26 March 1965, Volume 147, Number 3665 SCIENCE

Ultrahigh Vacuum

Application makes possible important advances in physics, chemistry, and engineering.

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Simulation of the environment of outer space, investigations of the physics and chemistry of surfaces, and experiments in high-energy nuclear physics and on thermonuclear fusion have been fostered by recent advances in vacuum science and technology. These advances have enabled research workers to attain extremely high vacuum—that is to say, extremely low pressures. Why is this so? Before answering this question and describing some of the techniques of ultrahigh vacuum let us consider some of the general features of vacuum.

The word "vacuum" describes any volume filled with gas at a pressure below atmospheric pressure; hence, pressure must be specified to characterize the degree of vacuum accurately. Atmospheric pressure is the pressure exerted by a column of mercury 760 mm high at 0°C under standard acceleration of gravity of 980.665 cm sec^{-2}. The most common units of pressure used in studies of vacuum are millimeters of mercury and torr; millibars are also used in some investigations. The torr is currently the preferred unit (1 torr = 1 mm-Hg = 1.332 mb).

The general classifications of vacuum are rough, medium, high, and ultrahigh. Each classification can be described in several ways: by a range of pressures, by the density of gas molecules, by the average distance a molecule moves before it collides with another molecule (the mean free path), and by the frequency with which molecules collide with the surface of the container. As pressure is lowered, density decreases, the mean free path increases, and molecules hit the container less frequently. The four general classifications of vacuum, together with the specific properties of each, are summarized in Table 1.

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Applications

The extremely high cost of space research has made it imperative to learn on earth how satellites and their component parts will behave in the environment of cold black space. Therefore, it is necessary to duplicate the conditions of space as best we can. We find one condition which must be duplicated is low pressure, for as we depart from the earth the pressure rapidly decreases. At 300 miles (480 km) altitude the pressure is about 10^{-8} torr (Fig. 1). A number of very large chambers have been constructed which can duplicate, at least to some extent, the conditions of outer space. A photograph of the exterior of the simulator at the Missiles and Space Division of the General Electric Company is shown in Fig. 2 (*1*). This chamber is 9.75 m in diameter and 16.5 m high and can achieve a vacuum of 10^{-9} torr. The design considerations necessary for these very large systems, which quite probably can achieve pressures as low as 10^{-13} torr in the near future, are well documented (*2*).

In the particle accelerators used in nuclear physics, the best results are obtained in high vacuum because the particles do not lose energy in collisions with gas molecules. In order to better probe the structure of the nucleus, physicists are requiring apparatus in which very high nuclear interaction energies (in the center-of-mass system) are available. In such experiments these energies are many billions of electron volts. Ultrahigh vacuum is helping to make this possible. The use of two intersecting beams of high energy particles rather than the impingement of one high energy beam with a stationary target offers a powerful new approach to this problem (*3*). It is impractical and inefficient to build two accelerators and have their beams intersect. The solution is to accelerate particles, alternately clockwise and counterclockwise, into two ring-shaped chambers located in a magnetic field. In this manner the particles may be stored in the rings and high particle densities can be achieved. The particle beams can be diverted so that they intersect and interactions can be investigated.

Because it takes hours to fill the rings to the desired density and the particles travel billions of miles as they circulate, it is necessary that there be few collisions with background gas in the rings. This demands that the pressure be less than 10^{-9} torr. In the case of stored electrons or positrons, emitted synchrotron radiation can cause photodesorption of gas from the inside of the chamber (*4*). Therefore, sophisticated processing and pumping techniques are required. The storage rings are immense. For example, the proposed proton storage ring facility at the European Center for Nuclear Research will consist of two intersecting rings each 150 m in radius with a volume of about

Table 1. Classifications of vacuum. The variation of gas density, mean free path, and collision frequency with pressure.

	Vacuum				
	←Rough→	←Medium→	←High→	←Ultrahigh→	
Pressure (torr)	760	1	10^{-3}	10^{-8}	10^{-13}
Density (mol/cm^3)	2.5×10^{19}	3.2×10^{16}	3.2×10^{13}	3.2×10^{8}	3.2×10^{3}
Mean free path* (cm)	6.6×10^{-6}	5×10^{-3}	5	5×10^{5}	5×10^{10}
Collisions with surface* (mol sec^{-1} cm^{-2})	2.9×10^{23}	3.8×10^{20}	3.8×10^{17}	3.8×10^{12}	3.8×10^{7}

* For air at 25°C.

6000 liters. Vacuum of 10^{-9} torr in the rings and 10^{-11} torr in interacting regions (*5*) will be required.

Very high vacuum conditions are also needed in some of the very long new accelerators. Stanford's 20-Gev linear electron accelerator should be completed in 1966 and is 2 miles (3.2 km) long (*6*).

Very high purity gases are required in thermonuclear devices in which elements like deuterium or tritium undergo thermonuclear fusion and produce energy. The presence of ionized impurities can lead to a large loss of power in the form of bremsstrahlung, the loss being proportional to the square of the nuclear charge of the impurity. Thus, since even a very small amount of an impurity can lead to an extremely large loss of power (*7*), every effort must be made to keep the gases very pure. This of course demands that very low residual pressure (that is, contaminant concentration) be obtained and that outgassing (desorption of gas) of walls must be slight. There is quite a variety of experimental approaches to a study of thermonuclear fusion (*8*). The C-Stellerator at Princeton, for example, can achieve a base pressure of 10^{-10} torr. It is about 12.2 m long, is made of stainless steel, and has a volume of about 400 liters (*9*).

Any experiment designed to study the physical or chemical properties of a surface whose composition is to be representative of the bulk material requires the preparation of a surface free of contamination and the maintenance of cleanliness for a time which is sufficient to perform the experiment. If the time is only a few seconds, then the surface may be generated in a vacuum of about 10^{-6} torr. If, as is usually the case, the time required is of the order of several minutes or hours, then the surface must be prepared and maintained in a vacuum of 10^{-9} torr or less. The time to contaminate an initially clean surface is plotted as a function of pressure in Fig. 3. It is assumed that every molecule which strikes the surface is adsorbed.

By using ultrahigh vacuum and associated techniques it is possible to produce clean surfaces (*10*) and to probe their physical and chemical properties. Techniques which have been used to study clean surfaces and their interaction with gases include field emission of electrons (*11*), field ionization of a gas such as helium (*12*), work functions (*13*), photoelectric emission (*14*), secondary electron yield (*15*), Auger electron emission (*16*), and catalytic reactivity (*17*). Figure 4 shows a field ion image of an atomically clean tungsten surface (*18*). All of these techniques have led to new understanding of surface structure and the chemical reactivity of clean surfaces.

Technological advances based on this information include high-intensity x-ray tubes which use field emission cathodes (*19*), the preparation of superconducting thin films (*20*), and the development of new lubricants for metals (*21*).

All of these scientific and technological advances are made possible by exploiting the properties of high vacuum, that is, low particle concentration, long mean free path, and few collisions with a surface. These then are some of the important areas of science and technology where ultrahigh vacuum is having quite an impact.

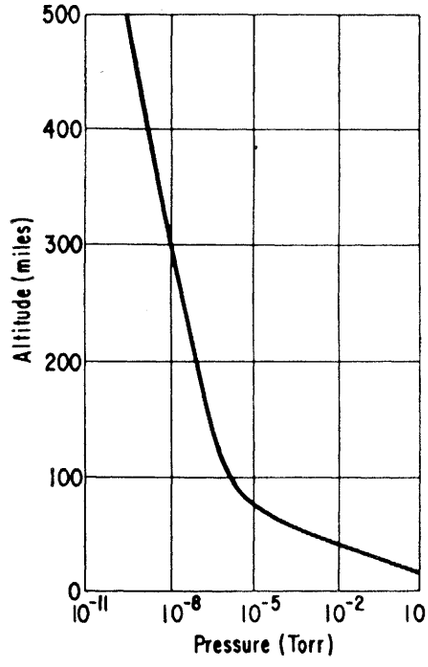


Fig. 1. Variation of total pressure with altitude above the earth. 100 miles = 161 km.

Ultrahigh Vacuum

The factors which determine the degree of vacuum or equilibrium pressure, P_{eq}, in a vacuum system include the rate at which gas enters the chamber and the rate at which it is removed. That is

$$P_{eq} = \frac{Q_T}{S} \text{ torr},$$

where Q_T is the total gas influx expressed in torr liters per second, and S is the pumping speed expressed in liters per second. Q_T is the sum of the quantities of gas entering the system from all sources and includes leaks in the system wall, permeation through the wall, desorption from the wall, back-streaming from the pump, and gas evolved by high vapor pressure materials in the system. These sources are shown schematically in Fig. 5.

There are two obvious methods for reducing the pressure in the system. One is to minimize the gas influx, Q_T, and the other to maximize the pumping speed, S. The choice of either of these methods or a combination of the two is dictated by the experiment to be performed in the vacuum system. Consider two examples: a study of the reaction of a gas with a metal film, and the ground testing of a space vehicle. The vacuum system used for the gas-film study is shown in Fig. 6 (*17*). It is constructed from glass and metal and can be heated to 300° or 450°C for outgassing of the parts. This results in a low influx of gas. Because of the low Q_T of this system, a 5-liter-per-second mercury diffusion pump trapped with liquid nitrogen can pump a 2-liter volume down to 10^{-10} torr.

Testing of a space vehicle in a space simulator is quite a different story. Here Q_T is very large and heating (bakeout) is impossible. Therefore

pumps with enormous capacities must be used. Cryogenic pumps, with capacities of many thousands of liters per second, are the primary means for removing gas in these simulators. These pumps are cooled surfaces which surround the test vehicle and remove from the gas phase a large fraction of molecules which strike the surface.

These two systems and the others discussed earlier are quite different, even though similar components are employed and even though both permit achievement of ultrahigh vacuum. Let us now dissect ultrahigh vacuum systems and consider some of the new techniques which have been developed to produce, measure, and use ultrahigh vacuum (*22*).

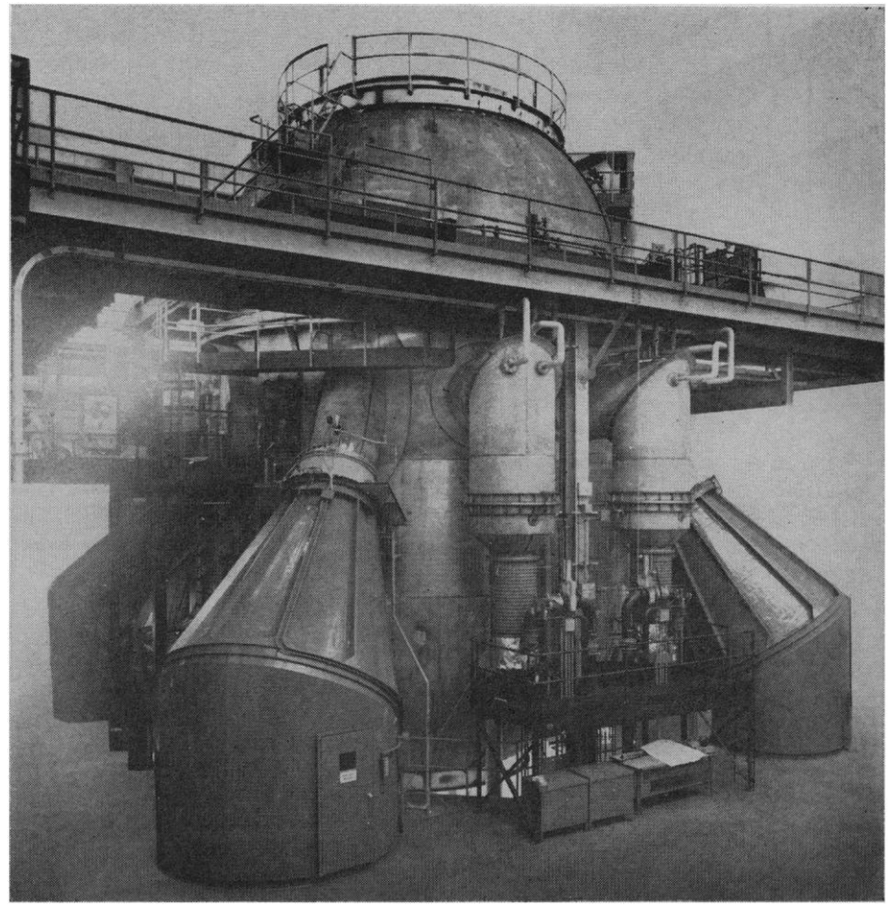


Fig. 2. Space simulation chamber at the General Electric Space Environment Simulation Laboratory, Valley Forge, Pennsylvania. This chamber can accommodate a spacecraft weighing as much as 20,000 kg (44,000 pounds).

Materials

One of the major considerations in the selection of materials for ultrahigh vacuum systems is to keep the gas influx to a minimum. Therefore, materials should have a low vapor pressure and should be impervious to the diffusion of gas through them at both the system operating temperature and the bakeout temperature (250° to 450°C). These criteria generally preclude the use of rubber O-rings, waxes, greases, glass stopcocks and joints, brass, and soft solder found in many laboratory vacuum systems (*23*).

Pumps

A vacuum is produced by removing molecules from gas in a chamber. Any device which accomplishes this is called a pump. There is a great variety of pumps. Pumping may be accomplished by displacement, transfer of momentum, condensation or adsorption, chemical reaction, ionization and acceleration into a surface, or diffusion through a semipermeable membrane. Many pumps involve combinations of these methods. The selection of the method of pumping for a given experiment or system depends on the kind and quantity of gas to be pumped as well as the ultimate pressure to be obtained.

Displacement pumps remove gas by using a piston and valves, and operate much like the way a bicycle tire pump would operate if it were hooked up backwards. The piston takes many forms. It can be a reciprocating piston, an eccentric rotor, or two counter rotat-

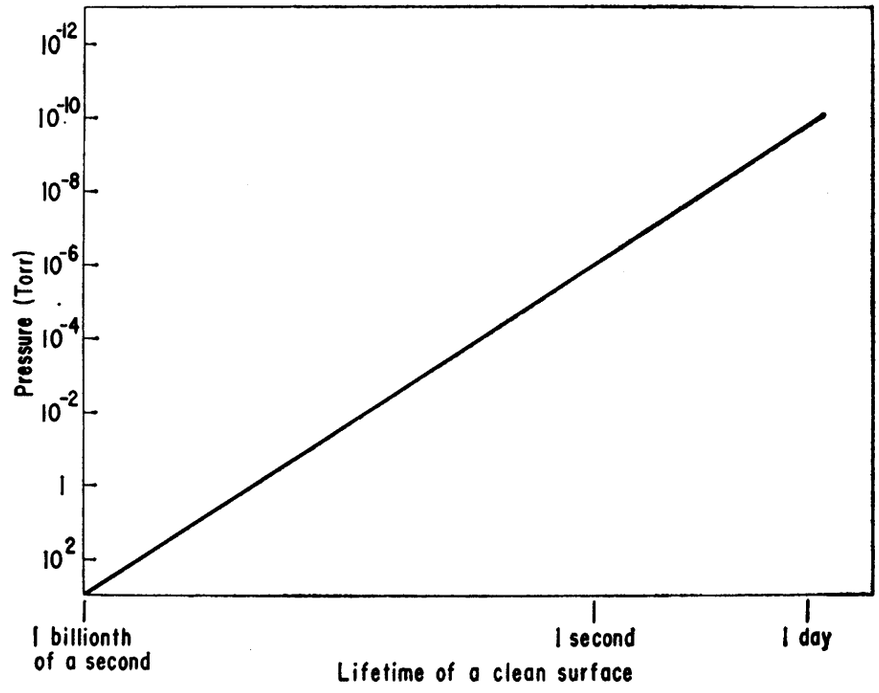


Fig. 3. Variation of clean surface time with pressure.

ing figure-eight-shaped lobes. Mercury can also be used as a displacement fluid piston. At best these pumps can achieve pressures of about 10^{-6} torr. They are often used in conjunction with some of the other pumps to be mentioned to produce pressures in the ultrahigh vacuum range.

A second class of pumps operates on the principle of momentum transfer. In diffusion pumps, momentum is transferred to the gas being pumped by a directed high speed vapor of mercury or oil. The working fluid can diffuse into the system being evacuated unless suitable traps are provided. This is called backstreaming. Zeolites (*24*) are an effective trap material for oil vapor, and liquid nitrogen traps remove mercury vapor. With proper trapping, a vacuum of 10^{-10} torr or less can easily be achieved.

Momentum can also be transferred to gas molecules by a rapidly rotating surface. In these "molecular" pumps there are no mechanical seals between the input and exhaust ports and traps are not required. This idea is an old one and dates back to Gaede in 1913 (*25*). The turbomolecular pump is a modern development of these ideas (*26*). It has been claimed that pumps based on this principle can produce pressures as low as 10^{-9} torr. This type of pump has the advantage that it contains no working fluid which can contaminate the vacuum system. It can also have a very large pumping speed.

In principle, one of the simplest methods for removing gas from a volume is to adsorb or condense it on the walls of the vessel by reducing the temperature. If the gas is removed by condensation on a very cold surface (such

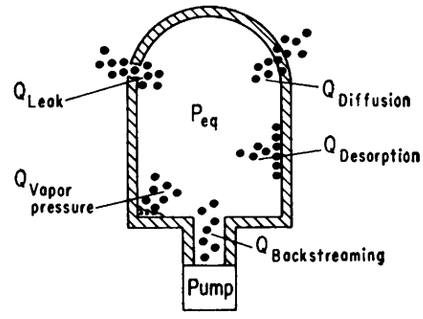


Fig. 5. Schematic representation of the various sources of gas influx in a vacuum system.

that its vapor pressure is negligible), the term cryogenic pumping or cryopumping is applied. If, however, the gas is physically adsorbed on a material of high surface area at a low temperature, the term sorption pumping is used.

Sorption pumps consisting of a tube of activated charcoal at liquid nitrogen temperature have been used for years. Today zeolites are used as well as charcoal. A typical zeolite sorption pump is shown in Fig. 7 (*27*). A pump of this design can be used in place of mechanical pumps in the initial pump-down of ultrahigh vacuum systems, or it can be used to produce pressures below 10^{-10} torr.

At 20°K (the boiling point of hydrogen at 760 torr) all gases except helium, hydrogen, and neon have a negligible vapor pressure (Fig. 8). At 4.2°K (the boiling point of liquid helium at 760 torr) all gases but helium and hydrogen have a very low vapor pressure. Therefore, cold surfaces at these low temperatures will act as pumps for most of the common gases.

The rate of removal of a gas depends on the condensation coefficient for the gas at the temperature of the surface and the cold surface area available. The condensation coefficient is the fraction of molecules that stick to the surface divided by the number that strike the surface. A simple calculation indicates that a liquid-nitrogen–cooled surface has a pumping speed of about 15 liter sec^{-1} cm^{-2} for water vapor. Because it is possible to achieve such enormous pumping speeds, cryogenic pumps are being used in large space simulators. To achieve these large speeds it is necessary to maintain a large conductance to the cold surface and to shield the cold surface from radiation from warmer surfaces. The approach to cryopumping taken in one space simulator is to line the interior

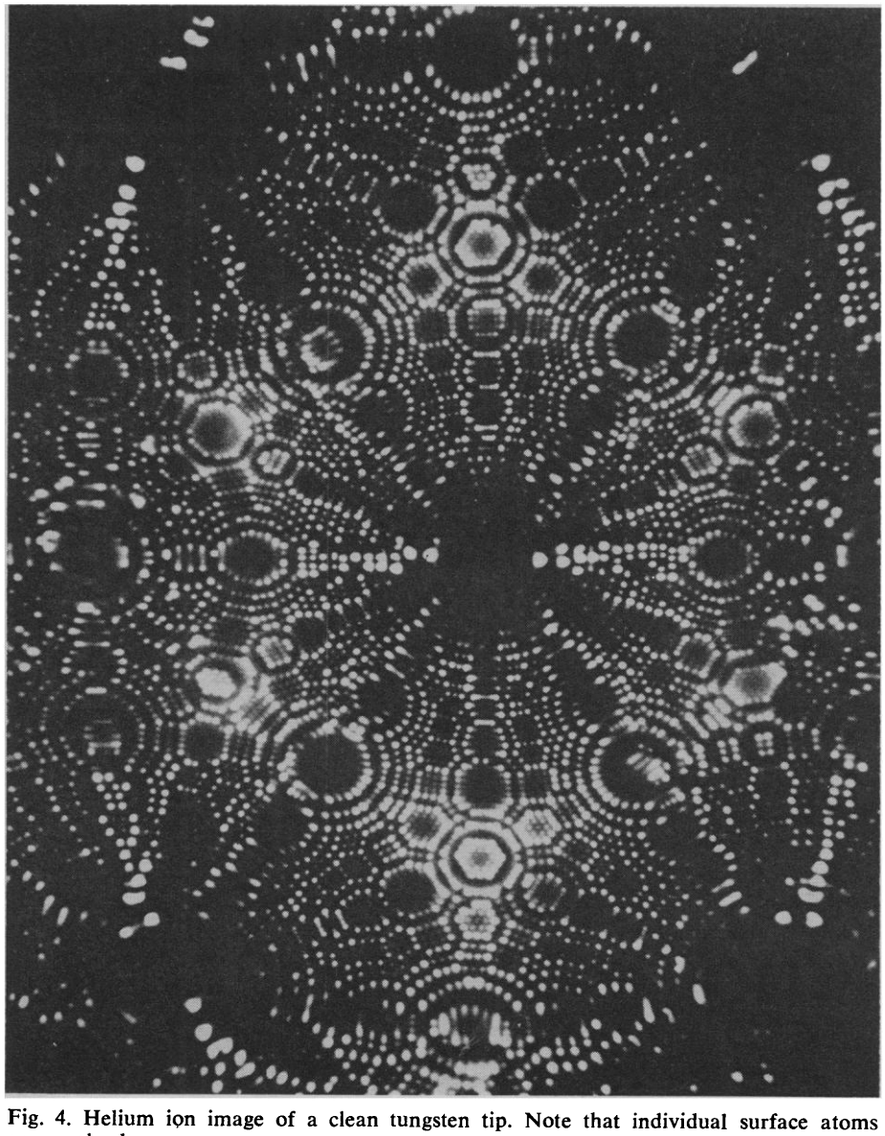


Fig. 4. Helium ion image of a clean tungsten tip. Note that individual surface atoms are resolved.

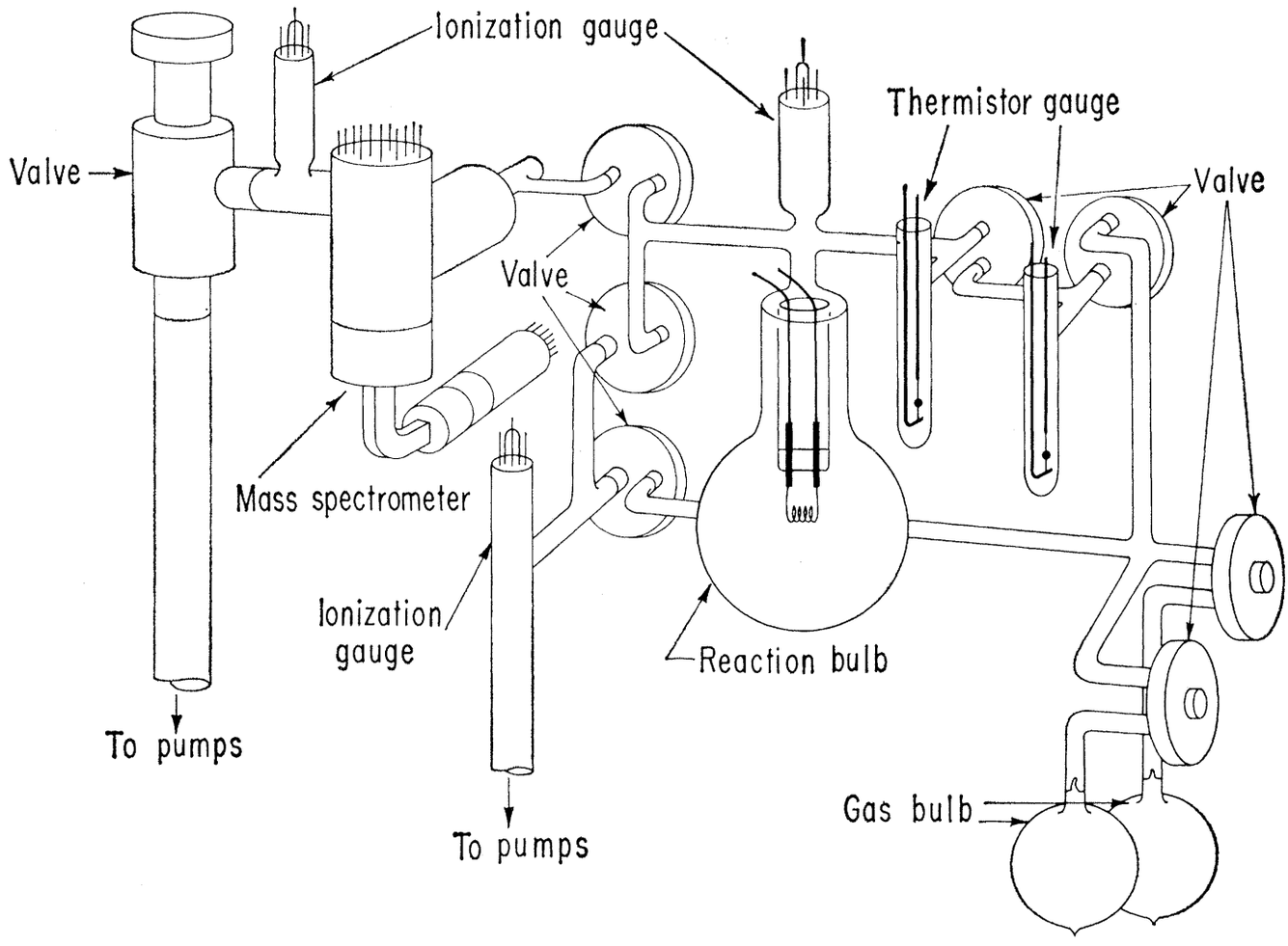


Fig. 6. An ultrahigh vacuum system used for studying the reaction of a gas with a clean metal film. This system is constructed from glass and metal and can be heated to a high temperature to aid in the production of ultrahigh vacuum.

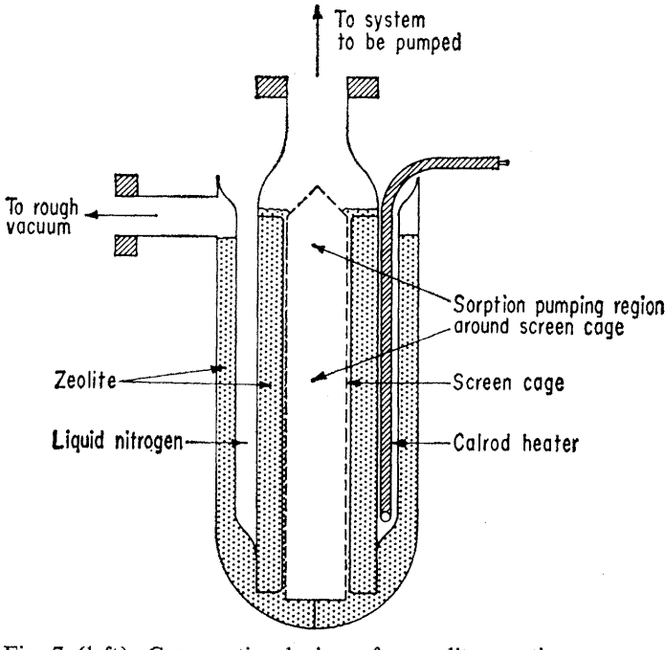


Fig. 7 (left). Cross-sectional view of a zeolite sorption pump. below room temperature.

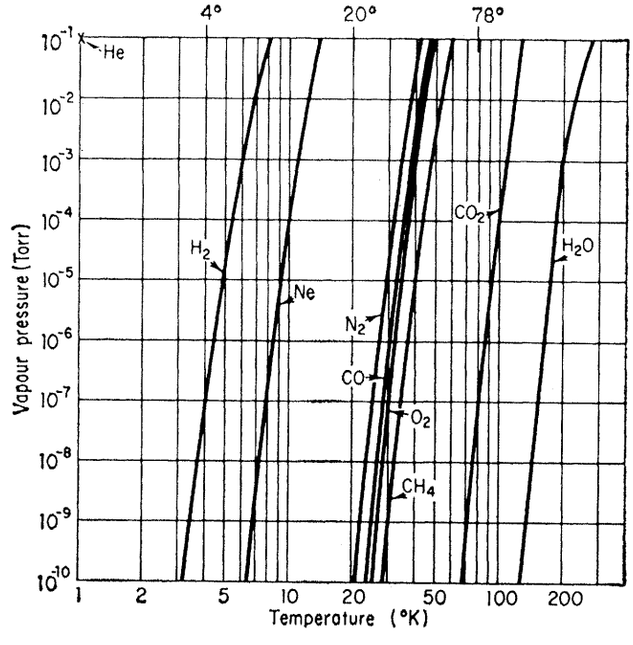


Fig. 8 (right). Vapor pressures of a number of common gases

Table 2. Classification of metals and semi-metals based on adsorption properties. A indicates adsorption, NA no adsorption. Parentheses indicate that a metal probably belongs to the group in which it is placed but that the behavior of films is not known.

Group	Metal	Gas						
		O_2	C_2H_2	C_2H_4	CO	H_2	CO_2	N_2
A	Ca, Sr, Ba, Ti, Zr, Hf, V, Nb, Ta, Cr, Mo, W, Fe, (Re)	A	A	A	A	A	A	A
B_1	Ni, (Co)	A	A	A	A	A	A	NA
B_2	Rh, Pd, Pt, (Ir)	A	A	A	A	A	NA	NA
C	Al, Mn, Cu, Au*	A	A	A	A	NA	NA	NA
D	K	A	A	NA	NA	NA	NA	NA
E	Mg, Ag, Zn, Cd, In, Si, Ge, Sn, Pb, As, Sb, Bi	A	NA	NA	NA	NA	NA	NA
F	Se, Te	NA	NA	NA	NA	NA	NA	NA

* Gold does not adsorb oxygen.

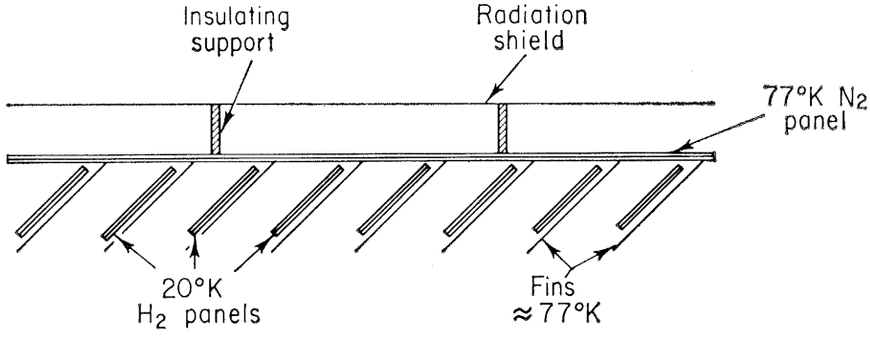


Fig. 9. Cryogenic wall of a space simulator. Panels at 20°K are shielded from room temperature radiation by surfaces at 77°K.

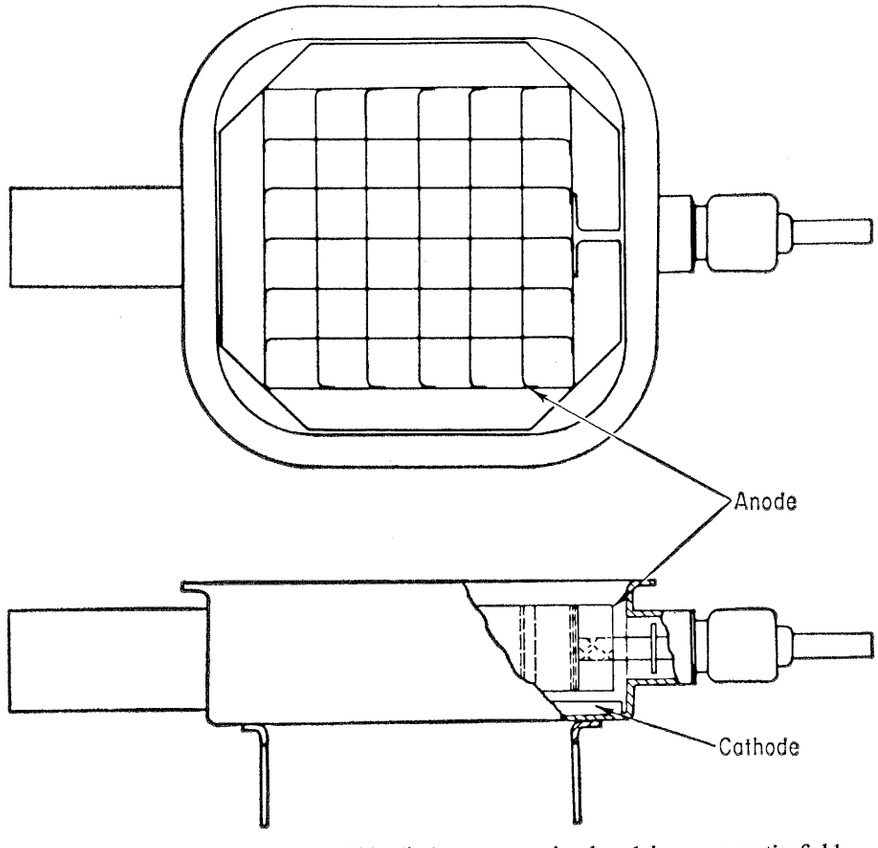


Fig. 10. A sputter-ion pump. This diode structure is placed in a magnetic field.

walls with 20°K panels shielded by a 77°K surface (Fig. 9) (*28*).

A third cryogenic method of pumping is called cryotrapping. In this pump a gas which would ordinarily have a high vapor pressure at the temperature of the cold surface is trapped in a matrix of a material with a low vapor pressure. For example, hydrogen can be pumped on a cold surface at 77°K if water vapor is admitted to the system (*29*). Similarly, hydrogen and helium can be pumped by a surface at 4.2°K if argon is admitted (*30*).

It is often convenient to remove chemically active gases from a vacuum system by causing them to react with an active metal to form a compound with a very low vapor pressure, or a strongly held chemisorbed layer of gas. Pumps of this type have been used for many years in the production of vacuums in radio tubes and other electronic devices and are usually referred to as getters (*31*). The process of chemisorption or reaction of the metal with the gas is called gettering. In these pumps, active metal films are usually generated by the evaporation of a wire or pellet by resistance heating, or by radio frequency induction heating (*32*). Of course, the bulk getter must be carefully outgassed before evaporation or it will contribute gas to the vacuum system.

Often a metal will adsorb only a limited variety of gases. Thus, by judicious choice of a getter material, trace impurities may be removed from a gas. Table 2 lists a number of metals and semimetals and the gases that they will chemisorb at room temperature (see *33*).

Clean metals can also alter the composition of gas in a system. For example, ethane, propane, and butane will decompose on clean films of rhodium and iridium to methane (*17, 34*), whereas on films of tungsten and molybdenum, ethane will decompose to hydrogen (*35*). It has been reported that when impure titanium getters were evaporated in vacuum, methane was formed from hydrogen and carbon impurities (*36*). In some gas-metal systems displacement reactions can occur. For example, nitrogen can displace hydrogen adsorbed on molybdenum, and carbon monoxide can displace hydrogen adsorbed on tungsten (*37*). It has been found that when barium films are exposed to carbon monoxide and water, hydrocarbons are generated (*38*). Thus great care must be given to the selection

of a getter metal for a particular system.

Titanium, molybdenum, and barium have been used extensively as getter materials. The speed of getter pumps depends primarily on the effective surface area of the getter and the sticking coefficient of the gas.

Electronic pumps remove gas from a system by ionizing gas molecules by electron impact and by accelerating the positive ions through several thousand volts potential difference into a metal or glass surface. The adsorption and re-emission of ions from metal and glass surfaces have been investigated by many workers (*39*).

Electronic-chemisorption pumps are very effective devices for evacuating a system. They can pump chemically active and inactive gases, often do not act as a source of contaminating gases, and do not require liquid nitrogen traps. These pumps contain a means for producing clean metal surfaces, a source of electrons, and an accelerating field. The potential of the metal surface is negative. Titanium and molybdenum are normally used for the getter material, although tantalum may have special advantages (*40*). Several means have been used for producing clean metal films. These include evaporation from a wire source, evaporation by electron bombardment, or sputtering.

Perhaps the most popular type of electronic-chemisorption pump is the sputter-ion pump. This pump is based on the cold-cathode or Penning discharge gauge. One typical form (Fig. 10) (*41*) is a diode structure consisting of two parallel-plate titanium cathodes separated by an "egg-crate" shaped anode and located in a magnetic field. Several thousand volts are applied between the anode and the cathodes. Because of the magnetic field, electrons produced travel long spiral paths and have an increased chance of ionizing the residual gas. Positive ions produced are accelerated into the cathodes and sputter titanium onto the anode. The pumping action is a combination of gettering, acceleration of ions into surfaces, and burial of atoms.

The operation of this type of pump is not as straightforward as it may first appear. For example, diode pumps exhibit a memory effect—that is, a rare gas previously pumped can be re-emitted. This is often observed if a rare gas has been pumped. For example, if argon is continuously admitted to a system which is solely pumped with a diode sputter-ion pump, pressure fluctuations

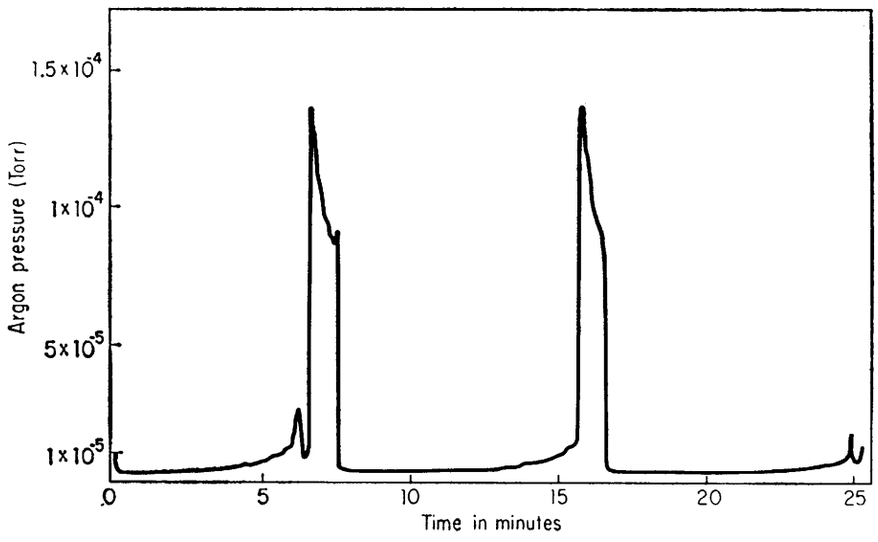


Fig. 11. Mass spectrometer measurement of argon pressure versus time for a system being pumped with a diode sputter-ion pump exhibiting argon instability.

will occur. The mass spectrometer record of pressure with time indicates that the pressure fluctuations are due to argon gas. Such a pattern of pressure versus time for a getter pump exhibiting argon instability is shown in Fig. 11 (*42*). It has also been observed that a rare gas can replace a previously pumped rare gas (*43*).

The pumping of hydrocarbons by ion pumps can lead to the generation of a wide range of new compounds which can diffuse back into the vacuum system. For example, when benzene was pumped with a diode pump, methane and hydrogen were generated (*44*). When the pump was disassembled, it was observed that the cathodes were covered with a carbonaceous deposit (Fig. 12). Various refinements of this pump have been developed which avoid some of the above-mentioned problems. These include the slotted cathode pump (*45*) and the third-electrode pump (*46*). These pumps are available commercially and have speeds which range

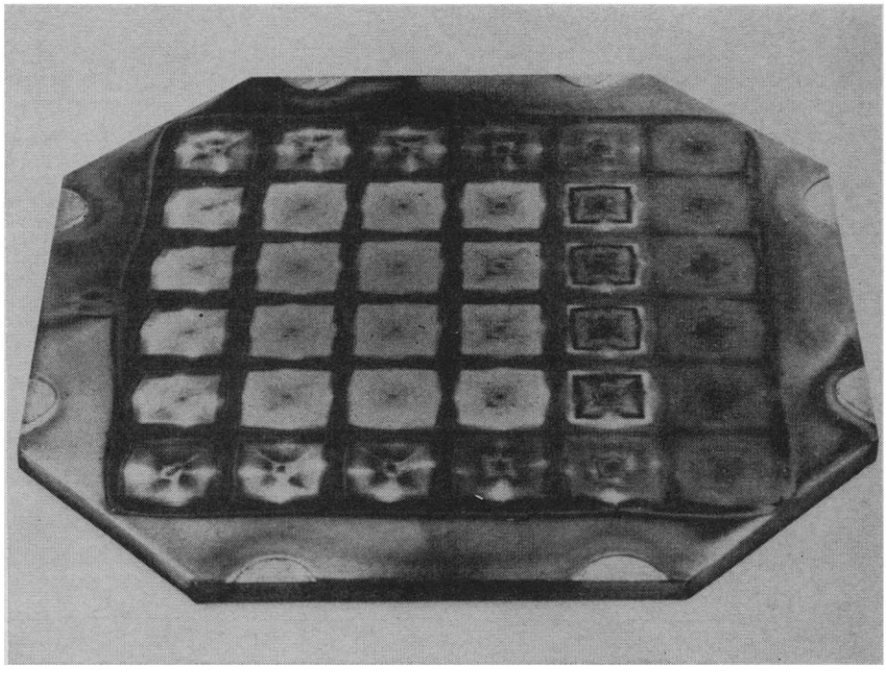


Fig. 12. Cathode of a diode ion pump after pumping benzene at a pressure of 10^{-5} torr. Note the presence of a carbonaceous deposit on the surface.

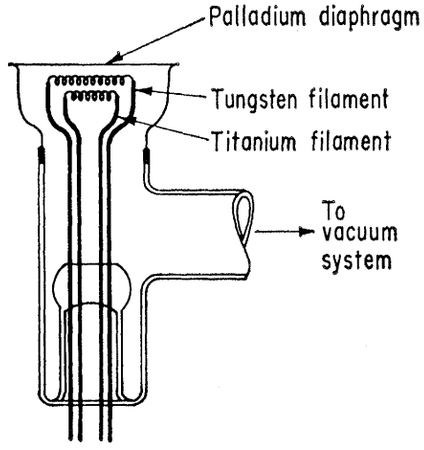


Fig. 13. Hydrogen pump, 5 cm in diameter, with a palladium diaphragm. It pumps 5 liter sec^{-1}.

from one to several thousand liters per second. There are a number of other pump designs which utilize getter-ion pumping (*47*).

The principle of selective diffusion of a gas through a metal diaphragm has been incorporated into a pump of novel design (*48*). This pump (Fig. 13) is specifically designed to remove hydrogen from a system. The diaphragm is made from palladium or a silver-palladium alloy and is heated to about 600°C by electron bombardment with a tungsten filament. Pumping occurs only if the outside of the diaphragm is in an oxidizing atmosphere (air) and the inside is in a reducing atmosphere or a good vacuum. It has been possible to pump down to a hydrogen pressure of 10^{-9} torr while the hot palladium diaphragm was exposed to air.

Gauges

In most systems the degree of vacuum is expressed in terms of the total gas pressure in the system. What we are really interested in, for the most part, is the particle density in the system. If the temperature and the pressure are known, the density may be easily calculated. The composition of the gas in the system is normally expressed in terms of the partial pressure of each component. There is a variety of gauges that will measure the total pressure, the partial pressure, or the density of gas in a vacuum system. The useful pressure ranges for a number of gauges are given in Fig. 14 (*49*).

The McLeod gauge is the standard device used to calibrate most vacuum gauges. In this gauge, the gas at an unknown pressure is trapped in a standard volume and is compressed to a known final pressure by a mercury piston into a capillary tube of known dimensions. The final volume is measured and the initial pressure is calculated from Boyle's law. McLeod gauges may be used to measure pressures down to about 10^{-5} torr. This pressure, however, is far from the ultrahigh vacuum region.

Since it is difficult to measure low pressures directly, an indirect approach is usually taken. The residual gas is bombarded with electrons. Positive ions produced are collected and the current, i_+, measured. The positive ion current can often be related to the gas density in the system, ρ, by the following expression:

$$i_+ = i_- lQ(E)\rho$$

where i_- is the electron current, l the distance the electron travels through the gas, and $Q(E)$ the probability that a gas molecule will be ionized by an electron of energy E.

Ionizing electrons can be produced by hot filaments, cold cathodes, or ultraviolet light–electron multiplier (*50*) sources. Alpha particles have also been used (*51*).

The triode ionization gauge is the device used most frequently to measure total pressures in high vacuum systems. This gauge contains a hot tungsten filament (0 volt potential) as a source of

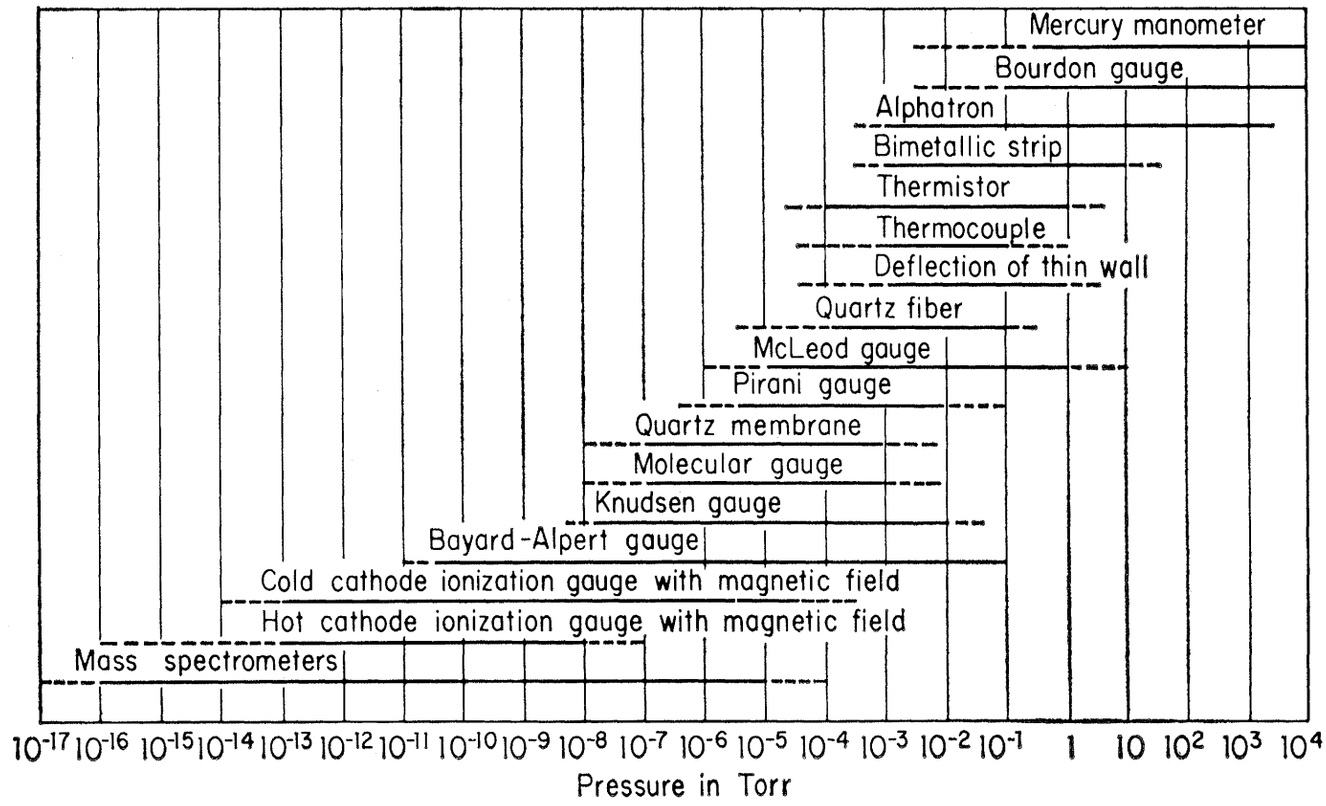


Fig. 14. Useful pressure ranges for a number of vacuum gauges.

electrons, a grid at about 150 volts, and a collector at about −20 volts. We find that the pressure in a system can be expressed as

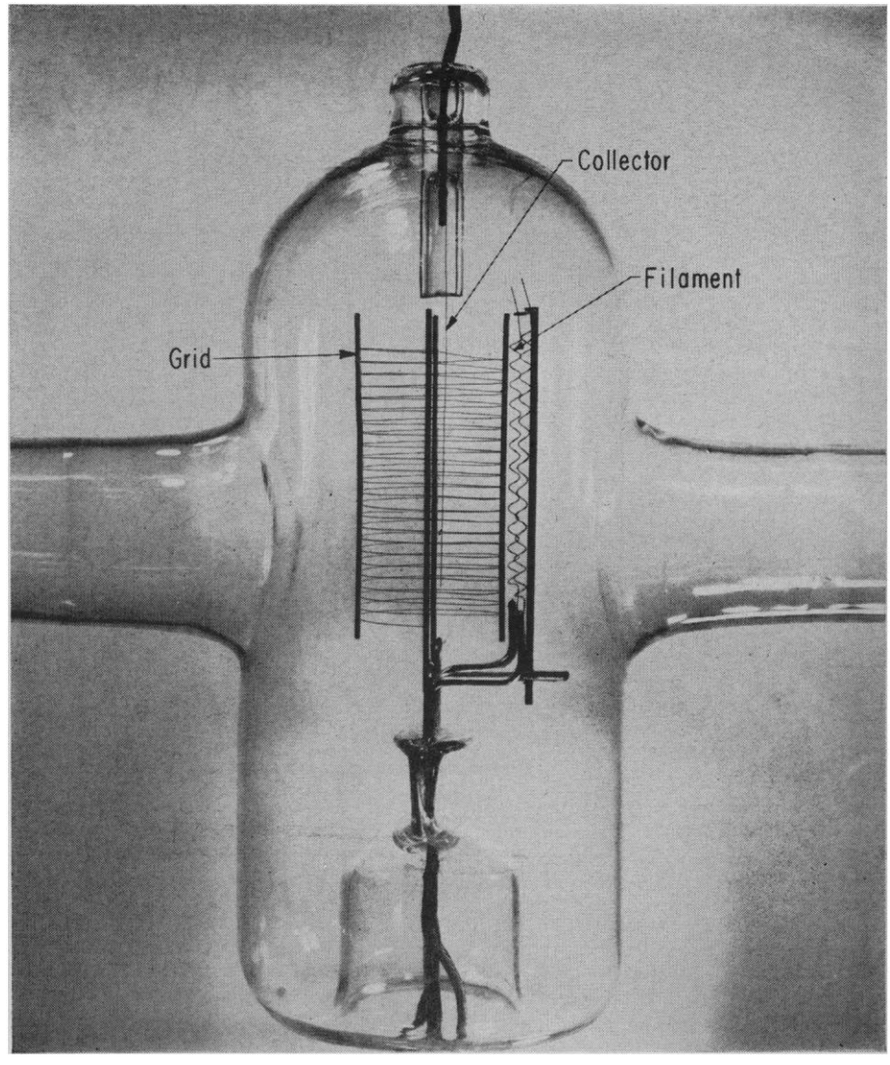
$$P = \frac{1}{S}\,\frac{i_+}{i_-},$$

where S is the sensitivity of the gauge for the particular gas.

One of the early forms of the triode ionization gauge took the form of a central filament surrounded by a cylindrical grid and a cylindrical sheet plate. The lower pressure limit of this structure, about 10^{-8} torr, was determined by the x-ray effect—that is, electrons that strike the grid cause the emission of soft x-rays. These x-rays strike the collector plate and produce electrons. The departure of an electron from the plate is electrically the same as the arrival of a positive ion at the collector. This limiting x-ray effect depends to a large extent on the physical shape of the collector. When the collector is massive and surrounds the grid, it intercepts practically all the soft x-rays generated at the grid.

Bayard and Alpert (*52*) changed the geometry of the ionization gauge to reduce this effect. They inverted the standard gauge structure. The collector takes the form of a wire, 0.013 mm in diameter, surrounded by a cylindrical grid. Tungsten filaments are located outside the grid. The collector intercepts only a small fraction of the soft x-rays, and the lower pressure limit is reduced to about 10^{-10} to 10^{-11} torr. This structure is called the Bayard-Alpert gauge or the inverted ionization gauge and is shown in Fig. 15. A typical nitrogen calibration curve for a Bayard-Alpert gauge made by the calibrated conductance method is given in Fig. 16 (*53*).

There are a number of factors which can cause errors in pressure measurement at all pressures (*54*). The gauge can act as a pump and remove gas; therefore the conductance of the tubulation between the gauge and the system must be kept large. The gauge operation may become unstable and go into oscillation. The hot filament of the gauge may interact with the gas being measured and alter it. For example, oxygen can react with carbon impurities in hot tungsten filaments to produce carbon monoxide (*55*); hydrogen can be dissociated on the filament and the atoms react with glass in the system to produce carbon monoxide, water, and methane (*56*); or hydrocarbons can decompose to methane and hydrogen (*57*). Some of these effects can be



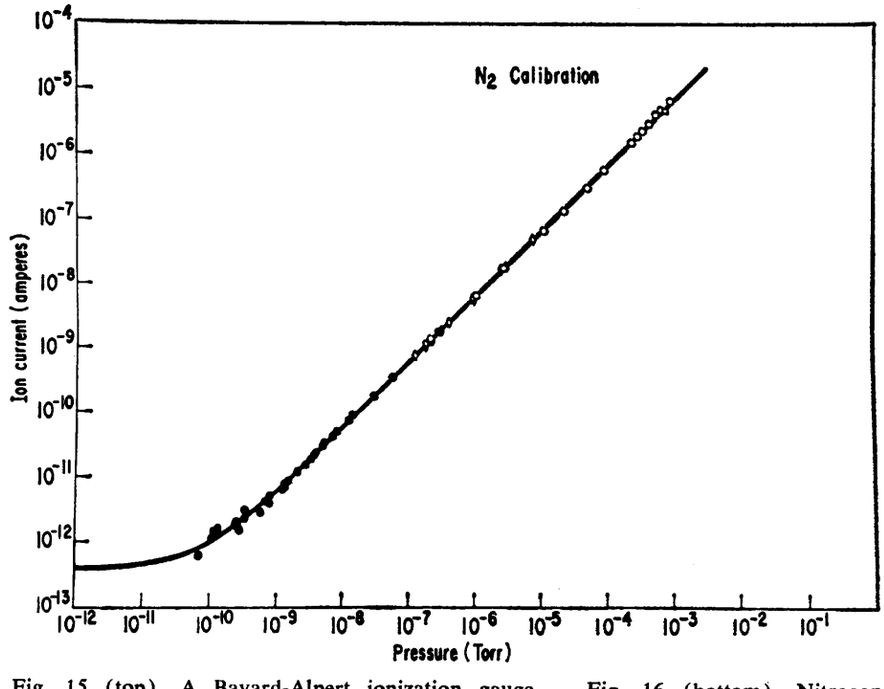


Fig. 15 (top). A Bayard-Alpert ionization gauge. Fig. 16 (bottom). Nitrogen calibration curve for a Bayard-Alpert ionization gauge. Electron emission current is 0.5 ma.

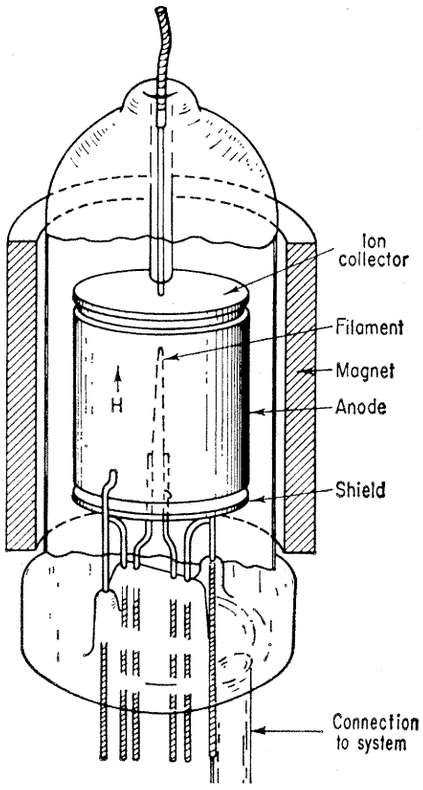


Fig. 17 (left). A hot-cathode magnetron ionization gauge.

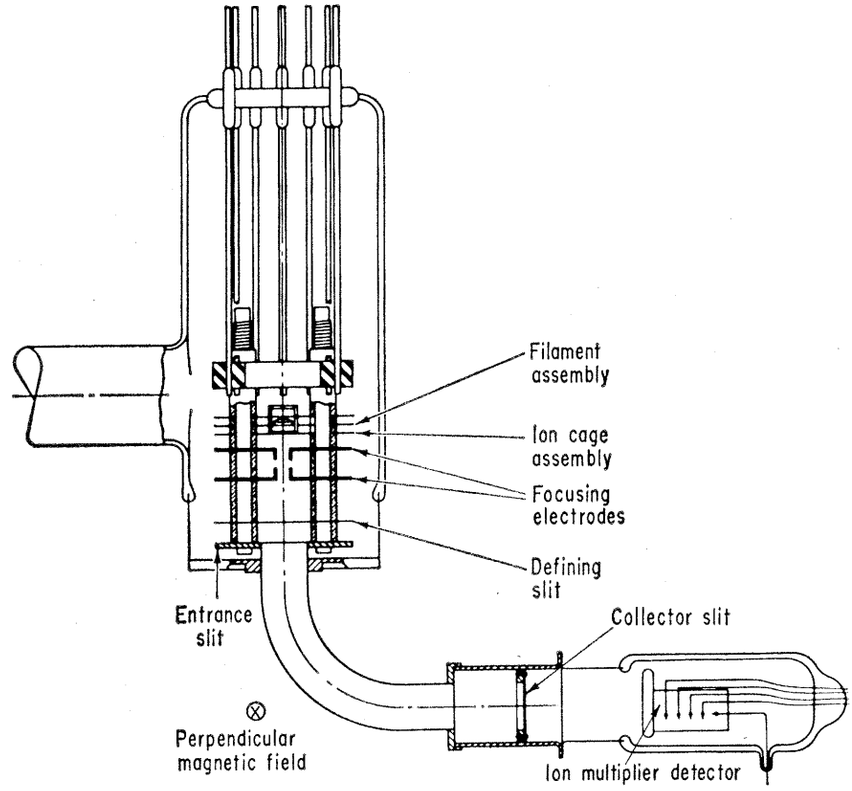


Fig. 18 (right). Magnetic deflection mass spectrometer.

reduced substantially by substituting a tantalum or rhenium filament coated with lanthanum boride for the tungsten electron source. This material has a lower work function than tungsten and consequently a lower operating temperature (*58*). Other effects affect the accuracy at low pressures. These include the x-ray effect already discussed, the reverse x-ray effect (*59*), positive ion emission from hot filaments (*60*), and desorption from the grid (*61*). A number of modifications have been made on the basic Bayard-Alpert ionization-gauge structure to reduce some of these effects (*62*).

The sensitivity of ionization gauges may be increased by increasing the effective path length of the ionizing electrons. This is often done with magnetic fields. One such gauge is the hot-cathode magnetron ionization gauge which is shown schematically in Fig. 17 (*63*). In this device electrons emitted from the filament spiral in long paths around the axial magnetic field. Since the magnetic field is adjusted so that very few electrons reach the anode, very low electron emission is used, and the pumping speed of the gauge is low. Pressures as low as 4×10^{-13} torr were measured with this gauge (*63*). It is suggested that the range of this gauge can be extended to about 10^{-17} torr by the addition of an ion multiplier detector (*64*).

A class of vacuum gauges eliminates the hot filament as an electron source and uses cold emission. The Penning

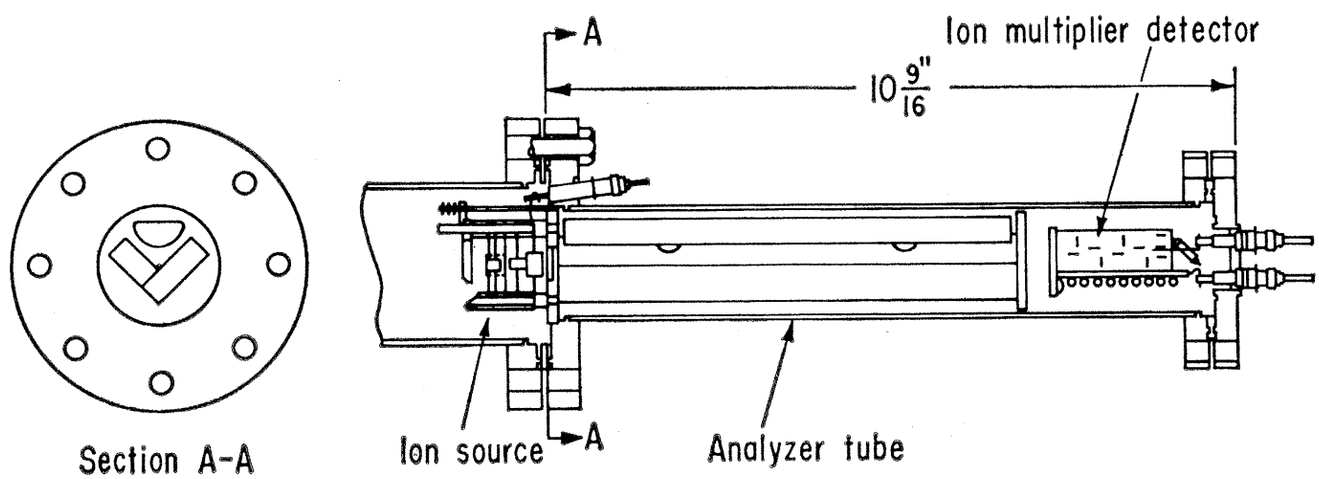


Fig. 19. Monopole mass spectrometer. Length of analyzer of tube, 26.8 cm.

gauge consists of two flat parallel cathodes separated by a cylindrical or ring-shaped anode located in an axial magnetic field. A potential of several thousand volts is applied between the electrodes. Because of the magnetic and electric field, electrons produced by cold emission oscillate in spiral paths between the cathodes. Positive ion current to the cathode is measured and is proportional to the pressure. Gauges of this type measure pressures from the ultrahigh vacuum range (about 10^{-12} torr) to about 10^{-4} torr. It is often difficult to start such gauges at low pressures. The addition of a small tungsten filament obviates this problem (*65*). A number of other cold cathode gauges have also been developed (*66*). These gauges have a fairly high pumping speed. This property was put to use in the sputter-ion pump described earlier.

There are many occasions when it is necessary to have accurate pressure-measuring devices in the range 10^{-5} torr to 1 torr. Typical experiments which require such gauges include the measurement of electrical properties of ultrapure gases and the investigation of catalytic reactivity of clean metal surfaces. Gauges to be used in this pressure range must be bakeable, retain their calibration after baking, and not act as a source of contamination to the vacuum system. There are a variety of absolute and secondary gauges which satisfy these criteria. There are several ionization gauges that can be used to measure pressures up to about 0.1 torr (*67*). A bakeable thermistor vacuum gauge for measuring pressures in the range 1 to 10^{-4} torr has recently been described (*68*).

All of the gauges described thus far give an indication of the total pressure in the vacuum system. If a number of gases are present, it is often important to know the concentrations or partial pressures of each.

Partial pressure gauges convert a portion of the gas into ions and measure the relative number of ions of each mass (or mass/charge, m/e) present. They are often called mass spectrometers. Mass spectrometers contain (i) an inlet system for the gas to be measured, (ii) an ionization region in which the gas is converted to ions, (iii) an analyzing region that separates the ions according to m/e, and (iv) a detector that measures the number of ions for each m/e.

Ionization is usually accomplished by bombardment with electrons from a hot filament, although cold cathodes, spark sources, field ionization, and other sources have been used. Detectors are either Faraday cage collectors or ion multipliers. The latter detector is generally used at very low pressures, that is, low ion concentrations. The types of m/e analyzers vary greatly. Mass spectrometers which have been used in ultrahigh vacuum applications include time-of-flight (*69*), omegatron (*70*), radio frequency (*71*), crossed field (*72*), magnetic deflection, quadrupole, and monopole. We will briefly discuss the last three types.

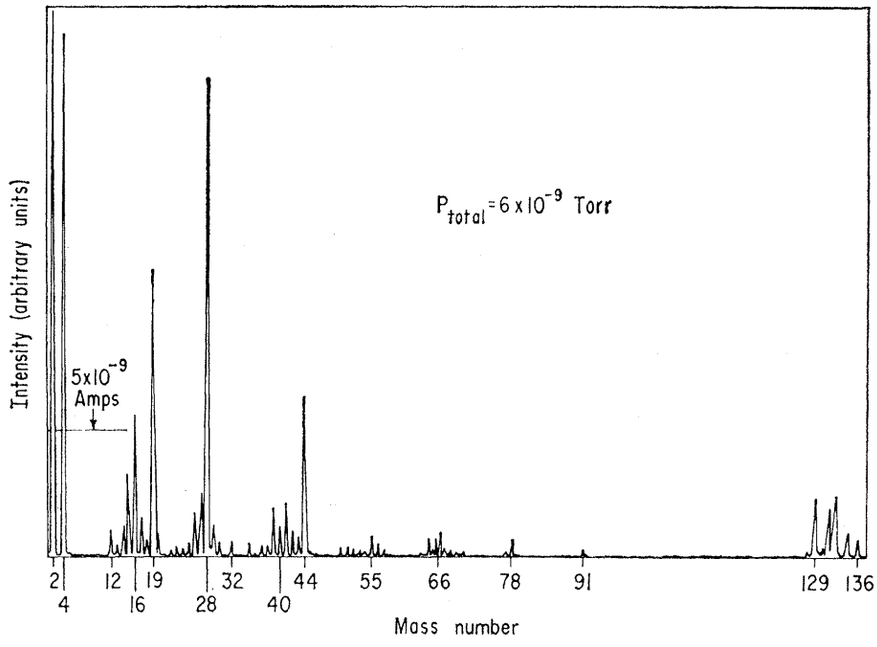


Fig. 20. Typical mass spectrum taken with the monopole mass spectrometer shown in Fig. 19.

The magnetic deflection mass spectrometer is one of the more widely used devices for partial pressure measurement in vacuum systems. A schematic diagram of a typical spectrometer is given in Fig. 18 (*73*). Ions produced by electron bombardment are accelerated through a potential difference and

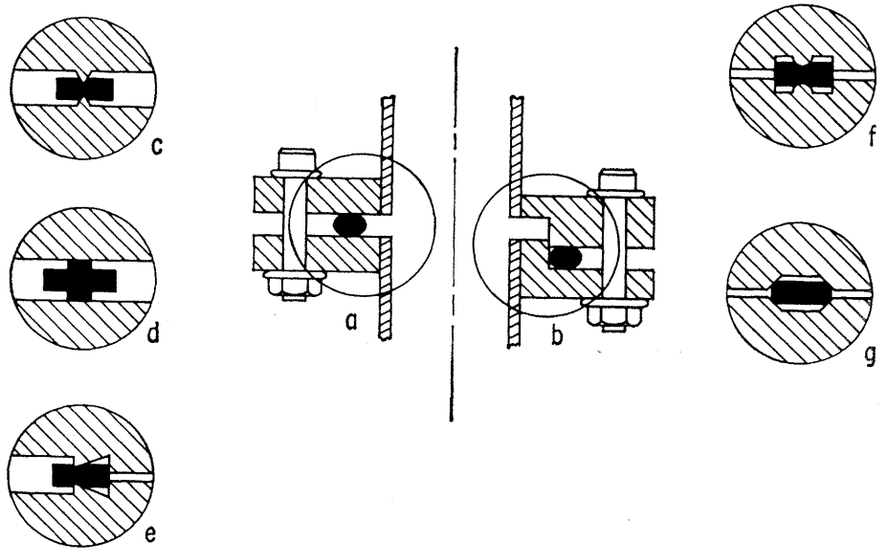


Fig. 21. Demountable metal gasketed flanges. A large number of flange-gasket configurations have been successfully used. Some of these are shown here. *a*, "O" ring; *b*, corner; *c*, knife edge; *d*, coined gasket; *e*, "conflat"; *f*, semi-toroid; and *g*, "andar."

analyzed with a magnetic field. When the ions enter the magnetic field, they experience an acceleration and have circular trajectories with a radius r. This radius of curvature is a function of the mass m of the ion, its charge e, its energy Ve, and the magnetic field strength H. Here V is the potential difference, and e is equal to ne', where e' is the charge of an electron and n the number of such charges. We find that the radius of curvature is given by

$$r = (1/H)(2\,Vm/ne')^{1/2}$$

or

$$r = (144/H)(VM/n)^{1/2}$$

where M is the molecular weight of the ion. With sensitive ion multiplier detectors and counting devices, these mass spectrometers will measure partial pressures as low as 10^{-16} torr (*74*). Modulated molecular beam sampling techniques have been used with this type of mass spectrometer to greatly increase its signal to noise ratio (*75*).

Within the last 5 years, the use of an electric quadrupole field as a mass analyzer has become quite popular (*76*). In this device ions are introduced along the axis of a quadrupole field which is produced by four parallel cylindrical rods. A simplified treatment of the equations of motion of ions in this field has recently been given (*77*). It has been demonstrated that it is possible to create a portion of the quadrupole field by means of two electrodes, a right angle and a circular rod (*78*). This is referred to as a monopole configuration and can be used as a mass analyzer. A schematic diagram of a monopole mass spectrometer is given in Fig. 19 (*79*), and a typical spectrum is shown in Fig. 20 (*79*).

Seals

Vacuum seals are generally classified as static or motion seals and may be permanent or demountable. Today, with the increasing use of high-temperature bakeout procedures, seals are often also classified as bakeable or nonbakeable. The type of seal used in a system must of course be tailored to the particular problem to be solved. Thus, while grease and rubber gaskets are widely applied in 10^{-6} torr systems, they are to be avoided in ultrahigh vacuum systems.

Permanent static seals are made by the direct joining of glass-to-glass, glass-to-metal, metal-to-metal, and so on by methods such as fusion and welding.

A perfect demountable seal is one in which continuous molecular contact between the two mating surfaces takes place. In practice such seals are approached with liquid metal and shear gasket seals. Solders and low vapor pressure elements like indium have been used to seal joints. Separation is accomplished by merely heating the joint.

Perhaps the simplest demountable static seal is one which utilizes the elastic or plastic deformation of a soft metal gasket. A number of variations of this type of seal exist. A number of these are shown schematically in Fig. 21 (*80*). All of these seals will withstand high-temperature bakeout. The flanges are made from stainless steel and the gaskets from gold or oxygen-free high-conductivity copper. It is necessary in these seals to generate extremely high pressures at the flange-gasket interface. By restraining the lateral motion of gaskets (capturing) it is possible to produce pressures up to ten times the yield strength of the gasket material (*81*).

A variety of seals has been developed for introducing rotary or translational motion into ultrahigh vacuum systems. Metal bellows have been used to introduce translational as well as low speed rotational motion into vacuum systems. Levers and gears may be used to amplify the motion. With materials of low vapor pressure, these seals can be used in bakeable ultrahigh vacuum systems. The transmission of motion by using the coupling of magnetic fields is another widely used technique. The vacuum wall must be made of a nonmagnetic material. Permanent magnets or electromagnetic induction drives may be used. Vibrational motion can be introduced by magnetically driving a reed at its resonant frequency (*82*) (Fig. 22). Motion may also be introduced in ultrahigh vacuum systems by using a low vapor pressure liquid to form a seal around a shaft going through the vacuum wall. A eutectic mixture of gallium, indium, and tin has also been

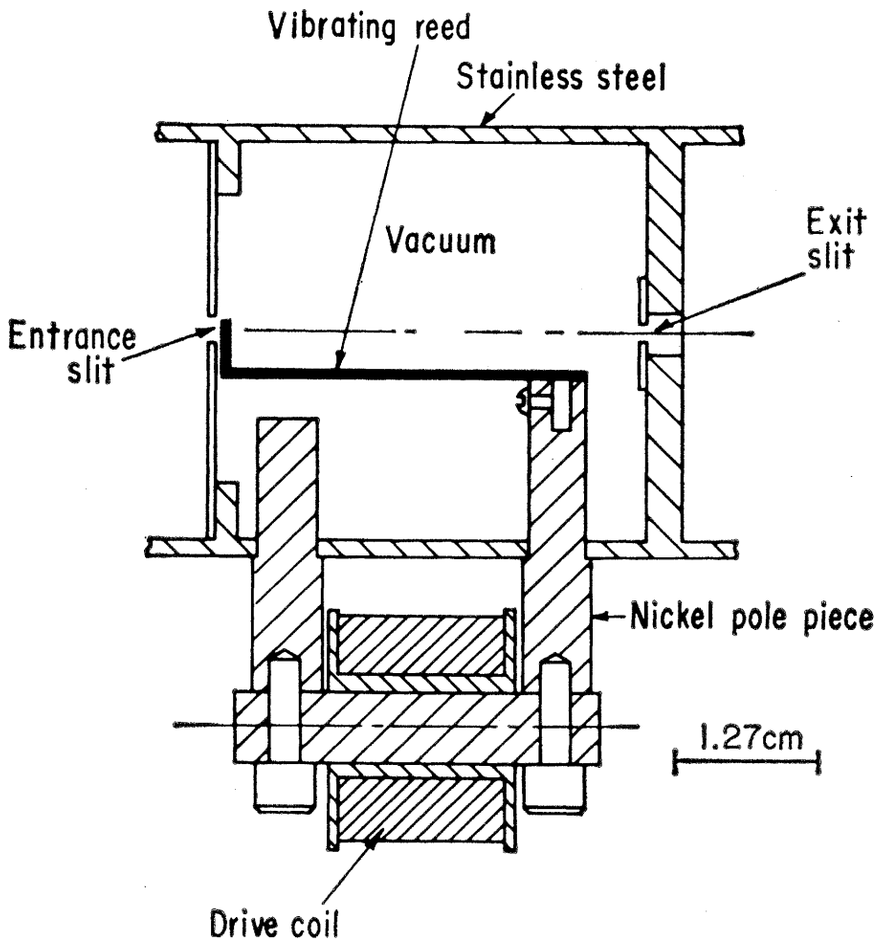
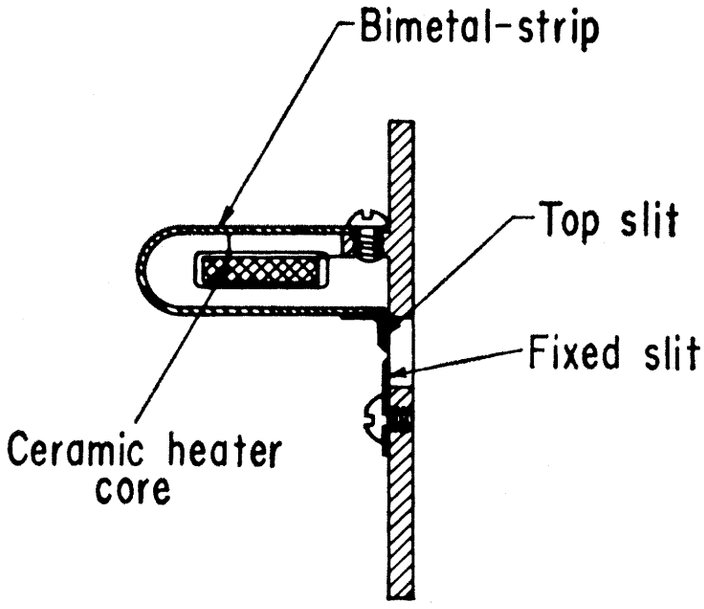


Fig. 22. Vibrating reed assembly. The reed is magnetically driven at its resonant frequency.



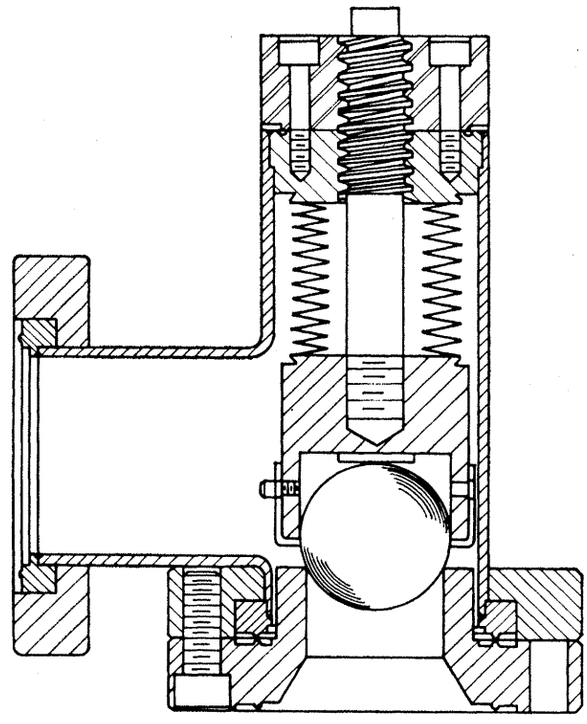


Fig. 23 (above). Adjustable slit assembly. The slit is actuated by heating a bimetallic element attached to one slit jaw. Fig. 24 (right). A metal valve 2.5 cm in diameter.

used with good results (*83*). This material melts at 10.7°C and has a vapor pressure of less than 10^{-8} torr at 500°C. Mercury, if trapped with liquid nitrogen, can also be used (*84*).

Another simple method, which has been largely neglected, is to heat a bimetallic strip in the vacuum system. An electric current is taken through the vacuum chamber wall by means of standard fittings and is used to heat a bimetallic strip. Figure 23 shows an adjustable mass spectrometer slit which operates on this principle (*85*).

Valves

Valves are an integral part of ultrahigh vacuum systems because they are used to vary conductances in the system and to isolate various parts of it. Valves with the smallest closed conductance are made from metal. The sealing is accomplished by compression of a metal, shearing of a metal, or by actual melting of a metal. Motion is achieved with a flexible bellows or diaphragm, which is welded or brazed to the valve body, or by magnetic coupling. A typical design for a small metal valve which employs a compression seal is shown in Fig. 24 (*86*). Many of the larger metal valves (15 to 35 cm in diameter) actually shear the valve seat material (usually copper). In this manner a fresh seat is generated at each closure (*87*).

Indium and other low melting, low-vapor-pressure metals have been used as valve seat materials. They have also been used as a working fluid in cutoffs (*88*). Glass valves generally take the form of carefully lapped planar or spherical surfaces. Magnetic coupling to an iron slug incorporated in the valve is the usual method of operation. A thin glass membrane or tube and a metal hammer enclosed in glass are commonly used separators. Once opened, this "valve" remains open.

A class of valves, called "diffusion leaks," depends on the rate of diffusion of a gas through a material. Porous ceramic rods have been used as leaks (*89*). The rod is partially surrounded with mercury and the leak rate controlled by varying the height of the mercury. It is well known that some gases will diffuse through metals and glasses and that the rate of diffusion is a sensitive function of temperature. A number of valves or leaks operate on this principle. They take the form of a thimble or closed tube connected to the vacuum system. The tube is surrounded by a heater wire so that its temperature can be varied. Palladium and nickel tubes have been used for hydrogen and deuterium leaks (*90*), silver for oxygen (*91*), and 96 percent silica glass for helium (*92*).

Advances in vacuum science and technology have made the attainment of pressures in the ultrahigh vacuum range ($P < 10^{-8}$ torr) commonplace. We have described in some detail the designs and principles of operation of vacuum pumps, gauges, seals, and valves. System design and component selection is dictated by the experiment to be performed. Examples are presented which cover the gamut from a small glass laboratory system (several liters in volume) to a large metal space simulator (several thousand liters in volume).

The use of ultrahigh vacuum techniques has made possible radical new approaches to problems in physics, chemistry, and engineering.

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