VACUUM TECHNOLOGY

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Summary

Vacuum is required in nearly all scientific and engineering laboratories. The subjects which have been compiled in this chapter include: gas-kinetic processes, physical principles and operation modes of vacuum pumps and vacuum gauges, calculation of pipelines, basics of gas composition measurment, leaks and leak detection techniques.

1. Introduction

A vacuum is a space from which air or other gas has been removed. The amount we need to remove depends on the application, and we do this for many reasons. At atmospheric pressure surfaces are constantly bombarded by molecules. These molecules can either bounce from the surface, or attach themselves to the surface, or perhaps chemically react with the surface. The air or other gas surrounding a surface quickly contaminates it as soon as it is cleaned.

Many effects become possible in high vacuum when molecules can travel long distances between collisions. Metals can be evaporated from a pure source without reacting in transit. Molecules or atoms can be accelerated to high energies to sputter away or be implanted in the bombarded surface. Electrons or ions can be scattered from surfaces and collected. The energy changes they undergo on scattering or release from a surface can be used to probe or analyze the surface or underlying layers.

The vacuum environment plays a basic and indispensable role in present day technology and is used by a wide variety of scientists including physicists, chemists, and biologists, and technologists and engineers who work in research, development and industrial production.

Pressures in the high vacuum range are needed for the manufacture of microwave, power, cathode ray and photomultiplier tubes, light bulbs, aluminizing of mirrors, glass coating, decorative metallurgy, gas display panels and ion implantation. The pressure must be reduced to a very high vacuum range for most thin-film preparation, electron microscopy, mass spectroscopy, crystal growth, and X-ray and electron lithography. Pressures in the ultrahigh vacuum range are necessary for surface analytical techniques and material studies. It is also necessary in particle accelerators, fusion machines, space simulation systems and systems used for the growth of films by molecular beam epitaxy.

2. Units and Ranges of Vacuum

The standard international (SI) unit of pressure is the pascal (Pa) which is equivalent to one newton (N) per square meter (m^2) but it is still quite common to use the millibar (mbar) which is not an SI unit. The millibar is quite convenient because, in the majority of applications of vacuum technology, the concept of force per unit area is not very relevant and we are more likely to be interested in the molecular density. Among other units the most common is the Torr. The relationship between these units and the atmosphere, that is a natural unit of pressure, is:

1 atmosphere = 760 Torr = 1013 mbar = 1.013×10^5 Pascal.

The variety of applications of vacuum technology demands that the range of pressures should extend over more than fourteen orders of magnitude and it is useful to divide the total pressure range into four regions, namely:

1. *low vacuum* – atmospheric pressure to 1 mbar (10^5-10^2 Pa),
2. *medium vacuum* – 1 mbar to 10^3 mbar (10^2-10^{-1} Pa),
3. *high-vacuum* – 10^{-3} mbar to 10^{-8} mbar (10^{-1}-10^{-6} Pa),
4. *ultra-high-vacuum* – 10^{-8} mbar to 10^{-11} mbar and less (10^{-6}-10^{-11} Pa).

These four divisions are somewhat arbitrary but it is interesting to note that in each case it is possible to relate them to different physical properties of the residual gases.

Low vacuum. In this region, in which the pressure is still a significant fraction of the atmospheric pressure, the main property is the force exerted by the atmosphere; this can be used for mechanical handling, vacuum forming, vacuum brakes, degassing of fluids and vacuum impregnation in certain electrical components where, for example, the air is replaced by a fluid to improve the electrical insulation.

Medium vacuum. The applications in this range are extensive and include processes such as vacuum drying and vacuum freeze drying for the food and pharmaceutical industries and vacuum distillation for the chemical industry. In many of these processes an important factor that has to be considered is the vapor pressure of the fluid which, of course, is often water and the effect of this on the vacuum pumps must also be taken into account. It is necessary that the pressure in the system should be less than the saturated vapor pressure of the fluid at the appropriate temperature. Thus in vacuum drying at room temperature the pressure must be less than about 1 mbar, whereas for vacuum freeze drying in the range -50^{o}C to -180^{o}C, the pressure must be in the region of 10^{-2} mbar.

High vacuum. The high vacuum region has very many applications and includes the production of special materials for the metallurgical, electronics and aircraft industries and other processes such as electron beam welding. In TV, X-ray and gas discharge tubes, electron microscopy and particle accelerators it is necessary to use high vacuum but perhaps the most important process in this pressure range is that of vacuum evaporation of thin films for lens blooming and many aspects of the semiconductor and high technology computer industries. Nearly all of these applications demand that the mean free path – the average distance traveled by the gas molecules between collisions – must be greater than the dimensions of the vacuum chamber. The appropriate value of the mean free path in terms of the pressure, p mbar, is given by the relation

The appropriate value of the mean free path λ (in meters) in terms of the pressure p (in millibars) is given by the relation λ=6.6x10^{-5}. Thus at a typical high vacuum pressure of 10^{-5} mbar the mean free path is about 6.6 m which is greater that the dimensions of most high vacuum chambers.

Ultra-high vacuum. The pressure in the atmosphere is about 10^{-10} mbar at a height of 10^{6} m so that some space simulators require ultra-high vacuum. On the other hand research in thermonuclear fusion uses ultra-high vacuum in order to achieve extremely high gas purity. However in this pressure range the mean free path is very large and it is more important to consider the molecule-surface collisions rather than the molecule-molecule collisions. If we assume that at a pressure of p mbar all gas molecules arriving at a surface remain on that surface, then the time t seconds to form a monolayer of, for example, nitrogen molecules, on that surface is given approximately by the expression

$t=3\times10^{-6}/p$.

Thus at 10^{-6} mbar the time is 3 seconds whereas at 10^{-10} mbar the time is several hours and this makes it possible to conduct measurements on atomically clean surfaces. Ultra-high vacuum is therefore an essential requirement in all surface studies such as electron microscopy, mass spectroscopy, in molecular beam epitaxy, in laboratory investigations of the processes in space and so on.

***p*(mbar)**	***n*(number m^{-3})**	**λ**	***J*(cm^{-2}s^{-1})**
10^3 – 1 atm	2.5×10^{25}	6.6×10^{-6} cm	2.9×10^{23}
1	2.5×10^{22}	6.6×10^{-3}cm	2.9×10^{20}
10^{-3}	2.5×10^{19}	6.6 cm	2.9×10^{17}
10^{-6} (HV)	2.6×10^{16}	66 m	2.9×10^{14}
10^{-10} (UHV)	2.5×10^{12}	660 km	2.9×10^{10}

Table 1. Number density *n*(number m^{-3}), mean free path (λ), and impingement rate (*J*) for nitrogen gas at 295 K.

It is instructive to tabulate the values of number density *n*, mean free path λ and impingement rate *J* for representative pressure values. Table 1 shows data for nitrogen gas N_2, the dominant constituent of air, at 22^{o}C=295 K.

(see *Physical Quantities and Units*)

3. Pumping Process

A vacuum system is a combination of pumps, valves and pipes which creates a region of low pressure. The most important gas which we pump is air, because it is in every system. It contains a lot of constituents, as seen from Table 2, and the main are nitrogen (78%), oxygen (21%), argon (0.94%) and carbon dioxide (0.033%).

Constituent	**Content**		**Pressure**
	(vol %)	**ppm**	**(Pa)**
N_2	78.084±0.004		79,117
O_2	20.946±0.002		21,223
CO_2	0.033±0.002		33.437
Ar	0.934±0.001		946.357
Ne		18.18±0.04	1.842
He		5.24±0.004	0.51
Kr		1.14±0.001	0.116
Xe		0.087±0.001	0.009
H_2		0.5	0.051
CH_4		2.	0.203
N_2O		0.5±0.1	0.051

Table 2. Components of Dry Atmospheric Air

To achieve the required vacuum is not simply a matter of removing a sufficient quantity of the air originally in the vessel. This indeed has to be removed but we then find that there are continuous sources which launch gas into the volume and which present the pump with a continuous gas load. The vacuum achieved at steady state is the result of a dynamic balance between the gas load and the ability of the pump to remove gas from the volume. We may divide these sources into broad categories, and denote the loads they present by the symbol Q.

(a) ***Leaks,*** Q_L. These may be real leaks due to passageways through the vacuum wall from outside the vessel or, more subtly, virtual leaks due to gas being trapped in localities from which it can emerge only slowly into the vacuum surroundings.

(b) ***Vaporization,*** Q_V. Often inadvertently, but sometimes of necessity, materials which exert a significant vapor pressure are present in a vessel, contributing a gas load. Water vapor from imperfectly dried components is particularly troublesome. Careless handing of components has to be strictly avoided in high-vacuum and ultra-high vacuum practice, for example.

(c) ***Outgassing,*** Q_G. This term describes the release of gas from the internal surface of the vacuum wall and the surfaces of components inside the vessel. It forms the principal source of gas in many systems and limits the degree of vacuum which can be achieved.

(d) ***Process generated gas,*** Q_P. Many processes carried out in vacuum cause the release of gas, often from materials which are heated. For instance in vacuum degassing applications metals are heated to high temperature to rid them of dissolved gas.

(e) ***Back-stream from the pump,*** Q_B. Pumps do not work perfectly and capture all molecules which enter them, so that some molecules from the vacuum chamber return to it. In the case of a diffusion pump, vapor of the working fluid causes the back-stream into the vacuum chamber.

Action of a pump is characterized by pumping speed S (in liter/s or m^3/h). Here S is the gas volume that is removed from a vessel at given pressure per unit of time. Consider the usual pumping system shown in Figure 1. The system can be divided into two parts: the pumping volume and the pump with connecting pipes and valves. As mentioned above, there are several sources of gas to the pumping volume, i.e. Q_L, Q_V, Q_G, Q_P, Q_E. and we can measure all these gas quantities in terms of pV that is equal to the mass of a gas with the accuracy of the factor RT/M (M is the mass of das molecule).

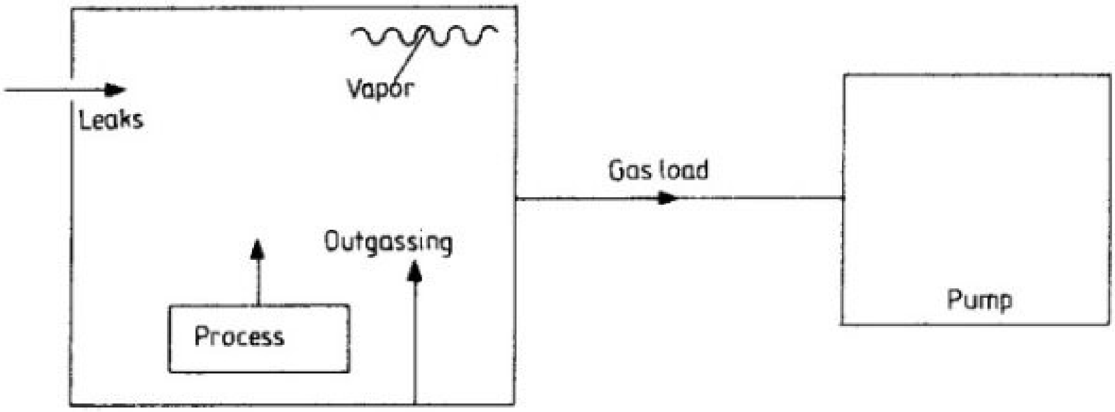

Figure 1. Schematic representation of a vacuum system.

The main equation which describes the pumping process has a form

$$-Vdp = (Sp - Q_L - Q_V - Q_G - Q_B - Q_P)dt. \quad (1)$$

The left part of this equation is equal to the gas loss in the pumping volume, and the right side determines the quantity of gas which is removed by the pump and incoming to the volume during the time *dt*. When the ultimate vacuum will be achieved (at the pressure p_{min}), the derivative *dp/dt*=0 and correspondingly

$$p_{min} S = \sum Q_i . \quad (2)$$

Thus we can express the pumping speed as

$$S = \frac{1}{p_{min}} \sum Q_i . \quad (3)$$

All loads are usually have constant values or are practically time-independent, so we can easy integrate the equation (1), and taking into account the relation (2) we have

$$p - p_{min} = (p_0 - p_{min}) \exp(-St/V), \quad (4)$$

where p_0 is the initial pressure. As a rule the initial pressure is much higher than the ultimate pressure, and we can write that

$$p = p_0 e^{-(S/V)t} + p_0 \quad (5)$$

We see that the time parameter $\tau = V/S$ characterized the efficiency of the pumping system.

A system filled with an ideal gas, which has no interaction with the walls other than reflection, exhausted by a pump of constant volumetric efficiency, should be expected to exhibit a pressure versus time performance as expressed by the above equation.

Theory predicts that the pressure should fall exponentially with time, at a rate determined by the 'time constant' of *V/S*. As it follows from a majority of observations, in many vacuum systems the pumping rate (liters per second) exceeds the volume (liters) by a factor of 10, resulting in a time constant of 0.1 s.

3.1. Flow in Pipelines

The pumping system is not only the pump itself but that includes also pipes and valves. Each flow in vacuum pipelines is expressed by the continuity equation

$$Q = p_1 S_1 = p_2 S_2 \tag{6}$$

according to which the same throughout of gas Q (in Torr liter per second) flows through each of a number of successive cross sections 1, 2, *etc*. up to n. The important formula

$$p_1 - p_2 = SpW. \qquad \text{(analogous to Ohm's law)} \tag{7}$$

is of equally general validity; it expresses that the difference of pressure between the ends of a pipeline $p_1 - p_2$ is proportional to the product of the throughput flowing through the pipelines (Sp) and the impedance (W) of the pipeline. On the other hand, the pumping speed (S) is not constant for all cross sections. The relation between the pumping speed at the inlet of a pipeline (S_1), at the outlet (S_2) and the impedance (W) or conductance (L) of the pipeline is given by the equation

$$S_1 = \frac{1}{\frac{1}{S_2} + W} = \frac{1}{\frac{1}{S_2} + \frac{1}{L}}, \tag{8}$$

which states that the pumping speed at the inlet of a pipeline S_1 is all the smaller the larger the impedance W of the pipeline. From this it follows that exact knowledge of factor W is necessary for estimating the efficiency of vacuum systems.

For calculating the pipeline impedances W in sec/liter and the rates of flow G in g/sec, the following expressions are applied:

$$Sp = \frac{G}{M} RT, \tag{9}$$

$$p_1 - p_2 = \frac{G}{M} RTW, \tag{10}$$

$$W = \frac{1}{G} \frac{(p_1 - p_2)M}{RT} = \frac{10^3 \rho}{G}, \tag{11}$$

Here the gaseous constant R=62.37 Torr×liter×deg^{-1}×mol^{-1}.

For long pipelines (*l>>r)* and small differences of pressure between the ends of the pipeline ($p_1 - p_2 \ll (p_1 + p_2)/2$) laminar flow is expressed by Hagen-Poiseuille's equation:

$$W = 12\frac{l\eta}{Fr^2(p_1 + p_2)}, \text{ s/liter.} \quad (12)$$

Here l,r in cm, $\eta(p),p$ in Torr, F cross section of pipe in cm^2, ρ gas density in g/cm^3.

Cases of turbulent flow do not occur in the ranges of medium-high and high vacuum. (see kinetic theory of gases)

4. Pumps

At the beginning of the twentieth century the highest vacuum attainable by air pumps was of the order of 10^{-4} mm of mercury. In the following brief period of some 45 years the ultimate vacuum obtainable has not only been pushed to a much lower pressure but, especially, the speed of pumping has been enormously increased, the simplicity and ease of the pumping operation have been greatly advanced.

A vacuum pump is a device for creating, improving and/or maintaining a vacuum. Two basic categories exist, the gas transfer and the entrapment groups. The gas transfer group can be subdivided into positive displacement pumps in which repeated volumes of gas are transferred from the inlet to the outlet usually with some compression, and the kinetic pumps in which momentum is imparted to the gas molecules so that gas is continuously transferred from the inlet to the outlet. In contrast, entrapment pumps are those which retain molecules by sorption or condensation on internal surfaces.

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Biographical Sketch

Tsipenyuk Dmitry Yur'evich, graduated from the Moscow Institute of Physics and Technology (MIPT) in 1985, becoming candidate of sciences in 1992. From 1985 until the present time he has worked at the General Physics Institute Russian Academy of Sciences, now are being the senior scientist of this Institute. His scientific interests include: lasers physics, interaction of light with matter, remote sensing, and theoretical aspects of unified description of interactions. D.Yu.T. has published more than 40 papers in scientific journals. In addition, he is the author of 7 patents concerning an application of laser breakdown for elemental analysis and an application of luminescence in different technological areas.