

# **Vacuum Principles and Applications**

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## Preface

It is often said that we live at the bottom of a sea of air. The atmosphere around us it is composed of a variety of molecules such as nitrogen, oxygen, water vapor, carbon dioxide, argon and a host of other natural and manmade substances. The density of this mixture of molecules is determined by such factors as the total depth of the atmosphere, ambient temperature, altitude and meteorological conditions. At the microscopic level, of the molecules are in motion and the combined force exerted by this vibrating mass of molecules on a surface is something we call pressure.

Vacuum technology is concerned with reducing the density of molecules to some predetermined level in a contained environment and, more often than not, replacing the mixture that we call air with some other mixture of gases. The resulting environment is dictated by what we want to do in that environment, be it operating a surface analysis instrument, depositing films, making a neon sign or developing the next life saving pharmaceutical. The ability to accurately and reproducibly measure pressure and control gas flow are keys to operating a successful vacuum application.

*“Modern atomic physics is the child of the vacuum pump”* - Karl K. Darrow, 1932

The development of vacuum pumps and systems capable of reaching very low pressures has been intertwined with most of the advances in physics since the mid-nineteenth century. The simple low pressure electrical discharge tubes developed by Geissler and others quickly progressed from curiosities to devices with significant implications. The discovery of x-rays by Roentgen in 1895 represented a watershed. The identification of the electron and the invention of the cathode ray oscilloscope tube happened at about the same time. Other developments quickly followed: the vacuum tube made the radio industry possible and vacuum coating processes led to new types of optical elements as well as to integrated circuits. The scanning electron microscope, mass spectrometer, laser, computer, microwave oven, compact disk and even plasma treated tire cords would all be fiction without vacuum and vacuum processes.

*“One man’s vacuum is another man’s sewer”* - N. Milleron, 1970

Vacuum technology covers a very wide range of pressures and conditions. Vacuum to a person doing fiberglass laminating is very different from the vacuum used by a neon sign worker. A thousand times better than this is the level of vacuum used in x-ray tubes and particle accelerators. And, a thousand to a million times better than this is the degree of vacuum used in atomic level research on the surfaces of materials.

Even though it pervades our technology and our lives, vacuum is a field that has not been very accessible to the the non-specialist, mainly due to a lack of information specifically targeted toward that audience. While Nature may not really abhor a vacuum, she does pose a number of challenges. It is hoped that this brief text will help the user of vacuum technology to better understand the principles that are involved in the creation and use of vacuum.

## A Note on Units, Conventions and Intent

Unit selection is based on common usage in the United States.

Pressure:	Torr (mmHg)
Length:	centimeter
Volume:	cubic centimeter or liter
Mass:	gram
Time:	second

Rather than specifying specific pressure ranges in the vacuum regime, the following terminology may be used:

Low or Rough Vacuum:	Between 1 Torr and atmospheric pressure
Medium Vacuum:	Between 1 milliTorr ( $10^{-3}$ Torr) and 1 Torr
High Vacuum:	Between $10^{-8}$ Torr and 1 milliTorr
Ultra High Vacuum:	Below $10^{-8}$ Torr

Standard conditions, unless otherwise noted, are:

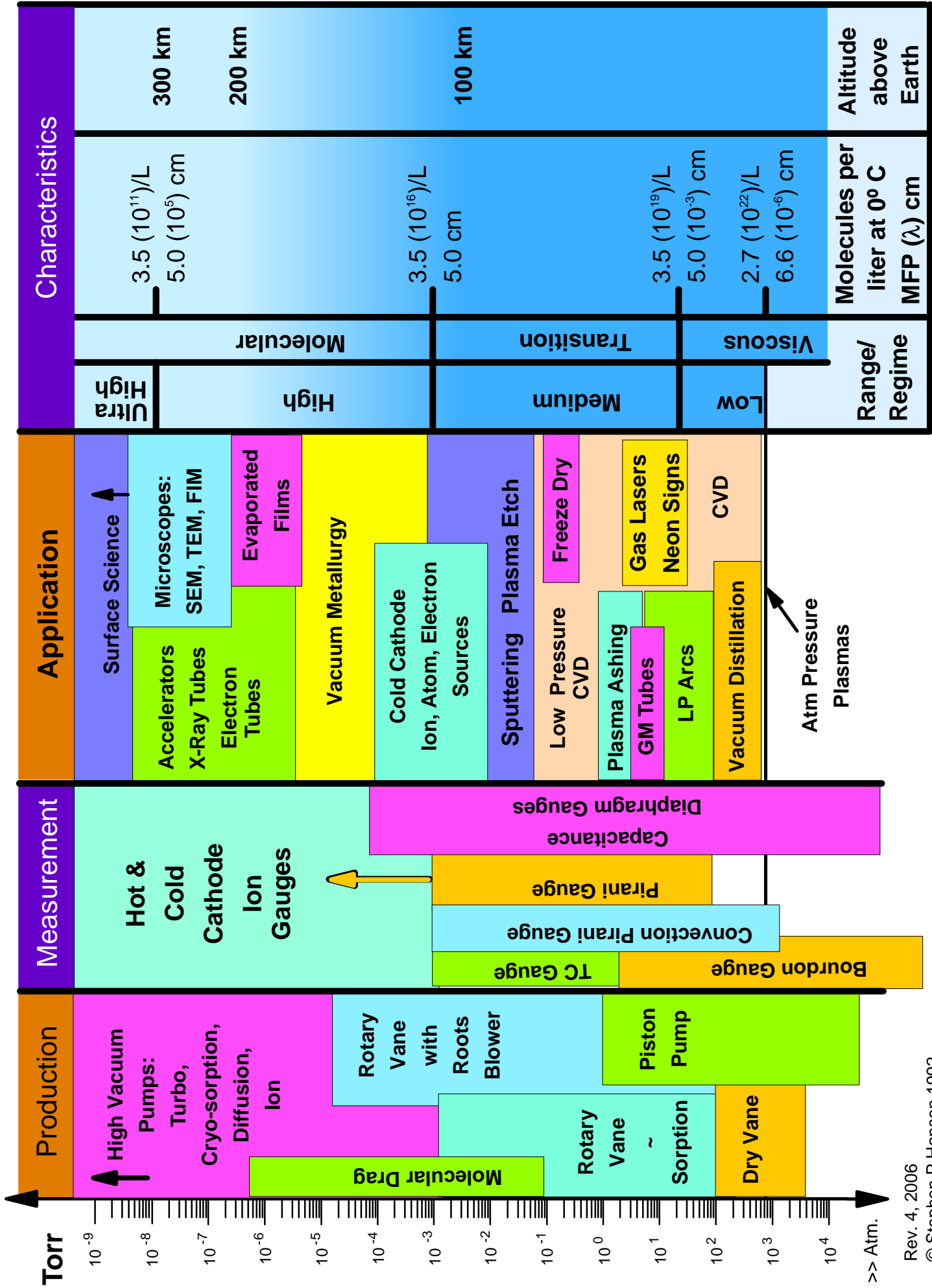
Temperature:	0 °C
Pressure:	760 Torr

This brief descriptive tutorial is primarily intended to serve as a supporting document for the VPAL-A and VPAL-B educational products that are provided by The Science Source of Waldoboro, Maine. It should be used in conjunction with the VPAL exercises and a standard physics textbook.

Used as a stand-alone text, the author has endeavored to provide a compact document that touches on the critical aspects of vacuum technology that should be understood by anyone using vacuum equipment.

For those who are interested in further reading, a list of recommended vacuum texts is provided in the Appendix.

The figure on the next page is a map of the various methods used to produce and measure vacuum along with a selection of applications and physical attributes of vacuum. Vacuum represents a range of conditions all dependent upon the density and motions of the gas molecules that comprise the environment. There is no one pump nor gauge that is usable across the continuum. Likewise, each vacuum process has a limited range of operating pressures.



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## **Appendix**

**Section 1**  
**Vacuum Principles**



# Chapter 1      Pressure and Pressure Measurement

## Pressure

Ask a variety of people what “normal” sea-level atmospheric pressure is and you will get answers such as 14.7 pounds per square inch, 760 Torr, 29.92 inches of mercury, 1 bar, 100,000 Pascal, etc. What do these units mean and where do they come from?

Just before the middle of the 17<sup>th</sup> century, Evangelista Torricelli (1608-1647) became the first person to “measure” atmospheric pressure. He did this with his invention, the mercury barometer. This device consists of a glass tube, nearly a meter in length, that is sealed at one end. To set up the barometer, the tube is first filled with mercury and then inverted with the open end placed in a cup of mercury. What is observed is that the mercury column drops to some level in the tube. Since the space at the top of the tube was originally full of mercury (that is, free of air), the space now contains a reasonably good vacuum. The actual height of the mercury column in the tube then represents the weight of mercury that can be lifted by the difference in pressure between the pressure exerted on the mercury in the cup by the atmosphere and the pressure at the top of the tube (about zero).

At sea level, Torricelli observed that the typical height of the mercury column was about 760 millimeters. This would rise or fall depending upon the weather (hence the application of the barometer in meteorology). Other investigators found that the height of the column decreased when a barometer was carried up a hillside.

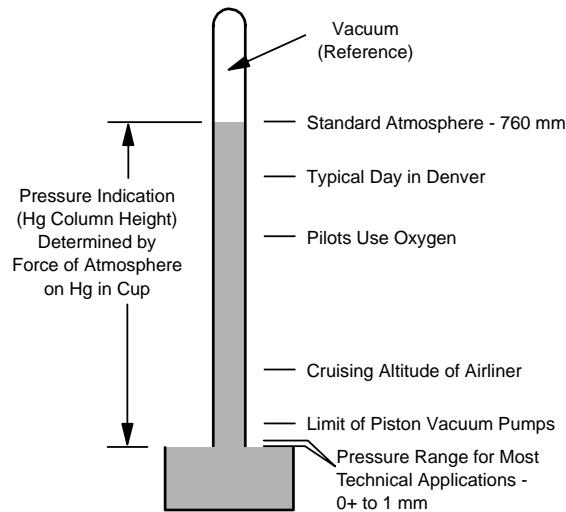
Analyzing this a bit more, we can observe three things:

The barometer is measuring the pressure difference between the ambient atmosphere and a zero pressure reference. (Of course, the reference is not quite at zero pressure but it is at a pressure that is negligible as compared to what is being measured.) Thus, the pressure measurement is termed *absolute* because it is referenced to zero. If the barometer were carried toward space, the column height would drop to zero as the atmospheric pressure dropped to zero.

It is the force of the atmosphere on the mercury as applied over the surface area of the cup, i.e. the force per unit area that is causing the mercury in the column to rise. This is the proper definition of pressure. Devices such as barometers where the pressure is measured by the action of the gas on a moveable element are termed direct gauges. The moveable element can be a liquid column, a diaphragm, a bellows, a coiled tube or anything else that will move in response to the pressure of the gas.

The reading is the height of the column (length) for the particular working material (mercury). The proper way of expressing the reading is mmHg for millimeters of column height for the given working fluid. If water, which has a specific gravity about 13.5 times lower than mercury were used, the column height for the same

pressure would be that much more, i.e. about 30 feet. That brings us to the issue of pressure measurement units. We'll examine these in the next section.



**Figure 1.1** The barometer

## Pressure Units

For the reasons noted above, the traditional way to express pressure in a vacuum system is in terms of the height of a column of mercury that is supported by the pressure in the system. This developed from the use of the barometer and later mercury column manometers (such as the McLead gauge) as the primary way of measuring sub-atmospheric pressures. To summarize, one standard atmosphere of pressure will support a mercury column 760 millimeters (29.92 inches) high. One millimeter of mercury (mmHg) is the same as 1 Torr, a unit named in honor of Torricelli. A thousandth of 1 mm Hg is referred to as 1 micron Hg or, in more current terminology, 1 milliTorr (mTorr). None of these is actually a scientifically proper measure of pressure.

As stated before, pressure is properly defined as the force per unit area that the gas exerts on a surface. Depending upon what set of units we are working with or are accustomed to we can, for example, express 760 Torr as equivalent to 14.7 pounds per square inch (psi). If we are using the cgs (centimeter-gram-second) system, the same pressure is about 1.03 bar (units of dynes/sq. cm). In the *SI (Système International)* system, units that are based on the meter and kilogram are used. Here, pressure is stated properly in terms of newtons/sq. meter or Pascal (Pa). In this system, 1 standard atmosphere is expressed as 101,300 Pa.

In the USA we tend to stay with torr as the unit has a long history and people are familiar with it. Just remember that, properly speaking, the torr isn't really a unit of pressure.

## Pressure References

Since we live our lives at this thing we call atmospheric pressure, we have a perception that vacuum is anything less than 1 atmosphere and pressure is anything above. Thus, to a person who lives in Boston, anyone who lives in Denver is living in a vacuum. (Normal atmospheric pressure at Denver's mile-high altitude is about 660 Torr.) When we express pressure with reference to our normal, but varying, atmospheric pressure we are using *gage* pressure. Gage pressure simply tells you how far away you are from the ambient reference. Common dial gauges are an example of an atmosphere referenced gauge. In process systems, gage pressure is useful in applications such as loadlock monitoring and control where it is important or necessary to open the chamber right at the prevailing atmospheric pressure.

As we've seen, the barometer measures absolute pressure. The perfect zero pressure reference would be a vessel with absolutely no gas molecules in it. If even one molecule is introduced, we have some pressure, albeit very small. Absolute pressure is generally used when it is important to know exactly how much "stuff" is in a given volume. Molecular densities, hence absolute pressure, are very important in vacuum apparatus and process tools as many properties of the vacuum environment are related directly to absolute pressure.

Differential pressure is another term that is often encountered. This is simply the pressure difference that exists between any two points in a system. For example, the differential pressure across an in-line filter might be monitored to insure proper air flow or to provide a warning in the event of clogging.

## How Pressure is Created

Pressure, as previously noted, is defined as the force exerted by the gas over the area that the force is being exerted. This gives us force per unit area. The force arises from the fact that molecules are always in motion. First we'll look at molecular velocity.

If we were to record the velocities of the molecules of a specific gas at a specific temperature, we would be able to plot a velocity distribution of the form shown in Figure 1.2 on the next page (curve A). What this says is that for any collection of molecules of the same type of gas, there is a distribution of velocities. This distribution is known as the Maxwell speed distribution.

If we increase the temperature of this particular gas sample, the average velocity will increase and the curve will shift to the right. Cooling will have the opposite effect. Gases with a lower molecular weight will have a higher average molecular speed distribution than that of a heavier gas. These effects are depicted in curve B.

The pressure that is exerted by a contained volume of gas results from the force of the molecules and is therefore a function of velocity and mass. Since average velocity varies inversely with mass, the resultant force is independent of the gas type. Therefore, equal numbers of molecules of any type in a given volume at the same temperature will exert the same pressure.

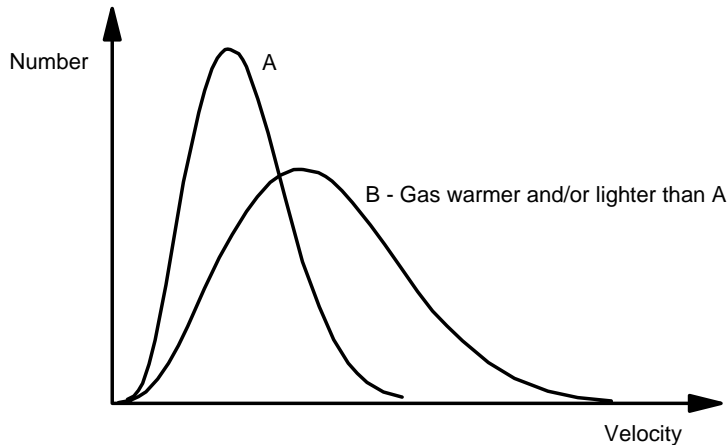


Figure 1.2 Molecular velocities

## Vacuum Pressure Measurement

Torricelli's barometer (and derivative liquid column gauges) are classified as direct gauges. That is, they respond directly to pressure as force/unit area. A frequently seen example of a direct gauge is the Bourdon tube gauge as exemplified by the common "dial" gauge that is used for compressed air applications, tire pressure measurement, etc.

While the barometer and related liquid column devices are great examples of the direct gauging principle, they have limited utility in today's industrial and scientific applications: resolution is poor, readings are intermittent in some arrangements, there are no provisions for interfacing with electronic controllers and mercury is hazardous.

As a result a number of differing types of gauges have been developed over the past century. The most prevalent are the so-called *indirect* gauges, named because they don't actually measure pressure but, instead, measure a physical property of the gas that changes predictably with pressure. Indirect gauges are widely used as they are (relatively speaking) fairly simple in construction as compared to today's electronic output direct gauges, as exemplified by the capacitance diaphragm gauge or CDG.

The table on the next page illustrates the operating principles and associated ranges of some of the more common vacuum gauges. Some of these are best used as rough indicators of pressure or they may be used as input elements of automatic control systems where fast response and a high degree of accuracy are required.

In this grouping, other than the Bourdon gauge, only the CDG (also referred to as the capacitance manometer) reads true pressure. This, plus the inherent accuracy, repeatability and speed of response of CDGs, makes them the preferred instrument for process pressure measurement and control.

Gauge Type	Class	Reference	Measurement Principle	Range	Accuracy
Bourdon	Direct	Atmosphere	Deflection of coiled tube	Low vacuum: 0-30 inches Hg	5% of Reading
Thermocouple	Indirect	Absolute	Thermal conduction of gas from heated filament. Thermocouple measures temperature of filament at constant current. Electronics relates temperature to pressure for a given gas type.	Medium vacuum: 1 mTorr to 1 Torr	15-25% of Reading. Accuracy will vary over the range.
Pirani	Indirect	Absolute	Thermal conductivity of gas. Power to a filament is varied so as to maintain a constant filament temperature. Electronics relates power to pressure for a given gas type.	Medium and low vacuum. Some will work to atmosphere and/or down to $10^{-5}$ Torr. Widest range in a single gauge.	10-25% of Reading. Accuracy will vary over the range. Typically 5% repeatable.
Ion Gauge	Indirect	Absolute	Number of gas ions produced by a constant flux of electrons. Electronics relates ion current to pressure for a given gas type.	High to ultrahigh vacuum: $<10^{-3}$ Torr	10-25% of Reading.
Capacitance Diaphragm Gauge (CDG)	Direct	Absolute, Atmosphere or Differential	Metal foil diaphragm and rigid conductor form a variable capacitor. Pressure causes diaphragm to curve, electronics relates curvature to pressure.	High vacuum ( $10^{-5}$ Torr) to atmosphere for the family. Any one CDG can cover 3 decades of pressure.	0.05% to 0.5% of Reading depending upon grade.

### Thermal Conductivity Gauges - Thermocouple and Pirani

Thermal conductivity-based gauges represent a large class of indirect gauge. These depend upon the variation of thermal conductivity of a gas through the medium vacuum range and, in some cases, up to atmospheric pressure. The two common thermal conductivity gauges, the thermocouple gauge and the Pirani gauge, both date from the early 20<sup>th</sup> century.

The lower limit of usefulness is determined by the very low thermal conductance of a gas at the lower end of the medium vacuum range and the increasing background thermal losses caused by radiation from the heater and conduction through the supporting wires and leads. This usually occurs at about 1 milliTorr.

The **thermocouple gauge** is the simplest of the indirect gauges. The usual analog or digital readout indicators are inexpensive and the construction of the gauge itself, usually with a nickel plated steel housing, is rugged and low-cost.

The mass of the thermocouple junction, the constant current heating method and the lack of temperature compensation provide for a slow response time (greater than one second) and poor temperature stability.

The simplicity does make for a low cost gauge that performs best as an indicator of vacuum line pressure where high accuracy is not required.

The **Pirani gauge** was developed by Manfred von Pirani in 1906.

All of today's Pirani gauges are operated in a constant temperature mode. While the circuitry is more complex than that of the variable temperature mode thermocouple gauge, the response of the Pirani gauge is faster (as fast as 10 ms) and the usable range is extended to about 100 Torr. With a typical filament temperature of 120 °C, the gauge is compatible with some otherwise reactive gases.

Parameters affecting the sensitivity of a Pirani gauge include the diameter of the filament wire, the length of the wire, thermal coefficient of resistance of the wire, thermal and emissive properties of the wire, operating temperature, properties of the support wires and diameter of the tube.

In the upper region of operation of a Pirani gauge the sensitivity declines as a hot, stationary boundary layer forms around the filament. If the filament is positioned coaxial to the shell of the tube and mounted horizontally, convection currents will form at about 100 Torr. By making use of the two modes of operation (normal Pirani mode plus convection mode), the convection Pirani gauge can operate over a six decade range of 1 milliTorr to 1000 Torr. Recently introduced micromachined Pirani sensors have extended the low end to  $10^{-5}$  Torr.

### **The Ionization Gauge**

The ionization, or ion gauge is another type of indirect gauge. The property it measures is the number of molecules of gas that are ionized (i.e. which acquire positive charges by having one or more electrons knocked away from them) by a current of electrons passing through the gas. The number of molecules that become ionized is dependent upon the number of molecules available to be ionized (determined by the molecular density of the gas), the strength of the electron current and the type of gas.

Electrons may be generated by thermionic emission from a filament or from a high voltage discharge that is sustained between two electrodes. The former type is called a hot cathode gauge, the latter a cold cathode gauge.

In practice, the source of electrons is a heated filament of a refractory material such as tungsten. The electron current is established and directed by a positively biased grid, usually a coarse mesh or spiral of fine wire. Ionization takes place within the volume of the gauge and the created ions are attracted to the negatively biased collector.

A number of electrode configurations are in use. The most common for general high vacuum purposes is the Bayard-Alpert configuration.

### **Gas Sensitivities in Indirect Gauges**

Different gases have varying thermal conductivities. Gases that are lighter than air, such as helium, are better thermal conductors because the molecules travel faster. Gases that are heavier than air, such as argon and xenon, are relatively poor thermal conductors. Since thermocouple and Pirani gauges work on the principle of thermal conductivity, their readings will change with gas type.

The errors are particularly large at higher pressures. For some gauges, usually Piranis, the manufacturer may provide calibration curves for various pure gases. Corrections for a few common gases (helium, argon) may also be incorporated in the electronics of the readout. When calibrating a thermal conductivity gauge (or any other kind of indirect gauge), the gauge must be calibrated with the gas that the gauge will be used with.

With regard to ion gauges, the pressure indication produced is dependent upon the quantity of ions that are generated and collected. For a given set of conditions the ion yield is proportional to pressure. The ion yield in an ion gauge is dependent upon two factors:

**Gas type:** for a given electron energy, it is more difficult to ionize the smaller atoms which have tightly bound electrons than it is to ionize heavier atoms. Thus a given density of helium atoms will produce fewer ions than the same density of argon atoms.

**Incident Electron Energy:** If the electron energy is low, it is more difficult to ionize the gas. However, as electron energies are increased, there is a diminished probability of an electron-molecule interaction. An electron energy has to be selected that optimizes the yield of ions. For the hot cathode gauge, this is usually in the range of 100 electron volts (eV).

### **The Capacitance Diaphragm Gauge (Capacitance Manometer)**

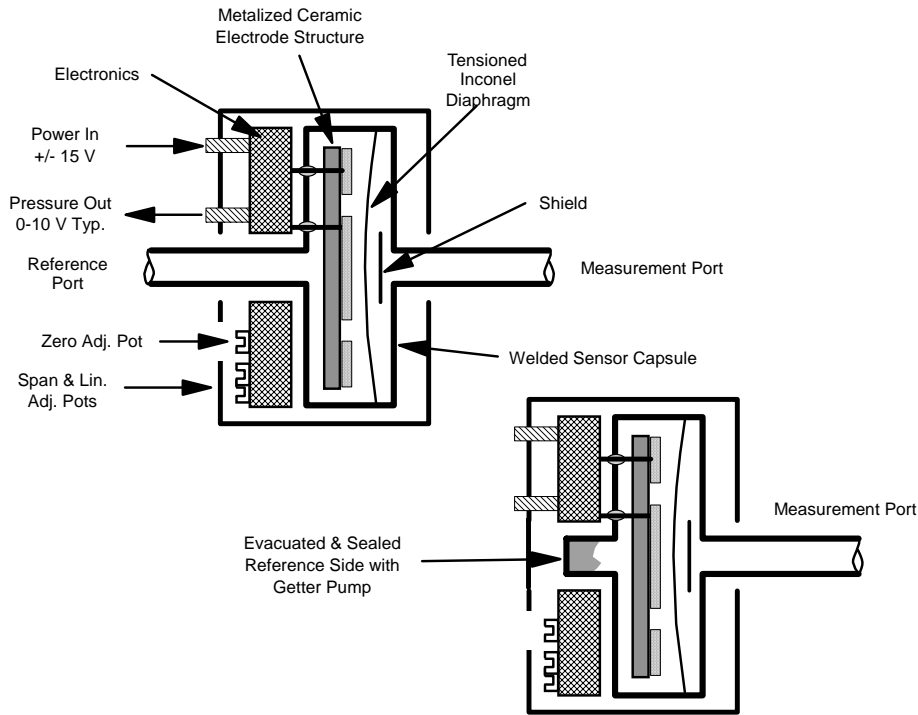
For process and other applications where high accuracy and gas independence are required, the CDG reigns supreme in today's lineup of vacuum gauges.

CDGs are stand-alone transducers that typically require a  $\pm 15$  volt power supply and deliver a 0-10 volt pressure signal that is directly proportional to pressure. In addition to this scaled analog output, most manufacturers are also providing CDGs with a variety of digital interfaces. Common protocols include RS-485, DeviceNet, Profibus and ethernet.

CDG's are ideal for process measurement and control applications in that they are fabricated from non-corrosive materials, provide accurate pressure readings independent of gas type and are very accurate and repeatable.

While the origins of the CDG can be traced to the late 1920s the first commercial device of this type was introduced in 1961. Commercialization was initially driven by research applications followed quickly by the increasing demands of the semiconductor industry. While any given process vacuum system may have dozens of thermocouple, Pirani or ion gauges, there is almost always one CDG on the process chamber to provide precision in measurement and control.

The figure on the next page shows the basic concept. The sensor consists of a welded capsule which is divided by the diaphragm. The pressure of the gas is applied to a tensioned diaphragm of inconel, an alloy chosen for its mechanical and chemical stability. Instead of measuring the deflection of the diaphragm by mechanical linkages, its changing curvature is sensed electrically by means of a capacitance bridge. (The deflection of the diaphragm is very slight - a few thousand Angstroms.)



**Figure 1.3** Capacitance diaphragm gauges: differential (top), absolute (bottom)

Behind the diaphragm is a ceramic substrate upon which there is a metal pattern in a form similar to that of a circular bull's eye surrounded by a ring. As a result of this geometry, between each electrode and the diaphragm there is a capacitance that is defined by the areas of each electrode, the dielectric constant of the intervening gas (usually 1: air or vacuum), and the separation of each electrode from the diaphragm. Each of these variable capacitors is a leg in a bridge circuit. When the diaphragm is flat, the bridge is balanced and there is an output of 0 volts. However, as the diaphragm becomes curved, the bridge is unbalanced and the degree of imbalance is reflected in the output voltage.

The displacements of the diaphragm are small and subtle. Total displacements over the range of the sensor are on the order of a few thousands of angstroms. The circuitry can resolve displacements on the order of the diameter of a hydrogen molecule.

In the case of a differential CDG, both sides of the diaphragm are brought out through tubulations. To make the CDG into an absolute gauge, the reference side is pumped by the manufacturer to high vacuum and then sealed off. In order to maintain high vacuum over the life of the instrument, a getter is fired during the pinchoff process, just as is done with vacuum tubes. The getter scavenges any gas that might evolve after pinchoff.

When a CDG's measurement port is open to any environment where the pressure is greater than the pressure within the reference volume, the diaphragm will be curved toward the electrodes and the output of the gauge will be some positive voltage. So, how do we translate that output to pressure?



Each CDG is specified according to its full scale (FS) range and it will be able to measure pressure for some number of decades below that full scale. As examples:

A 1000 Torr manometer will produce a 10 volt output at 1000 Torr, 1 volt at 100 Torr, 0.1 volt at 10 Torr, and so forth.

A 1 Torr manometer will produce a 10 volt output at 1 Torr, 1 volt at 0.1 Torr, 0.1 volt at 0.01 Torr, and so forth.

From this it is apparent that, at pressures significantly below the full scale pressure, the output voltage will fall to very low values. As a consequence of this, there is a resolution limit that is typically 1 part in 10,000. Thus the resolution of a 1 Torr FS manometer is 0.1 milliTorr, corresponding to an output of 1 mV.

The pressure range over which the manometer will provide a useful (accurate and repeatable) is smaller than the four decades between the resolution limit and the full scale rating. The reading range, where the manometer is used for measurement, is about 3-1/2 decades. When the manometer is used in a pressure control system, there is a control range that covers about 2-1/2 decades. This narrower range (50 mV floor) helps to assure immunity from system noise.

Families of capacitance manometers are manufactured with overlapping ranges. Some applications and processes may require multiple manometers

One fact of life with CDGs is that the zero has to be adjusted upon installation. It should also be checked on a regular basis. There is always an accessible zero adjust potentiometer on the instrument. All you have to do is pump the measurement side to a low enough pressure such that the output *should* be zero volts - usually about 5 decades below the full scale rating. Then you turn the pot until the output *is* zero volts. After doing this, all of the readings to the full scale should be within the manufacturer's specification. Zeroing does not affect the calibration of the instrument - it's analogous to zeroing a set of dial calipers before use. Under no circumstances should a CDG be adjusted to correspond to the reading of a less accurate gauge such as a Pirani or thermocouple gauge.

Zeroing differential gauges is a simpler process - just connect the ports with a piece of tubing to assure the same pressure across the diaphragm and set the zero pot.

Over long periods of time other parameters such as the full scale range (span) and linearity will wander out of spec. Then it's time to send the device back to the manufacturer for recalibration. Although these pots are accessible (usually you have to pry out a plug), they should never be touched unless you know what you are doing and have a full calibration stand. The generally recommended calibration period is 1 year.

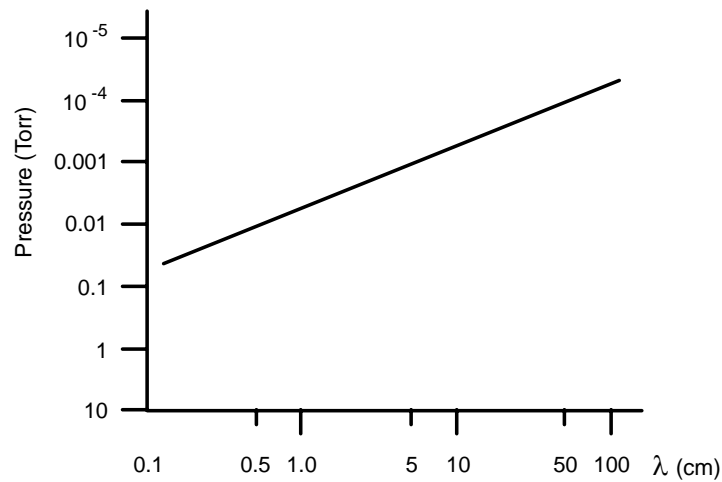
## Chapter 2 Important Vacuum Terms and Concepts

### Mean Free Path and Molecular Density

Mean Free Path ( $\lambda$ ) is defined as the average distance traveled by molecules between collisions. For air at standard temperature:

$$\lambda = 0.05/P$$

Where  $\lambda$  is in cm and  $P$  is pressure in Torr. The constant will change depending upon gas type or conditions.



**Figure 2.1** Mean free path

The long mean free path at low pressures is a key enabler for devices such as vacuum tubes and particle accelerators as well as for processes such as vacuum coating where microscopic particles such as electrons, ions or molecules must traverse considerable distances with minimal interference.

The lengthening of the mean free path is a natural consequence of decreasing molecular density (lower pressure). The following table gives the molecular density at standard temperature at several pressures. Note that there is a direct relationship between density and pressure.

Pressure (Torr)	Molecules/Liter
760	$2.7 \times 10^{22}$
1	$3.5 \times 10^{19}$
$1 \times 10^{-3}$	$3.5 \times 10^{16}$
$1 \times 10^{-8}$	$3.5 \times 10^{11}$
$1 \times 10^{-12}$	35 million

## Flow Regimes

Breezes and floating dust particles are common features of the air environment that we live in. Gases at very low pressures behave very differently from gases at normal pressures. As a reduction in pressure occurs in a vacuum system, the gas in the system will pass through several flow regimes. At higher pressures these include *viscous* flow where the molecular mean free path is substantially shorter than the size of the system's lines and chambers. Viscous flow may be either *laminar* where the flow is regular with no eddies (think of a wide river with a gentle flow), or *turbulent* where the flow is irregular (put a large rock in the middle of that same river). Turbulence is to be avoided in vacuum systems where cleanliness is important (for example, in semiconductor process equipment) as turbulent gas will pick up dust particles and deposit them on the product being processed.

Moving deeper into the vacuum environment, *molecular* flow occurs when the molecular mean free path exceeds the tubing diameter. Here the molecules behave statistically without regard to what their neighbors are doing. Here molecules can and will move against the general flow simply because there is no "herd" effect to direct the flow in one direction. Most of the flow will be from high pressure to low but only because there are more molecules present upstream and the statistics say that, in general, the motion will be from the higher density region to the lower. Air currents do not exist in molecular flow and, even when a line is connected to a large vacuum pump, small particles of dust will not move.

A third flow region, sometimes called *Knudsen* but more frequently *transition* flow, exists between the viscous and molecular regimes. This regime generally coincides with the medium vacuum range.

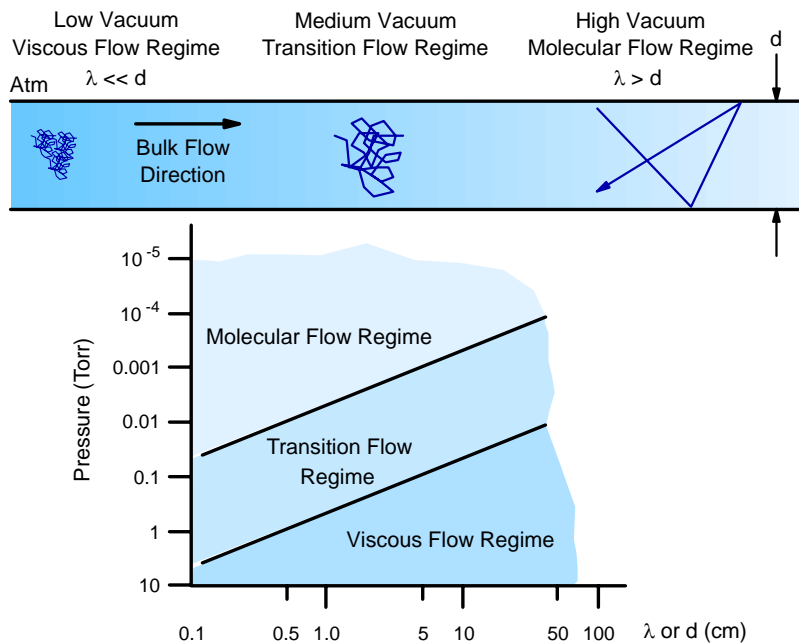


Figure 2.2 Flow regimes

Which flow regime the gas is in depends upon several factors including tube diameter and pressure. As a rule of thumb, when the ratio of the average mean free path in a tube to the diameter of the tube is less than 0.01, the flow is viscous. When this ratio is greater than 1 the flow is molecular. The ratio  $\lambda/d$  is referred to as the *Knudsen number*,  $Kn$ .

## Conductance

Conductance is defined as the volume of gas, at the prevailing pressure, that passes through a line or other system element per unit of time. Since conductance is volumetric it does not define how much mass is moving in a line. We'll address mass flow later.

The conductance of any element is a function of a number of parameters including the specific properties of the gas (viscosity, etc.) as well as system conditions (pressure, line diameters and lengths, and so forth). The following discussion is fairly cursory but several key rules and guidelines will be presented.

### Conductance in Viscous Flow

For the viscous flow regime, as would be the nominal case for mechanically pumped roughing lines we have, for room temperature nitrogen and a long, straight tube:

$$C = 188 \times d^4/L \times P_{ave} \text{ liters/sec}$$

where  $d$  is the line diameter in cm,  $L$  is the line length in cm and  $P_{ave}$  is the average pressure in the line in Torr. 188 is a computed factor that takes into account the physical properties of nitrogen.

The important points here are that the conductance is strongly influenced by diameter and much less so by length. Also, the conductance is a function of line pressure. Furthermore, the lower the pressure, the lower the conductance will be.

### Conductance in Molecular Flow

Because of the statistical nature of molecular flow and the very low absolute pressure gradients, pressure is not a factor in this flow regime where, for example, a high vacuum pump would operate. Here we have, for room temperature nitrogen and a long, straight tube:

$$C = 12.3 \times d^3/L \text{ liters/sec}$$

Note that in molecular flow the pressure term is not present. (The molecules are moving randomly, driven by statistics. There is no pressure gradient as such in molecular flow.) Line diameter remains the dominant factor but, for any given line, the conductance is lower in molecular flow than in viscous flow.

An example would be a 2 inch (5 cm) inlet high vacuum pump which has a specified inlet pumping speed of 100 liters/sec. The pump is connected to a small experiment chamber through 60 cm of 2.5 cm diameter tubing. Inserting the numbers, we find a line conductance of only 3.1 liters/sec. This may be adequate for the small chamber but it certainly throttles the pump significantly. If a 5 cm line of the same length were substituted, the conductance would rise to 25 liters/sec.

## Basic Guidelines for Optimizing Line Conductance

Lines should be as short and large as possible.

Diameter has the strongest influence

For any given line in viscous or transition flow, the lower the pressure, the lower the conductance

Conductance is lowest, but constant, in molecular flow

One additional point that is not entirely obvious from the discussion in this section is that bends in high vacuum lines should be avoided. When a wandering molecule hits a bend, it is nearly as likely to return along the path that it came in on as opposed to continuing past the bend. Bends also have a negative effect in the viscous flow regime but it is not anywhere near as significant.

## Avogadro's Law and Mass Quantity

Avogadro's Law states: "Under equal conditions of temperature and pressure, a given volume will contain the same number of molecules regardless of the type of gas." When quantified, we find that a mole ( $6.02 \times 10^{23}$  molecules) of any gas will occupy a volume of 22.4 liters under standard conditions of temperature and pressure (0 °C and 760 Torr). From chemistry we know that a mole of any material has a mass in grams equivalent to the atomic mass of the particular molecule as measured in atomic mass units (AMU).

As examples, a 22.4 liter vessel of nitrogen (atomic mass 28) at standard conditions will have a mass of 28 grams. The same container of helium will contain 4 grams of that gas.

If the temperature and pressure in the vessel are not standard, then the ideal gas law is invoked to adjust for the deviation from standard conditions. If the gas is at a higher pressure, but at standard temperature, there will be a proportionally larger number of molecules. If the gas is at a higher temperature, but at standard pressure, then there will be fewer molecules. (One caveat: make sure that temperature adjustments are made with Kelvin (absolute temperature) units.)

The bottom line here is that if volume, temperature and pressure are specified, it is then possible to determine how many molecules are in the volume. This gets us to terms that we can use in vacuum practice, as we usually talk in terms of volumes, pressures and temperatures.

For vacuum purposes, the above relationship is normalized to more convenient volume units, i.e. liters or cubic centimeters. What we end up with are *standard condition* volumes.

Using liters, the standard condition volume term is the *Torr-liter*. This represents the molecules contained in a one liter volume at a pressure of 1 Torr. Not stated but understood is a standard temperature of 0 °C. Adjusting from the relationship of 22.4 liters of gas at 760 Torr and 0 °C equals  $6.02 \times 10^{23}$  molecules, one Torr-liter would then contain about  $3.5 \times 10^{19}$  molecules.

Using cubic centimeters, the standard condition volume term is the *standard cc* (std. cc or scc). This represents the number of molecules contained in a 1 cc volume at a pressure of 760 Torr. A standard temperature of 0 °C is also understood. Going back to the pressure/temperature/volume relationship for a mole of gas, a std. cc contains about  $2.7 \times 10^{19}$  molecules.

The following table provides a summary:

Measure	Volume	Pressure	Quantity of Molecules
1 Mole at STP	22.4 liters	760 Torr	$6.02 \times 10^{23}$
Torr-liter	1 liter	1 Torr	$3.5 \times 10^{19}$
Std. cc	1 cc	760 Torr	$6.02 \times 10^{19}$

### Throughput (Standard Condition Volumetric Flow)

As noted in the paragraph on conductance, conductance is purely volumetric and does not account for gas density. In vacuum practice there is a term called *throughput* which is usually abbreviated as  $Q$ . Throughput is the mass quantity of gas that passes through a system element in a specified period of time. In other words it normalizes conductance (e.g. in liters per second) to a standard pressure.  $Q$  is the number of standard condition volume units that flow in a given period of time. The common units are Torr-liters/second (T-l/sec) and standard cubic centimeters per second (Std. cc/sec) or minute (sccm).

The practical convention for which set of units you pick is usually based on the application.  $Q$  within the vacuum system is usually specified in terms of T-l/sec since pressures are usually measured in Torr and volume in liters. Leaks are usually quantified in terms of Std. cc/sec.  $Q$  from intentional leaks (i.e. flow control devices) is usually specified as Std. cc/minute (sccm) or, for high flows, Std. liters/minute (slm). Some handy conversions are:

$$1 \text{ Torr-liter} = 1.32 \text{ Std. cubic centimeter (scc)}$$

$$1 \text{ Torr-liter/sec} = 79 \text{ Std. cubic centimeters/min (sccm)}$$

Another term that is used for leak rates is Atmosphere cc/sec (or Atm. cc/sec). This is about equivalent to sccm except that the pressure reference is the prevailing ambient pressure, usually near 760 Torr unless you live in the mountains.

Since all of these terms relate to molecular quantity, therefore to the actual mass of the gas, standard condition volumes are also called *mass quantities* and standard condition volumetric flow is also called *mass flow*.

### Pumping Speed and Throughput

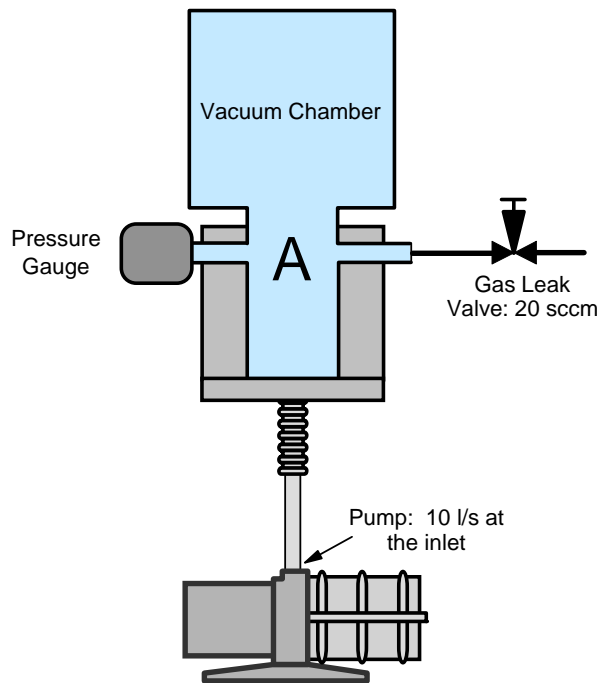
The speed of a pump ( $S$ ) is the volume of gas flow across the cross section of the tubing per unit time. It is the same as conductance except that conductance generally refers to passive elements, such as lines, in a system whereas the pump is an active participant in the action. The common units for  $S$  are liters/second. Throughput ( $Q$ ) is the product of pressure ( $P$ ) and speed ( $S$ ):

$$Q = P \times S$$

This is a very useful relationship in vacuum practice.

If you think of the vacuum system as an electrical circuit, throughput is like current flow and it is constant everywhere in the circuit. The various elements of the system (lines and pumps) are analogous to resistances except instead of voltage drops there are pressure differentials. In putting together a vacuum system you want minimal pressure differentials in the connecting lines and maximum throughput everywhere.

Consider the situation of Figure 2.3 below:



**Figure 2.3** Relationship between  $Q$ ,  $P$  and  $S$

The chamber has been pumped down to a steady state condition. The pressure gauge reads 2 Torr with the leak valve opened to admit 20 sccm of nitrogen. According to the manufacturer's literature, the speed at the inlet of the pump should be 10 liters/sec. We will assume that this is correct.

- What is  $Q$  at the inlet to the chamber in terms of Torr-l/sec?
- What is  $Q$  at the pump inlet?
- What is the speed  $S$  at the inlet to the pump line (point A)? Is it the same as the speed at the pump inlet? If not, why?
- What is the pressure at the inlet to the pump?
- Explain the effect of the pump line in this exercise.
- We want to raise the pressure in the chamber to 4 Torr. Suggest several methods to do this.

# Vapor Pressure

Consider an aluminum cup containing water at sea level and room temperature. The water molecules are exerting a pressure against the water to air interface. Under these conditions the pressure exerted by the water molecules is about 18 Torr. If the cup is left alone, the water will slowly evaporate. This occurs because some fraction of the molecules have enough energy to break through the interface and escape into the air as water vapor. The energetic molecules lower the average energy in the remaining water but, since the process is slow, the surrounding air keeps the water temperature at a constant value.

If we heat the cup of water, the molecules gain energy and move more quickly. The pressure that the water molecules are exerting at the interface increases and so does the rate of evaporation. Eventually the water is heated to a point where the water boils. At this point the *vapor pressure* of the water equals the ambient air pressure and water molecules are freely liberated.

We can accomplish the same task by reducing the ambient pressure around the cup. As the pressure decreases the evaporation rate increases. At some point the pressure is low enough for the water to boil at room temperature. If the water has remained at room temperature, this will occur when the pressure is lowered to 18 Torr.

A second observation that can be made from this is that nothing happened to the aluminum, at least over the temperature and pressure range that the experiment was run. However, aluminum also has a vapor pressure. Its characteristic is just very different from that of water. In fact, the large difference in vapor pressure characteristics is why the process of boiling water in a metal pan works. The water can boil away but the pan remains essentially unchanged. Figure 2.4 shows the vapor pressure characteristics of a variety of materials.

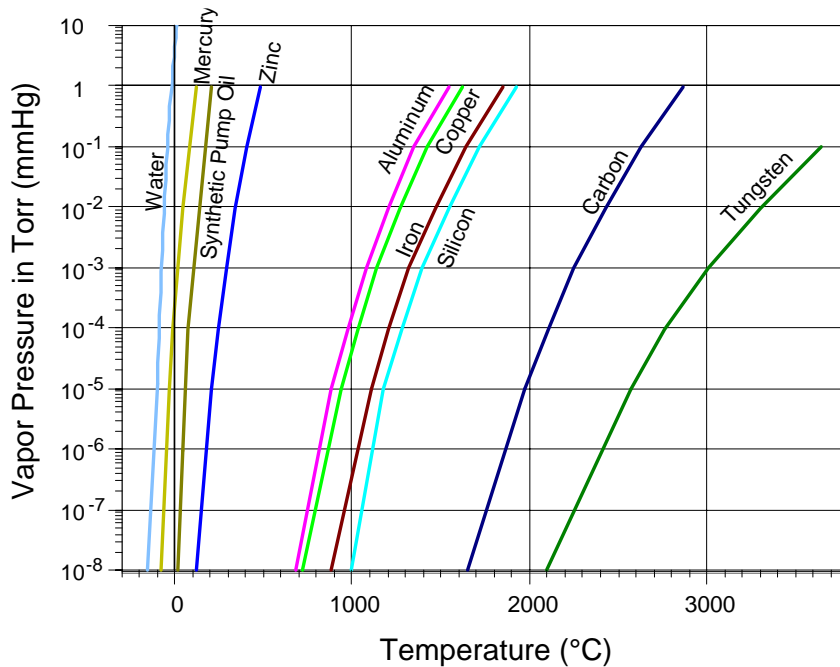


Figure 2.4 Vapor pressures of common materials



The chart shows clearly why some materials are to be chosen over others in the construction of vacuum apparatus. Low vapor pressure iron alloys, aluminum and copper tend to dominate. Brass is still used in some vacuum apparatus but it has to be avoided in high vacuum and high temperature applications because of its zinc content. At 1 milliTorr, the zinc will evaporate out of the alloy at under 300 °C.

Tungsten and carbon have very low vapor pressures along with high melting points. Mirror coating by means of vacuum evaporation is a long standing process. In this process aluminum is heated by tungsten filaments or held within a heated carbon crucible. The aluminum that had served as the container for our boiling water experiment now becomes the material that is boiled away, held by the even lower vapor pressure tungsten or carbon fixtures.

## Pumpdown Time Constant

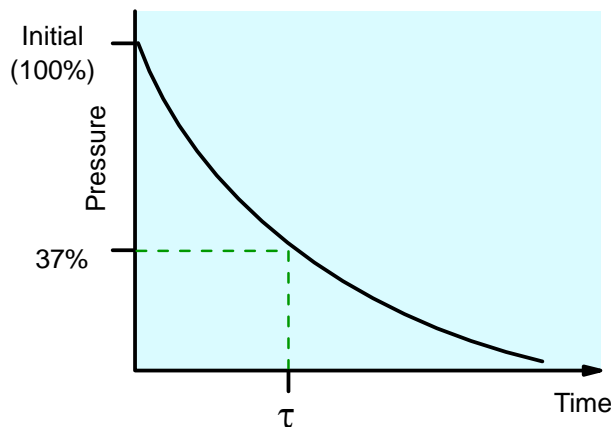
Pumping the air from a chamber is analogous to discharging a capacitor through a resistor. In the vacuum system, the chamber volume is like the capacitor and the pump and connecting lines is like the resistor. Just as there is a discharge time constant for the resistor and capacitor ( $\tau = RC$ ), for the vacuum apparatus the time constant is the chamber volume divided by the pumping speed:

$$\tau = V \div S$$

For a 10 liter chamber pumped by a 2 liter/sec pump, the time constant is 5 seconds.

This relationship is useful for determining the actual (effective) pumping speed of a system. Calculate the chamber volume, run the pumpdown curve and compute the speed from the results. It is also good practice to maintain a log of vacuum system performance. When something goes wrong there is a record of what things should be.

The exponential form of the pumpdown curve is shown in Figure 2.5 below.



**Figure 2.5** Pumpdown time constant

## Outgassing

Outgassing is the liberation of gases from the bulk of a material or from the surface of the material. Some materials are gassy by nature and it is best to avoid their use. Some polymers, such as the polyimides, absorb moisture but once they are dry (generally through baking) they are very acceptable vacuum materials provided that they are enclosed within the walls of the system..

Assuming that a system is tight, as the pressure gets lower most of the load is from gases evolving through outgassing. This becomes significant below pressures of around 100 mTorr. Outgassing will be the main limiting factor with regard to the *ultimate* (lowest) pressure which any particular system may reach, assuming that leaks are absent.

Water is one of the worst offenders in vacuum systems because our ambient air tends to have a high water content (humidity) and the polar nature of the molecule makes it rather “sticky.” When a vacuum system has been exposed to room air, water is the most difficult to remove and will comprise the bulk of the *residual* (remaining) gas in a pumped system.

If the construction of a system is appropriate to the practice, the accumulated layers of water vapor and other gases may be evolved by heating the system in an oven or with a hot air gun to a temperature of at least 150° C and usually more. For most applications down to about 10<sup>-5</sup> Torr this level of cleaning is not required. However, the system components should be kept clean (no fingerprints or other grime), dry and sealed off from room air. To achieve pressures below 10<sup>-8</sup> Torr a thorough baking of all components is required in addition to superlative vacuum practice.

The chart in Figure 2.6 below shows the outgassing characteristics of a number of materials. In Chapter 4 we will see how to apply this type of data.

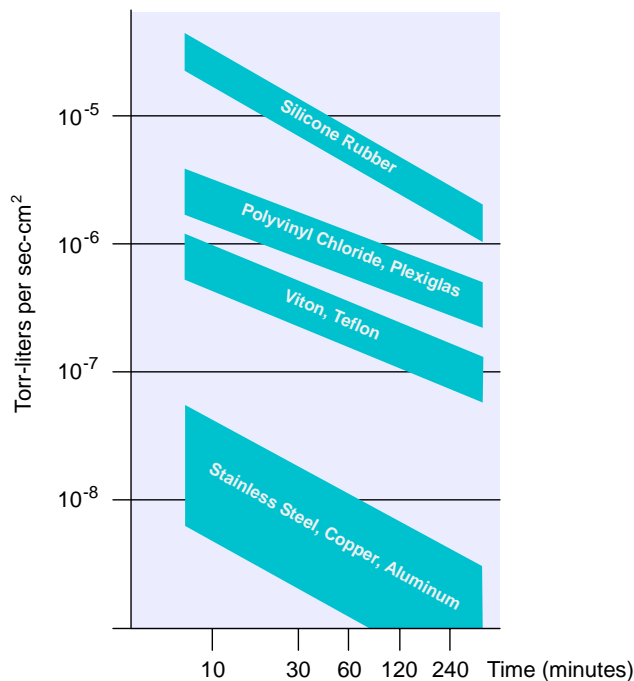


Figure 2.6 Outgassing rates

## Thermal Conductivity

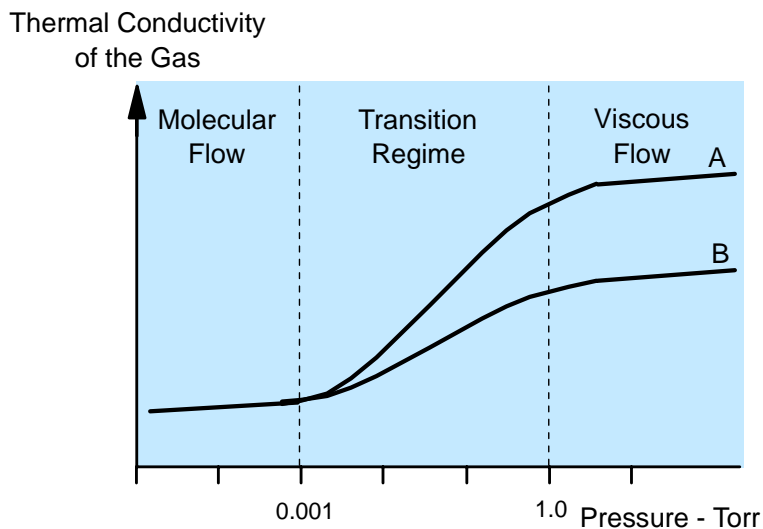
Molecules transfer thermal energy through collisions. Intuitively one would think that thermal conductivity would decline in concert with gas pressure (molecular density). However, molecule to molecule thermal conductivity is more of a function of the flow regime. Through the viscous flow regime, where collisions are frequent and over very short distances, thermal conductivity is essentially constant. Once we reach molecular flow, molecular collisions are nearly nonexistent and the gas becomes a very good thermal insulator. However, in the transition flow regime thermal conductivity varies almost linearly with pressure.

Gas type plays a role in this as well. The heavier molecules, such as argon, are poor heat transmitters while light gases, such as helium, are excellent conductors of thermal energy.

Some of the implications of this are:

- Dewar exploited the thermal insulating properties of high vacuum with his vacuum flask (Dewar flask or thermos bottle).
- The operating principle of the thermal conductivity gauges (thermocouple and Pirani) leverage the pressure varying thermal characteristics of gases.
- Helium at about 10 Torr pressure is delivered to the backsides of semiconductor wafers while the front surfaces are exposed to hot plasma processes. The cool helium is at low enough pressure that it doesn't leak into the main chamber but this pressure is still within the viscous flow regime so the cooling effect is as good as if the helium were at a much higher pressure.
- Double pane windows have argon between the glass panes to improve the insulating properties.

Figure 2.7 depicts the general characteristic of thermal conductivity vs pressure.



**Figure 2.7** Thermal conductivity vs pressure

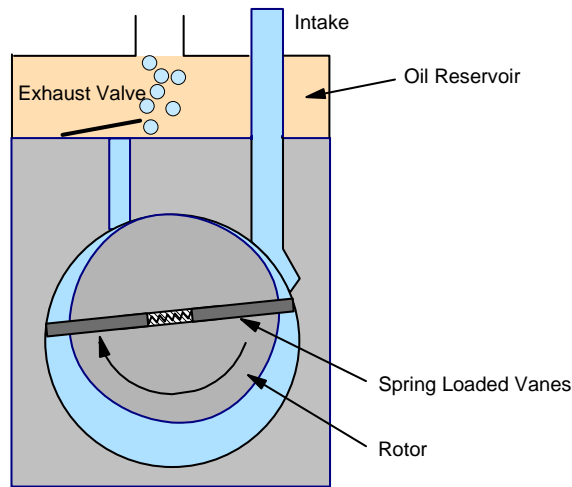
## Chapter 3 Pumps and Pumping Systems

### Mechanical Displacement Pumps for Low and Medium Vacuum

#### The Oil Sealed Rotary Vane Pump

At the heart of the typical vacuum system is a mechanical pump. While most common air pumps use reciprocating pistons to create pressure or vacuum, this type of pump is not suitable for high quality vacuum of the sort needed in scientific applications. The reciprocating motion, leakage past the piston and the dead space that exists above the piston all conspire to limit the level of attainable vacuum.

The type of pump that has dominated vacuum practice for most of the past 100 years is the oil sealed rotary vane pump. As shown in Figure 3.1 this type of pump has a rotating off-center cylindrical rotor that “sweeps” air through the housing in which the rotor is located. Air is kept from passing between the vacuum and pressure sides by means of a set of two vanes that are arranged across the diameter of the rotor. The entire mechanism is lubricated and sealed by immersion in an oil bath.



**Figure 3.1** Rotary vane vacuum pump

The mechanical pump may either be used by itself in applications where only a moderate degree of vacuum is needed or in conjunction with other types of pump where higher degrees of vacuum are required. In this latter case, the mechanical pump is referred to as a *fore* or *backing* pump. Here the purpose of the mechanical pump is to bring the pressure in the system down to a level which will permit the operation of the high vacuum pump.

One rotary pumping stage will achieve a vacuum of about 0.1 Torr in normal use. In order to get better vacuum it is standard practice to place two pumping stages in series, coupled by a common shaft. In the case of most industrial duty pumps, the specifications will usually state an ultimate

vacuum of 0.1 milliTorr. However, this level of vacuum is usually only attainable under ideal circumstances. A more realistic value is 5-10 milliTorr.

### Gas Ballasting and Blower Pumps

A standard feature of rotary vane pumps is a *gas ballast*. This is a low conductance leak valve that leads from the outside to the channel between the two pump stages. This valve is opened when pumping moisture laden air (as is often the case in initial pumpdown) or when pumping a process chamber that has a high content of other condensable gases. The function of the gas ballast is to dilute the condensate laden gas in the compression part of the cycle to help prevent the condensable from turning to liquid. Water and other condensable materials, when allowed to condense in the pump, will lead to degradation of the pump and oil.

Since the ballast is a leak, it will have the effect of limiting the pump's low pressure capabilities. If the condensable is only present during initial pump down, the gas ballast is closed once the moisture has been cleared. If condensables are continuously introduced, as is the case in some process applications, the gas ballast is left on. Examples of these processes include freeze drying and chemical vapor deposition (CVD). In these cases, if the poorer pressure performance cannot be tolerated, an auxiliary dry pump will be mounted on the inlet of the rotary vane pump. Typically these are of a Roots blower design as seen below. The blower pump has two counter rotating rotors that have small clearances between each other and the pump shell. There is no lubrication. Because of the clearances, on the order of a few thousandths of an inch, the pump will not work efficiently when exhausting to atmosphere but will work well in the medium vacuum regime.

Blower pumps will restore the ballasted wet pump's base pressure and will augment the pumping capacity (speed) of the wet pump.

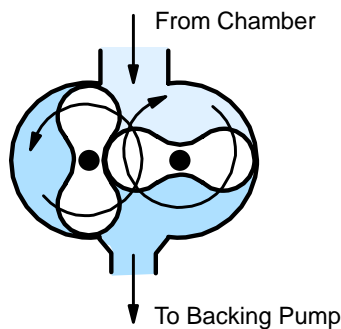


Figure 3.2 Roots blower pump

### Dry Mechanical Pumps

The oil sealed pumps are now losing favor within the industries that require very clean vacuum. This is because there will always be some back migration of oil vapor from a "wet" pump to the chamber. Common dry pumps include:

**Scroll pumps:** these use a pair of nested spirals. One spiral “wobbles” within the other stationary spiral essentially squeezing the gas along the spiral path.

**Blower pumps:** these are essentially a series of Roots blower stages (4 to 5 stages) on a common shaft. With this many stages it is practical to operate the pump directly to atmosphere.

**Diaphragm pump:** this is a dry piston style pump using elastomeric diaphragms arranged in one or two stages. These are low capacity pumps and are only capable of achieving a vacuum in the range of a few Torr.

## High Vacuum Pumps

### Momentum Transfer Pumps

Mechanical pumps are displacement devices and they only perform when the gas being pumped behaves like a fluid. Their efficiency is highest in the viscous flow regime. Performance will degrade through the transition region and the pump will cease to operate when it enters the molecular flow regime. This places a practical base pressure limit of about 1 milliTorr on this type of pump.

For high vacuum we have to look at other pumping mechanisms. The most common types of high vacuum pump use the principle of momentum transfer where molecules are directed away from the inlet of the pump toward the exhaust by giving them a mechanical “kick.”

It must be noted that high vacuum pumps do not “suck” gas into the inlet. In molecular flow there is no such action. A high vacuum pump is opportunistic: the molecules that happen to wander into the

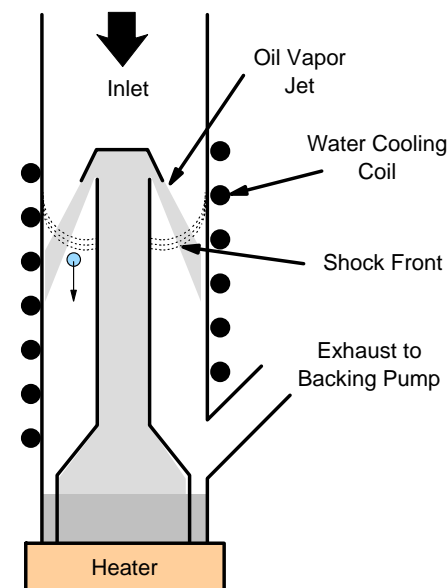


Figure 3.3 Diffusion Pump

pump in the course of their random walk are the only ones that the pump can act on. As a consequence the speed of the pump is directly related to the aperture of the inlet.

The original high vacuum pump is the oil diffusion pump. This pump, invented by Irving Langmuir in 1916, utilizes a jet of vapor that is generated by the boiling of hydrocarbon or synthetic oil. The high speed oil molecules force, by momentum transfer, the incoming molecules toward the outlet side of the pump. The everyday analogy is to just about any sport that is played by hitting a ball. The object is to impart energy to the ball to make it go very quickly in some specific direction.

An important operating aspect of the diffusion pump is that the supersonic stream of vapor creates a pressure rise which forms a steep and stable wave front - a shock wave. The shock wave acts as a seal across the inlet of the pump that prevents the pumped gas molecules from migrating (backstreaming) back toward the inlet. If the backing pressure is too high, the shock front will move closer to the nozzle and the wave will no longer extend across the width of the inlet. Thus the seal disappears and there will be a sudden increase in inlet pressure. The point at which this occurs is called the *critical backing pressure*. The critical backing pressure will usually be in the range of about 0.3 to 1.0 Torr for today's pumps and fluids.

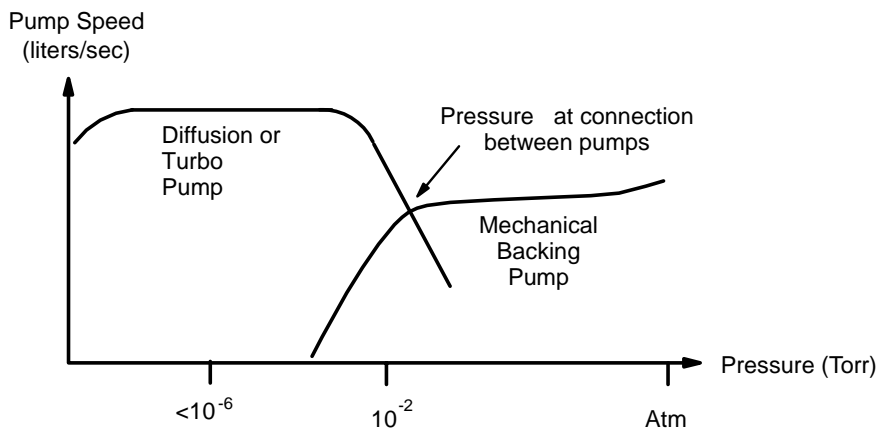
Unlike the illustration, most of today's diffusion pumps have 3 jets (stages), arranged along the length of the chimney. The multiple jets improve the compression ratio of the pump.

Diffusion pumps are simple (no moving parts) and robust. They are made in inlet sizes ranging from 2 inches to 4 feet. The use of oil as the working medium will lead to some level of backstreaming into the process chamber unless the system has effective traps (usually liquid nitrogen cooled baffles) and the system is operated properly. Improper venting or mechanical pump failure during operation will lead to oil contamination throughout the system. Diffusion pumps are now mostly used in large vacuum coating operations and materials processing. As with the oil sealed vane pumps, contamination sensitive applications have migrated away from pumps that use oil as a working fluid.

The turbomolecular pump (turbo) is the other example of a momentum transfer pump. The operating principle is essentially the same as that of the oil diffusion pump. However, instead of a fast moving jet of oil vapor the turbo is replaced by fast moving metallic blades. Looking into the inlet of a turbo pump is almost like looking into a small jet engine in terms of the appearance of the blade structure. That's where the similarity ends.

The blades are arranged on a rotor shaft that spins at anywhere from about 30,000 rpm to over 90,000 rpm. There are multiple rows of blades along the length of the rotor to improve the compression ratio of the pump. When a molecule enters the pump and comes in contact with a blade, it is given a boost in the direction of the outlet.

Turbo pumps are very clean and are found in applications ranging from analytical devices to semiconductor process tools. Because of the high shaft speeds and small clearances, they have to be treated well and not used in contaminating environments. Large particles entering the pump or build-ups of material on the blades will destroy the pump.



**Figure 3.4** Pump speed vs pressure curves

Unlike the diffusion pump, the blades have to be kept below the speed of sound. (The shockwave would destroy the blades.) As a result, turbo pumps aren't as efficient as diffusion pumps in terms of their ability to handle the light, fast moving molecules (hydrogen, helium).

The figure on the next page shows the pressure vs pumping speed relationships for mechanical and momentum transfer pumps. Note how, when used in pairs, there is a region of overlap in the medium vacuum (transition flow) region. This shows how complementary the two types of pump are.

### Capture Pumps

The other class of high vacuum pump are the capture pumps. These may be likened to a storage closet - there's no outlet.

In high vacuum, for many applications, there is very little actual throughput of matter. In clean systems or where gas flows are very low, capture pumps can offer high efficiency and a high degree of cleanliness. The following is a brief discussion of the various capture pumps that are found in industry and research.

**Cryo-sorption Pumps:** These are essentially refrigerated buckets that are attached to the vacuum chamber. These pumps have 3 stages as follows: The inlet baffle and shell wall of the pump are held at about liquid nitrogen temperature (77 K range). These surfaces freeze water vapor and other common condensables (oil vapor, etc.). An inner stem is held at about 15 K. This surface will condense all other molecules with the exception of the light gases (hydrogen, helium, neon). On the inside of this stem is a coating of activated charcoal. Only the molecules that aren't condensed on the other surfaces find their way to this region. The charcoal absorbs the light gases, much like a sponge absorbs water. While the name of the pump indicates that there are two operating modes, these pumps are commonly just referred to as cryopumps.

A helium refrigerator is used to cool the pump. After a period of use the pump will accumulate a sufficient load of ice that purging is required. In this *regeneration*



process, the pump is warmed to the point that the captured molecules return to their gaseous state and can be pumped away with a mechanical pump.

Cryo-sorption pumps are seen in high and ultra-high vacuum applications where there is little or no gas flow. Ion implanters and sputtering systems are common examples.

**Other Capture Pumps:** Ion and sublimation pumps work by burying gases under a deposited film. These are usually found on analytical instruments or as adjuncts to larger pumps. For example, a sublimation pump may be used to assist a cryo-sorption pump for improved hydrogen pumping. Getter pumps are chemical pumps that use an active material (usually a barium compound) to scavenge residual gases. Getter pumps are typically used in sealed vacuum components such as vacuum tubes (the getter is the shiny metal on the inside of the glass envelope), microwave oven tubes, etc.

### A Simple High Vacuum Pumping System

Figure 3.5 presents a layout of a typical vacuum system. Note that the line from the mechanical pump diverges into two lines: one (foreline) goes to the exhaust of the high vacuum pump; the second (roughing line) goes to the chamber. During initial pumpdown the entire system is evacuated through the roughing line. The high vacuum pump is then started while the roughing valve is closed (high vacuum and foreline valves are open). This establishes the normal path from chamber to high vacuum pump to mechanical pump.

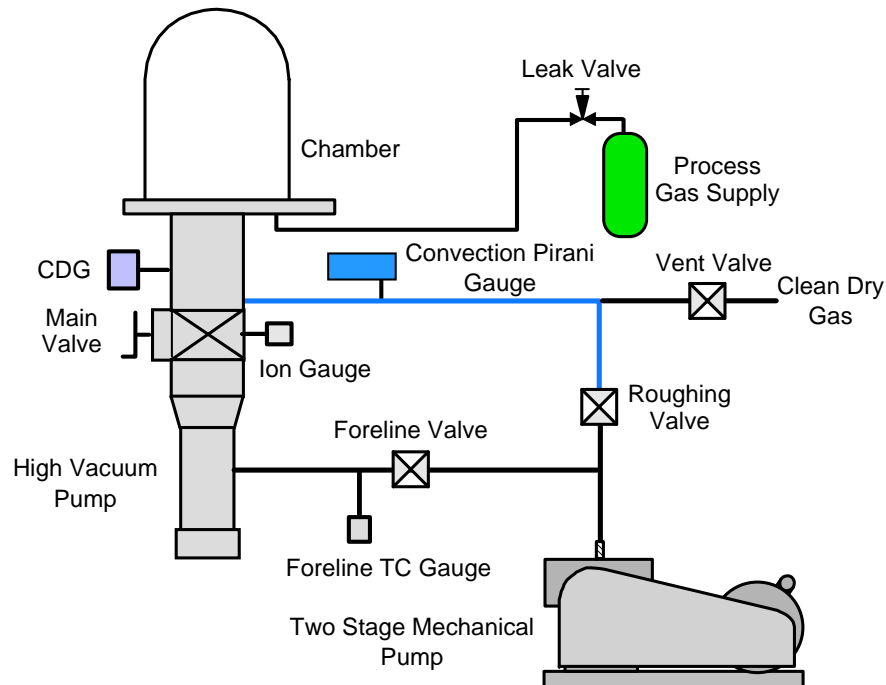


Figure 3.5 High vacuum pumping system

When the chamber is to be vented, the high vacuum valve is closed (roughing valve remains closed) and the vent valve is opened. This keeps the high vacuum pump under vacuum. It does not need to be shut down.

To repump the chamber, the foreline valve is closed (high vacuum pump is isolated under vacuum) and the roughing valve is opened. This connects the chamber directly to the mechanical pump. When the pressure is low enough for the high vacuum pump to take over (the “crossover” point), the roughing valve is closed and the foreline and high vacuum valves are opened.

A gas feed is shown for applications where a gas may be admitted to the chamber. Note the positions and types of gauges that are used.

## Chapter 4      Leaks: the Good, the Bad and the Ugly

### Introduction

When a vacuum chamber is evacuated with a pumping system, the rate of pressure decline will slow and eventually, for all practical purposes, cease. The minimum pressure that the system reaches is called the *base* or *ultimate* pressure.

The job of the vacuum pump is to remove gas molecules from the system. In theory, a high vacuum pump should be able to remove each and every molecule that wanders into its inlet. In practice, the system itself is continuously contributing a seemingly infinite number of molecules and the pump has to contend with this load.

What this means is that a 1 liter chamber has more gas than the 1 liter's worth. There is gas that has adhered (*adsorbed*) to the walls of the chamber. There is gas that is within some of the materials that make up the system and that will, at reduced pressures, evolve into the chamber. Some materials will turn to gas (vaporize) at low pressures. Some gas will enter the system through holes, cracks and other gaps in the system's walls.

In some cases gases are introduced intentionally into a vacuum system. This is the case with sputtering systems, ion sources and chemical vapor deposition systems, to name a few examples.

Each of these gas sources is a leak. The bad ones are termed real leaks. In these the offending gas is transmitted into the system from the outside world. The gas usually will come in through an actual hole or channel in the wall of the system. Examples would be a crack in a weld or a defective seal. In the case of the more subtle *permeation* leaks, gas molecules will migrate through the bulk of the vacuum system's walls. The elastomers that are used for seals are all subject to permeation and should be selected and used with care.

The ugly ones are a result of gas sources within the system. These may result from poor materials choices or contamination of the vacuum surfaces by finger prints. These internal sources of gas are termed virtual leaks. The intentional leaks, the good ones, are the ones that are used to introduce process gases into the system.

Good, bad or ugly, the common denominator is that leaks represent the ingress of gas molecules, at some given rate, into the vacuum system.

In this section we'll examine how to differentiate between real and virtual leaks, how leaks are sized, and how to make and size intentional, predictable leaks using orifices.

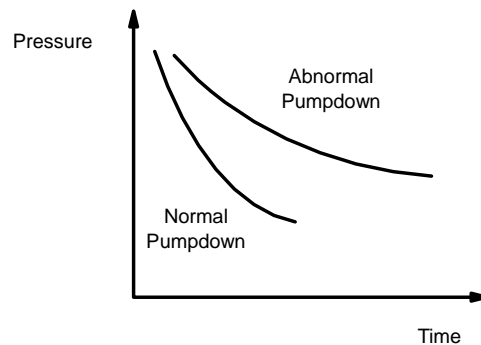
### Identifying Leaks

The performance of a vacuum system is dependent upon a host of parameters: the type and size of the pump, the size of the chamber, the length and directness of the lines between the pump and the chamber, and so forth.

If you have a system that you run on a regular basis, you will have an understanding of how quickly it pumps down and what the base pressure is. If you make a change to the system (perhaps it's just a matter of having opened the system or perhaps you put some new fixtures in or appendages on the system) you might notice that it doesn't behave as well as it did. What you might observe is a slower pumpdown and a poor base pressure. The differences are illustrated in Figure 4.1.

A lesson to be learned here is that it is useful and frequently important to keep notes of your system's performance. If there is no baseline, it is very difficult to determine when something is amiss.

So, at this point we know something is wrong with the system. However, the pumpdown profile



**Figure 4.1** Normal and abnormal pumpdowns

won't reveal what the problem is. It could be contaminated pump oil or it could be a partially closed valve. Or, it could be a leak.

Here is where an additional system feature is valuable. This feature is an isolation valve that is located at or near the pump inlet. If the system is pumped to its base pressure and the isolation valve is closed, the pressure in the system above the valve will rise at some rate. The rate and form of this pressure rise profile is indicative of the size and type of leak.

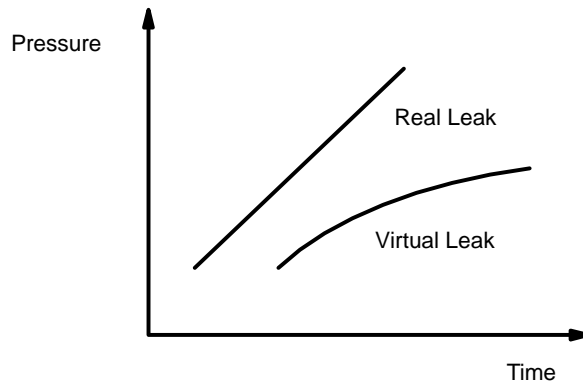
Here is another place where a baseline is important: every system will exhibit some pressure rise. The baseline will let you know when it is abnormally high. If the pressure rate-of-rise is not abnormal, then you will have to look elsewhere for the problem. This could be a pump problem or a leak below the isolation valve.

If the pressure is rising abnormally, use a watch and record the pressure at a succession of equal time intervals. Try to let the pressure rise a couple of orders of magnitude and then plot the results on a linear scale.

Referring to Figure 4.2, a real leak will yield a linear pressure rate-of rise curve. The slope of the curve is a function of the leak rate and the volume of the system: the larger the leak, the steeper the slope; the larger the volume, the shallower the slope. We will quantify this later in this section.

The leak can be through an actual hole or channel, say a crack in a weld, a badly seated gasket or some other aperture. Or the leak can be a result of the gas from the outside room permeating

through some component of the system. Elastomers, as are used in o-ring seals, are permeable to gases. There's no actual hole but gas molecules can work their way through the bulk of the material. Different elastomers have varying permeabilities: silicone is very permeable while viton is relatively non-permeable. Permeation is selective as well: helium will work its way through a gasket much more easily than nitrogen will.



**Figure 4.2** Differentiating leaks

Minimizing permeation leaks is a matter of selecting the correct elastomers (or other organics) and then minimizing the exposed areas of those permeable materials.

Permeation is not restricted to organics. At very low pressures, descending into the ultrahigh vacuum realm, permeation of hydrogen through glass and metals can start to become a problem.

Returning to the rate-of-rise test, if the slope gets shallower as time goes on, you probably have a virtual leak. For example, gas trapped in a threaded hole under a bolt will leak out slowly causing the pressure to rise. Eventually the gas will all leak out (since this is a finite source) and the leak will appear to go away. This sort of situation can be verified by repumping the system and then looking again at the pressure rate-of-rise. Since the gas has already been dissipated, the virtual leak will no longer be present. It is for this reason that you can buy special bolts and screws for vacuum use that have holes drilled through them.

Moisture and surface contamination (finger prints, etc.) will have a similar effect. Eventually these will cease to be gas sources if the system is pumped for a long enough period of time. (Depending upon the level of vacuum required, it may be a *very* long time.)

You have to be cautious of the vapor pressures of the various materials that make up the system components. Inappropriate choices can result in virtual leaks. Organics within the chamber will be sources of virtual leaks. If you are trying to get vacuum levels below  $10^{-6}$  Torr, you have to be concerned about metals and metal alloys that have high vapor pressure constituents.

If your virtual leak source is due to a basic materials incompatibility, then no amount of pumping is going to rid the system of that problem.

To conclude this section, what you'll probably find in your rate-of-rise test is a mixture of low-level real and virtual leaks that are harmless to your application (and which would probably take forever to fix anyway), and occasional nasty leaks that have to be fixed. These nasty things will also be combinations of real and virtual leaks. Now we'll look at how to size a leak.

## Sizing Leaks

As stated before, leaks represent molecules entering the system. Therefore, the proper way to specify a leak is in language that relates to how many molecules per unit time are being admitted. However, talking about  $x$  molecules per second is a bit inconvenient as no other common vacuum measurement uses molecules as a unit.

Since all of these terms relate to molecular quantity, thence to the actual mass of the gas, standard condition volumes are also called *mass quantities* and standard condition volumetric flow is also called *mass flow*. These are terms that have been discussed in Chapter 2.

Another term you will run into for leak rates is Atmosphere cc/sec (or Atm. cc/sec). This is roughly equivalent to sccm except that the pressure reference is the prevailing ambient pressure (usually near 760 Torr unless you live in Denver or Albuquerque).

## Determining the Size of a Leak

With that bit of grounding behind us, it is fairly straightforward to quantify the size of a leak in units such as sccm by using the pressure rate-of-rise measurement discussed previously. As noted:

- A pressure rise over time indicates a leak.
- A linear rate-of-rise indicates a constant leak rate, typical of real leaks.
- The slope of the curve is a function of the size of the leak and the volume of the system.

Knowing that the  $Q$  of the leak is related to pressure, volume and time, we can determine the size of the leak from the rate-of-rise curve (pressure change with time) assuming we know the volume of the system. Of course, since we are measuring the mass flow,  $Q$ , as standard condition volumetric flow, we also have to adjust for these standard conditions. The relationship that does this is:

$$Q(\text{sccm}) = 79 \left[ \frac{273}{273 + T} \right] \left[ V \frac{\Delta P}{t} \right]$$

The guts of this equation is in the right hand set of brackets where  $\Delta P$  is the change in pressure in Torr,  $t$  is the time in seconds over which the change in pressure occurred, and  $V$  is the volume of the chamber in liters. The units here are Torr  $\times$  liters  $\div$  seconds. The left hand brackets give us the temperature adjustment factor where 273 is 0 °C in Kelvin units and  $T$  is the temperature of the gas in the chamber, also in K. Finally, the factor of 79 is the conversion from T-liters/sec to sccm.

The largest uncertainty here is the volume of the system. With a rule and a bit of time, a reasonable volume determination can be made.

## Intentional Leak Sources

Now we'll discuss the good leaks, the intentional sort that are used to admit gases into vacuum systems. The gases may be used to control pressure or, in addition, they may be used to produce a plasma or promote a chemical reaction. There are various forms of leaks that are suitable for doing this. Needle valves are one common form. The main problem with needle valves or other simple low conductance leaks is that it is not easy to determine with any sort of precision the  $Q$  of the leak at any particular setting.

Precision leaks are very important in research and industry. Semiconductor vacuum process equipment, as one example, would not produce anything but junk were it not for the accurate and repeatable leaks that are used to admit the reaction gases to the systems.

The first type of leak device to be discussed is the simple passive orifice leak. Orifices may be found at many points in a vacuum system where it is desired to restrict or control flow in a reproducible manner.

The second leak device is the thermal mass flow controller, an active flow control device that can be set to provide a specific flow rate.

### Orifice Leaks

Consider the configuration of Figure 4.3. Here there is a piece of tubing with an orifice mounted in the tube.  $P_1$  and  $P_2$  refer to the pressures to the left and right of the orifice respectively.



**Figure 4.3** Orifice in a tube

Obviously, if  $P_1 = P_2$  there will be no flow of gas through the tube. If  $P_1$  is increased slowly (with  $P_2$  remaining constant) gas will flow through the orifice. The amount of gas flow (the mass flow) will now be a complex function of gas properties, orifice geometry, temperature,  $P_1$  and  $P_2$ .

The orifice, since it has a cross-sectional area less than that of the tubing, serves to increase the velocity of the gas that is flowing through the tube. Bernoulli found that as velocity increases, pressure falls ( $\rho$  is the gas density):

$$\frac{P}{\rho} + \frac{V^2}{2} = \text{Constant}$$

This velocity/pressure relationship can be shown with the familiar experiment where one blows air between two parallel sheets of paper and the sheets move toward each other.

Bernoulli's law is only valid for constant density flow and for gases. If the flow is not too great the gas is, for all practical purposes, incompressible.

Returning to the orifice, if the pressure  $P_1$  continues to increase relative to  $P_2$ , a point will be found where the velocity of the gas through the orifice becomes sonic. That is, the gas velocity reaches the speed of sound. This condition occurs when  $P_1$  is about twice  $P_2$ .

It turns out that the gas velocity cannot exceed the speed of sound. Further increases in  $P_1$  only increase the density of the gas going through the orifice. Since the density is directly proportional to the pressure, the mass flow becomes directly proportional to  $P_1$ :

$$Q \text{ (mass flow)} = kP_1 \text{ when } P_1 \geq 2P_2$$

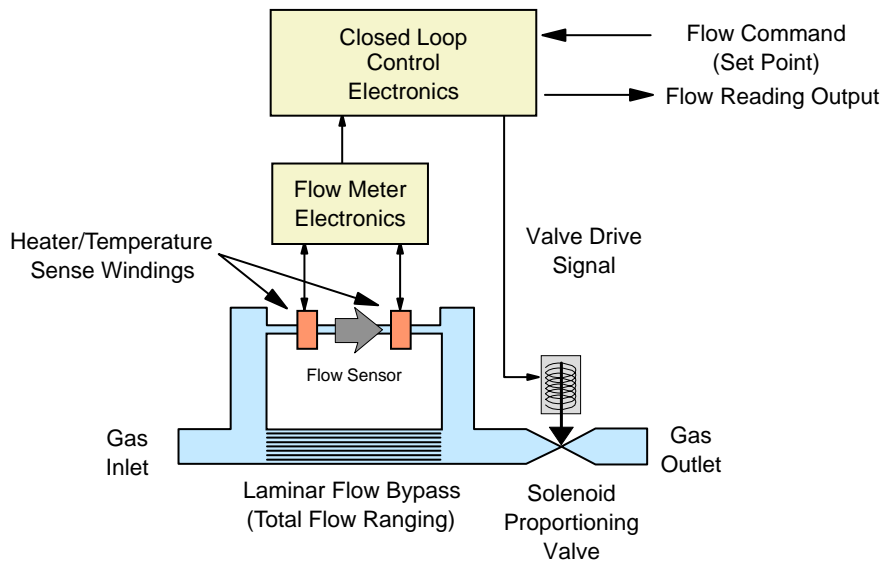
Because the density is now increasing, the gas has become compressible.

This sonic flow condition, also called choked flow, can be used as the basis for some types of mass flow meters and controllers. The value of  $k$  can be calculated or it can be experimentally derived.

### Thermal Mass Flow Controllers

The thermal mass flow controller (T-MFC or simply MFC) is a very common device for the active control gas flow into vacuum process systems. As the name implies, these rather clever instruments work by measuring the rate at which the molecules flowing through a sensor tube transport heat. Figure 4.4 is a schematic diagram of the internal workings of an MFC.

There are 4 major components in an MFC: the mass flow sensor, the flow bypass, a control valve and the electronics.



**Figure 4.4** Elements of a thermal mass flow controller



The mass flow sensor consists of a short length (on the order of an inch) of small-bore capillary tubing. The small diameter of the tube ensures that the gas traverses the tube in a viscous, laminar flow condition. This simply means that the molecules flow parallel to the axis of the tube without turbulence. The avoidance of turbulence is key to making the device repeatable.

Placed around the tube are a pair of windings that act as heaters and, by monitoring the resistances of the windings, the temperature of the tube at those locations. Each of the windings is attached to a bridge circuit that maintains each of the windings at a specific temperature (i.e. each winding is held at a constant resistance). When the gas enters the sensor tube, the first heater warms the gas to some temperature. The molecules of the gas transport the heat down the tube.

At any given gas flow, each of the heaters will have some amount of power delivered to it to maintain the desired temperature. If the gas flow rises, the first heater will require more power to maintain the constant temperature. From this, more heat will be transported to the second heater (more molecules per unit time) and its power will be decreased. The bridge circuit looks at how much power is required by the heaters and provides an output that, after linearization and scaling, is directly proportional to the gas' mass flow.

The small bore sensor tube can only transport so much gas, usually just a few sccm. To handle larger mass flows a bypass element is placed in parallel to the flow sensor tube. The bypass is just another laminar flow path and it is sized so that there is a constant splitting ratio between the two paths. The bypass serves exactly the same function as a shunt does in an ampere meter. By using bypasses of various sizes, MFCs can be produced that have full scale flows from one to thousands of sccm.

What has been described thus far is the metering aspect of the device. To actually control the mass flow, a proportioning control valve is introduced along with the appropriate closed-loop circuitry.

In use, the inlet gas supply is usually in the pressure range of 15-30 psig. (A constant pressure is important to the operation of an MFC. Pressure pulses will have a direct effect on the flow output.) The outlet can go directly to the vacuum chamber. Flow control is linear with a 0-5 volt input (setpoint).

MFCs are available in full scale flow ranges from 1 sccm to over 500k sccm. The operating range of a typical MFC is from 100% of full scale to lower than 20% of full scale. Standard accuracy is 1% of the full scale range.

Since MFCs work on the basis of the thermal properties of the gas, they are sensitive to the gas type. Users have to specify not only the sccm range but also the gas type. Manufacturers provide correction factors for hundreds of different gases. Each correction factor is a constant that is referenced to nitrogen = 1. As examples, argon has a correction of 1.39 (higher flow than nitrogen) while silane ( $\text{SiH}_4$ ) is 0.60 (lower flow than nitrogen).

**Discussion:** In addition to the thermal MFCs there also exists a class of flow control device called the pressure-based MFC. These devices use sonic orifices. Modify Figure 4.4 as a pressure-based flow controller with closed loop control.

# Chapter 5 Leak Detection

## Leak Detection Methods and Equipment

While the pressure rate-of-rise method that was described in Chapter 4 will provide an indication of the type of leak and overall size of the leak, it only serves as a first level troubleshooting tool. To actually localize the leak we have to probe the system with a tracer gas and then use a gas-type selective instrument to pinpoint the leak.

Figure 5.1 shows a vacuum system with a leak. Air is drawn into the system via the leak and causes an increase in the system's normal base pressure. The standard practice for detecting leaks in a vacuum system is probe the various fittings and welds of the system with a tracer gas that comes from a small hand-held nozzle or wand. When the wand is placed in proximity to the leak, the tracer gas will be drawn into the system. To detect the tracer gas the system must be equipped with a measurement device that is sensitive to the tracer gas. Helium is the most frequently used test gas for several reasons:

- Helium molecules are small and fast providing high sensitivity in leak detection.
- Helium is easy to differentiate from other gases by the instruments used for leak detection.
- Helium is not found in any significant quantity in the environment. This reduces the amount of “noise” in measurements.
- Helium is inert and not harmful to equipment or humans.

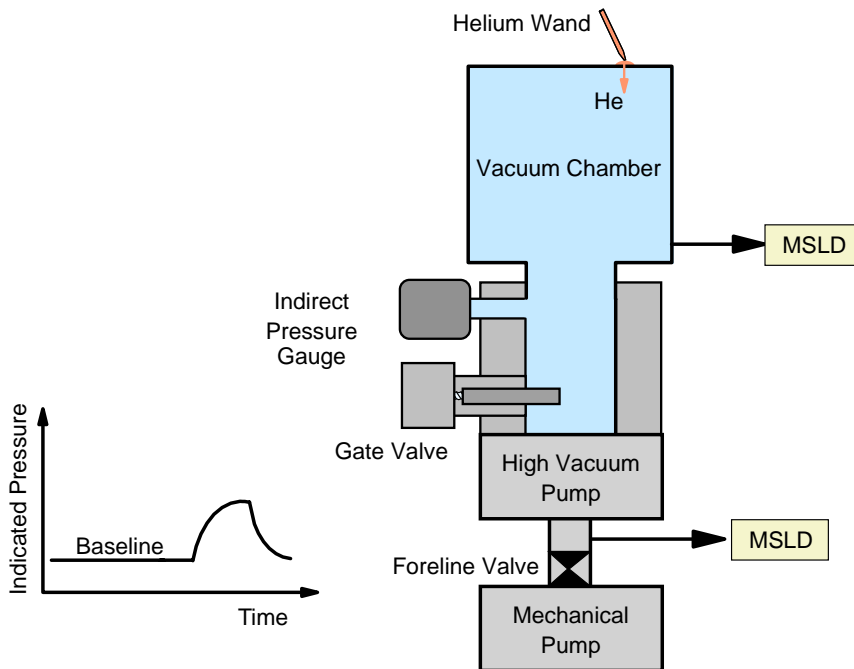


Figure 5.1 Leak detection of a vacuum system

## Use of an Indirect Gauge

Since indirect gauges are sensitive to gas type, the introduction of a tracer gas into a pumped system through a leak will cause a change in the gauge's pressure indication. Pirani (medium vacuum) and ion (high vacuum) gauges are useful for this method of leak detection. When using this method, a peak (or dip, depending on the characteristics of the gauge) in the baseline pressure will be seen when the wand is passed over the location of the leak.

The sensitivity and response time for this method depends upon the chamber volume, pumping speed, pressure and the gauge sensitivity to the tracer gas versus air. This method is generally usable for intermediate size leaks down to the  $10^{-4}$  to  $10^{-5}$  Atm. cc/sec range. The simplicity of this method is that one or more of the gauges that are normally installed on a particular system may be used.

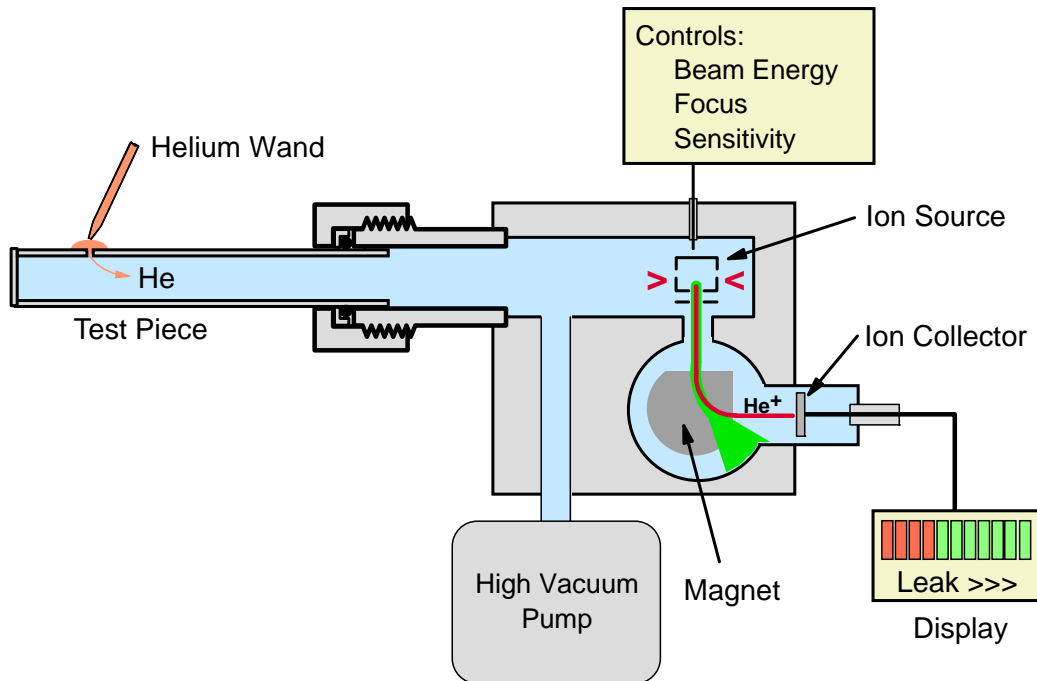
## The Mass Spectrometer Leak Detector

To achieve higher sensitivity we must use an instrument that can discern even very small quantities of the tracer gas. This can be achieved with a very special type of gauge that is based on mass spectrometry of the molecules in the system. The mass spectrometer leak detector (MSLD) is a device that is sensitive to only the tracer gas. The more generalized partial pressure analyzer is a mass spectrometer that is designed for the analysis of the specific gases that are contained in the vacuum chamber. The MSLD will be covered in this section and the chapter will conclude with a brief discussion of mass analyzers.

Figure 5.2 is a simplified schematic diagram of a typical MSLD. The MSLD has to operate under high vacuum conditions to provide a long enough mean free path for the ions to traverse the distance from the ion source to the collector without scattering from collisions with other ions. All such leak detectors have an integral vacuum pumping system.

The principle of operation is straightforward. The ion source contains one or more filaments that act as a source of electrons. The several electrodes in the ion source constrain the ions to the axis of the source and then extract them toward the magnetic analyzer. As the ions exit, they are focused into a fine beam and given a specific amount of energy by an acceleration electrode. As the ions enter the magnetic field they curl around the field lines. The radius of curvature is determined by the leak detector parameters of ion energy and magnetic field and by the ratio of the mass to charge ( $m/e$ ) of the ion. The MSLD is set up such that only singly charged helium ions ( $\text{He}^+$  with  $m/e = 4$ ) hit the collector. The nearest non-helium ions would be hydrogen,  $m/e = 1$  or  $2$  ( $\text{H}^+$  or  $\text{H}_2^+$ ), or nitrogen,  $m/e = 7$  ( $\text{N}^{++}$ ).

In order to quantify the leak rate, a *calibrated leak* is attached to the instrument and the MSLD's sensitivity control is adjusted to correspond to the marking on the calibrated leak. Calibrated leaks consist of a small reservoir of helium that has a very fine capillary (usually fabricated from metal or fused silica) as the leak source. The reservoir is pressurized by the manufacturer to an amount that provides the correct leak rate.



**Figure 5.2** Mass spectrometer leak detector

To perform a leak check of a small volume test piece, that piece can be attached directly to the leak detector as shown in Figure 5.2. The small pumping system of the typical MSLD cannot evacuate a large system.

### Using a Mass Spectrometer Leak Detector

For leak detection in vacuum systems the leak detector and its pumping system are used in conjunction with the vacuum system's pumps. The MSLD can be used to find leaks as small as  $10^{-10}$  Atm. cc/sec.

Referring to Figure 5.1, the MSLD may be connected in either of two locations:

**Connected to the main chamber.** The helium that enters through the leak will be pumped by both the large system pump and the leak detector's pump. This lowers the sensitivity of the MSLD by the ratio of the pump speeds. Some improvement can be gained by partially closing the chamber's gate valve.

**Connected in the foreline between the high vacuum and backing pumps.** In this case the backing pump is isolated by closing its valve and the leak detector's pump does the work of the mechanical pump. (Remember, at low pressures, the actual throughput of the backing pump is very small.) This technique provides a very fast response time as all of the tracer gas is drawn into the MSLD.

## **Proper Use of the Wand**

The flow of helium from the wand does not need to be very strong. Indeed, too much helium flow will saturate the environment and lead to false or confusing results. If the tip of the wand is placed in a cup of water, there should only be a couple of bubbles per second.

The motion of the helium wand has to be slow and deliberate. Begin on the parts of the system furthest from the MSLD and work toward it. Since helium is lighter than air, the probing should also move from higher regions to lower. When probing a specific fitting or weld, bring the wand to the area and then withdraw it. Pause to see if there is a response in the leak detector before moving on to the next connection.

When leak detecting linear assemblies such as lines, take into account the time constant for any helium to get from the probe area to the MSLD.

Make use of the leak detection ports on the fittings. VCR fittings have small holes in the connectors which provide a good path to the seal area. CF flanges have diametrically opposed grooves toward which the helium should be directed. For KF flanges, insert the wand into the gap in the compression ring.

When performing leak detection around complex components, it is often helpful to place a bag around that component. The bag may then be filled with helium. This serves to concentrate the helium in the area of interest.

Since helium can readily permeate elastomers (some types much more readily than others), such permeation can appear to be a real leak. The difference is that the time constant of a real leak is much faster than that of a permeation leak. Also, once an elastomer has become permeated with helium, it can take a substantial amount of time for the permeated helium to dissipate. During this period, leak detection will be difficult.

## **Partial Pressure Gauges**

### **The Mass Analyzer**

The gauges that were covered in Chapter 1 provide a *total* pressure reading. Most gaseous environments consist of a mixture of gases and there are a number of circumstances where it would be desirable to know how much of what is in the chamber.

The Law of Partial Pressures states that the total pressure in a system is the sum of the pressures exerted by each of the constituent gases. In our environment roughly 78% of the air we breathe is nitrogen and 20% is oxygen. The remaining 2% consists of water vapor, argon, carbon dioxide and lesser amounts of hundreds of other gases. In the case of nitrogen, that 78% corresponds to 593 Torr of the normal 760 Torr sea-level pressure.

Partial pressure gauges are a relative of the ion gauge. In the case of the ion gauge, gas molecules are ionized by the impact of incident electrons. All of the generated ions are collected to provide an

ion current from which is inferred the total pressure. The partial pressure gauge has a spectrum analyzer inserted between the ion source (where the ions are generated) and the collector electrode.

The spectrum analyzer can operate by any of several methods:

**Magnetic sector:** Ions from the ion source are accelerated to a specific energy. The energy, expressed in electron volts (eV) is directly related to the voltage that is applied to the acceleration electrode of the ion source. The ions pass through a magnetic field that is perpendicular to the path of the ions and deflected from their original path by an amount determined by the ions' energy, mass and charge. A collector placed at the proper position will detect ions of a specific mass and charge.

By varying the ion energy, different ionic species will hit the collector. Synchronizing the scan energy with the collector output will yield a plot of intensity (pressure) versus mass/charge. Magnetic sector mass analyzers have excellent accuracy and resolution and are usually found in laboratory environments. The previously discussed mass spectrometer leak detector (MSLD) is a simplified magnetic analyzer system that is tuned for helium ions as a dedicated leak detection tool.

**Time of flight (TOF):** Ions of constant energy are timed as they pass through timing electrodes. Heavier ions will travel more slowly than fast ions. TOF is mainly used in laboratory and spaceflight applications.

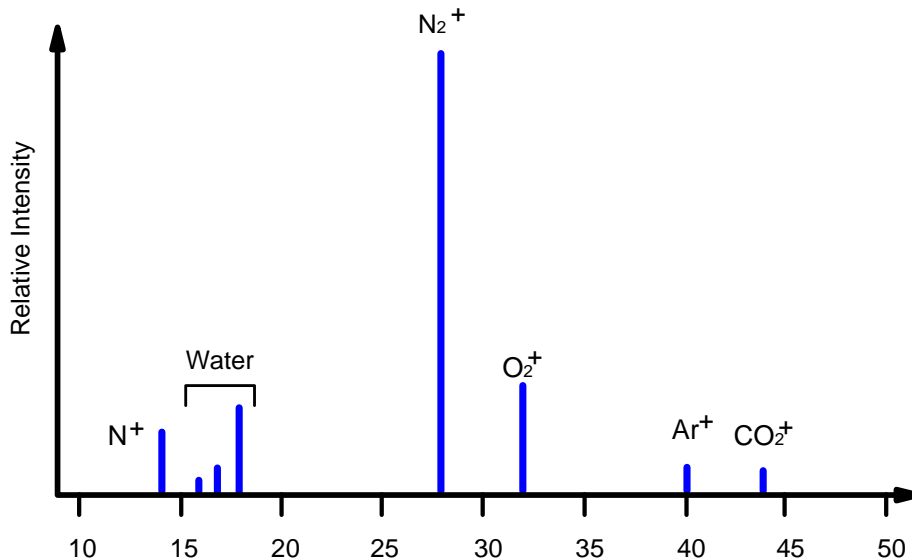
**Electrostatic:** Electrostatic segregation of ions is found in the quadrupole mass analyzer (QMA), also referred to as the residual gas analyzer (RGA). This instrument uses a parallel array of 4 rods that have varying DC and RF potentials applied to the rod structure to perform the filtering function. The ion beam is directed down the axis of the system and only those for which the field conditions are correct will pass through. RGAs are found on vacuum process tools where they are used to monitor, and sometimes control, the gas mixture environment in the chamber. They also perform double duty as leak detectors.

## The Mass Spectrum

Figure 5.3 depicts the spectrum of air as might be seen on the display screen of a partial pressure analyzer.

The vertical scale shows relative pressure. In the example the scale is linear. Since the indication of any ion gauge is dependent upon not only the actual partial pressure of the gas but also the sensitivity of the gauge to that gas, the pressure scale is only approximate unless the peaks are calibrated for the specific gases.

The horizontal axis shows the various ionic species according to  $m/e$ . The important thing to note is that the spectrum doesn't really display the various gases in a system but rather the molecular ions and various ionic fragments of those gas molecules. Interpreting a complex spectrum can sometimes involve a fairly detailed investigative process.



**Figure 5.3** Mass spectrum of air

Note that nitrogen and oxygen, being the major constituents of air, have large peaks corresponding to their molecular masses with a single charge ( $m/e = 28$  and  $32$ ). These are called major peaks. There is a smaller peak at  $14$  for  $N^+$ . Water splits into 3 fragments with a very easy to recognize pattern. Here we have  $H_2O^+$  at  $18$ ,  $OH^+$  at  $17$  and  $O^+$  at  $16$ .

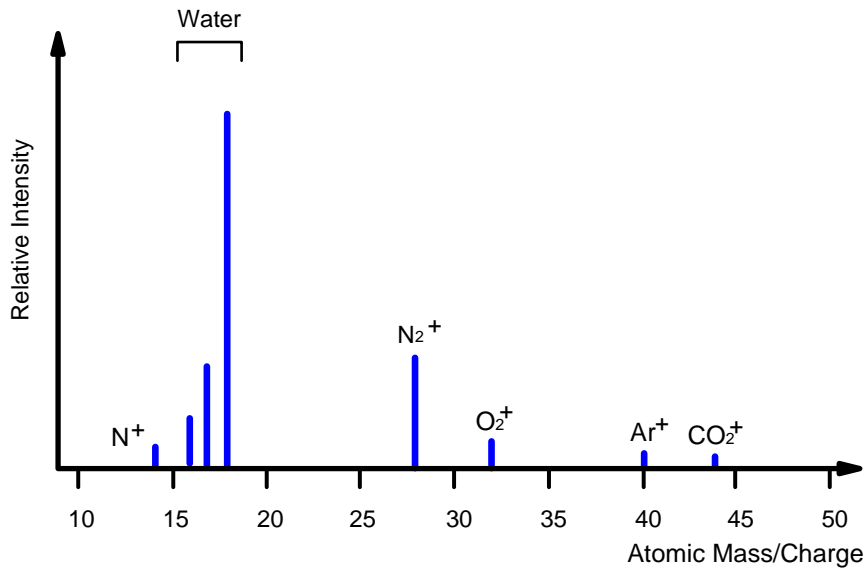
Is there any CO in the gas mixture? For CO we would expect a major peak at  $28$  ( $CO^+$ ). The problem is that  $28$  is also the major peak of nitrogen. To identify CO we would also have to look for  $C^+$  ( $12$ ),  $CO^{++}$  ( $14$  - same as  $N^+$ ) and  $O^+$  ( $16$ ). While  $14$  and  $16$  are also present, in this scan there is no  $12$ . Therefore either there is no CO or the concentration of CO is much lower than that of nitrogen.

This can all seem hopelessly confusing but, when applied to vacuum systems, the operator is generally looking for specific species as opposed to trying to identify everything in the chamber. Also, the software that is supplied with mass analyzers will have libraries that show typical scans of various gases.

Figure 5.4 shows what the spectrum might be for a system that has been evacuated into the medium vacuum range. Note that water now dominates the spectrum. This is because water is difficult to pump from a chamber and, as pumping proceeds, water becomes the dominant gas.

If the system were pumped for a very long period of time or the chamber was baked, the water peaks would diminish or disappear. Growth in the nitrogen and oxygen peaks would suggest that the system has a leak.

Oil contamination from backstreaming would be identified by  $CH_2$  fragments at intervals of  $m/e = 12$ . These are most prevalent toward the right side of the scan, at  $m/e$  over  $40$ .



**Figure 5.4** Mass spectrum of a chamber at medium vacuum

To look at changes in the gas composition, the software can establish a baseline. This might be done at base pressure when the system is known to be performing normally. The spectrum scan then shows only changes (increase and decrease) over time. This mode helps to eliminate the clutter of too much data and permits the operator to concentrate on what the system is doing.

Finally, the software can be set to show the variation in specific  $m/e$  over time as a trendline. As an example, for a sputtering system using argon as the process gas it may be desirable to monitor the major peak of argon (40) as well as any expected contaminants. Example peaks to monitor would be the water peak at 17, nitrogen at 28 and oxygen at 32.

Why would the 17 peak of water of water be selected as opposed to the more obvious major peak at 18? While argon's major peak is at 40, if argon is the dominant gas because of its use in the process, there is also a strong Ar<sup>+++</sup> peak at 18. The water 17 peak cannot be confused with any other gas in the system so it is the appropriate one to monitor in this situation.



## Chapter 6 Common Vacuum Flanges and Seals

### Introduction

Connections between components in vacuum systems may either be permanent connections made typically by welding or brazing or demountable connections using a variety of flanges and fittings.

Most vacuum systems tend to be modular, using standard components wherever possible. The manufacturers of vacuum components such as valves, gauges, flow controllers, etc. generally offer their products with a selection of standard fittings so that the user can match those items to their specific hardware and application. There is also a wide variety of standard vacuum component hardware including straight tubes, bends, tees, crosses, reducers and so forth. These come in varying diameters and lengths, all equipped with an assortment of standard flanges.

Any kind of connection poses an opportunity for leaks so it is important that the person working with vacuum hardware knows how to properly use and care for the various fittings.

There are too many specific configurations of separable connector to cover each one. This chapter will concentrate on the principles behind the two major classes of demountable fitting: the o-ring sealed flange as exemplified by the ISO-KF system and the metal sealed flange as exemplified by the CF system. Other sealing systems will vary in terms of their physical manifestations but their principles are the same. The reader who is interested in specific dimensional data is invited to visit the various manufacturers' catalogs. A selection may be found in the Appendix.

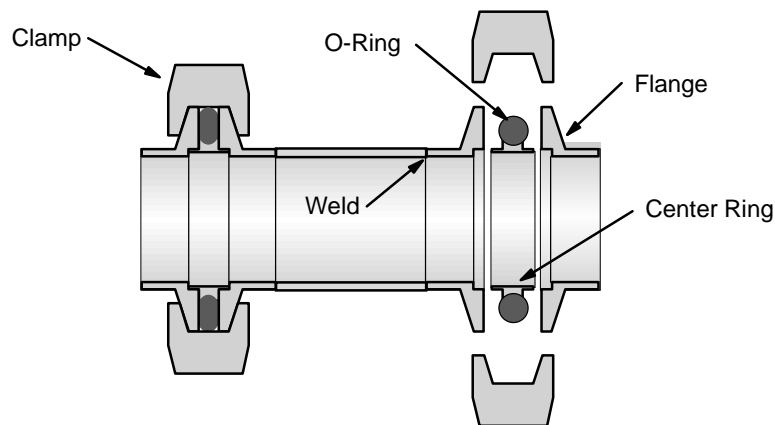
### ISO-KF O-ring Sealed Flanges

The ISO-KF series is widely used in low and medium vacuum applications and may be used selectively in high vacuum equipment. "KF" refers to the original name "klein Flansch" or "small flange." KF flanges may also be referred to as QF ("quick flange"), NW or DIN. The KF system is standardized and is recognized by ISO, DIN and Pneurop.

Each flange is sized according to the largest tube ID that can be welded to it. The outside diameter is a standard North American tube dimension, in inches. The standard KF flanges are KF16 (0.75 inch OD tube), KF25 (1 inch OD tube), KF40 (1.5 inch OD tube) and KF50 (2 inch OD tube).

Figure 6.1 on the next page shows the design features of the KF series flanges. Each flange set consists of a pair of similar flanges and a metal ring (center ring) and o-ring assembly. The center ring has three functions: it positions the o-ring on the flange seal area, it maintains the ID of the o-ring against the force of atmospheric pressure and it determines the amount of o-ring compression when the flanges are drawn together.

In addition, a clamping assembly holds the flanges together. The clamps usually consist of two hinged aluminum half-rings that encircle the flanges and are closed with a thumbscrew or toggle. When tightened the clamp draws the flanges together against the center ring.



**Figure 6.1** KF flange system

The seal is created by the compression of the o-ring. The amount of compression is important. Over the life of the seal the o-ring has to maintain an adequate amount of force on the flanges. Too little compression and the seal will be poor or unreliable. Too much and the seal may fail over time if the o-ring material takes a “set” and the degree of force declines. The shoulder on the center ring has a width that assures a proper amount of o-ring compression (about 25-30 percent).

KF seals are designed to work with finger tightening of the clamp. The o-rings are used dry - no grease or lubricant. (Greases are traps for contaminants and should never be used with anything but sliding seals.)

There are a wide variety of materials choices for the o-rings. The default is viton but other materials may be used depending upon application (temperature, chemicals, radiation, etc.).

Periodic inspection of o-ring seals is advisable. Issues to look for include excessive set, flow of the o-ring, embrittlement, tackiness and cracking.

In the figure, note the weld. Proper vacuum welds are made from the inside of the tube and are generally in the form of a butt weld. This minimizes the possibility of trapped areas that can create virtual leaks. The standard welding process for flanges and tubing is orbital tungsten-inert gas welding.

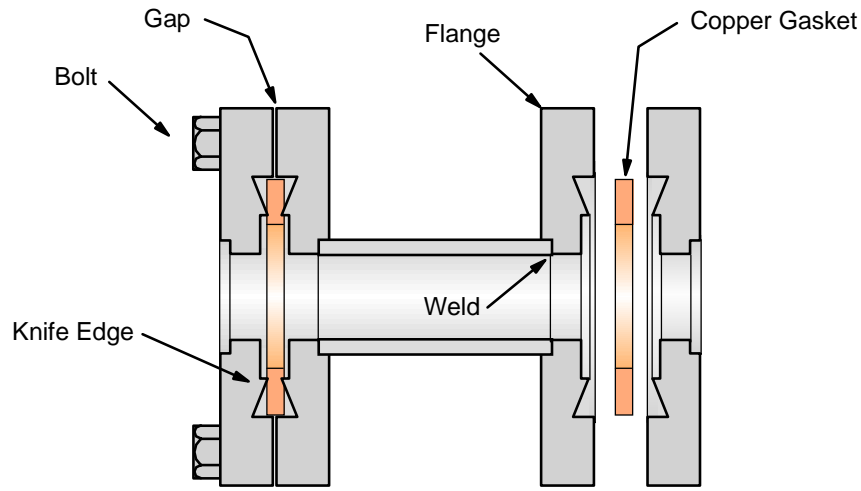
For larger sizes of tubing there are ISO extensions to the KF series, for example the LF (“large flange”) series. This uses the same general sealing geometry but with bolts or clamps to draw the flanges together. Bore sizes range from 63 to 1000mm.

## **CF Metal Sealed Flanges**

O-ring seals such as the KF system are very easy to use. Since they do use elastomers, there are applications limitations at high and ultrahigh vacuum and with some process materials. In these cases an all-metal configuration is required.

The most commonly used all-metal flange system is the CF. The CF is derived from Varian Corporation's trademark ConFlat design. The CF is not formally standardized but the equivalent CF flanges from other reputable manufacturers are interchangeable.

Referring to Figure 6.2, the CF flanges use a knife edge that bites into a soft gasket. The gasket material is usually OFHC copper. The knife edge has a shallow angle on the outside and a slightly off-vertical angle on the inside. The seal is made in the area where the steeper side of the knife edge has cut into the gasket.



**Figure 6.2** CF flange system

In assembling a CF flange the bolts are sequentially tightened so that the force on the gasket is uniform. In the tightening process the perimeters of the flanges are slightly deformed. This creates something equivalent to a hairpin spring that extends from the perimeter of one flange, through the knife edges and back to the perimeter of the other flange. This effect produces a very strong and enduring force on the seal area.

When a CF flange set has been properly tightened there will be a uniform gap between the flanges (purists will use a feeler gauge to set the proper gap). Tight bolts and a uniform gap are signs that the flanges have been coupled correctly. No gap usually means that the assembler has forgotten to insert a gasket.

Never reuse a metal gasket. The resulting seal may leak immediately or it may fail at some random time. Given the number of bolts that have to be tightened the cost of the gasket is small compared to the time and effort required to redo the job.

The CF flange system is very robust and represents essential hardware for high and ultrahigh vacuum. Mated flanges will survive the large temperature excursions that are required for bakeout and they are free of the outgassing and permeation issues that render o-ring systems inappropriate.

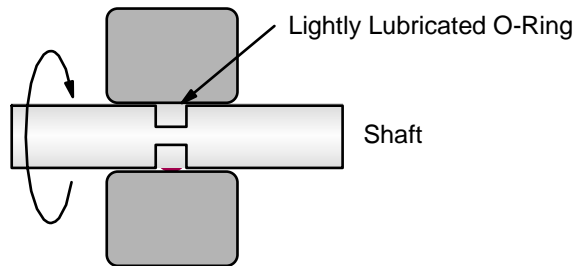
CF flanges are commonly available in sizes from 1-1/3" to 10" OD. Single piece as well as two piece rotatable flanges are manufactured.

### Other Metal Sealed Connectors

For smaller lines the VCR system developed by Swagelok Corporation is widely used. These will be found on some vacuum gauges, MFCs and instruments that deliver gases to vacuum systems. These connectors use screw male-and female fitting pairs and a small metal gasket. Made for tubing sizes from 1/4 to 1/2 inch these are essentially miniature versions of the CF.

### Dynamic Seals

The seals described above are static seals. In many cases it is necessary to transmit rotary motion into a vacuum system for moving a fixture or rotating a valve "flapper." This is generally done with an o-ring that is placed in a groove on the rotating shaft as shown in Figure 6.3.



**Figure 6.3** Dynamic shaft seal

The o-ring is placed in a groove that has been machined into the shaft. The dimensions of the groove are such that the o-ring will receive the correct amount of compression and will stay within the confines of the groove when the shaft is inserted into the housing.

Dynamic seals are one place where the o-ring should be lubricated. The lubrication is not related to the seal itself but is required to ensure that the o-ring can slide in the housing without sticking or tearing. The amount of lubricant required is very small - just enough to give the o-ring a shiny surface appearance.

# Chapter 7 Pressure Control in Vacuum Systems

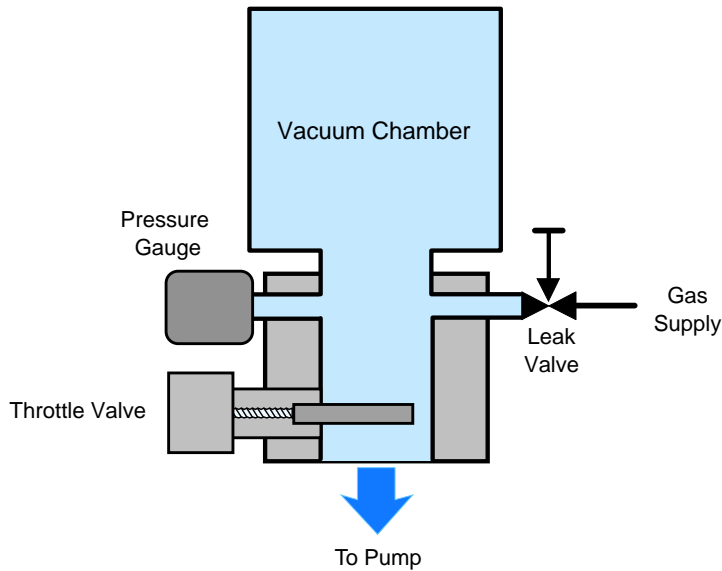
## Introduction

In the throughput discussion in Chapter 2 and the leak discussion in Chapter 4 we saw how leaks (the bad or ugly kind) could degrade the base pressure of a vacuum system and that the system's equilibrium pressure represents a balance point between pump throughput and  $Q$  into the chamber.

This chapter will pull together these elements and will describe the methods that are used to drive a vacuum system to a specific set of conditions of gas flow and pressure as required by a specific process.

## Manual Pressure Control

Consider the vacuum system shown in Figure 7.1 below.



**Figure 7.1** Manual pressure control elements

When the throttle valve (a variable conductance) is open and the leak valve (another variable conductance) is closed, the chamber will settle at a base pressure determined by the pump's throughput and the sum of the sources of gases within the system. These would be from real and virtual leaks, permeation and outgassing and represent the overall gas load presented to the pump.

If we want the chamber to reach a specific pressure then we would open the leak valve by some amount, perhaps just under the desired pressure. Then we could close the throttle valve by some amount to get the system to the desired pressure.

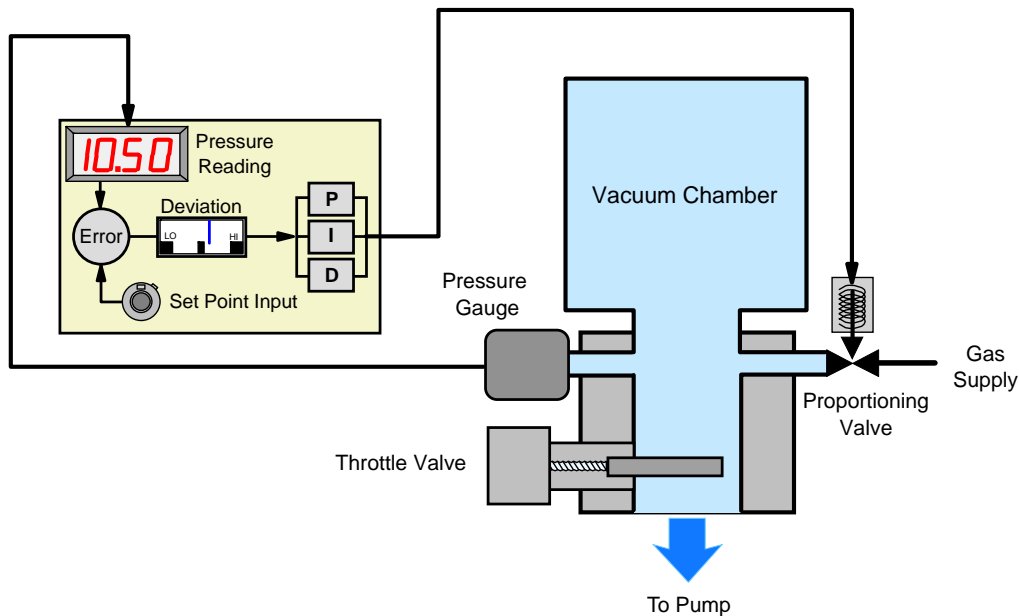
While this technique will achieve the result of realizing a specific pressure in the chamber, there are a number of deficiencies:

- As system conditions change it will be necessary to continuously monitor and adjust the pressure control elements.
- The process requirements may require multiple gases as would be the case for a reactive process. Simple metering (conductance) valves cannot perform this task.
- It may be necessary to independently control the flow of each of the incoming gases while also controlling pressure.

## Closed Loop (PID) Pressure Control - Upstream Control Mode

### General Configuration

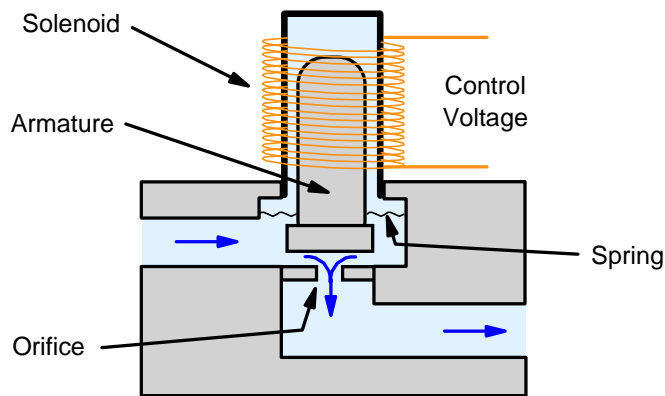
In Chapter 4 we saw how closed loop control is applied in a mass flow controller. The same approach can be applied on a larger scale to a vacuum system. Figure 7.2 represents an extension to the previous example with closed loop pressure control. This particular configuration is referred to as upstream pressure control. This is because we are driving a valve that is upstream of the chamber.



**Figure 7.2** System with upstream PID pressure control

### The Proportioning Valve

Proportioning valves are an important element in vacuum systems. As we saw in Chapter 4, a proportioning valve is incorporated into mass flow controllers as an element in the control loop. Figure 7.3 depicts the general configuration of a solenoid proportioning valve.



**Figure 7.3** Solenoid proportioning valve

The solenoid proportioning valve uses the magnetic field developed by a solenoid to move a magnetic armature that is within the valve. A flat spring centers the armature and also provides a countering force to the magnetic field. The usual arrangement is normally closed - when no current is applied to the solenoid the face of the armature rests on the orifice. This face may have an elastomeric material to seal the valve or it may be a metal-non sealing configuration. It should be noted that control valves of this type are not meant to be positive shut off devices. If a positive shutoff is required, there should be a dedicated isolation valve downstream of the control valve.

The action of the valve is as a variable conductance with very high resolution. Proportioning valves are designed to be used as part of a closed loop control system with constant feedback. They are not designed as “set and forget” valves as the armature position will wander without constant correction.

The orifice is sized to the flow range required by application, and manufacturers will offer a range of flows and guidelines for selection. A typical offering would be several ranges from 10 sccm to 10,000 sccm. Note that the flow rating is based on the orifice size under typical upstream and downstream pressure conditions with nitrogen gas (see the orifice section of Chapter 4).

## **PID Control**

PID stands for Proportional - Integral - Derivative. Referring back to Figure 7.1, the box on the left hand side is the pressure controller. This controller has two inputs. The first is for the setpoint. This is the adjustment for the desired system pressure. The second is the actual pressure input from the vacuum system’s pressure gauge. The application usually requires a CDG due to the need for accuracy, response speed and gas-type insensitivity.

These two signals feed into an error circuit. The error circuit determines the difference between the two inputs and then provides an output related to the error. To the left of the error element is a deviation indicator that provides the operator with a graphical representation of the magnitude of the error and whether the error is on the high side of the setpoint or below.

The error signal then progresses to the PID circuitry. The PID elements function as follows:

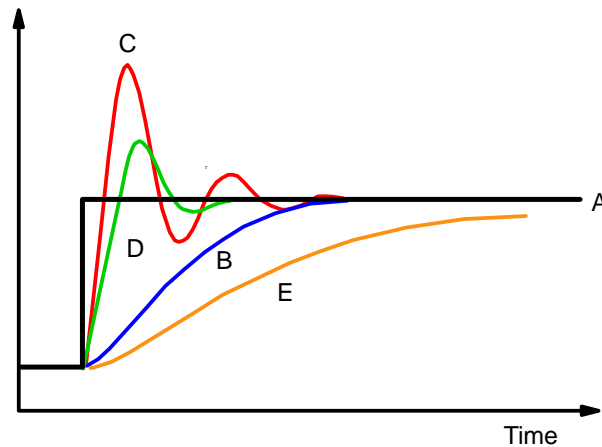
**Proportional:** This element provides a response that is instantaneous and is a directly proportional to the amount of error. It is also called gain. If the proportional setting is too low, there will only be a weak response to a large error. Conversely a high proportional setting will produce too large a response to a small error with the result that the system may become unstable and oscillate.

**Integral:** This element provides an additional signal that is directly related to the length of time that an error signal has existed. For example, if the pressure almost reaches the setpoint but there exists a persistent error, the integral element will integrate the error with time and provide a correction.

**Derivative:** This element provides a signal that is proportional to the rate of change of the error signal. It slows down the response as the system approaches the setpoint. This element is used to reduce the amount of overshoot that may be produced by the other elements but will also slow down the overall time for the system to reach the setpoint.

As a thought experiment, think about your actions when you step into a small shower stall and turn the water on after entering. You become part of a closed loop control system and unconsciously are using each of the elements described above. Depending upon the temperature of the water, your proportional element may be set very high.

Some representative response curves are shown in Figure 7.4.



**Figure 7.4** System response to various PID controller settings

“A” represents the ideal response to the setpoint change. The response in “B” is slow with no overshoot. In this case the derivative element would be fairly strong. In the case of “C” the system reaches the setpoint quickly but overshoots and oscillates about the setpoint for a few cycles. Increasing the proportional term would probably drive the system into oscillation. “D” also



overshoots but the system settles fairly quickly. Finally, “E” represents a very slow response system with a persistent error that requires attention from the integral element.

The type of response that is applicable to a given vacuum system is highly dependent upon the application. In some cases overshoot is not desirable but on the other hand, time is money and getting to setpoint quickly can be very important in terms of manufacturing efficiency.

Upstream control is the simplest pressure control mode and can permit a single gas (or a premix of gases) to enter the system. Common examples of systems using upstream control include freeze dryers and single gas sputter deposition. Both require accurate pressure control and a specific gas for the process - nitrogen for freeze drying and argon for sputtering.

### Ratio Upstream Control Mode

This configuration is required where it is necessary to control pressure and also introduce multiple gases where the mass flows of the gases have to be supplied in a specific ratios due to reaction requirements.

One of the most common examples of a process that uses ratio control is reactive sputtering. The parameters that must be controlled are the chamber pressure and the ratio of argon to the reactive gas (oxygen or nitrogen). A typical ratio for this application might be 95% argon to 5% oxygen.

The solution is to replace the simple proportioning valve with a pair of mass flow controllers. Each gas is controlled by one of the MFCs in such a way that the proper mass ratios are maintained.

Referring to Figure 7.5, the output of the pressure controller is fed to a ratio control device. This

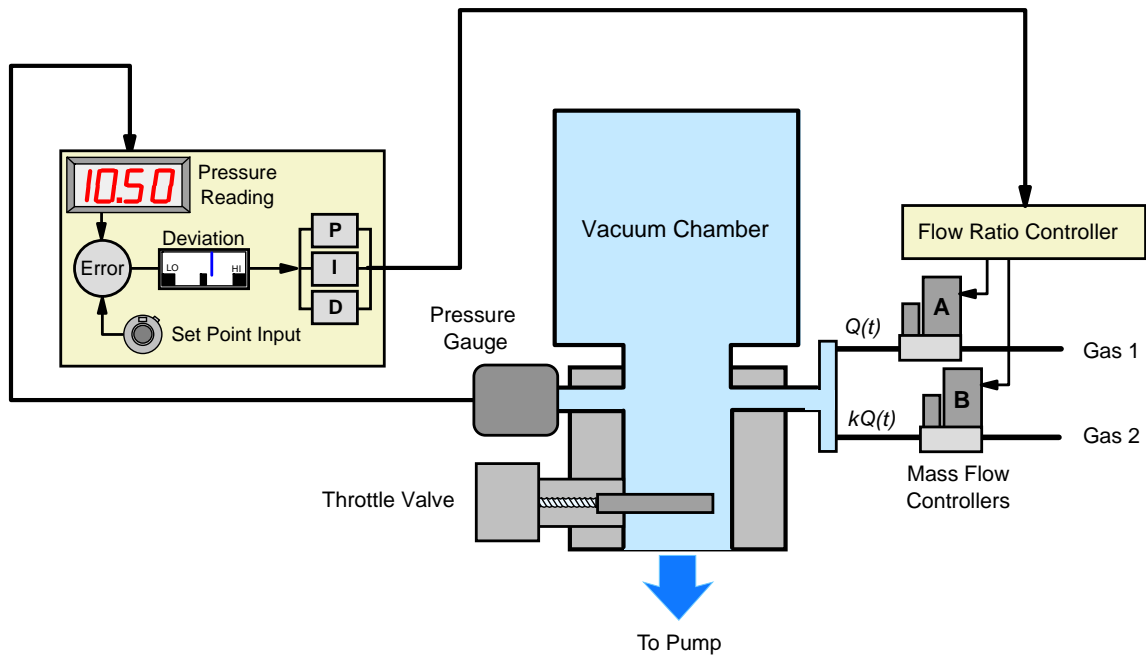


Figure 7.5 Ratio upstream control

simply apportions the total signal required between the two MFCs. If MFC “A” is defined as the master controller that is delivering some time varying  $Q$ , then MFC “B” will deliver some constant fraction of what “A” is delivering. The net result is a constant split or ratio of  $Q$  between the two MFCs but the total flow is adjusted in response to the pressure controller.

## Downstream Control Mode

This is the most complex and flexible of the pressure control methods. Downstream control is utilized when it is necessary to control the absolute quantities of each gas admitted to the system while also controlling pressure. Each gas will be controlled by an MFC, each one of which is providing a specific  $Q$ . This setup only controls the gas input to the system, not the pressure.

The pressure is controlled with a motorized throttle valve that is located between the chamber and the pump. The name of this configuration is derived from the fact that the pressure control element is downstream of the chamber.

Figure 7.6 depicts this configuration.

Downstream control is commonly used with processes such as chemical vapor deposition (see Chapter 10) where the reactant gases must be admitted not only in a specific ratio but also at a specific rate. This precludes ratio control as a method. In addition to this, the process pressure also has to be controlled within tight limits. Some processes may also change the gas flow rates during the process or introduce other gases through additional flow controllers.

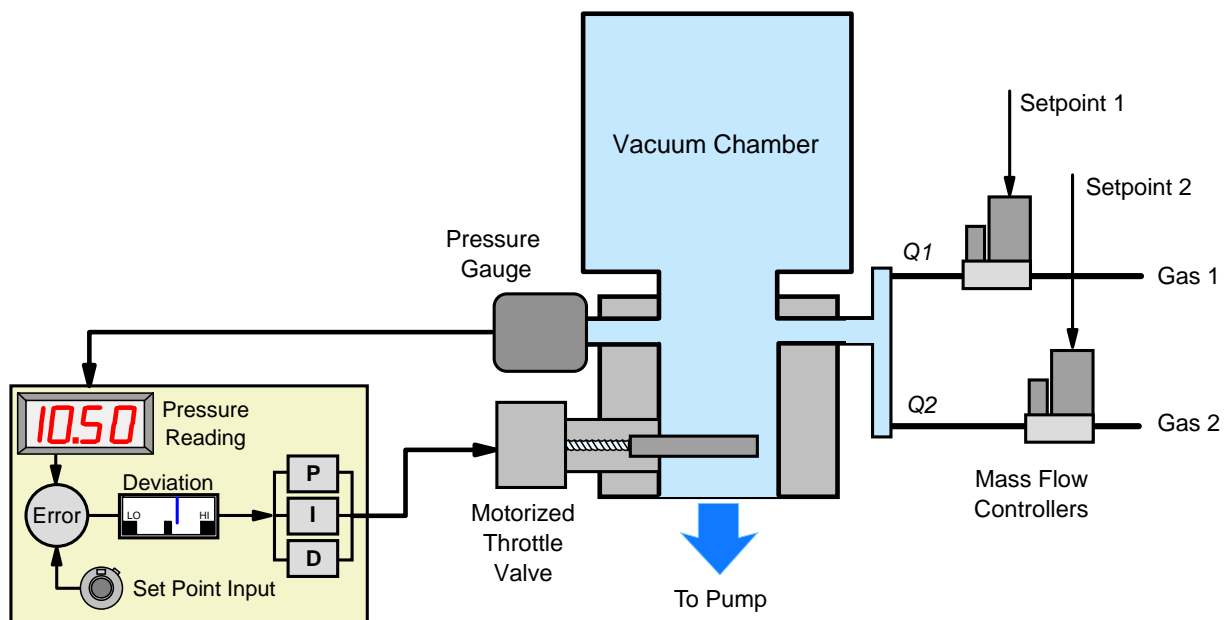


Figure 7.6 Downstream control

# **Section 2**

# **Vacuum Applications**

## Chapter 8      The Freeze Drying Process

### Introduction

Freeze drying (more formally known as *lyophilization*) is a very interesting process and one with a set of fascinating applications. Preserving the freshness of coffee and making “survival” food are couple of the well known examples of freeze drying. Freeze drying is widely used in the manufacture of pharmaceuticals. Museums use freeze drying as an alternative to taxidermy for the preservation of animal specimens ranging in size from near-microscopic organisms to alligators. Soft-shelled animals such as worms and spiders are particularly good subjects for freeze-drying. Medical schools are now using freeze-dried organs for study purposes. After drying, the tissue is impregnated with silicone resin. This gives the organ a “natural” feel without the foul smell and handling problems associated with formaldehyde-soaked tissue matter.

The freeze-drying process itself is quite instructive in that it vividly demonstrates such physical processes as adiabatic cooling, sublimation and pumping by condensation.

### Freeze Drying and the Phases of Water

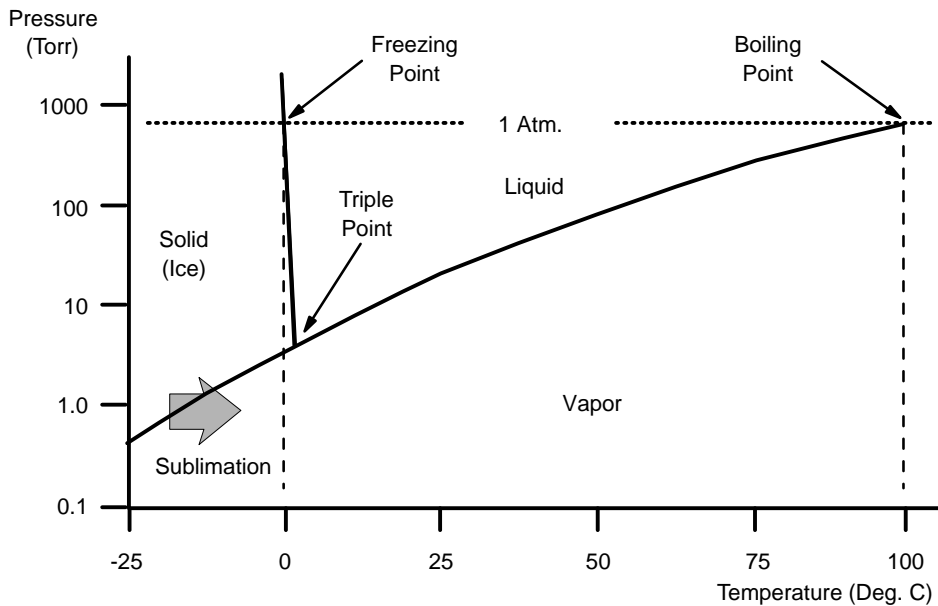
Freeze drying is a way of preserving organic matter. The basic idea is to stabilize a substance by removing essentially all of the water from that substance. This is done by lowering the temperature of the material until it is in a totally solid state, placing it in a low pressure environment, and then extracting the water by sublimation. By subliming the water (that is, by establishing a set of conditions that allow the solid ice to turn directly to water vapor without going through a liquid phase), the structure of the material is maintained.

Some products are also preserved by evaporative drying where the water is removed by simple evaporation at atmospheric pressure. This tends to collapse the cellular structure, the result of which is a distorted (microscopically and macroscopically) version of the original. The production of dried fruit is an example of this type of process.

The conditions which result in sublimation are shown in the phase diagram of water. This is depicted in Figure 8.1. At normal atmospheric pressure it can be seen that, as the temperature rises from sub-freezing, water goes from a solid state (ice) through water and then to a vapor as the water boils. If, however, the water is frozen and then the pressure is dropped to some level below about 4.5 Torr, the water will avoid the liquid phase as the temperature is increased. This is the pressure-temperature regime in which freeze drying operates.

The intersection of the three phases is the triple point. This exists when the pressure is about 4.5 Torr and the temperature is just above 0 °C. If you were holding a glass of ice water while floating along under a high altitude balloon at 50 km above sea level, the water would be boiling. No conjectures are offered on what would be happening to you!

Since there is a phase boundary between the solid and vapor phases at temperatures below freezing, sublimation can occur at higher pressures. This is simply a function of the vapor pressure of water. However, the process will be slow. This effect can be observed by placing an ice cube in a freezer



**Figure 8.1** Phase diagram of water

and noting that the size of the ice cube decreases over a period of days or weeks. There is no melting, it just evaporates. In freeze drying, the low pressure by itself speeds up the process. Additionally, it actually permits the sample to be heated, further speeding up the process.

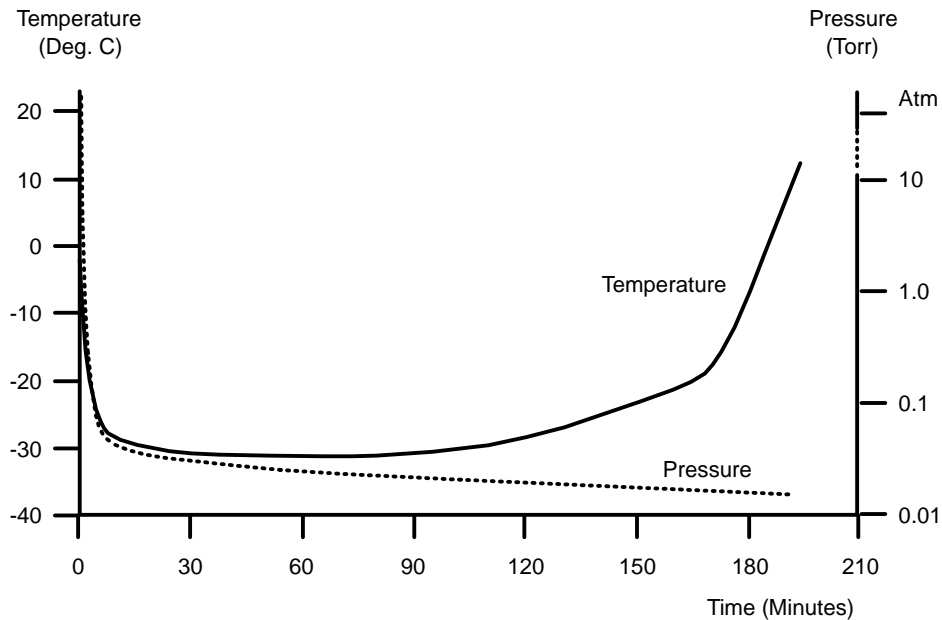
### Adiabatic Cooling

If a gas undergoes a rapid expansion, the temperature of the gas will drop. Likewise, if a gas is compressed rapidly, the temperature will rise. This effect is seen in tire pumps and is the principle behind refrigerators and air conditioners. This is called an adiabatic process and is related to the rate at which energy is added to, or taken away from, a system. Were the process to go slowly (e.g. slowly pumping up a tire), we have an isothermal process where the temperature remains at the ambient value.

In the case of freeze drying, we have a frozen sample that is introduced into a vacuum system. At low pressures, the evaporation rate increases resulting in adiabatic cooling which serves to keep the temperature of the sample low or, even reduce the temperature even further.

This effect can be demonstrated by placing a wet bulb thermometer (a standard glass thermometer with cotton wrapped around the bulb and moistened with water) into a vacuum chamber and pumping the system down. Once pumping has started, record pressure and temperature over time.

A typical curve is shown in Figure 8.2 on the next page. With regard to pressure, it should drop fairly quickly to below 1 Torr provided the pump has sufficient capacity. Close attention to the initial pumpdown will reveal various irregularities in the pressure decline as the water transitions from boiling to the frozen state. Once the water has frozen, the pressure will decline to an initial plateau and the temperature will stabilize at its lowest value.



**Figure 8.2** Adiabatic cooling experiment

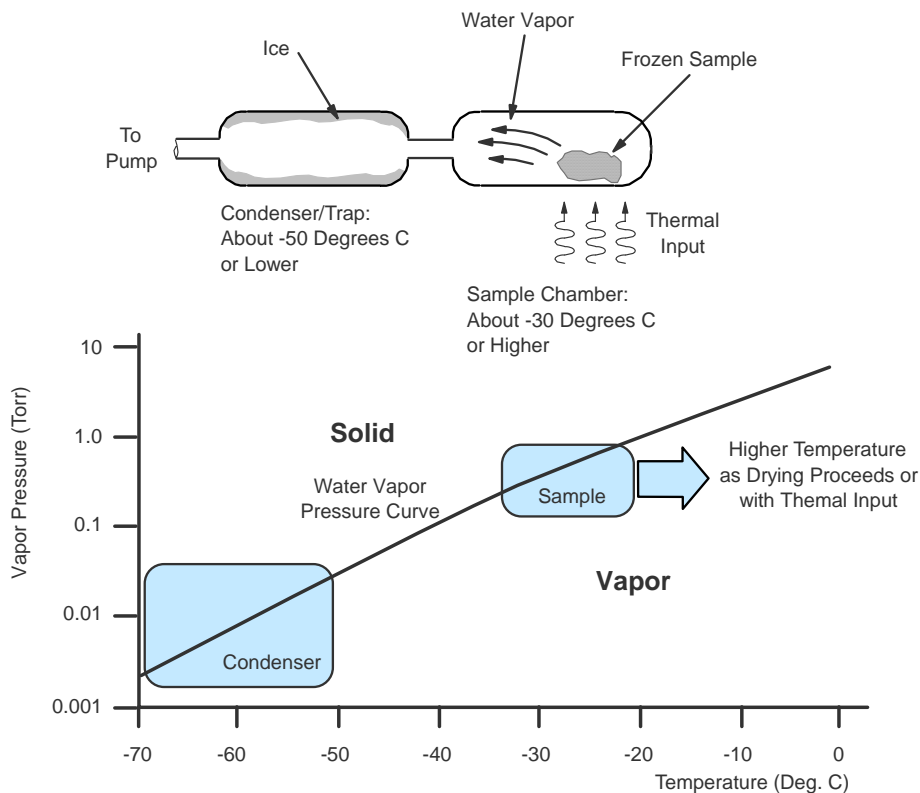
Over time, as the water evaporates, the temperature will rise and the pressure will fall. Eventually, the wick will be completely dry and the temperature will rise to ambient and the pressure will decline to the system's normal base pressure.

From this it can be seen that, once the sample is frozen and is placed in a low pressure environment, adiabatic cooling keeps the sample frozen. If we wanted to speed up the process, we could even heat the specimen without concern of melting. While there may be other reasons to avoid additional heating, pharmaceuticals are usually warmed to about +40 °C in the final stages of drying in order to drive off the more tightly bound water. This is called secondary drying.

### The Freeze Dryer

What was described above is a basic freeze dryer. Its major deficiency is how it handles the outgoing water vapor. With a large vapor load, the pump will have a hard time reaching base pressure and the oil will eventually become grossly contaminated with water, even with the gas ballast engaged. Thus, a "real" freeze dryer will have some sort of trap, or condenser, to catch the water before it gets to the pump. The condenser is an in-line device that is chilled to a lower temperature than that of the sample. This is usually in the range of -50 to -70 °C. Such temperatures can be reached with an alcohol/dry ice slurry for laboratory scale freeze dryers or with a mechanical refrigerator for industrial scale freeze dryers.

What the condenser actually does is to set up a pressure differential between the sample and the condenser. This creates a pumping action that draws the water away from the sample. Were the sample colder than the condenser, the migration would be in the wrong direction. This is also exactly how high vacuum liquid nitrogen traps and cryo pumps work. Figure 8.3 shows in schematic form the overall structure and operation of a freeze dryer in the context of the system elements, the associated temperatures, and the vapor pressure curve for water. Heating of the sample, as noted earlier, is usually done in the latter stages of drying.



**Figure 8.3** Freeze dryer schematic (top) and vapor pressure to temperature relationships (bottom)

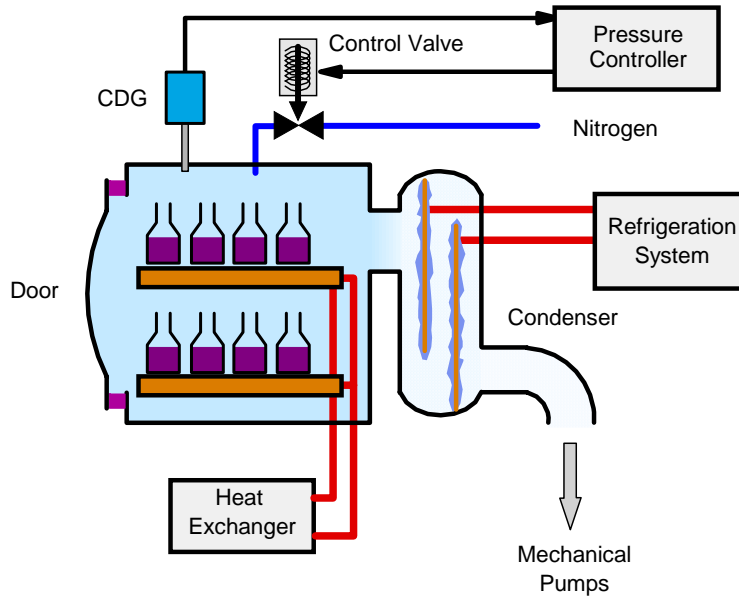
## An Industrial Freeze Dryer

Figure 8.4 depicts a freeze drying system as might be used in the pharmaceutical industry. The typical chamber is either a cylinder or a cube, about 1 meter across, with a full size door at the front. Inside the chamber are a number of trays that hold vials of serum. The trays are connected to a heat exchanger which is used to cool the trays for the initial freezing of the serum and then to heat the vials during secondary drying.

At the back of the chamber is a condenser with a number of chilled baffles. The condenser output connects to a high capacity mechanical pumping system. The condenser actually does most of the pumping as the vast majority of gas produced is water vapor.

To fully control the process the system incorporates upstream pressure control as described in Chapter 7. A CDG is used as the pressure gauge. It provides a high level of accuracy and is gas-type insensitive. This is especially important as the residual gas composition is a mix of water vapor and nitrogen with the ratio changing constantly during the process cycle. Gas admission is controlled by a solenoid proportioning valve and the pressure control elements are tied together with a PID pressure controller. Typical process pressures are in the range of 0.1 to 0.2 Torr.

At the end of the process the condenser is brought to room temperature in order to melt the ice and prepare the freeze dryer for the next run.



**Figure 8.4** Industrial freeze dryer



## Chapter 9      The Glow Discharge

### Introduction

“Of all the new phenomena wherewith the world has been enriched by modern physics, there is none more beautiful than the glow in a suitably rarefied gas. In the tube between the electrodes, the *viewless air* takes form and color; it seems to condense into luminous mists, to gather itself into islands of variously tinted cloud, which zones of darkness divide.

“The charm of these phenomena is nothing lessened by remembering that in history they make their first appearance as precursors of the conquest of our present wisdom. Modern atomic physics is the child of the vacuum pump. When pressures of the order of a millimetre were attained, the glow revealed itself in its greatest splendor. Attracted by the sight, physicists forged onward to lower and ever lower pressures as fast as better pumps were made. The splendor waned; but the change was simplification. Ultimately the glory of luminous clouds was gone; but by that time there were sharp and clearcut beams of radiation in the tube: one proceeding from the cathode into the zone of the discharge, others in the opposite sense....and a radiation arising from the places where the beam of the first mentioned hit the wall or any other obstacle. It then transpired that the rays of the first type were free electrons, those of the second were free ionized atoms, the last were photons of a frequency higher than any yet known. So came about the discovery of electricity released from matter, of ionized atoms freely wandering in space, and of the X-rays.”

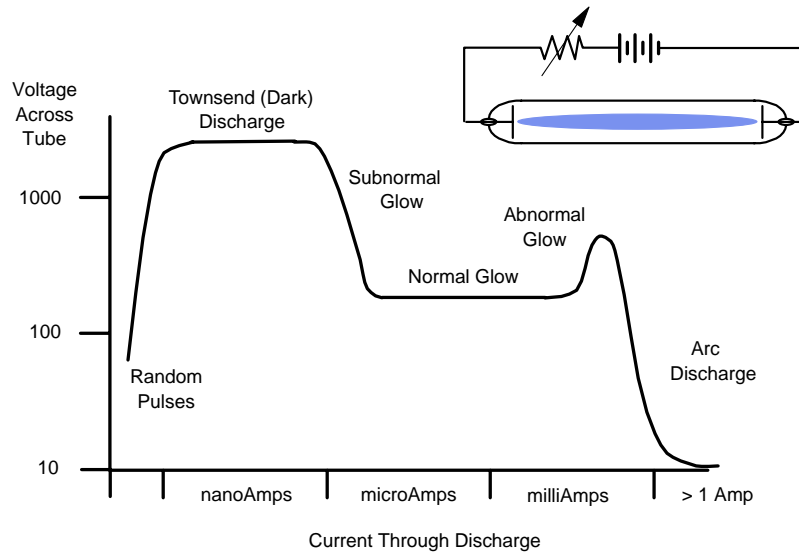
This is how Karl K. Darrow begins the chapter on the self-sustaining glow discharge in his 1932 book “Electrical Phenomena in Gases” (Williams & Wilkins Co., Baltimore). These two paragraphs poetically summarize the study of electrical discharges in vacuum beginning with Jean Picard’s observation in 1676 of luminous flashes in the “empty” space of a barometer tube to Wilhelm Roentgen’s discovery of the x-ray in 1895.

While the glow discharge phenomenon is distinct from vacuum technology, many of today’s most useful vacuum processes are dependent upon the glow discharge. In the next section we will examine the sputtering process, just one of the many low pressure plasma processes that are in use today.

### Evolution of the Glow Discharge

A glow discharge takes place in an evacuated tube with two electrodes. When a suitably high voltage is impressed across the tube, there will be a breakdown and the gas will form into a *plasma*, a neutral mix of positive ions and electrons.

Figure 9.1 depicts the voltage vs current relationship for a glow discharge tube of the dimensions that an amateur experimenter might work with 1 inch diameter electrodes placed about 18 inches apart. This tube would be operated at a pressure where the characteristic forms of the discharge will be revealed, about 1 Torr.



**Figure 9.1** Glow discharge voltage-current relationship

If the current is slowly ramped by decreasing the value of the resistor, the gas in the tube will go from a non-conducting state to one in which there are very low current random pulses (induced by, in the absence of anything else, the passage of naturally occurring ionizing rays) which create a very dim, sporadic illumination. Through this region the current rise is small compared with the increase in voltage drop. This eventually changes to a region where the voltage rise slows as current increases more drastically. In this region, the discharge is called *self-sustaining* as the current becomes independent of the external ionizing source, the applied voltage.

Increasing the current further leads to what we usually see as the typical glow discharge. There are three regions: the sub-normal glow, the normal glow, and the abnormal glow discharge. A discharge tube in the normal condition will show a bright region in close proximity to the cathode, the *negative glow* (see Figure 9.2). At first this sheath will only cover a portion of the cathode area. As the current through the tube rises, the negative glow will expand to cover the entire cathode. When the cathode is covered with the sheath, further current increases will drive the discharge into the abnormal region and the voltage will again rise.

Note that the voltage across the electrodes is very constant while the discharge is in the normal state. Finally, with higher levels of current, the tube will rise and then, with enough current, will go into a very low voltage arc discharge mode. This happens when the current density is high enough to vaporize material from the electrodes, causing a drastic drop in resistance as the resulting vapor plasma fills the tube.

The relatively low voltage that exists across the tube in the normal mode is a surprise to many, particularly when devices such as neon signs are driven by transformers in the 5 to 10 kV range. However, such transformers are really constant current devices: the high voltage capability is required to initiate the discharge but, as the current rises, the voltage drops to maintain a normal glow. If the transformer were not of special construction, the current would escalate resulting in arc formation and the destruction of the tube and transformer.

In other applications, such as in cold cathode electron guns, very high voltages can be sustained across the gap by supplying the potential in brief pulses. This drives the discharge into the abnormal region but keeps electrode heating to a low enough level that vaporization, and hence arc formation, does not occur.

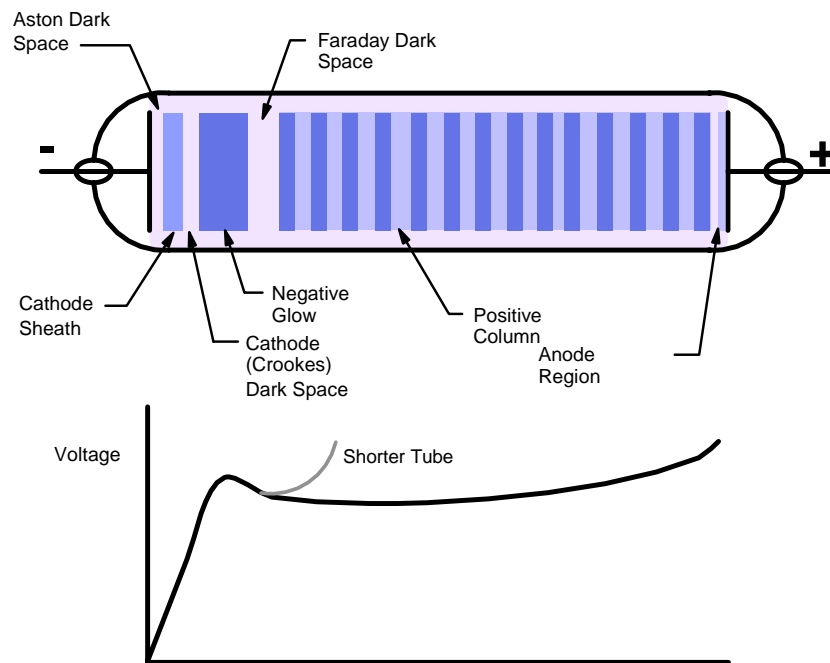
## Glow Discharge Regions and Characteristics

When a fully formed glow discharge is viewed, its appearance is similar to that of Figure 9.2. The details will change according to pressure and type of gas present but suffice it to say that there are two main groupings: the regions associated with the cathode (up to and including the Faraday dark space) and the regions associated with the anode.

The voltage distribution across the glow discharge is interesting. As shown in Figure 9.2, it is not linear. Instead, most of the voltage drop is near the cathode. This results in a high electrical field and this is the region where the electrons that are emitted from the cathode are accelerated. The fast electrons enter the negative glow where there is a high degree of ionization and this area tends to have a high positive space charge. In the positive column, the field strength is constant (as indicated by the gradual rise of voltage over the length of the column) and there is an equal distribution of positive ions and electrons.

At any given set of conditions (voltage and pressure) the negative region is a constant entity. If the pressure is lowered, the negative region will expand and the positive column will shrink. Also, as is shown in the lower illustration of Figure 6.2, if the electrodes are moved closer, the positive column will shrink to the point where it may almost completely disappear.

A long and bright positive column is needed in neon sign tubes. Such devices are operated at pressures in the vicinity of 5 Torr where the negative region is small. On the other hand, the

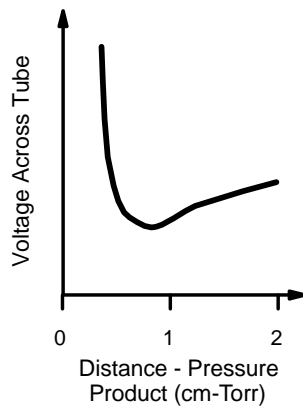


**Figure 9.2** Regions of the glow discharge and voltage distribution

sputtering process that will be discussed in the next section relies on the activity that occurs in the negative region and the dimensions and pressures are adjusted such that the positive column is of minimal length.

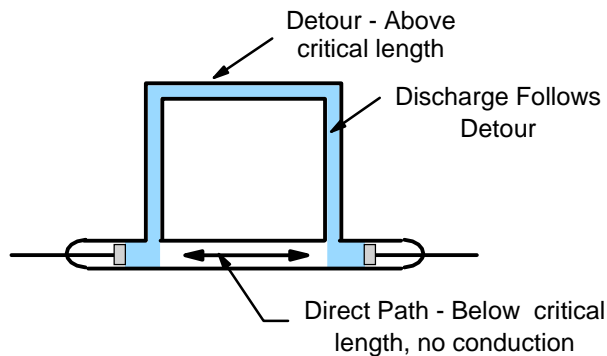
### The Obstructed Discharge

If the anode is moved even closer to the cathode, such that it closely approaches then enters the negative region, the voltage will have to rise in order to maintain a discharge. This is because there is less space for ionization to occur. The general form of the relationship is shown in Figure 6.3. Note that the sustaining voltage relates to the product of the pressure (in Torr) and the separation (in cm). A discharge that takes place in this regime to the left of the voltage minimum is called an *obstructed* discharge.



**Figure 6.3** Cathode fall voltage vs  $D \times p$

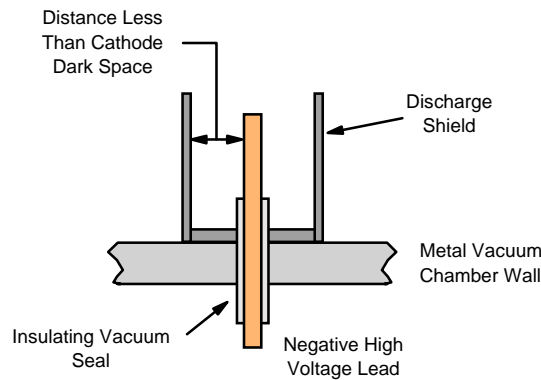
An interesting device that demonstrates this is the so-called *detour* tube developed by Hittorf in 1884. (See Figure 9.4.) Here the direct path between electrodes becomes shorter than the cathode region as the pressure in the tube is lowered. When that occurs, the discharge takes the longer route through the side tube where a normal glow discharge can form.



**Figure 9.4** Hittorf's detour tube

The occurrence of the voltage minimum in Figure 9.3 and the action of the Hittorf tube illustrates the *Paschen effect*, also known as the minimum sparking potential.

One practical use of this effect is for high voltage feedthroughs into vacuum chambers. (See Figure 9.5.) Instead of trying to insulate the negative connection (which will more than likely result in virtual leaks and other problems), if the lead-in is surrounded by a coaxial anode with a spacing that is smaller than the negative region, the gap will hold off a high voltage.



**Figure 6.5** High voltage feedthrough

## Visual Characteristics of the Glow Discharge

To complete this section, the tables below provide some details on the appearance of glow discharges based on gas type and pressure.

Discharge Color with Various Gases	
Air	Red to pink, paling at lower pressures
Water	Faint blue
Mercury	Blue
Carbon dioxide	Bluish green
Oxygen	Pale yellow & pink
Nitrogen	Red
Helium	Red/violet
Neon	Brick red
Argon	Dark red
Decomposed oil	Gray/green

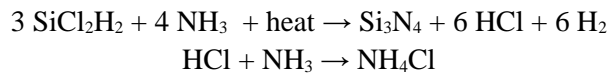
Appearance of Discharge at Various Pressures	
50 Torr	Thin red/violet streamer
10	Streamer fills tube
2	Striations in positive column
0.5	Striations spaced 10mm
0.1	Crookes dark space forms
0.01	Green fluorescence of glass from x-rays
0.001	Black out (gas becomes non-conductive)

# Chapter 10 Physical Vapor Deposition by Sputtering

## Introduction: Chemical and Physical Vapor Deposition

Vacuum deposition of substances on substrate materials may be performed by either of two broad classes of process.

In *chemical vapor deposition* (CVD) a mixture of reactive gases is made to flow over a heated substrate. When the gases contact the substrate a reaction occurs. The result of the reaction is the deposition of a thin film of material. Byproducts of the reaction are then drawn off by the pumping system. An example of CVD is the deposition of silicon nitride films from the reaction of dichlorosilane and ammonia. The deposited film is silicon dioxide. The primary byproducts are hydrogen chloride and hydrogen although there is a secondary (and undesirable) reaction between the ammonia and hydrogen chloride resulting in ammonium chloride, a high vapor pressure material that, while gaseous in the heated chamber, will condense on the vacuum lines upon exit from the hot zone. The condensate is generally handled with a cooled trap in the exhaust line of the chamber.



CVD is widely used for the deposition of dielectrics and hard coatings (for example the wear resistant coatings on cutting tools).

*Physical vapor deposition* (PVD) is purely or primarily a physical process. There are two widely used PVD processes: vacuum evaporation and sputtering.

Evaporation is a physical deposition process that involves heating a material up to the point where the vapor of that substance is freely evolved. At low pressures (typically below  $10^{-4}$  Torr) the molecules of that substance travel directly away from the source and then condense on the article which is to be coated. Evaporation is a good technique for many metals and some inorganic compounds (aluminum, copper, magnesium oxide, etc.) but is not useful for organics, refractory materials and substances that either have too low a vapor pressure or which decompose or react at the elevated temperatures required for evaporation. Evaporation is a very fast and economical process. Common vacuum evaporated films include the aluminum coatings on mirrors and tinted coatings on architectural glass. In the 1920s and '30s vacuum evaporation revolutionized the optical industry.

Sputtering is also a physical process but relies on a different mechanism. In this brief overview we'll look at the evolution of the understanding of the sputtering mechanism and will discuss some configurations of sputtering systems.

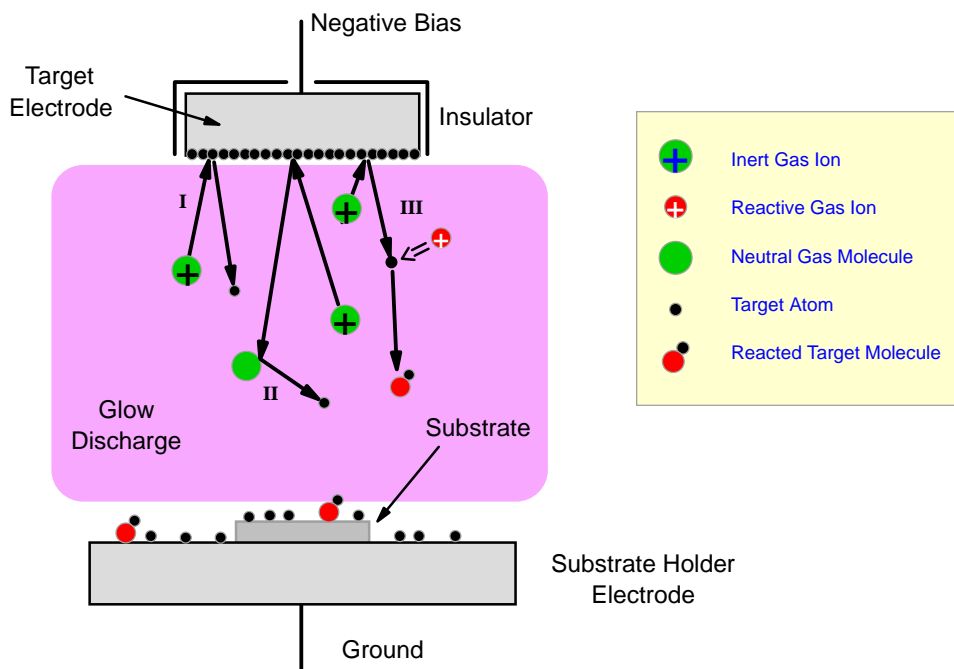
## What is Sputtering?

Sputtering is a material transfer process that occurs between electrodes in a glow discharge system. The process was first observed by W.R. Grove in 1852. Sputtering was first thought to be an electrochemical process, where the biased electrodes and glow discharge were analogous to anode, cathode and electrolyte of a plating cell. A true understanding of the fundamental processes behind sputtering took the better part of a century after Grove's early studies. The process worked, even if it wasn't understood.

The term "sputtering" is believed to have its origins in the Dutch word "sputteren" which means "To spit out in small particles and with a characteristic explosive sound" and the word is commonly used in English to indicate confused and uncontrolled speech, as in "to sputter and stutter." Applied to the vacuum deposition process, the word is an attempt to describe the way that matter is ejected from the cathode of the discharge apparatus.

## The Sputtering Process

Figure 10.1 shows the situation in a simple two electrode configuration. A glow discharge is established between two electrodes. The proper condition is in the abnormal glow region where there is sufficient current to cover the cathode with the glow. (This is facilitated by shielding the unused - back and sides - portion of the cathode with an insulator as shown.) The glow discharge plasma consists of electrons and gas ions. The ions, positively charged, will be accelerated toward the cathode. When an ion hits the cathode, an atom of the material comprising the cathode will be ejected. This ejected atom will then propagate at high speed away from the cathode with some of them arriving at the substrate. (See item 'I' in the figure.) In a sputtering system, the cathode surface is called the target as this is what the ions aim toward.



**Figure 7.1** The sputtering mechanism

Ideally, the target atoms that impinge on the substrate will get there without any intervening collisions with gas molecules. This requires a mean free path on the order of the target to substrate distance. If the mean free path is short, a situation as shown in item 'II' will result with the target atom being deflected and slowed down by the collision. Target to substrate separations are usually on the order of a few cm so pressures in the range of a few milliTorr are generally selected. Remember, too low a pressure and the discharge will go out.

In some cases these collisions can be desirable. For example, if an irregular substrate is to be coated, a non-collimated cloud of target atoms will provide a more uniform coating. This is often employed when coating samples for examination under a scanning electron microscope. Here the pressures may be in the tenth of a Torr range where mean free path is on the order of a millimeter.

The choice of the operating gas is important. It should provide a fairly massive ion as the sputtering rate increases with heavier target ions. It should be easy to ionize and it should be non-reactive (more on that attribute later). Finally it should be fairly plentiful and inexpensive. Argon meets these requirements well and it is pretty much the standard sputtering gas.

Concerning the non-reactive aspect, if a reactive gas such as oxygen or nitrogen is present in the system, there will be reactive ions in the space between the target and the substrate. A target atom such as aluminum could react with the gas ions and form the compound aluminum oxide or aluminum nitride. The reactions can happen in the target-substrate gap (see item 'III' in the figure), at the target surface, or at the substrate surface. To avoid this it is important to reduce the amount of reactive residuals to very low levels. The simplest approach is to pump the chamber down to base pressure (if only a mechanical pump is used, this would be on the order of a few milliTorr), purge with argon, repump and repeat the process several times. This is usually good enough for non critical applications. For high quality films it is necessary to pump the system to very low base pressures (at least  $10^{-6}$  Torr and, for semiconductor processes, more like  $10^{-9}$  Torr).

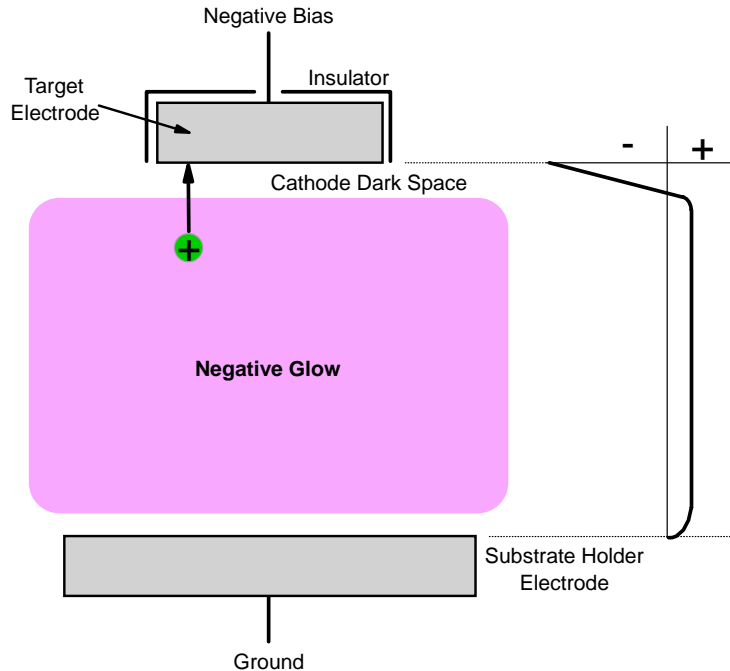
In some cases this ability to produce reacted species is important and is one of the reasons why sputtering has become an essential process in the electronics industry. For example, if it is desired to produce a thin film capacitor, a pure film of aluminum (one plate of the capacitor) can be produced by sputtering in pure argon. When enough thickness has been built up, a small but controlled amount of oxygen (usually around 5%) is bled in with the argon to produce an aluminum oxide dielectric. When enough of the insulating layer has been deposited, the oxygen is shut off and the process reverts to depositing pure aluminum, forming the other plate of the capacitor. By just changing the gas mixture, a three layer structure is produced. This variant of sputtering is called reactive sputtering.

Finally, if the polarity of the electrodes is reversed, the argon ions will impinge on the substrate and will cause some of the substrate to be removed. This "ion milling" process is often used to clean a substrate prior to the sputter deposition step. Ion milling is also used to thin specimens as part of the preparation process for transmission electron microscopy (TEM).



## Potential Distribution in a DC Plasma

Sputtering plasmas are operated in the abnormal glow regime where the plasma sheath covers the full area of the electrodes. Furthermore, the plasma is in the obstructed configuration where there is only a negative glow and a large cathode voltage drop. This provides high ion energies with vertical incidence. Figure 7.2 shows the potential distribution of a DC plasma in this state.



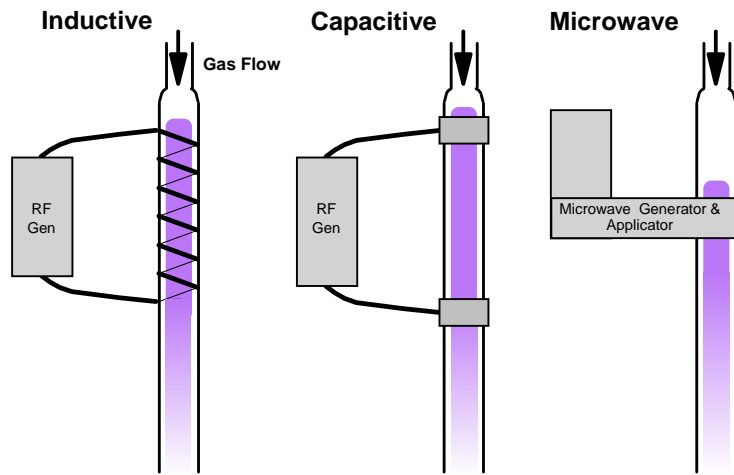
**Figure 10.2** Potential distribution in a sputtering discharge

## The RF Plasma

DC glow discharges draw a true electrical current from the power supply in the form of electrons and ions. The physical processes are dominated by the axial DC electric fields. In the case of AC fields, the ions and electrons will oscillate with the changing field. When the power supply frequency is raised to the MHz range the period of the oscillations will become comparable to the time that it takes the electrons, and especially the ions, to traverse the sheath between the plasma and the electrodes. At such frequencies the power supply interacts with the plasma by displacement rather than by real currents.

The net effect of this is that the power can be delivered to the plasma without the requirement for electrodes that are in contact with the plasma. The absence of current carrying electrodes in contact with the plasma also tends to improve the reliability, reproducibility and lifetime of the plasma reactors themselves and of the products made with them.

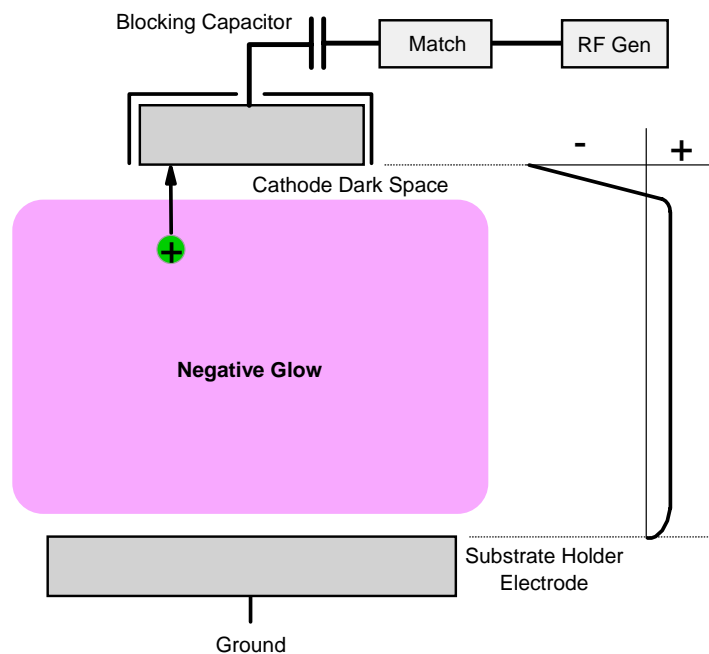
There are a number of methods that are used to couple radio frequency power to plasmas and these are pictorially depicted in Figure 10.3.



**Figure 10.3** RF and microwave coupling mechanisms

Because of the high electron mobility in a plasma (RF or DC), an RF diode system tends to develop large electron currents during the positive portion of the applied RF cycle. Usually a large variable capacitor (500-2000 pF) is placed in series between the RF power supply and the powered electrode. This capacitor allows a significant negative bias to develop on the cathode, typically half of the value of the applied peak-to-peak RF voltage. This bias is then the acceleration voltage for ions from the plasma, which move much too slowly to respond to the applied RF potentials. A major use of RF plasmas is in the sputtering of dielectrics where DC supplies are ineffective.

In addition to the series blocking capacitor, it is important to use two other tuning components to help match the complex impedance of the plasma to the 50 ohm output impedance of the RF power supply. These components usually consist of a variable shunt capacitor (connected to ground) and a series fixed inductor. They are located with the series capacitor adjacent to the cathode to minimize interconnect length. A schematic depiction of an RF processing system is shown in Figure 10.4.



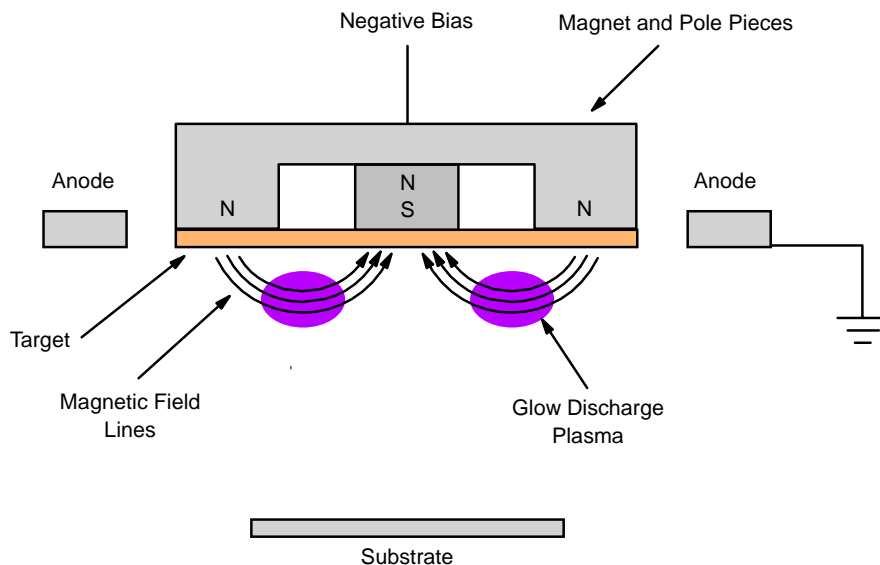
**Figure 10.4** RF sputtering configuration

## Magnetron Sputtering

One of the most commonly encountered sputtering configurations is the magnetron configuration. This is depicted schematically in Figure 10.5. As the name implies, a magnetic field is introduced by placing one or more permanent magnets behind the target. This arrangement produces a planar magnetron - opposing poles are present across the surface of the target. Assuming a circular configuration, the south pole appears as a centrally located disk, the north pole is a ring that surrounds the south pole.

The magnetic field serves to concentrate the electrons, hence the ionization, to a toroidal region in close proximity to the target. This improves the stability of the discharge, especially at low pressures, and increases the flux of ions thereby increasing the efficiency of the process.

Figure 10.5 shows another change from the simple arrangement of Figure 10.1. Here the anode is moved away from the substrate where it is now coplanar with the target/cathode. The substrate is now completely out of the plasma, a feature that can be important if the substrate is sensitive to high densities of charged ions.



**Figure 7.5** Planar magnetron electrode configuration

## Sputter Yield

The deposition rate of the sputtering process is dependent upon the type of incident ion, the energy of those ions. It is also dependent upon the material being sputtered, specifically the binding energy of the atoms that comprise the target. Relative to aluminum and other conditions being constant, silver has a yield of 2.8, copper is 2.0, gold is 1.7 and silicon is 0.50.

# Appendix

## Appendix: Further Reading

### **Vacuum Science & Technology**

John O'Hanlon, *A User's Guide to Vacuum Technology*, 3<sup>rd</sup> edition, Wiley-Interscience, 2003

Alexander Roth, *Vacuum Technology*, 3<sup>rd</sup> edition, North Holland 1990

James M. Laferty (Editor), *Foundations of Vacuum Science and Technology*, Wiley-Interscience, 1998

Marsbed H Hablanian, *High-Vacuum Technology*, 2<sup>nd</sup> edition, Marcell Dekker, 1997

T.A. Delchar, *Vacuum Physics and Techniques*, Springer, 1993

### **Plasma Processes**

Brian Chapman, *Glow Discharge Processes*, John Wiley & Sons, 1980

J. Reece Roth, *Industrial Plasma Engineering: Principles*, Vol. 1, Taylor & Francis, 1995

J. Reece Roth, *Industrial Plasma Engineering: Applications*, Vol. 2, Taylor & Francis, 2001

### **History of Vacuum Science & Technology**

Paul A Redhead (Editor), *Vacuum Science and Technology: Pioneers of the 20th Century*, American Institute of Physics, 1997

### **Web Resources (on line catalogs of vacuum hardware and components)**

Kurt J. Lesker Company <http://www.lesker.com>

MKS Instruments <http://www.mksinst.com>

