

DESIGN CRITERIA FOR ZERO-LEAKAGE CONNECTORS FOR LAUNCH VEHICLES. VOL. 2, LEAKAGE FLOW

Edited by T.P. GOODMAN

CONTRACT NAS 8-4012

MARCH 15, 1963



FINAL REPORT FOR FIRST CONTRACT PERIOD

(March 1962 through February 1963)

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Contract NAS 8-4012

VOLUME 2

LEAKAGE FLOW

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March 15, 1963

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- SPONSORED BY: Missile and Space Division General Electric Company Philadelphia, Pennsylvania

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CR-50,558

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21. STANDARDS FOR "ZERO LEAKAGE"

Ъу

T. P. Goodman

21.0 Summary

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"Zero leakage" has a much more stringent meaning in rocket propulsion systems than in the more conventional technologies such as steam-power-plant design. In a rocket propulsion system, it is not sufficient simply to have "stable leakage" or "leakage by molecular diffusion only". In rocket systems, zero leakage as determined from a bubble test means leak rates less than 10^{-4} atm cc/sec. or 10^{-6} lb. of air per hour; however, laboratory measurements with mass-spectrometer leak detectors can detect leaks of one-millionth of this magnitude. Leakage by diffusion through solid walls is generally well below zero leakage as determined by a bubble test, though still readily detectable by mass-spectrometer measurements.

21.1 Introduction

The development of rocket propulsion systems imposes requirements of hitherto unknown severity on the leak-tightness of fluid connectors. Until recently, most of the technology of fluid connectors was developed for fluids such as steam, for which leak-tightness was primarily a matter of the cost of the escaping fluid. In rocket systems, the leakage of fuels and oxidizing agents can cause explosions. Moreover, when fuels are stored in space satellites for later use in returning to earth, leakage must be reduced to an absolute minimum to economize on the payload of the satellite and to increase reliability of operation.

There are two traditional interpretations of "zero leakage" which are useful for steam power plants but are wholly inadequate for rocket propulsion systems. The first interpretation of "zero leakage" might better be called "stable leakage". Roberts (Ref. 1) has shown that a gasketed flange joint having uniform gasket pressure is theoretically stable when the ratio

m = gasket pressure/internal pressure

is greater than 1, and unstable when m is less than 1. When m 1, the internal pressure tends to blow the joint apart. In practice, some joints are found to be stable when when m 1, but Roberts attributes this to sticking of the gaskets. In a stable joint, there is still a small amount of leakage between the gasket and flange surfaces, but in steam-power-plant practice this stable leakage is often interpreted as "zero-leakage" because the cost of the steam that escapes is negligible. In a rocket propulsion system, even this leakage through a "stable" leak can be unacceptable.

A second interpretation of "zero leakage" might better be called "leakage by molecular diffusion only". Flow through a large passage under large pressure differential is generally turbulent; as the passage size decreases, flow becomes laminar. When the clearance in the passage is decreased to the same order of magnitude as the mean free path of the fluid molecules, the laminar flow changes to a flow by molecular diffusion. When the passage clearance is so small that flow is by molecular diffusion, the resulting flow is often thought of as "zero leakage"; however, even the small flow by molecular diffusion may be too much for a rocket propulsion system. Indeed, as shown in Section 22 of this report, the rate of molecular diffusion flow through a given passage is greater than the rate of laminar flow would be through the same passage.

21.2 Quantitative Measurements

For rocket propulsion systems, since both the "stable leakage" criterion and the "leakage by diffusion only" criterion are inadequate, it is important to specify <u>quantitatively</u> the leakage that is acceptable in a given connector. Then appropriate measurement techniques must be used to determine whether this leak-tightness has been achieved. Some highly accurate means are available for laboratory measurement of leak-tightness, but unfortunately, field measurement techniques are much less precise. Nevertheless, the laboratory measurements can be employed to evaluate new design principles.

To obtain an idea of the order of magnitude of leakage required in rocket launch vehicles, we have attempted to arrive at a quantitative working definition of "zero leakage." From our discussions in Huntsville we concluded that a good working definition of "zero leakage" is a leakage rate such that no bubbles can be detected in half an hour when a fully pressurized connector is immersed in water. Information obtained from the ATL Vacuum Center indicates that the threshold leakage rate at which no bubbles would appear in 5 to 15 minutes is approximately $2x10^{-6}$ 1b/hr of air at room temperature and atmospheric pressure. Thus, for no bubbles in half an hour, the threshold flow rate may be taken as 10^{-6} 1b/hr of air at standard conditions. For this flow rate, a single bubble appearing every half hour would have a mass of $0.5x10^{-6}$ 1b and a diameter of 0.28 in. at standard conditions. Since some air would dissolve in the water rather than appearing in bubbles, the bubbles for this leak rate would actually be somewhat smaller. Thus, 10^{-6} 1b/hr appears to be a reasonable definition of "zero leakage" as detectable from a conventional bubble test.

Table 21.1 compares the leakage rates that can be discerned from a conventional bubble test with leak rates that are discernible by other measurement techniques. Since many different units are used in describing leakage rates, all the measurements are shown in several different systems of units. It is convenient to remember that for air, 1 1b/hr is very nearly equal to 100 atm cc/sec. An "atm cc" (atmospheric cubic centimeter) is the mass of fluid that would occupy a volume of one cubic centimeter at room temperature and atmospheric pressure. A "micron-ft³" is the mass of fluid that would occupy a volume of one cubic foot at a pressure of one micron of mercury (1/760,000 atmosphere) at room temperature.

From Table 21.1 it can be seen that by standard mass-spectrometer flow measurements, flows in the "zero leakage" range can be quantitatively evaluated with high precision. The mass spectrometer was used to make the flow measurements described in Volume 3 of this report.

With lower sensitivity 0.96×10^{-5} 1×10^{-9} 1.27×10^{-10} 0.97×10^{-11}

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21.3 References

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- 3. H. S. Scheffler, <u>Final Report on Task 511</u>, <u>Leak Detection</u>, Battelle Memorial Institute, 1959.
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22. FLOW CALCULATIONS FOR SMALL PASSAGES

by

B.T. Fang and P.N. Le Fort

22.0 Summary

An analysis of a one-dimensional channel flow model of leakage is developed and leakage flow calculations are made for the gases:

- 1. helium
- 2. hydrogen
- 3. oxygen
- 4. nitrogen

and for the liquids:

- 1. hydrogen
- 2. oxygen
- 3. RP-1

for pressures up to 1000 atmospheres and clearances up to 200 microinches.

The results are plotted as graphs for a representative flow passage. Equations are given for converting the results to flow passages of other dimensions.

22.1 Introduction

Since it is impossible to obtain perfectly smooth surfaces, there will be minute clearances between the two surfaces of flanged connectors. Fluid under pressure will tend to seek its way through these small clearances. For a certain fluid at a given pressure and temperature, the amount of leakage depends on the geometry of the clearance. As a first step toward the understanding of the leakage process, we shall replace the actual situation of leaking through the many_small irregular passages by a model of onedimensional flow through a channel of length ℓ , width w and thickness h. In the simplest representation we can take

- l = width of flange (in the direction of leakage flow)
- w = mean perimeter of flange
- h = clearance between flanges

A more sophisticated approach is to take l, w, h as some kind of statistical averages of the geometry, surface finish and clamping pressure of the flanges. The proper selection of these values for a given fluid connector will be the subject of Section 33. The purpose of the present section is to determine the amount of leakage when the dimensions l, w, h of the passage are known and to present values for the flow of various gases and liquids through a passage of typical size.

22.2 Parameters Defining Flow Regions

The leakage may have the character of turbulent flow, laminar flow, or molecular flow. Roughly speaking, turbulent flow occurs at the highest rate of leakage and molecular flow occurs when the leakage rate is very small. Since we are primarily interested in achieving a very small amount of leakage, the leakage process will generally be in the laminar-flow and molecular-flow regions.

The parameter which defines the laminar and turbulent flow regions is the Reynolds number $\underline{\rho\,\nu h}$

where

ρ = density of fluid
 ν = velocity of fluid
 μ = viscosity of fluid

For a cylindrical tube (h = diameter, d) the critical Reynolds number is about 2000, beyond which turbulent flow may occur. For a channel, the critical Reynolds number will be somewhat smaller. A good estimate is to take the critical Reynolds number as 2000 based on an equivalent diameter (Ref.9) defined as

 $d = 4 \times \frac{Cross Sectional area of Channel}{Perimeter of channel}$ $\swarrow \frac{4 \text{ wh}}{2 \text{ w}} = 2 \text{ h}$

This would give us a critical Reynolds number of 1000 based on the clearance h.

Notice that for steady isothermal channel flow the Reynolds number is approximately constant throughout the length of channel. This means that it is very unlikely that part of flow in the channel is laminar while the rest is turbulent.

The parameter which defines the laminar and molecular flow regions is the Knudson number λ/h , where λ is the mean free path of the gas molecules. Molecular flow dominates when the mean free path is of at least the same order of magnitude as the thickness i.e., when the Knudson number is of the order of 1 or greater. Since the mean free path is inversely proportional to the pressure at a given temperature, Knudson's number increases in the direction of flow. It often happens that at the entrance of the channel the flow is laminar while at the exit it becomes molecular.

22.3 Rate of Leakage in Laminar Flow

The equations which govern the flow rate are

- (1) Conservation of mass
- (2) Momentum equation
- (3) Energy equation

In steady flow, conservation of mass requires the mass flow rate across any cross section of channel to be a constant. The energy equation comes in when the flow is compressible. The momentum equation is from the well known Navier-Stokes equations which assume a simple form for one-dimensional channel flow.

- In our application the flow velocity is small so that
- (1) Inertia effect can be neglected (Section 22.9.2)
- (2) Normal viscous stress can be neglected.
- (3) Turbulence does not occur.

Under these conditions the pressure gradient which causes the flow is balanced by the retarding viscous friction at the walls of the channel. It is well known that the velocity profile across the channel is parabolic (the consideration of entrance length is given in Section 22.9.1) and that the mass rate of flow is

$$Q = -\frac{\rho_{dP}}{dx} \frac{wh^3}{12\mu}$$
(1)

where $-\frac{dP}{dx}$ = pressure gradient in the direction of flow

 ρ = density of fluid

 μ = viscosity of fluid

For incompressible fluids such as liquids, Eq.(1) can be integrated, assumming ρ and μ are not pressure dependant, to give

$$Q = \frac{\rho \, wh^3 \, \Delta \mathbf{P}}{12\mu l} \tag{2}$$

where

△P = pressure difference

For compressible gases, thermal effects come into play and Eq.(1) cannot be integrated directly. Since the flow is very slow and the thickness of channel very small, the flow is approximately under isothermal conditions (Section 22.9.2). For a gas behaving essentially as a perfect gas Eq. (1) can be integrated to give.

$$Q = \frac{wh^{3}(\triangle P) \bar{P}}{12 \ \mu \ \ell \ P/\rho} = \frac{m \ wh^{3}(\triangle P) \bar{P}}{12 \ RT \ \mu \ \ell}$$
(3)

where

- \bar{P} = mean pressure
- T = absolute temperature
- R = universal gas constant
- m = molecular weight

It is readily seen from Eq. (2) and (3) that for both gases and liquids the mass rate of leakage is

- (1) Proportional to
 - (a) Width of channel
 - (b) Cube of thickness of channel
- (2) Inversely proportional to
 - (a) Viscosity of fluid
 - (b) Length of channel

For liquids the rate of leakage is further proportional to the pressure difference and to the density of liquid. For gases the rate of leakage is proportional to the pressure difference and to the mean pressure. For large pressures it is therefore approximately proportional to the square of internal pressure. The leakage rate is also inversely proportional to the absolute temperature and directly proportional to the molecular weight of the gas. It is evident that in the leakage of either liquids or gases the most effective means of reducing leakage is to decrease the clearance between the flanges.

22.4. Molecular Flow Correction

In order to achieve a very small leakage rate, the clearance between sealing surfaces has to be very small. When the clearance becomes so small that it is of the same order of magnitude as the mean free path of the gas molecules, leakage by the process of random molecular motion becomes of importance. The essential correction is that the no-slip condition at the channel walls is no longer true. Therefore, the leakage rate will be greater than that computed from the laminar-flow equation.

Knudson proposed the following formula for the leakage rate in free molecular flow (Ref.1,2):

$$\frac{4V_{a}\rho(\Delta P)/P}{3\int_{0}^{l}(H/A^{2})dl}$$
(4)

where

H = perimeter of channel A = cross section of channel ℓ = length of channel V_a = mean molecular speed = $\sqrt{8 \text{ RT}/\pi \text{m}}$

This equation can also be rewritten as

$$Q = \frac{8\sqrt{2} (\Delta P)}{3\sqrt{\pi RT/m} \int_{0}^{\beta} (H/A^{2}) d\ell}$$
(5)

which shows that the leakage rate is

- (1) proportional to the pressure difference.
- (2) inversely proportional to the square root of absolute temperature.
- (3) proportional to the square root of the molecular weight of the gas.

The effect of geometry of the channel on the leakage rate is represented by the factor ℓl 2

$$\int_0^\ell (H/A^2) d\ell$$

which shows that

 For the same channel cross sectional area, the greater the perimeter-to-area ratio, the smaller the amount of leakage. Therefore, the circular tube is the least desirable shape.

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(2) For a channel of given shape (H/A = constant) but changing cross-sectional area, the smaller cross sections have a choking effect on the amount of flow. For instance, a channel of changing cross section allows less flow than a constant-area channel having the same cross-sectional area as the mean cross-sectional area of the changing channel.

It is convenient to put the leakage-rate equation into yet another form. From kinetic theory of gases we have the mean free path of gas molecules as

$$\lambda \cong 2(\mu/P) \sqrt{RT/m}$$
 (6)

Substituting in the leakage-rate equation, we have

$$Q = \frac{4}{3} \sqrt{\frac{2}{\pi}} \frac{1}{\int_{0}^{\ell} H/A^{2} d\ell} \frac{\rho}{\mu} \bar{\lambda} \bar{\mathbf{F}} \frac{\Delta P}{P}$$
(7)

where $\bar{\lambda}$ is measured at the mean pressure .

For the rectangular channel we have been considering, the above formula becomes

$$Q = \frac{2}{3} \sqrt{\frac{2}{\pi}} \frac{\rho wh^2}{\mu \ell} \quad \tilde{\lambda} \bar{P} \frac{\Delta P}{P}$$
(8)

If we compare this with the leakage rate as computed from the laminar flow equation (3), we obtain

$$\frac{Q \text{ free molecular flow}}{Q \text{ laminar flow}} = 8\sqrt{\frac{2}{\pi}} \frac{\overline{\lambda}}{h}$$
$$= 8\sqrt{\frac{2}{\pi}} \times (\text{Knudson's number})$$
(9)

which also shows that Knudson's number is our measure of whether the flow would be essentially laminar or molecular.

Strictly speaking, Knudson's equation applies only to free molecular flow where the mean free path is very large compared to the clearance h. For transition flows the following modified equation has been proposed (Ref.2).

 $Q = Q_{\text{laminar flow}} + \epsilon Q_{\text{free molecular flow}}$

where ϵ is of the order of magnitude unity. The exact value of ϵ depends on the gas and properties of passage walls. For a capillary tube Adazumi (Ref.3) showed that

 $\epsilon \stackrel{\sim}{=} 0.9$ for a single gas $\epsilon \stackrel{\sim}{=} 0.66$ for gaseous mixtures such as air 15

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We shall base our calculations on these values, which should give us a correct order of magnitude of the leakage rate. We have shown (Eq. 9) that it is possible to express Q_{free} molecular flow in terms of $Q_{laminar}$ flow and Knudson's number. Therefore, it is convenient to write the total leakage rate as $Q_{laminar}$ times a correction factor. Thus for a circular cross-section

$$Q = Q_{1\min}\left(1 + \epsilon \frac{16}{3}\sqrt{\frac{2}{\pi}} \frac{\bar{\lambda}}{r}\right)$$
(10)

and for the rectangular channel we have been considering

$$Q = Q_{\text{laminar}} \left(1 + \epsilon \ 8 \sqrt{\frac{2}{\pi}} \frac{\tilde{\lambda}}{h} \right)$$
(11)

The second term in the parenthesis represents the molecular flow correction factor.

We shall illustrate the result with a few numerical examples.

Consider the leakage of air at 60° F from a pressure of 24.5 atm. to the atmosphere. The leaking passage has length l = 0.1 in., width w = 5 in. and clearance 1 microinch. The laminar-flow equation would give us a leakage rate of

For air at 60° F and atmospheric pressure the mean free path is 3.5 microinches. At a mean pressure of (24.5 + 1)/2 = 12.75 atm., the mean free path is

$$\bar{\lambda} = 3.5/12.75 = 0.27$$
 microinch

The molecular correction factor is

$$(.66)(8)\sqrt{2/\pi}$$
 $(\bar{\lambda}/h) = 1.15$

Therefore, the leakage rate should be (1 + 1.15) or 2.15 times that computed from the laminar-flow equation.

If the gas is helium instead of air, we can get an approximate molecular correction factor in the following way. Kinetic theory of gases gives that the mean free path of gas molecules is proportional to viscosity and inversely proportional to the square root of molecular weight (see Eq. 6). The viscosity of helium is not too much different from that of the air while the molecular weight is about 1/7 of that of air. Therefore the molecular flow correction factor for helium is about $\sqrt{7}$ times that for air, or about 3. This shows that leakage of helium through the passage is dominated by molecular flow effect. 16

22.5 Gas Calculations

It has been shown that for a gas behaving essentially as a perfect gas leaking through an annular passage between two flanges

$$Q = \frac{wh^{3}(\Delta P)\bar{P}}{12\mu\ell(P/\rho)} \left[1 + \epsilon \ 8\sqrt{2/\pi} \frac{\bar{\lambda}}{h} \right]$$
(12)

where w = mean perimeter of the flange

- h = clearance between flanges $\Delta P = \text{ pressure difference}$ $\bar{P} = \text{ mean pressure } = \frac{P \text{ internal } + P \text{ external}}{2}$ $\mu = \text{ viscosity of the gas}$ $\ell = \text{ width of the flange (in the direction of leakage flow)}$ $(P/\rho) = \text{ pressure-density ratio of the gas (a constant for a particular gas)}$ $\epsilon = \text{ molecular correction factor ($$$$$$$$$$$$$$$$$.9)}$
 - $\bar{\lambda}$ = mean free path of the gas molecules at the mean pressure, \bar{P}

In order to obtain a quantitative idea of leakage rate a sample channel of length $\ell = 0.1$ in. and width w = 5 in. (corresponding approximately to a 1.5 in. diameter tube) will be considered. Various gases (helium, hydrogen, oxygen, and nitrogen) will be used to calculate leakage at 20°C (68°F) from high pressure to atmospheric pressure for different clearances, h.

Gas properties are readily vailable (Refs. 1 and 10-14) although corrections are necessary to obtain values at 20° C and 1 atm. These were made using the formulae

$$\frac{\rho_1}{\rho_2} = \left(\frac{P_1}{P_2}\right) \mathbf{x} \left(\frac{T_2}{T_1}\right)$$
(13)

and

$$\frac{\lambda_1}{\lambda_2} = \left(\frac{P_2}{P_1} \right) \quad T = \text{const.}$$
(14)

Property values used are tablulated below.

TABLE 22.1

Gas	$\rho(\#/in^3)$	λ(micro- inches)	µ(centi- poise)
He(20 ⁰ C & 1 atm)	6.01×10^{-6}	10.66	.0194
H ₂ (20 [°] C & 1 atm)	3.03×10^{-6}	6.78	.00931
$0_{2}^{-}(20^{\circ}C \& 1 atm)$	4.81×10^{-5}	3.86	.0206
N ₂ (20 [°] C & 1 atm)	4.21×10^{-5}	3.61	.0184

For the case considered, w = 5 in. and $\ell = 0.1$ in., $T = 20^{\circ}$ C, with an external pressure of one atmosphere,

$$Q_{\text{He}} = .4706 \times 10^{-9} h^{3} (\Delta P) \bar{P} \left[1 + 61.19 / h \bar{P} \right]$$

$$Q_{\text{H}_{2}} = .4946 \times 10^{-9} h^{3} (\Delta P) \bar{P} \left[1 + 38.89 / h \bar{P} \right]$$

$$Q_{0_{2}} = 3.549 \times 10^{-9} h^{3} (\Delta P) \bar{P} \left[1 + 22.14 / h \bar{P} \right]$$

$$Q_{N_{2}} = 3.478 \times 10^{-9} h^{3} (\Delta P) \bar{P} \left[1 + 20.72 / h \bar{P} \right]$$
(15)

where

Q = flow in lb/hr

h = clearance in micro-inches

 ΔP and \tilde{P} = pressure difference and mean pressure respectively in atmospheres

Figs. 22.1 through 22.4 present the results of the calculations for laminar flow only and for the combined laminar and molecular flow as expressed by equation 12.

Fig. 22.1 Leakage rate for Helium at 20° C (68° F) for an annular passage of 5 in. perimeter and 0.1 in. length, with 1 atmosphere external pressure.









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22-14 22

22.6 Liquid Calculation

Equation 1 is also the basis for the calculated leakage of liquids,

$$Q = - \frac{\rho w h}{12 \mu} \frac{^3 dP}{dx}$$

where ρ and μ are pressure-dependent. The density relation that results from the definition of bulk modulus is

$$\rho_2 = \rho_1 \left[\frac{K}{K - (P_2 - P_1)} \right]$$
(16)

where K == bulk modulus

 $P_1 = atm. pressure$ $\rho_1 = density at atm. pressure$ $P_2 = pressure of interest$ $\rho_2 = density at P_2$

On the other hand, little is known concerning the pressure dependence of viscosity (Ref.15). Of the many suggested equations, the one presented by Frenkel (Ref.16) was chosen, since it does not involve emperical constants or liquid properties as yet unknown, and it has been corroborated, to some degree, by experiment. The viscosity relation is, therefore, assumed to be

> $\mu_{2} = \mu_{1} \exp(P_{2}/P_{K})$ (17) where μ_{1} = viscosity at atm. pressure P_{2} = the pressure of interest μ_{2} = viscosity at P_{2} P_{K} = that pressure which corresponds to an e(2.71...)-fold increase of viscosity $P_{K} = \frac{\alpha KT}{\gamma}$ (18) where α = coefficient of thermal expansion K = bulk modulus T = absolute temperature

 γ = an empirical constant ≈ 5

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The substitution of these relationships into equation (1) results in a series solution for Q; however, an approximation can be made by first assuming ρ and μ as constants.

The result is equation 2. Now the pressure dependence is invoked by using values of ρ and μ as determined at the mean pressure. When the external pressure is one atmosphere, this yields

$$Q = \frac{\rho_1 wh^3}{12 \mu_1 \ell} \left(\frac{K}{K - \bar{P}} \right) \frac{\Delta P}{\exp(\bar{P}/P_K)}$$
(19)

Calculations were carried out for liquid hydrogen, liquid oxygen and rocket fuel, RP-1, using the properties (Refs. 1, 10-14, 17) listed in Table 22.2.

TABLE 22.2

Liquid	<u>p(#/in³)</u>	μ(centi- poise)	$\alpha \left(\frac{1}{\circ \mathbf{C}} \right)$	K(atm)
H ₂ (-252.7 [°] C & 1 atm)	.00256	.0130	13×10^{-3}	17,000
0 ₂ (-183 [°] C & 1 atm)	.0412	0.189	4.1×10^{-3}	15,000
RP-1(20 ⁰ C & 1 atm)	.0291	1.10	.92x10 ⁻³	13,600

Again for the case of w = 5 in. and l = .1 in.;

$$Q_{H_{2}} = 29.9 \times 10^{-8} \left(\frac{17,000}{17,000 - \bar{P}} \right) \frac{h^{3} \Delta P}{\exp(\bar{P}/880)}$$

$$Q_{0_{2}} = 33.1 \times 10^{-8} \left(\frac{15,000}{15,000 - \bar{P}} \right) \frac{h^{3} \Delta P}{\exp(\bar{P}/1100)}$$

$$Q_{RP-1} = 4.02 \times 10^{-8} \left(\frac{13,600}{13,600 - \bar{P}} \right) \frac{h^{3} \Delta P}{\exp(\bar{P}/850)}$$
(20)

where the quantities are as defined for equation 15. The results are presented as Figs. 22.5, 22.6, and 22.7.

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22.7 Conversion to Other Conditions

While, for the calculated leakage rates, specific dimensions and operating conditions were chosen, the resultant curves may be converted to leakage through other passages under other conditions. A convenient nomographic method of conversion can be found in Ref. 4.

- (1) In all cases:
 - a. To convert to other passage widths, multiply the leakage rate by

b. To convert to other passage lengths, multiply the leakage rate by

- (2) For gases when molecular correction is small:
 - a. To convert to leakage rate at other temperatures, multiply the leakage rate by

$$\left(\frac{293}{273 + \text{temperature}}, {}^{\circ}C\right)^{x} \text{or} \left(\frac{528}{460 + \text{temperature}}, {}^{\circ}F\right)^{x}$$

where values of (x) for the different gases are given in Table 22.3.

b. To convert to leakage rate at other outside pressures, P_{o} , read the ordinate in units of

$$\frac{\frac{P_{i}}{P}}{\frac{P_{i}}{P}}$$

and multiply the leakage rate as found from the graph by

- (3) For gases when molecular flow correction is not small:
 - a. To convert to leakage rate at other outside pressures, after applying the conversion in (1) and (2) to the given laminar flow curve, multiply the leakage rate by

$$1 + C/h\bar{P}$$

where values of (C) for the different gases are given in Table 22.3

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- (4) For liquids:
 - a. To convert to leakage rates at other temperatures, multiply the given rate by

$$\frac{\rho_2^{\mu_1}}{\rho_1^{\mu_2}} \bullet \exp\left[\frac{\overline{P}}{P_K} \left(1 - \frac{T_1}{T_2}\right)\right]$$

where ρ_1, μ_1 , and T_1 are the density, viscosity and absolute temperature for which the curves are valid and which are available from Table 22.2. ρ_2, μ_2 , and T_2 are the density, viscosity and absolute temperature at the temperature of interest, while the values of P_K of the liquids are given in Table 22.4

b. To convert to leakage rate at other outside pressures, read the ordinate in unit of

and multiply the leakage rate as found from the graph by
$$\frac{P_{o}\left[K - \frac{\overline{P}}{P_{o}}\right]}{\left[K - \overline{P}\right] \exp\left[\frac{\overline{P}(P_{o}-1)}{P_{K}P_{o}}\right]}$$

 $\frac{P_i}{P_i}$

Values of K, bulk modulus, and P_K for the different liquids will be found in Table 22.4. Due to the complexity of this conversion, in most cases it would be advisable to carry through the computation in its entirety starting with equation 11.

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	Table 22.3	
Gas	Exponent, x	<u>Constant, C</u>
He	1.653	61.19
н ₂	1.678	38.89
N ₂	1.702	22.14
°2	1.721	20.72

Table 22.4				
Liquid	Bulk Modulus, K(atm)	P _K (atm)		
H ₂	17,000	880		
0 ₂	15,000	1,100		
RP-1	13,600	850		

22.8 Extension to Other Models

It is obvious that the channel-flow leakage model we have been considering is far from being realistic. The actual leaking passages are probably more like many capillary tubes, of different and changing cross-sections and intersecting each other at many places. On the one hand, the present model can take these into consideration by properly choosing the geometrical dimensions of the channel passage, based on statistical consideration of the actual surfaces. On the other hand, the result of the present model can be extended to such other models which describe in more detail the shapes of the actual leaking passages. For instance, the essential difference in flowing through tubes rather than channels is the bounding effect of the sides. In tubes, the laminar-flow velocity profile becomes a paraboloid instead of a parabola, resulting in smaller leakage rate for the same cross-sectional area. Many of these flow analysis properties can be carried over from the result of the present model by using the idea of equivalent diameter mentioned previously. A similar situation appears in the molecular flow region.

For flow through channels of changing cross-section and interconnecting passages, we may conceive of the flow as going through a series of very short channels, each of constant cross-section. For flow in each short channel, Eqs. (2) and (3) apply and we can define the impedance of the channel to the flow as

$$\frac{\Delta P \bar{P}}{Q} = \frac{12\mu\ell}{\rho wh^3} \qquad \text{for liquids}$$

$$\frac{\Delta PP}{Q} = \frac{12\mu \, \ell RT}{m w h^3} \quad \text{for gases}$$

By properly combining these impedances (in series, in parallel, etc.) we can obtain the picture of over-all leakage process. As an example consider the flow of a certain liquid through a channel of gradually changing thickness. Let us divide the channel into n short channels each of thickness d_1 and length \mathcal{L}_i . The flow impedance of each short channel is

$$12\mu\ell_{i}/P d_{i}^{3} = c\ell_{i}/d_{i}^{3}$$

From the condition that the same amount of liquid flows through all the channels and the total pressure drop is the sum of the pressure drop across each channel, we can easily show that the over-all impedance to flow is

$$c \sum_{i=1}^{n} {l_i} / {d_i^3}$$

Since l_i/d_i^3 is inversely proportional to the cube of the thickness d, it can be seen that channels of smaller thickness have greater influence in impeding the leakage flow.

22.9 Appendix

22.9.1 Entrance Length

The leakage rate equation derived in section 22.3 is based on the assumption that in laminar flow, the velocity profile is parabolic at each section along the passage. The actual situation is that at the immediate entrance the boundary layer thickness is zero and the velocity profile is uniform. The thickness increases with distance until the two layers connect together and the velocity profile becomes parabolic. The distance from the entrance to the point where the two boundary layers come together is called the entrance length ℓ_e . It is apparent that in computing the leakage rate by Eqs. (2) and (3) we should replace ℓ by $\ell^{-}\ell_e$ and take the internal pressure as the pressure at the end of the entrance length.

Boussinesq (Ref.5) obtained theoretically the entrance length of a cylindrical tube as

 $l_{e} = 0.26 \text{ aR}$

a = radius

where

R = Reynolds number based on the radius

For a channel passage the entrance length will be somewhat longer. A good estimate can be made from our knowledge of the growth of boundary layer thickness δ for a flat plate (Ref.6).

$$\delta \sim \sqrt{\mu x/\rho v}$$

where x = distance from the entrance.

When the boundary layers come together, the boundary layer thickness = h/2. From the above relations we have

h/2
$$\sqrt{\mu \ell_e / \rho v}$$

or $l_e \sim Rh/4$

For leakage rates of interest to us, the leakage velocity is small and the clearance h very small so that the entrance length is also very short. It is generally permissible to neglect this entrance length in computing the leakage rate.

We have stated that the correct internal pressure to be used in computing leakage rate is that at the end of entrance length. As long as the entrance length is very short and the leakage rate is small, the difference between the pressures at the entrance and at the end of entrance length is negligible.

22.9.2 Consideration of Inertia and Temperature Variation

Most of the leakage rates of interest to us belong to the group treated previously. For compressible gases at large leakage rates the effect of inertia and temperature variation may become of importance. We shall treat these effects in this section and illustrate that with a large pressure difference the leakage speed as well as the leakage rate can become considerable even for small clearances.

When inertia is taken into consideration, the equation of motion of the fluid becomes

$$\rho v \frac{dv}{dx} = -\frac{dP}{dx} - \frac{12\mu}{h^2} v$$
(21)

which says that the change in momentum is equal to the difference between pressure gradient and the viscous friction at the walls. The last term is obtained by assuming that the velocity gradient at the wall is the same as the incompressible parabolic velocity profile. Dividing the equation by ρv^2 and integrating, we obtain

1

$$\ln \frac{\mathbf{v}_1}{\mathbf{v}_2} = -\frac{1}{\rho \mathbf{v}} \int_2^1 \frac{d\mathbf{P}}{\mathbf{v}} - \frac{12\mu}{\rho \mathbf{v} d^2} \quad (\mathbf{x}_1 - \mathbf{x}_2)$$
(22)

where 1 and 2 are two sections along the channel. By making use of the constant mass flow condition

$$Q = \rho vwh = constant$$
(22a)
we can express the integral $\int_{2} dP/v$ as $(1/\rho v) \int_{2} \rho dP$.

Still we cannot evaluate this integral until we know the relation between the density and pressure along the channel from section 1 to section 2. This relation depends on the heat transfer between the gas and the channel walls. We shall consider two extreme cases:

- (1) Infinite heat transfer so that the leakage is under isothermal conditions.
- (2) No heat transfer at all.

Actual cases lie in between these two extremes. In case (1) we have the additional isothermal condition.

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 $P/\rho = RT/m = constant$

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and in case (2) we have the conservation of energy relation

$$c_p T + v^2/2 = constant$$

where c_n = specific heat under constant pressure.

With these relations we are able to integrate Eq. (22). The results are, for case (1)

$$\ln \frac{M_1}{M_2} + \frac{1}{2\gamma} \left(\frac{1}{M_1^2} - \frac{1}{M_2^2} \right) = \frac{12\mu}{\left(\frac{Q}{Wh}\right)h^2} (x_2 - x_1)$$
(23)

and for case (2)

$$\frac{\gamma+1}{2\gamma} = \ln \frac{M_1 \sqrt{1 + \frac{\gamma-1}{2} M_2^2}}{M_2 \sqrt{1 + \frac{\gamma-1}{2} M_1^2}} + \frac{1}{2\gamma} \left(\frac{1}{M_1^2} - \frac{1}{M_2^2}\right) = \frac{12\mu}{\left(\frac{Q}{wh}\right)h^2} (x_2 - x_1)$$
(24)

where M = Mach number

γ = ratio of specific heats at constant pressure and at constant volume.

These equations involve two unknowns which are Mach numbers at sections 1 and 2. An additional equation which gives the relation between M_1 and M_2 is

$$\frac{M_2}{M_1} = \frac{P_1}{P_2}$$
 for case (1) (25)

and

$$\frac{M_2 \sqrt{1 + \frac{\gamma - 1}{2} M_2^2}}{M_1 \sqrt{1 + \frac{\gamma - 1}{2} M_1^2}} = \frac{P_1}{P_2} \text{ for case (2)}$$
(26)

The leakage rate Q is given by the following formula

$$Q = (wh) \left(\frac{M_1 P_1}{\sqrt{\gamma m/RT_1}} \right)$$

$$= (wh) \left(\frac{M_2 P_2}{\sqrt{\gamma m/RT_2}} \right)$$
(27)

We can compute the leakage rate from the above equations by taking section 1 and 2 as entrance and exit of the channel.

An important consequence of taking inertia into consideration is that it can be shown from Eq. (23) and (24) that the flow in the channel can never become supersonic, whatever the pressure difference may be. The same conclusion is true for the flow of liquids.

Generally speaking the Mach number at entrance is much smaller than the Mach number at exit. Under this condition both Eq. (23) and (24) simplify to

$$\frac{1}{2\gamma} \left(\frac{1}{M_1^2} \right) = \frac{12\mu}{(x_2 - x_1)} \left(\frac{Q}{wh} \right) h^2$$
(28)

Substituting in Eq. (27) we obtain the leakage rate as 3^{2}

$$Q = \frac{mwn \ p_1}{24\mu \ (x_2 - x_1)RT}$$
(29)

which is in agreement with Eq. (3) of section 2.3 when the internal pressure is large. This extends the validity of Eq. (3) derived previously to the present case when the temperature and inertia effect are considered.

We shall illustrate the results with the following numerical example:

```
Geometry of leakage channel
length = 0.1 in.
width = 5 in.
clearance = 50 \times 10^{-6} in.
```

Air at 60[°]F is leaking out from a pressure of 100, 50, 20, 10, 5, 2 atm. to atmospheric pressure.

Properties of air at 60° F are density = 1.15 x 10^{-7} lb-sec²/in⁴ at atmospheric pressure viscosity = 2.62 x 10^{-9} lb-sec/in² Ratio of specific heats $\gamma = 1.4$

The numerical result is summarized in the following table:

Entrance		Exit		Lookogo Poto
Pressure Atm.	Mach No.	Pressure atm.	Mach No.	leakage kate
100	0.0438	3.98 (adiabatic)	1	2.33
50	0.0219	1.99 (adiabatic)	1	5.83×10^{-1}
20	0.00876	1	0.175	9.36 x 10^{-2}
10	0.00438	1	0.0438	2.37×10^{-2}
5	0.00210	1	0.0105	5.58×10^{-3}
2	0.00656	1	0.0 013 1	6.98×10^{-4}

These examples show that

(1) For large pressure difference both the leakage speed at exit and the leakage rate are considerable. For instance, more than two pounds of air will leak out in an hour if the internal pressure is 100 atm. (1470 psi)

(2) Even at the leakage rate of 2.33 lb/hr, the corresponding Reynolds number can be computed as only 128. Turbulent flow does not occur.

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23. GAS PERMEATION THROUGH SOLIDS*

Ъy

H. J. Sneck

23.0 Summary

The gases used in rocket propulsion systems can permeate solid walls of metals or polymers by a molecular diffusion process. The rate of permeation flow is very slow compared to leak rates encountered in conventional engineering practice and is well below zero leakage as determined by a bubble test (Section 21). However, permeation flow can be readily measured by a mass-spectrometer leak detector. To achieve the ultimate in leaktightness, especially in long space voyages, and to make accurate measurements of leakage, the possibility of flow through solid pipe walls and solid gaskets must be considered.

This section presents a compilation of permeation flow data obtained by Dr. F. J. Norton of the General Electric Research Laboratory and others who have worked in this field. The order of magnitude of the permeation effect is illustrated by numerical examples at the end of this Section.

^{*} This material was originally prepared under Contract NAS 7-102, "Study of Dynamic and Static Seals for Liquid Rocket Engines."

23.1 The Permeation Process

The process whereby gases pass through sound solid membranes is described by Norton (Ref. 1) as follows:

1. The gas on the high-pressure side is first adsorbed and dissolved in an external surface layer on the membrane surface. Surface pretreatment is important here.



FIGURE 23.1 Permeation of Gas Through a Solid Membrane

- 2. The gas then diffuses through the "solid" driven by the concentration gradient according to Fick's Law. The diffusion of the gas may be as atoms of a dissociated molecule if the membrane is metallic, or molecules if the membrane is a polymer.
- 3. When the gas reaches the low-pressure side of the membrane, it undergoes a transition from a dissolved state to an absorbed state and is desorbed at the surface and passes into the surroundings (where it will reassociate if the diffusion was atomic). Again surface pretreatment may be important.

The overall steady-state process described above is termed "permeation", and the speed of the permeation process is controlled by the slowest of the above steps.

There are very few generalities one can make for the permeation process as a whole. Norton (Ref. 2) gives the known generalities as follows:

Gas Permeation Through

Metals	Polymers
No rare gas passes through any metal (and Halogen gases to no marked degree - Ref. 11)	All gases permeate all polymers
H ₂ permeates most metals, especially Fe	H_2O rate apt to be high
O ₂ permeates Ag	
H ₂ through Fe by corrosion, electrolysis	Many specificities
Permeation rates vary as pressure	Permeation rates vary as pressure

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where S = solubility.

$$-\frac{\partial c}{\partial x} = -s\frac{\partial p}{\partial x} = s\frac{\Delta p}{L}$$

Under this condition the diffusion coefficient is, in general, constant so that equation 2 may be written for a membrane of uniform thickness as

$$Q = DSA \frac{\Delta p}{L}$$
(4)

where $\triangle p$ = pressure drop across the membrane. If the permeation rate is defined as

$$P = DS$$
(5)

equation 4 becomes equation 1.

It should be noted that equation 1 is strictly applicable only if the diffusion is molecular in nature, as in polymers. Some exceptions to Henry's Law have, however, been noted for rubber by Jost (Ref. 3). If the diffusion process is atomic in nature, as with diatomic gases permeating through metals, Equation 1 is not strictly applicable. Dushman (Ref. 4) indicates that when the diffusion is atomic

$$Q \propto (\sqrt{p_1} - \sqrt{p_2})$$
 (6)

The permeation rate (P) used in Equation 1 is exponentially temperature dependent according to Equation 7, as illustrated in Figure 23.2 (Ref. 5).





$$P = P_{o} \exp\left(-\frac{E}{jrT}\right)$$
(7)

²³⁻⁴ 40 where

- P_{o} = constant, independent of temperature variation
- E = activation energy of permeation
- j = dissociation number (2 for diatomic gases permeating metals)
- $R = 1.986 \text{ cal/K}^{\circ}$
- T = degrees Kelvin

This equation is particularly useful in extrapolating permeation rates to higher or lower temperatures.

23.3 Permeation Rates and their Application to Calculations

The calculation of leakage rates due to permeation hinges on the experimental determination of the permeation rates (P) as defined by Equation 1. For polymers, experimental permeation rates can be extrapolated to different temperatures using Equation 7 and then applied directly in Equation 1.

The problem is not quite so straight-forward for metals where Equation 6 applies rather than Equation 1. One finds that the literature contains permeation rates for metals as defined by Equation 1. These rates are still exponentially temperature dependent as given by Equation 7, but they are correct only for the specific pressure difference given in their units (e.g., 1 torr or 1 mm Hg, 1 cm of Hg, 1 atmosphere). If the experimental values were determined when p_2 was very nearly equal to zero, and the case to be calculated also has p_2^2 very nearly equal to zero, then pressure extrapolations can be made using the simplified proportion.

$$\frac{Q_{actual}}{Q_{experimental}} = \sqrt{\frac{p_{1 actual}}{p_{1 experimental}}}$$
(8)

Otherwise extrapolations must be made according to Equation 6. Equation 8 is particularly useful when comparing, roughly, the results of different experimenters (Ref. 4).

Considerable care must be exercised when collecting permeation rate data from the literature for the following reasons:

23.2 Permeation Equations

The volumetric flow rate (S.T.P.) for the molecular permeation of a gas through a "solid" is described by the equation

$$Q = PA \frac{\Delta p}{L}$$
(1)

where

Q = volumetric flow rate at STP A = area normal to the flow Δ_p = pressure drop along the flow path L = Length of the flow path P = permeation rate

This equation can be derived from Fick's Law of diffusion

$$Q = -DA \frac{\partial c}{\partial x}$$
(2)

where

D = diffusion coefficient
$$\frac{cm^2}{sec}$$

$$\frac{\partial \mathbf{c}}{\partial \mathbf{x}}$$
 = concentration gradient

if the solubility is small. The concentration for very small solubility is related to the pressure through Henry's Law

$$\mathbf{c} = \mathbf{S}\mathbf{p} \tag{3}$$

- Some of it is in error (as pointed out by Dayton, Ref. 5) particularly that given by Jost (Ref. 3) and Barrer (Ref. 6).
- .2) The temperature must be noted because of the exponential dependence.
- At least four different sets of units have been used for the permeation rates. They are

a)
$$\frac{cm^{3} - mm}{sec - cm^{2} - torr}$$

b)
$$\frac{cm^{3} - cm}{sec - cm^{2} - atm}$$

c)
$$\frac{cm^{3} - mm}{sec - cm^{2} - atm}$$

d)
$$\frac{cm^{3} - mm}{sec - cm^{2} - atm}$$

This complicates the comparison of permeation rates of gases through metals for the reasons given above.

4) There are many specificities with regard to the membrane material (especially polymers) or pretreatment of materials (metals). This complicates the comparison of permeations for the same apparent material-gas combination.

Table 23.1 represents the consensus of data gathered from the references already cited as well as References 7 and 8. (It is worth noting here that when searching German-language literature the key word, permeation, is translated as "Gasdurchlässigkeit"). All data are for approximately $25^{\circ}C$ (68°F).

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TABLE 23.1 Permeation Rates (cm³-mm/sec-cm²-atm) at Room Temperature

	Hydrogen	Oxygen	Nitrogen	Helium
Iron	4 to 15 x 10^{-9}		4.2×10^{-19}	
Steel (low carbon)	3×10^{-10}			
Steel (27% chrome)	1×10^{-12}			
Aluminum	3.1×10^{-22}			
Natural rubber	1.5 to 4.0 x 10^{-6}	1.8×10^{-6}	1.0×10^{-6}	3.0×10^{-6}
Neoprene	1.0×10^{-7}	0.3×10^{-7}	0.1×10^{-7}	0.6×10^{-7}

The following permeation-rate data for air were obtained from References 9 and 10.

Perm	eation rate P in	<u>cm³-mm</u> at Tempe sec-cm ² -atm	erature
	<u>75°F</u>	<u>176⁰F</u>	<u>350°</u> F
Butyl Rubber	0.02×10^{-6}	0.32×10^{-6}	6.1×10^{-6}
Silicone Rubber	22.0×10^{-6}	45×10^{-6}	112×10^{-6}
Kel-F		0.8×10^{-6}	

While the data above appear skimpy, they represent the result of gleaning many different sources and careful comparison of the data for consistency.

The only gases cited in the literature surveyed were H_2 , O_2 , N_2 , Xe, Ar, He and Air. None of the exotic gases or vapors, particularly fluorine compounds, were encountered in the references. Cupp (Ref. 11) indicates that the halogen gases do not permeate metals to any marked degree; and if this is true, then they might be lumped in with the rare-gas generalization.

The only metals encountered were Fe (composition of alloying elements sometimes specified), Cu, Ni, Ag, Au, Al. Here wide variations in permeability are encountered, due to differences in composition, surface condition, heat treatment, etc. Private conversations with F.J. Norton at the General Electric Research Laboratory brought out the fact that hydrogen permeates metals at the highest rate of any of the gases. Hydrogen data can therefore be used to predict the upper limit of leakage loss for any metal in question.

A considerable amount of data is available for polymers. Some of the more common types have been chosen to indicate the order of magnitude of permeation rate and to indicate that the losses can be great if the polymers are not chosen properly (see Numerical Example). In a current article by Frank (Ref. 12), he indicates that recent work on polymers has taken two directions:

- Development of new polymers of low permeability (Mylar, Saran (Ref. 1), polyvinyl alcohol).
- 2. Treatment of polymers already available to decrease their permeability (impregnation with fillers, metal-coating the surface).

23,4 Numerical Example

In order to get a "feel" for the flow losses attributable to permeation, consider the following example:

A gasket 0.1" thick, 0.1" wide, and 5" nominal diameter with a 1 atmosphere pressure difference across it.



Fig. 3

A =
$$5(2.54) \pi (0.1)(2.54) = 10.13 \text{ cm}^2$$

L = 0.254 cm = 2.54 mm
 $\Delta p = 1 \text{ atm.}$

Equation 1 becomes

$$Q = PA \frac{\Delta p}{L} = \frac{10.13}{2.54} P = 3.99 P$$

For a natural rubber gasket at $25^{\circ}C$

	P	Q	þ	Loss
	$\frac{\mathrm{cm}^3 - \mathrm{mm}}{2}$	atm cc	<u>1b.</u> 3	<u>lb.</u> vear
	sec - cm ⁻ atm		cm	Jeur
^H 2	4.0×10^{-6}	15.96×10^{-6}	1.83×10^{-7}	92.0×10^{-6}
02	1.8×10^{-6}	7.18×10^{-6}	29.2 x 10^{-7}	661.0×10^{-6}
N ₂	1.0×10^{-6}	3.99×10^{-6}	25.5×10^{-7}	321.0×10^{-6}
Не	3.0×10^{-6}	11.97×10^{-6}	3.65×10^{-7}	137.7×10^{-6}

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For a Neoprene gasket at $25^{\circ}C$

	$\frac{\text{cm}^3 - \text{mm}}{\text{sec} - \text{cm}^2 - \text{atm}}$	Q <u>atm cc</u> sec	ρ <u>1b.</u> cm ³	Loss <u>lb.</u> year
н ₂	10×10^{-6}	3.99×10^{-6}	1.83×10^{-7}	2.30×10^{-6}
0 ₂	0.3×10^{-6}	1.2×10^{-6}	29.2×10^{-7}	110.0×10^{-6}
N ₂	0.1×10^{-6}	0.399 x 10 ⁻⁰	53.65×10^{-7}	32.1×10^{-6}
He	0.6×10^{-6}	2.4×10^{-6}	3.65×10^{-7}	27.6×10^{-6}

By way of comparison, a 10-foot-long steel pipe of the same diameter and thickness would lose Hydrogen at a rate comparable to those given above.

In conventional engineering practice, gas losses of the order shown above would, of course, be of no practical concern. When these figures are multiplied by perhaps 100, to allow for the presence of many such sealing devices in a space-craft, however, the order of magnitude begins to have significance. This is particularly true of vehicles for very long missions, where launch weight will be critical and a minimum amount of fuel will be carried.

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24. WELDED, BRAZED, AND SOLDERED JOINTS

by

G. W. Hume

24.0 Summary

Permanent joints between metal pipes and tubes can be made by the conventional joining techniques such as welding, soldering, brazing, and swaging (cold welding). When properly designed and fabricated, such joints can have the same leak tightness as the base metal itself. Welded, soldered, and brazed joints can be designed for a limited number of disassemblies and reassemblies, but their main application is for permanent joints such as the pipe-to-flange joints in a flange connector.

This brief discussion of metal joining techniques is included to give a comparison between these types of joints and gasketed joints, and to provide design information on the permanent joints (such as pipe-to-flange joints) used in separable fluid connectors.

24.1 Introduction

Extensive research and development of true hermetic seals has been made on other programs; however, permeation of the joints by gases and liquids over long periods of time and temperature cycling has not been evaluated, although the permeation through the joints should be in the same order of magnitude as that of the metal members. Liquid or gas permeation through solid metals is a function of time and temperature as well as the chemical makeup of the system (Sec. 23). Hydrogen and oxygen are the two gases most likely to create a permeation problem, as hydrogen permeates most metals if high-temperature cycling conditions are of long duration. Permeability of gases (Ref. 1) generally increases with plastic deformation; therefore stresses should be minimized. Each application will require a specific gas permeation study once the materials and processes for the application are established. The variables are many in these applications and because of this, the author will generalize in order to give the designer a broad look at the problem of fabricating metal joints.

In the welding field a joint is considered vacuum tight if it can pass the mass spectrometer leak test. In critical applications a pressure of 50 psi of helium is placed on one side of the joint and a vacuum on the other and allowed to stand for four hours under this condition. A reading is then taken on the mass spectrometer which will detect leaks of helium in the order of 1×10^{-10} atm. cc/sec. A joint is considered leak tight if it passes this test (i.e., if no leakage is detected by the mass spectrometer) and passes non-destructive tests such as x-rays, ultrasonic, and dye penetration. The applications to be considered here would require the extension of the above test to include the thermal cycling of the system through the temperature range needed for the application and testing it during the operation. In studying the different methods of making such junctions to stainless steel and aluminum, the following factors are considered prime requirements of an ideal hermetic seal:

- 1. Reliability
- 2. Ease of non-destructive leak testing
- 3. High strength
- 4. Ease of repair
- 5. Fabricated with minimum effort
- 6. Equally easy to fabricate in field
- 7. Ability to withstand extremes of temperature, vibration, and shock

The temperature range of -420° F to $+2500^{\circ}$ F encountered in launch vehicles is restrictive for these applications in that a number of joining processes are likely to be needed to cover every application.

Thermal expansion of the metals in the system is another important consideration when the temperature limits are as wide as in these applications. Ideally all members should expand at the same rate over the entire temperature range in order to avoid building up internal stresses which subsequently result in cracks in the system.

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24.2 Welding

Welding is not normally considered for use in an application requiring many separations during its life; however, with proper design it has been accomplished. There are a number of fusion welding processes available; however, the applications of tubing and pipes lead one to consider the nonconsumable inert arc welding process as the chief source of heat.

In the case of inert arc welded seals (Ref.2), typical designs are shown in Fig.24.1 which can be machined or sawed apart. No oil or grease should be used during cutting since these materials are detrimental to the reweld operation. Since no heat is used to reopen the weld, the two mating faces can be rewelded a number of times depending on the length of the lip preparations.

The inert arc welding process eliminates the expansion problem to a large degree as no foreign metal need be added to the metals in question. Differences in expansion can arise, depending on the rate of heating or cooling thin and thick sections of the system.



INERT ARC WELD





REWELD

AFTER WELDING



BEFORE WELDING



CUT OFF

AFTER WELDING



BEFORE WELDING



AFTER WELDING

FIG. 24.1 Typical Designs of Inert Arc Welded Seals

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24.3 Brazing and Soldering

It is apparent that brazing, soldering, and the base metal aluminum could not be used for sealing in the higher temperature range applications because of their low melting points and poor strength at elevated temperatures.

Brazed and soldered filler metals inherently have large differences in expansion from the base metals; however, in the lower temperature ranges they exhibit good ductility which overcomes the stress problem. Oxygen permeates silver easily; therefore in selecting braze alloys containing silver, one must use discretion as to the time and temperatures involved with oxygen in the system.

Reusable seals are difficult to design because each opening creates a reliability problem. In the case of brazing and soldering, the seal is reheated and separated, which causes oxidation to occur on the two mating surfaces. Care must be taken to remove the flux used to reduce this oxide in resealing, as it is readily trapped in the filler metal which can and has caused leaks in various systems, as shown in the typical x-ray of Fig. 24.2.

Typical designs for brazed or soldered joints in tubes and pipes are shown in Fig. 24.3. It is doubtful that brazing can be successfully used in separable seal applications because of the oxidation and diffusion problems encountered in reheating (1100 to 1900° F). Soldering, on the other hand, can be and has been employed in low temperature applications. The increase in strength of solders at very low temperatures (Ref. 3) can be noted on Fig. 24.4, which shows two typical solders and their mechanical behavior at various temperatures.



FIGURE 24.2 X-Ray of Soldered Copper Flange Joint

This full-size x-ray photograph, looking in the direction of flow, shows several voids (light areas) that are largely filled with flux from the soldering operation. In this application, a water line intended for "infinite" life, the joint leaked after 3764 hours of service. With improved soldering techniques and increased attention to removal of flux, the joint was made leak-tight. For satisfactory leak-tightness, the void area must be made less than 15% of the total flange area.



Good Tube to Tube Sheet Designs



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24.4 Swaging(Cold Welding)

It is well known that when metal surfaces are pressed together, the oxide film will fragment if sufficient force is applied. When the high spots touch, the metal behind them suffers plastic deformation. However, oxides are brittle, and the oxide layer must fragment to conform to the changed surface contours. Because of this fragmentation, some metal-to-metal contact occurs which acts as true metallurgical bonds at these sites. An excellent illustration of this characteristic is seen when fresh surfaces of lead or very soft indium are twisted together and the resulting weld has a macro strength approaching that of the base metal. Any metal can be made to weld measurably by shearing the two surfaces together at sufficiently high pressure. However, the results are difficult to produce consistently.

Techniques for achieving consistently satisfactory pressure welds at room temperature were developed for aluminum and copper. These are embodied in the Koldweld^{*} process. These techniques require as much as 33 per cent reduction in aluminum to obtain the desired metallurgical bond. These seals would not be reusable. In the case of tubes and pipes, a new mechanical seal (not metallurgical) called Swagelok^{**} has been developed which provides a leak-tight seal at three points on the tube by slightly deforming the tube itself. These seals are excellent for applications not requiring wide temperature cycling. However, it is doubtful that they would meet either the high or low cycling applications being considered here.

Koldweld Corporation, New York, New York

** Swagelok, Crawford Fitting Co., 884 East 140th St., Cleveland, Ohio

24.5 Conclusions

- 1. Each application will require a specific theoretical study of permeation rates once the materials and processes are established for the given system.
- 2. The inert arc welding of vacuum-tight tubes and pipes of aluminum and stainless steel are well established processes for one-time sealing applications. Reusable welded designs as outlined in this report have been made, although they have not been widely tested in the welding industry as of this date; however, welding is the only process capable of handling both high and low temperature applications.
- 3. Brazing can be successfully accomplished for applications in the range of room temperature to 1000°F. Data on brazed junction performance at -450°F are not readily available. The use of brazing in reusable seals will at best be very difficult to develop, and reliability will be in question due to excessive diffusion and oxidation problems on reheating.
- 4. Soldering has been successfully used in room temperature to -450°F applications. The choice of solder and method of perfecting the junctions will be dictated by the specific application. The higher temperature applications cannot be accomplished with solder seals.
- 5. Swaging (cold welding) could possibly be used in applications utilizing aluminum as the base material. It is not likely that these seals could be reused. No mechanical seal will meet the wide temperature ranges involved in these applications, due to relaxation and oxidation of the different members during its life.
- 6. In joints properly made by any of these processes, the leakage should be no greater than the permeation through the base metal.

24.6 References

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- 3. A. B. Kaufman, "Selecting Solders for Low-Temperature Service," <u>Materials in Design Engineering</u>, November 1958.

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