A User's Guide to Vacuum Technology

Third Edition

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WILEY-
INTERSCIENCE

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Published by John Wiley & Sons, Inc., Hoboken, New Jersey.
Published simultaneously in Canada.

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Library of Congress Cataloging-in-Publication Data:

Library of Congress Cataloging-In-Publication Data is available:
0-471-27052-0

Printed in the United States of America.

10 9 8 7 6 5 4 3 2 1

For Jean, Carol, Paul, and Amanda

Preface

This book is intended for the vacuum system user—the university student, technician, engineer, manager, or scientist—who wishes a fundamental understanding of modern vacuum technology and a user's perspective of modern laboratory and industrial vacuum technology.

Vacuum technology is largely secondary; it forms part of other technologies that are central to analysis, research, development, and manufacturing. It is used to provide a process environment. Many advances in vacuum technique have resulted from the demands of other technologies, although scientists and engineers have studied vacuum for its own sake. The average user is process-oriented and becomes immersed in vacuum technique only when problems develop with a process or when new equipment purchases become necessary.

A *User's Guide to Vacuum Technology, 3rd Edition* focuses on the operation, understanding, and selection of equipment for processes used in semiconductor, optics, and related technologies. It emphasizes subjects not adequately covered elsewhere, while avoiding in-depth treatments of topics interesting only to the designer or curator. Residual gas analysis is an important topic whose treatment differs from the usual explanation of mass filter theory. Components such as the turbomolecular and helium gas refrigerator cryogenic pumps are now widely used but not well understood. The discussion of gauges, pumps, and materials is a prelude to the central discussion of systems. System designs are grouped according to their function. Current designs are either single-chamber or multichamber; the details of each design are determined by the requirements of an industrial or research application.

In this edition, the discussion of gauges, pumps, and materials has been updated, where relevant, to reflect changes in practice. Spinning rotor gauges are no longer a laboratory curiosity. Ultrahigh vacuum gauges, though limited in their availability, will be a necessity in next-generation production deposition systems. Ultraclean, low dead volume metrology and valves, along with superior materials and cleaning techniques, have made contamination-free manufacturing a reality.

Ultraclean vacuum, once the domain of the researcher, is now routinely used for high-volume production of semiconductor chips and storage media. However, methodologies for reaching low pressures in a clean manner have changed significantly. No longer are single-chamber systems baked for twenty-four hours. Rather, cassette-based load/unload chambers serve as high-volume interfaces between atmosphere and ultraclean process chambers. These chambers, which can be accessed in serial or random order, are only exposed to atmosphere during maintenance.

Large, efficient multichamber medium and highvacuum systems are used in high-speed coating of numerous consumer products such as window glass, solar cells, video tape, printer paper, eyeglass lenses, automobile headlamps, plastic films and security devices.

The gap in knowledge and training between those who manufacture and those who use vacuum equipment continues to widen. It is from this perspective that the previous edition of this book has been revised. Important formulas have been denoted with a ▶ for emphasis. Easy questions have been emphasized with a †.

Thanks are due to countless researchers who, individually and collaboratively, have advanced this field by creative solutions to real problems; I also thank Dr. Bruce Kendall for his insightful comments and thoughtful review.

J. F. O'Hanlon

Tucson, Arizona

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Its Basis

An understanding of how vacuum components and systems function begins with an understanding of the behavior of gases at low pressures. Chapter 1 discusses the nature of vacuum technology. Chapter 2 reviews basic gas properties. Chapter 3 describes the flow of gases at reduced pressures, and Chapter 4 discusses how gas is evolved from the surfaces of materials. Together, these chapters form the basis of vacuum technology.

CHAPTER 1

Vacuum Technology

Torricelli is credited with the conceptual understanding of the vacuum within a mercury column by 1643. It is written that his good friend Viviani actually performed the first experiment, perhaps as early as 1644 [1,2]. His discovery was followed in 1650 by Otto von Guericke's piston vacuum pump. Interest in vacuum remained at a low level for more than 200 years, when a period of rapid discovery began with McLeod's invention of the compression gauge. In 1905 Gaede, a prolific inventor, designed a rotary pump sealed with mercury. The thermal conductivity gauge, diffusion pump, ion gauge, and ion pump soon followed, along with processes for liquefaction of helium and refinement of organic pumping fluids. They formed the basis of a technology that has made possible everything from light bulbs to space simulation. The significant discoveries of this early period of vacuum science and technology have been summarized in a series of historical review papers [2–7].

A vacuum is a space from which air or other gas has been removed. All gas cannot be removed. The amount removed depends on the application, and is done for many reasons. At atmospheric pressure molecules constantly bombard surfaces. These molecules can bounce from surfaces, attach themselves to surfaces, or perhaps chemically react with surfaces. Air or other surrounding gas quickly contaminates a cleaned surface. A clean surface—for example, a freshly cleaved crystal—will remain clean in an ultrahigh vacuum chamber for long periods of time, because the rate of molecular bombardment is low.

Molecules are crowded closely together at atmospheric pressure and travel in every direction much like people in a crowded plaza. It is impossible for a molecule to travel from one wall of a chamber to another without colliding with many molecules. By reducing the pressure to a suitably low value, a molecule from one wall can travel to another without a collision. Many effects become possible if molecules can travel long distances between collisions. Metals can be evaporated from a pure source without reacting in transit. Molecules or atoms can be accelerated to a high energy and sputter away, or be implanted in the bombarded surface. Electrons or ions can be scattered from surfaces and be collected. The energy changes they undergo on scattering or release from a surface can be used to probe or analyze the surface or underlying layers. For convenience the subatmospheric pressure scale has been divided into several ranges. Table 1.1 lists these ranges.

The required vacuum level depends on the application. Epitaxial growth of semiconductor films (reduced pressure epitaxy) and laser etching of metals are two processes that are performed in the low vacuum range. Sputtering, plasma etching and deposition, low-pressure chemical vapor deposition, ion plating, and gas filling of encapsulated heat transfer modules are examples of processes performed in the medium vacuum range.

Pressures in the high vacuum range are needed for the manufacture of traditional low- and high-tech devices such as microwave, power, cathode ray and photomultiplier tubes, light bulbs, architectural and automotive glazing, decorative packaging, degassing of metals, vapor deposition, and ion implantation. A number of medium technology applications including medical, microwave susceptors, electrostatic dissipation films, and aseptic packaging use films fabricated in a vacuum environment [8]. Retail security, bank note security, and laser and inkjet paper have joined this group.

The background pressure must be reduced to the very high vacuum range for electron microscopy, mass spectroscopy, crystal growth, and x-ray and electron beam lithography, and storage media production. For ease of reading, we call the very high vacuum region "high vacuum" and call the pumps "high vacuum pumps."

Pressures in the ultrahigh vacuum range were formerly the domain of the surface analyst, materials researcher, or accelerator technologist. Critical high-volume production applications, such as semiconductor devices, thin-

Table 1.1 Vacuum Ranges

Degree of Vacuum	Pressure Range (Pa)^a
Low	$10^5 > P > 3.3\times10^3$
Medium	$3.3\times10^3 \geq P > 10^{-1}$
High	$10^{-1} \geq P > 10^{-4}$
Very high	$10^{-4} \geq P > 10^{-7}$
Ultrahigh	$10^{-7} \geq P > 10^{-10}$
Extreme ultrahigh	$10^{-10} > P$

Source: Reprinted with permission from *Dictionary for Vacuum Science and Technology*, M. Kaminsky and J. M. Lafferty, Eds., American Vacuum Society, New York, 1980.

[a] 101323.3 Pa = 1 atmosphere.

film media heads, and extreme UV lithography systems, require ultrahigh vacuum base pressures to improve yield by reducing gaseous impurity contamination. Additionally, processes carried out in these systems must be free of particle contamination, so we call them ultraclean vacuum systems.

A vacuum system is a combination of pumps, valves, and pipes, which creates a region of low pressure. It can be anything from a simple mechanical pump or aspirator for exhausting a vacuum storage container to a complex system such as an underground accelerator with miles of piping that is maintained at ultrahigh vacuum.

Removal of air at atmospheric pressure is usually done with a displacement pump. A displacement pump is one that removes the air from the chamber and expels it to the atmosphere. Rotary vane and piston pumps are examples of pumps used to exhaust gases at atmospheric pressure. Liquid nitrogen capture pumps or sorption pumps have also been designed for exhausting gases at atmospheric pressure. They are used only on small chambers because of their finite gas sorption.

Rotary vane, piston and sorption pumps have low-pressure limits in the range 10^{-1}–10^{-3} Pa. Pumps that will function in a rarefied atmosphere are required to operate below this pressure range. Several displacement and capture pumps can remove air at these low pressures. The diffusion pump was the first high vacuum pump. It is a displacement pump. Its outlet pressure is below atmosphere. The turbomolecular pump, a system of high-speed rotating turbine blades, can also pump gas at low pressures. The outlet pressures of these two pumps need to be kept in the range 0.5–50 Pa, so they must exhaust into a rotary vane or piston "backing" pump, or "fore" pump. If the diffusion or turbomolecular pump exhaust gas flow would otherwise be too great, a lobe blower will be placed between the exhaust of the diffusion or turbomolecular pump and the inlet of the rotary pump to pump gas at an increased speed in this intermediate pressure region.

Capture pumps can effectively remove gas from a chamber at low pressure. They do so by freezing molecules on a wall (cryogenic pump), chemically reacting with the molecules (getter pump), or accelerating the molecules to a high velocity and burying them in a metal wall (ion pump). Capture pumps are more useful as high vacuum pumps than as atmospheric exhaust pumps because the number of molecules to be captured at high vacuum is less than the number removed during initial evacuation from atmosphere.

Air is the most important gas to understand, because it is in every vacuum system. It contains at least a dozen constituents, whose major constituents are described in Table 1.2. The differing ways in which pumps remove air, and gauges measure its pressure, can be understood in terms of the partial pressures of its components. The concentrations listed in Table 1.2 are those of dry atmospheric air at sea level (total pressure

Table 1.2 Components of Dry Atmospheric Air

Constituent	Content (vol. %)	Content (ppm)	Pressure (Pa)
N_2	78.084 ± 0.004		79,117
O_2	20.946 ± 0.002		21,223
CO_2 [a]	0.037		37.5
Ar	0.934 ± 0.001		946.357
Ne		18.18 ± 0.04	1.842
He		5.24 ± 0.004	0.51
Kr		1.14 ± 0.01	0.116
Xe		0.087 ± 0.001	0.009
H_2		0.5	0.051
CH_4		2.	0.203
N_2O		0.5 ± 0.1	0.051

Source: Reprinted with permission from *The Handbook of Chemistry and Physics*, 59th ed., R. C. Weast, Ed., copyright 1978, The Chemical Rubber Publishing Co., CRC Press, Inc., West Palm Beach, FL 33409.

[a] Carbon dioxide data from Mauna Kea, Hawaii, 2000. Data since 1955 are available as: http://stratus.mlo.hawaii.gov/Projects/GASES/co2graph.htm.

101,323.2 Pa or 760 Torr). The partial pressure of water vapor is not given in this table, because it constantly changes. At 20°C a relative humidity of 50% corresponds to a partial pressure of 1165 Pa (8.75 Torr), making it the third largest constituent of air. The total pressure changes rapidly with altitude, as shown in Fig. 1.1, whereas its proportions change slowly but significantly. In outer space the atmosphere is mainly H_2 with some He [6].

In the pressure region below 10 Pa, gases evolving from material surfaces contribute more molecules per second to the total gas load than do the gases originally filling the chamber. The correct pump is not the only requirement needed to reach low pressures—the materials of construction, techniques for joining components, surface cleaning techniques, and operational procedures are all critically important. In the remaining chapters the pumps, gauges, and materials of construction and operational techniques are described in terms of fundamental gas behavior. The focus is on the understanding and operation of vacuum systems for a variety of technological applications.

1.1 UNITS OF MEASUREMENT

Units of measurement present problems in many disciplines and vacuum technology is no exception. The use of noncoherent vacuum units has been common in the US long after the adoption of System International.

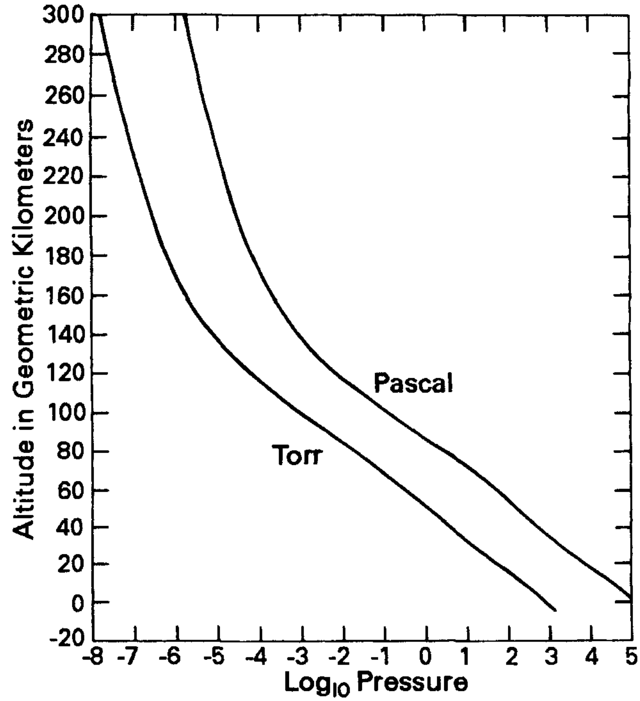

Fig. 1.1 Relation between the atmospheric pressure and the geometric altitude. Reprinted with permission from *The Handbook of Chemistry and Physics*, 59th ed., R. C. Weast, Ed. copyright 1978, The Chemical Rubber Publishing Co., CRC Press, Inc., West Palm Beach, FL 33409.

The meter-kilogram-second (MKS) system was first introduced over a half-century ago; its use became commonplace only after a decade or more of classroom education by instructors committed to change. In a similar manner, those who teach vacuum technique will lead the way to routine use of SI units. Instruments are manufactured for use in a global economy and their readings can be displayed in several formats. The advantages of using a coherent unit system are manifold. Calculations become straightforward and logical and the chance for error is reduced. Incoherent units such as permeation constant, the volume of gas (at standard temperature and pressure) per material thickness per material area per sec pressure difference, are cumbersome. Additionally, these permeation units mask their relation to solubility and diffusion. Ultimately, SI units will be routinely used. To assist with this change, dual labels have been added throughout the text. Basic SI units for pressure (Pa), time (s) and length (m) will be assumed in all formulas, unless noted differently within a formula statement.

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CHAPTER 2

Gas Properties

In this chapter we discuss the properties of gases at atmospheric and reduced pressures. The properties developed here are based on the kinetic picture of a gas. Kinetic theory has its limitations, but with it we are able to describe particle motion, pressure, effusion, viscosity, diffusion, thermal conductivity, and thermal transpiration of ideal gases. We will use these ideas as the starting point for discussing gas flow, gauges, pumps and systems.

2.1 KINETIC PICTURE OF A GAS

The kinetic picture of a gas is based on several assumptions. (*i*) The volume of gas under consideration contains a large number of molecules. A cubic meter of gas at a pressure of 10^5 Pa and a temperature of 22°C contains 2.48×10^{25} molecules, whereas at a pressure of 10^{-7} Pa, a very high vacuum, it contains 2.5×10^{13} molecules. Indeed, any volume and pressure normally used in the laboratory will contain a large number of molecules. (*ii*) Adjacent molecules are separated by distances that are large compared with their individual diameters. If we could stop all molecules instantaneously and place them on the coordinates of a grid, the average spacing between them would be about 3.4×10^{-9} m at atmospheric pressure (10^5 Pa). The diameter of most molecules is of order $2–6\times10^{-10}$ m and their separation distances are ~6–15 times their diameter at atmospheric pressures. For extremely low pressures, say 10^{-7} Pa, the separation distance is about 3×10^{-5} m. (*iii*) Molecules are in a constant state of motion. All directions of motion are equally likely and all velocities are possible, although not equally probable. (*iv*) Molecules exert no force on one another except when they collide. If this is true, then molecules will be uniformly distributed throughout the volume and travel in straight lines until they collide with a wall or with one another.

Using these assumptions, many interesting properties of ideal gases have been derived. Some elementary properties are reviewed here.

2.1.1 Velocity Distribution

As the individual molecules move about they collide with elastic collisions. Elastic collisions conserve energy, whereas the colliding particle's velocity is changed after each collision. We stated that all velocities are possible, but not with equal probability. The distribution of particle velocities calculated by Maxwell and Boltzmann is

$$\frac{dn}{dv}=\frac{2N}{\pi^{1/2}}\left(\frac{m}{2kT}\right)^{3/2} v^2 e^{-mv^2/(2kT)} \tag{2.1}$$

m is the particle mass and *T* is the Kelvin temperature. The relation between the Kelvin scale and the Celsius scale is $T(\text{K}) = 273.16 + T(°\text{C})$. In (2.1) *N* is the total number of particles, and *k* is Boltzmann's constant. Figure 2.1 illustrates (2.1) for nitrogen molecules (air) at three temperatures. It is a plot of the relative number of molecules between velocity v and $v + dv$. We see that there are no molecules with zero or infinite velocity, and that the peak or most probable velocity v_p, is a function of the average gas temperature. The particle velocity also depends on the molecular mass, the peak velocity can be expressed as $v_p = (2kT/m)^{1/2}$. The arithmetic mean or average velocity v is useful when describing particle flow.

$$v=\left(\frac{8kT}{\pi m}\right)^{1/2} \qquad ►(2.2)$$

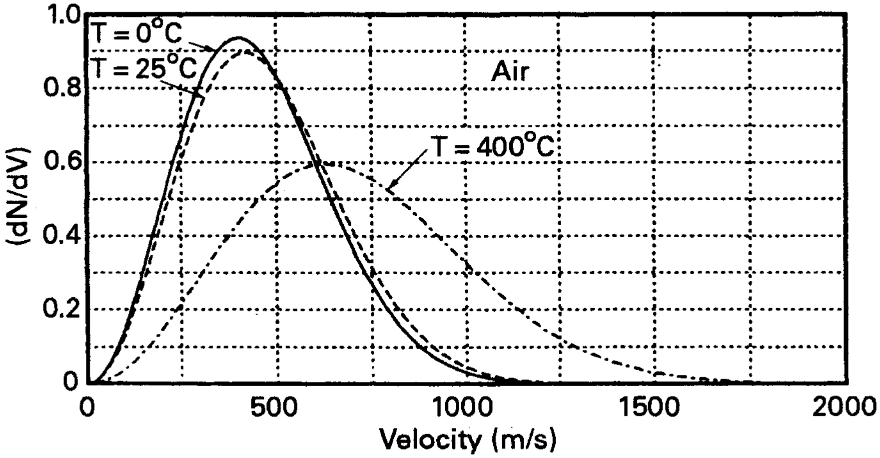

Fig. 2.1 Relative velocity distribution of air at 0°C, 25°C, and 400°C.