. It follows, therefore, that for a given boiler pressure, a high-speed jet is one of low forepressure tolerance, and a high forepressure jet is one of low pumping speed. The use of several jets of progressively lower speeds, but increasingly higher maximum forepressure, yields a pump with the high speed of the first stage and the high forepressure of the final stage.

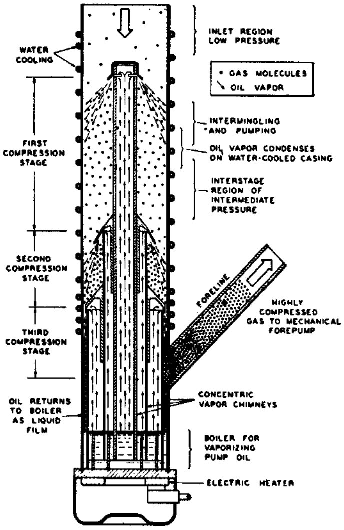

FIG. 4. Cross section of a typical diffusion pump.

Although thermodynamically the diffusion pump is highly inefficient, it can achieve a speed as high as 40% to 45% of the ideal pump, which pumps out all molecules streaming to the mouth of the pump by random kinetic motion. The difficulties with breakdown of the pumping fluid noted in connection with the vapor ejector, also apply to the diffusion pump. Silicone oils which are less susceptible to chemical breakdown are now gaining a wider use as diffusion pumping fluids.

There are two types of diffusion pumps: (a) the so called diffusion pump, which operates at a boiler pressure of about 0.5 to 1 mm. Hg and is capable of reaching pressures in the 10^{-6} mm. range; and (b) the booster diffusion pump, which has a boiler pressure of about 3–6 mm. Hg, and has an ultimate pressure of approximately 10^{-4} mm. The difference in performance characteristics of these two types of diffusion pumps is illustrated in Fig. 5.

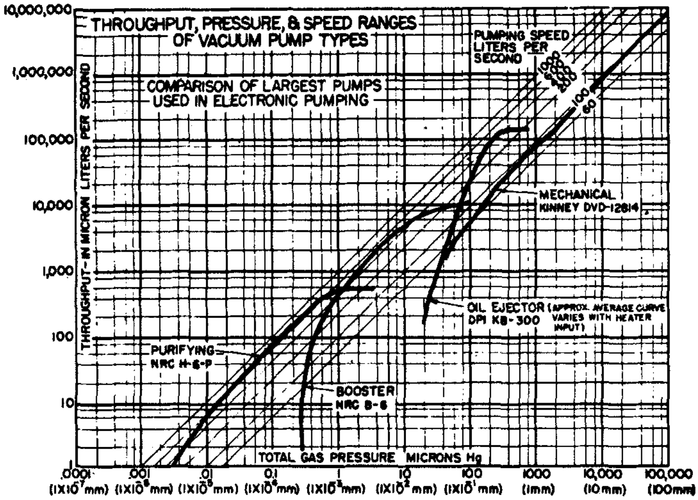

FIG. 5. Throughput *vs.* pressure characteristics of vacuum pump types.

In most applications more than one type of vacuum pump is used. For example, a mechanical vacuum pump might be used in conjunction with an oil ejector and a diffusion pump. In such instances it is necessary to adapt the valving arrangement so that each pump can be opened directly to the vacuum system. By this method it is possible to evacuate the system down to about 2 mm. with the mechanical pump alone, during which time the ejector and diffusion pumps are under no load and are kept under vacuum by a small "holding" mechanical pump. In the range 2 mm. to about 0.050 mm. the ejector pump is opened to the vacuum chamber and the mechanical pump is used for backing the ejector in series. Below 0.050 mm. the diffusion pump might be opened to the system and the ejector and mechanical pumps used to back the diffusion pump in series.

B. VACUUM GAGES

Gages which depend on mechanical force for response, such as liquid manometers, Bourdon tubes, and aneroid bellows, are very useful in vacuum measurements, but their range is generally limited to pressures above 1 mm. For lower pressures, several types of gages are available of which the following are the most commonly used.

1. *The McLeod Gage*

This is the only absolute gage available for pressure measurement below 1 mm. Other gages must be calibrated against an absolute standard, at least at one point before their readings can be meaningful.

Its principle of operation is based on Boyle's Law, in that a fixed large volume of the gas at the unknown pressure is compressed to a fixed small volume. The pressure of the gas following compression is measured by a mercury manometer, and the initial pressure is calculated from this pressure and the volume ratio. Pressures as low as 10^{-5} mm. Hg can be accurately measured by this gage. A major cause for error is the presence of condensable vapors in the gas being measured. Under such conditions the pressure obtained with this gage is closer to the partial pressure of noncondensables than the total pressure. It is not exactly equal to the partial pressure of noncondensables, however, because the condensable vapors may exert a significant vapor pressure after condensation.

2. *The Alphatron Gage*

In this gage a radium plaque is the source of alpha particle radiation which ionizes the gas to be measured in proportion to the gas density. The positively charged, ionized gas is collected on a cathode plate. The resulting electric current is directly proportional to the collection rate of ionized molecules and hence to the gas molecular density, and to the pressure. By proper amplification of this current and calibration at one or more points, a microammeter needle can be made to read pressure directly. The latest Alphatron model is capable of measuring pressures accurately from 1000 to 10^{-4} mm. in six linear scales on a single instrument. The pressure obtained is a total pressure reading. However, the reading depends on the composition of the gas, as different gases vary in susceptibility to ionization.

3. *The Hot-Filament Ionization Gage*

The basis of operation of this gage is similar to the Alphatron, except that an incandescent tungsten filament is the source of the ionizing agent. Electrons emitted from the filament, oscillate between a positively charged grid and an ion-collector plate held at a negative potential. The number of ionized molecules is proportional to the molecular density. The same dependence on composition applies for this gage as for the

Alphatron. The hot filament ionization gage is the most accurate gage for pressure measurements between 0.005 and 10^{-7} mm. A modification of this gage, attributed to Bayard and Alpert, has recently enabled accurate pressure readings down to 10^{-13} mm. Hg. Exposure of the gage to atmospheric pressure results in burnout of the tungsten filament. A protective relay circuit is, therefore, employed to turn off the filament current as a pressure rise takes place. An iridium-coated tungsten filament was recently introduced in an attempt to make the gage burnout-proof.

4. *The Philips Gage*

The Philips gage is a cold-cathode ionization gage depending on high voltage rather than high temperature as the source for high velocity electrons. It consists of an anode in the shape of an open-ended pill box and two cathode disks placed a short distance from either end of the anode. A differential of about 2000 volts is maintained between the electrodes, and a strong magnetic field is applied to cause the electrons emitted at the cathodes to move in directly to the anode. They take long spiral paths before reaching the anode, thereby increasing the chances of hitting and ionizing the molecules of the gas whose pressure is to be measured. The main advantage of the Philips gage is that it is burnout-proof and hence permits accidental exposure to atmosphere, but its main drawback is nonlinearity. Furthermore, the action of high voltage discharge in the presence of certain vapors, results in contamination of the electrodes and therefore in erroneous pressure readings.

5. *The Thermocouple Gage*

This gage makes use of the dependence of thermal conductivity on pressure. It is very similar to the hot-wire anemometer, except that the gage is calibrated against gas pressure rather than gas velocity. The thermal conductivity of gases becomes pressure-dependent, when the mean free path is significant in comparison to the dimensions of the apparatus. This gage is insensitive to pressure variations above 1 mm. but is useful in the range between 0.05 and 0.005 mm. Below 0.005 mm., heat loss by conduction through the gas is negligible compared to heat losses through the leads of the hot wire and by radiation. This is a very inexpensive gage and is very widely used for monitoring pressure variation where high accuracy is not of great importance.

Among other gages in use for high vacuum measurements are the Knudsen gage and the Pirani gage. A description of these gages and further detail on the gages discussed above, are given in reference (L3).

V. Conclusion

To say that high vacuum is a useful chemical engineering tool of long standing, is to overlook the absence of a chemical engineering approach in a majority of vacuum applications. High vacuum technology is now making the transition from research laboratories to properly engineered installations. It is the task of the chemical engineer to review the accumulated technology in the light of chemical engineering tools; namely, mass balance, energy balance, rate and equilibrium considerations, and to rewrite this wealth of information in a language familiar and useful to chemical engineering practice.

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