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CALCULATION OF THE REQUIREMENTS FOR OPERATION OF REFRACTORY-METAL COMPONENTS AT HIGH TEMPERATURE IN VACUUM

R. E. Clausing

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COMPONENTS AT HIGH TEMPERATURE IN VACUUM

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CALCULATION OF THE REQUIREMENTS FOR OPERATION OF REFRACTORY-METAL
COMPONENTS AT HIGH TEMPERATURE IN VACUUM

R. E. Clausing

ABSTRACT

Equations based on kinetic theory relate the contamination of refractory metals in vacuum to the appropriate variables. Several examples are given for which the allowable system pressures are calculated. The practical examples given illustrate the effect of varying several parameters.

The importance of the sticking factor for active gases on hot refractory metals and its effect on the system design are discussed. The experimental data for estimating the sticking factor for oxygen on columbium are given, along with some estimated values.

Experimental data on the composition and rates of outgassing of ultrahigh-vacuum systems and their importance in system design are discussed. Several methods of reducing contamination rates and the relative ease and effectiveness of these methods are presented. It was concluded that (1) tests of 1000 hr or longer will probably require system pressures of between 10^{-9} and 10^{-6} torr, the particular pressure depending upon the residual gas composition, test duration, allowable contamination level, and the other variables discussed; (2) since the most important source of contamination in a properly designed ultrahigh-vacuum system will be outgassing, systems should be designed to be bakable, to operate with walls as cool as practical, and to have a minimum of surface area and outgassing materials inside the system; (3) considerable added protection may be obtained by incorporating sacrificial getter surfaces in the system, or, alternatively, higher pressures may be tolerated if proper getter design is used.

THE PROBLEM

The mechanical properties of the refractory alloys columbium, titanium, molybdenum, tantalum, and tungsten are all markedly affected by contamination with oxygen, nitrogen, or carbon, and their resistance to corrosion by alkali metals may be significantly reduced. Testing of components for space power systems and other uses must be done in environments that do not cause appreciable contamination, for the test results to be meaningful.

The important variables determining the amount of contamination acquired by the metal during test are the test temperature and duration, the metal section thickness, and the environmental atmosphere and pressure. An equation can be derived to relate these variables for simple systems. Another important variable is the fraction of those molecules striking a surface that stick onto the surface, called the "sticking factor." Unfortunately, this factor has been determined for only a few combinations of gases, metals, and temperatures. However, a reasonable value can be assumed for this term in order to approximate the vacuum conditions required for the desired contamination control. The assumptions used in the derivation of these relations must be reconsidered when a complex system is involved.

ANALYTICAL EXPRESSION OF THE RELATION BETWEEN CONTAMINATION AND THE EXPERIMENTAL PARAMETERS

In the following derivation, which is based on the kinetic theory of gases, it is assumed that the rate of arrival of gas at the metal surface is the rate-controlling step in the contamination process. This assumption is not valid at low temperatures and relatively high gas pressures, but is valid for many combinations of contaminants and refractory alloys at low gas pressures and high refractory-metal temperatures (for instance, pressures of $\sim 10^{-8}$ or 10^{-9} torr and temperatures of $\sim 2000^\circ\text{F}$).

The number of molecules N that strike a unit area of surface per unit time is given by the relation

$$N = \frac{1}{4} nv \quad , \quad (1)$$

where n = the number of molecules per cubic centimeter, and v = the arithmetic average velocity of the molecules. From kinetic theory,

$$n = 9.656 \times 10^{18} \frac{P}{T} \text{ molecules cm}^{-3} \quad , \quad (2)$$

$$v = \left(\frac{8RT}{\pi M} \right)^{\frac{1}{2}} \text{ or } 14,551 \left(\frac{T}{M} \right)^{\frac{1}{2}} \text{ cm sec}^{-1} \quad (3)$$

where

P = the gas pressure (torr),

T = the absolute temperature of the gas ($^{\circ}\text{K}$),

R = the universal gas constant,

M = the molecular weight of the gas.

By substituting Eqs. (2) and (3) into Eq. (1),

$$N = 3.513 \times 10^{22} P(MT)^{-\frac{1}{2}} \text{ cm}^{-2} \text{ sec}^{-1} \quad . \quad (4)$$

The mass of gas incident upon a unit of surface area per unit time, defined as G , is equal to mN , where m is the mass of each gas molecule and is equal to the molecular weight divided by Avogadro's number.

Substituting for m and N , it follows that

$$G = 5.833 \times 10^{-2} P(M/T)^{\frac{1}{2}} \text{ g cm}^{-2} \text{ sec}^{-1} \quad . \quad (5)$$

Now if a certain fraction of the gas incident on the surface is absorbed and distributed throughout the thickness of the specimen, the resulting increase in the concentration of this gas in the specimen is given by

$$\Delta C = \frac{sGAt_{\text{sec}}}{V\rho} \quad , \quad (6)$$

where

ΔC = the increase in the concentration of the contaminating gaseous species in the metal (g/g),

t_{sec} = time,

A = area exposed to contamination (cm^2),

V = volume of uniform contamination (cm^3),

ρ = density of the refractory metal (g/cm^3),

s = sticking factor.

If we assume that a thin sheet (or thin-wall tube) of uniform thickness is being contaminated from only one side, A/V reduces to $1/x$, where x is the thickness of the sheet in centimeters. Substituting for G and converting t_{sec} to t_{hr} and x_{cm} to $x_{\text{in.}}$ results in the more convenient form

$$\Delta C = 82.5 \frac{P s t_{\text{hr}} M^{\frac{1}{2}}}{x \rho T^{\frac{1}{2}}}, \quad (7)$$

or, solving for pressure,

$$P = 0.01205 \frac{\Delta C x \rho T^{\frac{1}{2}}}{s t_{\text{hr}} M^{\frac{1}{2}}} \quad (8)$$

Thus the allowable pressure of contaminating gases is directly proportional to the permissible contamination level, the thickness of the specimen, and the square root of the absolute temperature of the gas. This pressure is inversely proportional to the sticking fraction, the test duration, and the square root of the molecular weight of the contaminating gas. Several examples that illustrate the interaction of these variables follow.

Sample Calculations

In the calculation of simple vacuum systems for the protection of refractory metals, the tolerable partial pressure for a given gas can be calculated if a value for the sticking fraction s is assumed. For example, the partial pressure of oxygen that can be tolerated is approximately 8.7×10^{-11} torr, if $s_{\text{O}_2} = 1$ and if a 100 ppm increase in the oxygen content of a columbium tube with an 0.030-in. wall thickness is permissible during a 10,000-hr test. If we had chosen the conditions that x equals 0.060 in. and t equals 2000 hr and if ΔC is allowed to equal 1000 ppm, the allowable P_{O_2} would have been 1×10^{-8} torr. If the more probable value of $s_{\text{O}_2} = 0.1$ (see next section) is assumed, the allowable oxygen pressure becomes approximately 1×10^{-7} torr.

In (real) vacuum systems the atmosphere is, of course, composed of several gases. In order to estimate the oxygen contamination due to a partial pressure of water vapor, for example, the worse case would be

to assume that $s_{\text{H}_2\text{O}} = 1$. The molecular weight of water is 18, but only 16/18 of this weight is oxygen. We use $M = 18$ to calculate $P_{\text{H}_2\text{O}}$, but set $\Delta C_{\text{H}_2\text{O}} = 18/16 \Delta C_{\text{O}_2}$. The net result is that if $s_{\text{H}_2\text{O}} = s_{\text{O}_2}$, the water vapor pressure that can be tolerated is 1.5 times higher than the oxygen pressure for the same allowable oxygen pickup.

At temperatures above 600°C hydrogen is not expected to be absorbed by refractory alloys to any appreciable extent; however, both nitrogen and carbon would be picked up from gases likely to be present in some vacuum systems. If the atmosphere contains carbon monoxide and it is assumed that $s_{\text{CO}} = 1.0$, $x = 0.060$ in., $t = 2000$ hr, and $\Delta C = 1000$ ppm O_2 , then $P_{\text{CO}} = 1.9 \times 10^{-6}$ torr. However, 750 ppm C will be absorbed in addition to the oxygen, providing that the carbon absorption is not limited by diffusion rates or other processes. Calculating the allowable partial pressure of nitrogen with $s_{\text{N}_2} = 1.0$, $x = 0.060$ in., $t = 2000$ hr, and $\Delta C = 1000$ ppm N_2 , we find that $P_{\text{N}_2} = 9.2 \times 10^{-9}$ torr.

The sticking fractions s_{N_2} , s_{CO} , and $s_{\text{H}_2\text{O}} = 1.0$ were used only for illustration and to estimate the most pessimistic boundary conditions. These factors, which can be determined only by experiment, are known only for a few combinations of gas, metal, and temperature. However, their values cannot exceed unity, which can be used, as above, to establish safe partial pressure limits for a given exposure.

EXPERIMENTAL DATA

Sticking Factor

The data given in Table 1 for the initial sticking fraction of gases on tungsten and molybdenum are typical of the only information available. These data were obtained under carefully controlled conditions on clean surfaces in ultrahigh vacuum, but represent only a few systems and do not provide the information required for high temperatures. However, they do illustrate the range of values to be expected and the typical decrease in sticking fraction with temperature. At low temperatures the surface is soon covered with adsorbed gas and the sticking fraction decreases, eventually going to zero. At higher temperatures the adsorbed gases diffuse into the metal, and the sticking fraction

Table 1. Initial Sticking Factors (s) for Several Gases on Tungsten and Molybdenum

Metal	Gas	Temperature (°K)	s	Reference
W	N ₂	300	0.55	J. A. Becker, p 165 in <u>Advances in Catalysis</u> , Vol 7, ed. by W. G. Frankenburg, V. I. Komarewsky, and E. K. Rideal, Academic Press, New York, 1955.
		600	0.17	
W	O ₂	300	0.14	J. A. Becker, E. J. Becker, and R. G. Brandes, <u>J. Appl. Phys.</u> <u>32</u> , 411 (1961).
	CO	300	< O ₂	
	CO ₂	300	} Quite small	
	H ₂ O	300		
Mo	N ₂	305	0.7	R. A. Pasternak and H. U. D. Wiesendanger, <u>J. Chem. Phys.</u> <u>34</u> , 2062 (1961).
		450	0.4	
		710	0.2	

remains finite until the metal will absorb no more oxygen and the surface layers of adsorbed gas again build up. As the temperature is increased and the pressure decreased, the diffusion of gases into the metal becomes more rapid and the arrival rate of gas at the surface decreases until the contamination rate is no longer limited by diffusion but by the gas pressure and the sticking fraction on an essentially clean surface.

There is no reliable way to predict the sticking fraction for the conditions of interest from existing data; therefore, experimental measurements must be made. There seem to have been no attempts thus far to directly determine the sticking fraction on refractory metals at high temperatures. However, Inouye¹ has measured the oxidation rates of columbium in oxygen at pressures between 5×10^{-4} and 3×10^{-5} . The sticking fraction calculated on the basis of Inouye's data is about 0.03 for oxygen on columbium at temperatures of 1200°C, which, because of the relatively high pressure during these tests, is somewhat lower than

¹H. Inouye, pp 649-65 in Columbium Metallurgy, ed. by D. L. Douglass and F. W. Kunz, Interscience, New York, 1961.

would be expected in an ultrahigh-vacuum system. The value of 0.03 should be accepted only with reservation because at high pressures the diffusion of the oxygen from the surface of the metal to its interior may not have been rapid enough to prevent some surface effects. It is also possible that the s_{O_2} value was influenced by impurities or gases other than oxygen adsorbed on the columbium surface.

Experiments are needed for obtaining the sticking probability of oxygen, nitrogen, carbon monoxide, carbon dioxide, water vapor, and methane on columbium and columbium alloy surfaces at temperatures from about 500 to 1500°C in order to predict contamination from these sources in vacuum environment. The sticking fractions for these gases over the same range of temperature for tantalum, titanium, zirconium, and other materials that might be considered as getters would also be valuable.

Gases in Ultrahigh-Vacuum Systems

The composition of gases present inside a vacuum system is dependent upon the past history of the system, the materials of construction, and the method of pumping. For example, in unbaked systems water vapor is often predominant. In ultrahigh-vacuum systems the most prominent gases are usually hydrogen, carbon monoxide, and water vapor, with methane and sometimes oxygen and nitrogen also present.^{2,3} If an oil diffusion pump is used, hydrocarbons may also be present, but they can be reduced to a very low level through the use of suitable trapping.

Because of the likelihood of large getter surfaces in systems of the type we are considering, the major constituents are likely to be the inert gases and hydrogen and perhaps methane. The active gases, which are desorbed from the system or leak into the system, will be rapidly removed by the getter surfaces, while the other (chemically less reactive) gases can be removed only by a diffusion pump or some other means. Gases that are evolved from the walls of the chamber, from the

²Quart. Progr. Rept. Oct. 1-Dec. 31, 1961, Ultrahigh Vacuum Techniques, NYO-9808.

³D. Alpert, "Production and Measurement of Ultrahigh Vacuum," pp 609-63 in Handbook der Physik, Vol. XII, Springer Verlag, Berlin, 1958.

gasket materials, or from leaks into the chamber can be monitored even at very low partial pressures by means of a mass spectrometer attached to the system. If the pressure of potentially harmful gases is known, corrective action can be taken before the components fail or serious contamination occurs.

Outgassing Rates

There is very little information in the literature on outgassing rates of baked surfaces, probably because of the difficulty with which such information is obtained. Apparently, no one has yet successfully designed and operated an apparatus for measuring these outgassing rates from the surface of easily replaceable samples. The only reliable data are those obtained from experimental systems constructed entirely of the materials of interest and instrumented with mass spectrometers. Most of these experimental systems in the past have been of glass.

Unbaked systems commonly have outgassing rates of about 10^{-7} torr-liters/sec-cm² for metal or glass surfaces.⁴ Gasket materials quite commonly have much higher outgassing rates; as an example, the rate for neoprene may be several hundred times this value.⁴ Baked systems, both glass and metal, have outgassing rates as low as 10^{-15} torr-liters/sec-cm².³ A value that should be easily obtained for clean, polished, baked stainless steel surfaces is about 10^{-10} torr-liters/sec-cm².⁵ Baking temperature and time, surface roughness, and cleaning procedure are perhaps the most important considerations in obtaining low outgassing rates.

The outgassing rates are temperature-sensitive, probably following a relation similar to that for vapor pressure: $\log P = A - (B/T)$. When the predominant gas in the system was water vapor, the outgassing rate was observed to become approximately halved for each 10°C decrease in temperature near room temperature. This observation fits the prediction given by the vapor pressure equation for water. This relation has also

⁴B. B. Dayton, "Relationship Between Size of Vacuum Chamber, Outgassing Rate, and Required Pumping Speed," p 101 in American Vacuum Society. National Symposium on Vacuum Technology Transactions, Pergamon Press, 1960.

⁵J. Lewin, private communication with author (Jan. 1962).

been observed for systems using elastomer gaskets in which the gas desorbed was thought to be noncondensable at liquid-nitrogen temperatures.⁶

CONSIDERATIONS FOR THE DESIGN OF EXPERIMENTS

Methods of Reducing Contamination

An obvious way of reducing the contamination level would be simply to increase the thickness of the specimen walls or to cover the walls with a superficial cladding through which the oxygen could not diffuse to the base metal. However, increasing the wall thickness is not generally practical unless the sections were very thin to begin with or the amount of material required is very small. The cladding, while offering some advantages, is difficult to apply. It can be simulated by wrapping the refractory metal with a foil of a more reactive material (see below).

A second and very important method of reducing the contamination is to reduce the outgassing or influx of gases from all sources to very low levels. As mentioned previously, baking temperatures and system operating temperatures affect the outgassing rates. These rates can be improved by a factor of 10 by the relatively easy method of increasing the bakeout time and/or temperature or by lowering the temperature of the outgassing surface as stated previously. A 10°C decrease in the temperature of the outgassing components (near room temperature) may decrease the outgassing rate by a factor of about 2; for example, cooling the wall of a vessel from 50 to 10°C might reduce the outgassing rate by a factor of 10 or more.

A third method of reducing contamination is to decrease the pressure in the system by increasing the pumping speed for active gases, by diffusion pumping, ion pumping, getter pumping, or possibly cryogenic pumping. A 10-in. oil-diffusion pump with a well-designed liquid-nitrogen-cooled trap has a pumping speed of between 1000 and 1500 liters/sec. If the sticking factor for active gases on a hot metallic getter is assumed to be about 0.05, each square foot of such surface area, adequately exposed

⁶I. Farkass and E. J. Barry, "Improved Elastomer Seal Designs for Large Metal Ultrahigh Vacuum Systems Permitting Pressures in the Low 10^{-10} Torr Range," p 35 in American Vacuum Society. National Symposium on Vacuum Technology Transactions, Pergamon Press, 1961.

to the vacuum, would have a pumping speed of about 500 liters/sec for gases having a molecular weight of 28. If the sticking factor is higher than 0.05, which seems likely for zirconium or titanium surfaces, this speed would be increased in proportion to the increase in sticking factor, again assuming that the conductance to the getter surface is not limiting. It is quite evident then, even with the pessimistic assumption that the factor is 0.05, that gettering on hot metallic surfaces offers a very attractive means for removing the chemically active gases from such a system.

A fourth method for reducing contamination rates would be through control of the sticking fraction - a high sticking fraction on getters and a low sticking fraction on the surfaces to be protected - but little information is available to indicate the extent to which this factor can be controlled. Experimental data by Inouye¹ suggest that nitrogen reduces the sticking fraction of oxygen on columbium at pressures of 1×10^{-4} torr. The effect of additives to a vacuum atmosphere might reasonably be expected to be different for different surfaces, so that the sticking fraction of oxygen could be reduced on columbium surfaces but increased, or at least not seriously decreased, on the getter surface. It seems likely, however, that considerable research will be required to develop useful techniques of this kind.

Getter Pumping

The role of getter materials in maintaining the required vacuum is illustrated by a large system such as that used to protect refractory-metal loops circulating alkali metals. Such a chamber might be 10 ft tall and 4 ft in diameter. Let us assume that an outgassing rate of 10^{-10} torr-liters/sec-cm², or 1.5×10^{-13} g of gas per square centimeter per second, may be obtained from the walls after baking and that the gasket surfaces contribute an amount of outgassing equal to that from the walls. (This last assumption may be either optimistic or pessimistic, depending upon the gasket materials, seal design, temperature, and total area of exposed gasket surface.) If the tank is a right cylinder, its interior surface area is about 1.5×10^5 cm². In 10,000 hr the outgassing from this area is 1.5×10^{-13} g/cm²-sec $\times 1.5 \times 10^5$ cm² $\times 3.6 \times 10^7$ sec = 0.7 g.

Adding an equal amount of outgassing from the gasketed materials makes a total of 1.4 g. If this amount of impurity could be uniformly distributed through 100 lb of columbium (45,400 g), then the contamination level would be only 30 ppm, which obviously is acceptable since commercial columbium is likely to have between 100 and 300 ppm O₂ present. If about 10 lb of a tantalum or zirconium getter is distributed in the system so that it absorbs nearly all of these impurities, it will be contaminated to only about 300 ppm O₂ even if it absorbed all of the outgassing of the system. If the getter is placed in the form of an outside radiation heat shield surrounding the loop, it might have an area of about 10 ft² of surface exposed to the vacuum in a geometry that would be favorable for the selective reaction of the impurities on this shield rather than upon the loop. The 10 ft² of surface area would have a pumping speed of about 5000 liters/sec, assuming the conservative sticking fraction of 0.05. Thus it is seen that getters can have adequate speed and capacity to handle the influx of gas.

The equilibrium pressure in the system under these conditions (~5000 liters/sec pumping speed and an outgassing rate of 10⁻¹⁰ torr-liters/sec-cm²) is calculated to be 6 × 10⁻⁹ torr. (This would be obtained, of course, only if no inert gases accumulated in the system or if they were pumped by a diffusion pump or ion getter pump and the air leaks and permeation were negligible.)

A large diffusion pump with a liquid-nitrogen-cooled trap would be necessary to compete with this getter pump for as much as half of the gas. It therefore seems unprofitable to attach a diffusion pump of large capacity to such a system, since the majority of the gas would be absorbed in the getters anyhow. A small diffusion or ion pump would be necessary to prevent the accumulation of chemically inert gases.

Since the sticking fraction on the getter is assumed to be fairly low, some gas can be expected to find its way past the getter material into the loop unless the getter material is carefully arranged to impede the flow of gas to the loop; that is, the vacuum conductance to the loop should be kept low in order to ensure that the active gas molecules have a much greater probability of striking the getter surface than the columbium.

If the pressure indicated by the ion gage is due largely to chemically active gases and becomes too high, corrective action must be taken immediately in order to avoid excessive contamination. If, on the other hand, high pressures are encountered but are due to inert gases, loop operation will not be endangered and the experiment can continue as planned. The use of a mass spectrometer on such a system also greatly facilitates the analysis of vacuum troubles.

Summary of Considerations for System Design

Some of the more important considerations in the design of a vacuum system for protection of refractory metals at high temperature are listed below:

1. The most important single factor to be considered in specifications for vacuum systems of this kind is control of the outgassing rate of the walls. Improvements as high as a factor of 10 can be obtained easily only through proper understanding of the factors affecting outgassing.

2. Systems for the protection of refractory alloys should be designed to be bakable. The baking temperature should be as high as possible, at least 250°C, and, if practical, 400 or 500°C. The baking time should be 16 hr or longer (longer times are required at lower temperatures).

3. The system should be designed so that the walls and as many of the internal components as possible can be maintained at least at the temperature of cooling water obtained from plant mains since small increases in temperature cause relatively large increases in outgassing rates.

4. Careful attention must be given to the design of vacuum seals. Elastomers have high outgassing rates, but can probably be tolerated for many systems if the seal design and operating techniques are proper.

5. Large surfaces of sacrificial getters in the system can be quite useful and are likely to be a practical way of obtaining large pumping speeds to compete with gettering action of large, hot, refractory-metal alloy surfaces. Since the information on sticking fractions available indicates values of 0.03 to 0.5 for oxygen and nitrogen on hot refractory metals, we must assume that molecules will bounce several times on hot

surfaces. Optically opaque one-bounce baffle arrangements (which do not permit the molecules to come directly to the hot refractory loops) do not prevent the gases from reaching the surface to be protected. The sacrificial getters should be designed so that gas molecules moving toward the loop must bounce many times on hot getter surfaces before contacting the loop - in other words, the vacuum conductance to the protected surface should be small in comparison with the getter pumping speed.

6. Diffusion pumps on these systems are useful primarily during bakeout and for removing inert gases to a reasonable level, but their continuous operation might contaminate the system with hydrocarbons and quite possibly accidentally damage the system through vacuum system failure or power failure. A small getter ion pump will not present this hazard and should be capable of handling the small quantity of inert gases present.

7. Thick sections in areas likely to be contaminated are useful in decreasing the level of contamination.

8. A mass spectrometer will keep the operators informed as to the nature of the vacuum atmosphere in the tank, permit a prediction of possible contamination levels, and aid in the determination of the causes of contamination and in the evaluation of vacuum troubles.

9. To permit more accurate estimation of contamination rates, the sticking fractions for oxygen, nitrogen, carbon monoxide, water vapor, and carbon dioxide on hot refractory-metal and getter surfaces must be measured over the range 500 to 1500°C.

SUMMARY AND CONCLUSIONS

Equations relating contamination rate to the system parameters have been developed which, with some assumptions, allow the calculation of maximum contamination rates or maximum permissible pressures, thus providing a safe system design.

Additional experimental data for the sticking fraction of active gases on hot refractory-metal alloy or getter surfaces are needed for the accurate design of protective vacuum systems.

For tests of 1000 hr or longer, system pressures between 10^{-9} and 10^{-6} torr will very likely be required. The allowable pressure will depend upon the residual gas composition, test duration, allowable contamination level, and the other variables discussed. (A properly designed system using sacrificial getters might operate satisfactorily at even higher pressures.)

Since the most important source of contamination in a properly designed ultrahigh-vacuum system will be outgassing, systems should be designed to be bakable, to operate with walls as cool as practical, and to have a minimum of surface area and outgassing materials inside the system.

Considerable added protection may be obtained by incorporating sacrificial getter surfaces in the system, or, alternatively, higher pressures may be tolerated if proper getter design is used.

The use of a mass spectrometer on protective systems should prove quite valuable.

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