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Design and Operation of Evapor-Ion Pumps*

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Abstract

A new type of high vacuum pump has been developed which makes possible the elimination of organic vapors and high vapor pressure materials from vacuum systems, without using coolant traps or baffles. This pump utilizes the gettering action of continuously evaporated titanium in conjunction with ion pumping. Some pumping speeds achieved are: 7000-8000 liters/sec at 3×10^{-6} mm of Hg for hydrogen, 6500-7500 liters/sec at 1.5×10^{-6} mm for nitrogen and oxygen, approximately 9 liters/sec at 4×10^{-6} mm for argon, and approximately 4 liters/sec at 3×10^{-5} mm for helium. The lowest pressure obtainable is about 2×10^{-7} mm of Hg. Dependence of pumping speeds on the temperature of the gettering surface, the pressure and the rate of evaporation of titanium is given.

I. INTRODUCTION

In recent years, the need for high vacuum systems free from organic vapors and other high vapor pressure components has become acute in many applications. In a conventional vacuum system, there are many sources of vapors which condense on the inner surfaces and form chemical impurities or change the properties of the surfaces. Sources of organic vapors, like rubber "O"-rings can be eliminated

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in a properly designed system by incorporating recently developed techniques such as the use of copper or aluminum gaskets. The working fluids of diffusion pumps and mechanical pumps used for evacuating a system are major sources of undesirable vapors. Baffles and liquid air or dry ice traps, which are costly to maintain, reduce the rate of contamination by vapors, but they cannot eliminate contamination completely. A bake-out of a vacuum system drives off condensed vapors from the interior surfaces and thus cleans up the system; however, this is only a temporary clean-up. A high vacuum pump which contains no organic substances or other high vapor pressure components would solve the problem of maintaining a clean vacuum system.

Such pumps have been developed by Foster et al.¹ and Schwarz²

1 Foster, Lawrence, and Lofgren, Rev. Sci. Instr. 24, 388 (1953).

2 H. Schwarz, Rev. Sci. Instr. 24, 371 (1953).

using the idea of ion pumping. Alpert³ has succeeded in achieving

3 D. Alpert, J. Appl. Phys. 24, 860 (1953).

pressures of about 10^{-10} mm of Hg using the pumping action of an ion gauge. Various types of getters have also been used for obtaining high vacua, but the use has been confined to obtaining a pumping action over a short interval of time and in small vacuum systems.

A contaminant free high vacuum pump was needed in this laboratory for use on the accelerating tube of a high voltage electrostatic accelerator now under construction. About three

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years ago, development work was started on a pump which would use a large scale continuous or intermittent gettering, alone or in combination with ionization for obtaining a pumping action.

II. PRELIMINARY INVESTIGATION⁴

⁴ Herb, Davis, Divatia, and Saxon, Phys. Rev. 89, 897 (1953).

Titanium metal was chosen as the getter because of its great chemical activity particularly with the more common gases, hydrogen, nitrogen, and oxygen. In addition, the vapor pressures of titanium and its compounds are all low, and the compounds are very stable solids at room temperature. The evaporation of titanium in large quantities is difficult since very few crucible substances can withstand the chemical activity of molten titanium. Graphite has proven to be the most suitable crucible substance up to the present.

In pump No. 1, shown in Fig. 1, a resistance heated graphite crucible C is used for the evaporation of titanium. Titanium wire W is fed into the hot crucible; the metal evaporates and condenses onto the walls of the cylindrical container. A feeding mechanism moves the wire continuously into the crucible at a controlled rate. Continuous condensation onto the pump wall maintains the activity of the gettering surface, and a pumping action results.

In order to pump gaseous components that do not respond to the gettering action of titanium, the molecules or atoms must be ionized and driven into the active surface. The ionization apparatus consists of a hairpin filament F and two concentric

cylindrical grids, G_1 and G_2 . Electrons emitted by the filament are attracted to the inner grid which is maintained at a positive potential with respect to the filament. The outer grid is maintained at the same potential as the inner grid. Hence, the electrons coast through the field free region (neglecting space charge effects - see Sec. V) between the two grids. When they emerge from the outer grid cylinder, they are subjected to a reverse field in which they are stopped and accelerated back in the opposite direction without change in energy. Thus the electrons oscillate through both grids and ionize the gas in the pump. Positive ions formed between the grids move to the outer grid because of thermal motion. Upon passing through the outer grid cylinder, they are subjected to a field which drives them into the pump wall, where they are buried by the condensing metal. In order to prevent electrons from striking the pump wall, the filament is biased to a positive voltage with respect to the pump wall which is at ground potential.

The lowest pressure achieved by pump No. 1 is 3×10^{-7} mm of Hg. The pumping action was investigated for various gases and it was found that H_2 , N_2 , O_2 , CO_2 , SF_6 , CCl_2F_2 , C_2H_4 , NH_3 and CH_4 are readily pumped, whereas the inert gases argon and helium are pumped rather slowly. Pumping speeds are given for various gases in Table I.

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TABLE I

Pumping speeds achieved by pump number one with the pump wall
water cooled.

Gas	Pumping Speed liters/sec	Ion Gauge Pressure mm of Hg
Hydrogen	150 - 350	1.5×10^{-5}
Nitrogen	60 - 120	"
Oxygen	60 - 100	"
Carbon dioxide	40 - 80	"
Sulfur hexafluoride	30 - 60	"
Freon	30 - 40	"
Air	20 - 50	"
Argon	6 - 12	"
Helium	1 - 3	"

Pump No. 1 has a number of disadvantages. The graphite crucible deteriorates very rapidly due to the formation of a non-volatile residue, probably titanium carbide, and finally the crucible breaks. The geometry of evaporation is poor, and a large amount of titanium is deposited on the surfaces of parts near the crucible rather than the container wall. Because of the 2.5 kw power dissipation of the crucible, the nearby surfaces, some of which are heavily coated with titanium, are much hotter than the wall. Thus temperature control of the gettering surfaces is difficult, and in all mechanisms (see Sec. V) which might contribute to the pumping action, the temperature of the titanium

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layer is important. Pump No. 1 also contains rubber "O"-ring seals, and thus any special characteristics of a system completely free of organic components can not be observed in this system. These and other limitations led to the construction of a new model which was designed to incorporate improvements indicated by pump No. 1 and to permit a closer examination of the pumping action involved.

III. DESIGN AND OPERATION OF PUMP NO. 2 ⁵

5 Divatia, Davis, and Herb, Bull. Am. Phys. Soc. 28, No. 6, 22 (1953).

The second model of the evapor-ion pump, shown in Fig. 2, is an all metal system free of organic and other high vapor pressure components. Copper or aluminum gaskets are used on all demountable joints. The pumping chamber is a stainless steel cylinder C. The evaporation apparatus consists of a $\frac{1}{2}$ " diameter graphite rod surrounded by a spiral filament F made from .025" diameter tungsten wire. Electrons emitted from the hot filament bombard the graphite post which is at a positive potential with respect to the filament and heat it to about 2000°C. Titanium wire W is fed down onto the post from which it evaporated and condenses on the pump wall. The geometry is such that most of the evaporated titanium reaches the wall. The same filament F supplies electrons for ionization and the process of ionizing and driving the ions into the wall is similar to that of pump No. 1. The inner grid G_1 is made of 0.018" diameter molybdenum wire and the outer grid G_2 is made of 0.020"

diameter nichrome wire. Stand off ceramics used for insulating the various components like the filament, the grids, and the post, are properly shielded from incident titanium vapor.

In order to feed the titanium wire onto the graphite post, motion must be transmitted through a vacuum tight seal made without the use of organic gaskets or grease lubricated joints. Sliding friction is likely to lead to abrasion and so ordinary bearings should be avoided.

These requirements are met by a wire feeder R, shown in Fig. 3. A thin-walled, flexible kovar tube T, plugged off at one end with a driving plug D, is copper brazed onto a shoulder S of a hole bored in the main feeder block B. Aluminum foil gaskets G₁ and G₂ provide the necessary vacuum seal between the feeder block and the wall. Ceramic insulators I₁ and I₂ are necessary to insulate the feeder from the pump wall because the titanium wire frequently touches the graphite post, which is at a high potential. A wobble stick W is pivoted at P as shown. The end of the wobble stick which is outside the vacuum system is made to move eccentrically in the hole in the feeder block by the driving shaft M which is powered with a variable speed motor. Since the tube T is flexible, the driving plug D follows the motion of the inner end W₂ of the wobble stick and would move in a circle if there were no constraints. As shown in the schematic of operation, the titanium wire is gripped between the driving plug D and the driving leaf L and thrust forward about 1/16". The wire is then released as the driving plug moves away from the wire, and the locking arm A, which is lifted from the wire during the

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stroke, presses on the wire preventing it from slipping back. The driving plug would move in a circular path except for the constraining leaves C_1 , C_2 , and C_3 which force the plug to move along a rectangular path. The length of the thrust is defined by C_1 and C_3 , while C_2 prevents the plug from swinging too far out to properly engage C_3 . While the plug is still riding on C_3 , the wire is gripped again and the cycle repeats. The titanium wire is stored on a spool O inside the pump. The rate of feed varied from .35 mg/min, i.e., 0.4 mm of wire/min, to 17 mg/min, i.e., 19 mm of wire/min.

The line to the mechanical roughing pump consists of a 3/4" diameter copper tube shown in Fig. 2, a rubber hose, a liquid air trap, and another rubber hose leading to the mechanical roughing pump. In the initial tests made with this pump, the roughing pump was cut off from the main system by pinching closed the copper tubing. The roughing pump was allowed to pump on the trap side of the pinch in the copper tube since it was difficult to obtain a vacuum tight seal by totally pinching off the tubing, probably because the inside of the tubing was contaminated with roughing pump oil and a cold weld was prevented. Later it was found that merely clamping off the rubber hose, so as to disconnect the liquid air trap and the roughing pump from the main chamber, did not alter the ultimate pressures or the pumping speeds significantly. In subsequent runs, the mechanical pump and the trap were disconnected by pinching the rubber hose.

To start the system, a rough vacuum is established with the roughing pump. The pump chamber below the gasket seal S is then

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enclosed in a bake-out furnace and heated to $\sim 300^{\circ}\text{C}$ for about 6 hours with the roughing pump connected through a liquid air trap. At the end of the bake-out, the furnace is removed and the system starts to cool rapidly. This cooling produces a pumping action, yielding a pressure of about 10^{-4} mm of Hg. The roughing pump no longer serves a useful purpose and the rubber hose is clamped off. The pressure is low enough so that filament F can be heated and grid voltage V_G can be applied. This starts the ion-pumping. Post voltage V_P is increased gradually until the post appears white hot (a temperature of about 2000°C), as seen through a system of mirrors and a glass window. Titanium wire is then fed onto the hot graphite post. The wire touches the post and melts back about $1/16"$. The titanium on the post evaporates, and the continuous feeding mechanism moves the wire into contact again. As soon as some titanium has been evaporated, the pressure drops rapidly. The main pump chamber is then immersed in a water bath, and a moderate flow of tap water maintains the temperature at about 20°C . The pressure drops to about 2×10^{-7} mm of Hg in less than a half hour after the beginning of evaporation, and the pump is in operation.

At present, the bake-out seems to be an essential step in starting the pump. If the system is not baked out, the lowest pressure obtainable with a mechanical roughing pump is about 1×10^{-3} mm of Hg. If the filament is turned on at this pressure, without a bake-out, various components start to outgas and the pressure goes up, creating a condition unfavorable for long filament life. Also, as soon as the post or the grid voltage is

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turned on, a glow discharge sets in because of the high pressure and it becomes impossible to achieve efficient ion pumping or efficient electron bombardment of the post. Titanium cannot be evaporated and the pump cannot be started. It may be possible to eliminate the bake-out and start the pump in a shorter time by providing a primer charge of titanium in a crucible which may be heated by conduction current. The pumping action of the evaporated primer charge should reduce the pressure to a value at which electron bombardment may commence.

Standard operating conditions are listed in table II. These have been chosen so as to yield the best pumping action compatible with stable operation and long lifetime.

Table II

Standard operating conditions.

The rate of evaporation of titanium was selected to give the pumping speed desired (see Sec. IV).

Post voltage	V_p	1000 volts
Post current	I_p	500 milliamperes
Grid voltage	V_g	1000 volts
Grid current	I_g	200 milliamperes
Filament bias voltage	V_b	100 volts
Positive ion current to ground at a pressure of 2×10^{-7}	I_b	10 microamperes*
Filament heater current		25 amperes
Filament heater voltage		12 volts
Total power consumption		1 kilowatt
Wall temperature		20°C
Ultimate pressure		2×10^{-7} mm of Hg

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* The significance of this measurement is questionable. Secondary electrons from the wall would contribute to this current. It may be changed by changing voltage V_b , with no appreciable influence on the ion pumping.

When operating under standard conditions, the wire moves down, contacts the post and melts back for a short distance. Just after the contact breaks, the post and grid current increase by about a factor of two, and the post and grid voltages drop by a factor of two. The positive ion current increases considerably, and the graphite post appears to be hotter than usual. This behavior may be due to ionization of titanium vapor as it leaves the post.

IV. INVESTIGATION OF PUMPING SPEEDS

Pumping speeds were measured by the constant pressure method.⁶ From a supply tank gas is bled into the main pump

6 Saul Dushman, Scientific Foundations of Vacuum Techniques (John Wiley and Sons, Inc., New York, 1949) p. 159.

chamber through a needle valve. By adjusting the needle valve and thus the rate of flow into the main chamber, the pressure is raised from the ultimate pressure to a convenient value and held constant. The rate of flow or the leak rate of gas into the pump chamber is determined from the known volume of the gas supply system and the rate of change of pressure in the gas supply system. Since the pressure in the main pump chamber is

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held constant, the quantity of gas bled into the system per unit time is the quantity of gas pumped per unit time. Knowing the constant pressure at which the pumping takes place, the pumping speed is computed from the leak rate.

Let: V = volume of the gas supply system,

P_g = pressure in the gas supply system,

$\frac{\Delta P_g}{\Delta t}$ = rate of change of pressure in the gas supply system,

P = ultimate pressure obtainable in the pump,

p = constant pressure maintained in the pump while gas is leaking into it, or total equilibrium pressure,

L = leak rate of gas through the needle valve,

P_{atm} = atmospheric pressure,

S' = pumping speed of pump corrected for ultimate pressure,

S = pumping speed of pump assuming the ultimate pressure is zero.

S is computed from the total equilibrium pressure in the pump, making no distinction between the total equilibrium pressure and the partial pressure of the gas being pumped. Since only the total equilibrium pressure is of interest in actual pumping applications, S is the practical measure of pumping speed. S' is computed from the partial pressure of the gas being pumped and is of use in understanding the mechanism of pumping action.

The quantities defined above are related as follows:

$$\begin{aligned} \text{Quantity of gas bled per unit time} &= \\ (\text{Leak rate}) \times (\text{atmospheric pressure}) &= \\ \text{Quantity of gas pumped per unit time.} \end{aligned}$$

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$$\text{or } V \frac{\Delta P_g}{\Delta t} = P_{\text{atm}} \times L = (p - p_u) S',$$

Thus

$$L = \frac{V}{P_{\text{atm}}} \frac{\Delta P_g}{\Delta t} \quad (1)$$

$$S' = \frac{V}{p - p_u} \frac{\Delta P_g}{\Delta t} \quad (2)$$

If V is given in cm^3 and t in hours, then L is given in cm^3/hr at N.T.P. If pressures are expressed in mm of Hg and L in cm^3/hr (N.T.P.) then

$$S' = \frac{2.1 \times L \times 10^{-4}}{p - p_u} \text{ liters/second.} \quad (3)$$

If $p \approx 10 p_u$, p_u may be neglected. Then

$$S' = S = \frac{2.1 \times L \times 10^{-4}}{p} \quad (4)$$

The pressures used in the gas supply system ranged from ~ 200 mm of Hg to ~ 750 mm of Hg. Before filling the gas supply with a gas, it is evacuated with a mechanical pump and flushed out with the gas to be pumped. All gases were taken directly from commercial tanks. Gas to be pumped emerges at E in the central region of the container shown in Fig. 2. Since the gas emerges in the pumping region, it meets little impedance in traveling to the pumping surface. Thus pressure gradients due to gas flow are confined to the main pumping chamber and do not influence the readings of the ion gauge located as shown in Fig. 2.

An RCA 1949 ion gauge is used, and since the sensitivity of an ion gauge varies for different gases, pressure readings

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must be corrected for each gas. If p_i = ion gauge reading and p = corrected pressure, then

$$p = k p_i \quad (5)$$

where k is the sensitivity correction factor, k . Approximate values of k are listed in Table III.

Table III
Ion Gauge Sensitivity Correction Factors for Different Gases^{7,8}

<u>Gas</u>	<u>k</u>
Hydrogen	2
Nitrogen	1
Oxygen	1
Air	1
Argon	0.8
Helium	6

7 Saul Dushman, Scientific Foundations of Vacuum Technique (John Wiley and Sons, Inc., New York, 1949) p. 352.

8 A. Guthrie and R. K. Wakerling, Vacuum Equipment and Techniques (McGraw-Hill Book Company, Inc., New York, 1949) p. 129.

When measuring S^i at pressures $p \leq 10 p_u$, p_u cannot be neglected, and the correction factor to be applied to p_u must be estimated. The residual gases responsible for the ultimate pressure p_u have not been identified, but $k \simeq 1$ is assumed for the following reason. The ultimate pressure is probably due to gases which are difficult to trap and hold in the titanium layer. If air is outgassed from metal parts or from the titanium wire when it is heated, the residual gas could be a mixture of argon and air with a high percentage of argon due to the low pumping

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speed for inert gases. Argon would also be the most likely gas to escape the titanium layer after being trapped. Thus from table III the sensitivity/correction factor for a mixture of argon and air is about one. Substituting Eq. (5) in Eq. (3) and Eq. (4)

$$S' = \frac{2.1 \times L \times 10^{-4}}{kp_1 - p_u} \text{ liters/second} \quad (6)$$

for $p > 10 p_u$

$$S = S' = \frac{2.1 \times L \times 10^{-4}}{k p_1} \text{ liters/second} \quad (7)$$

For discussing the mechanism of the pumping action, it is convenient to define the following quantities:

- n_g = the number of hits per stick, that is, the number of times a gas molecule hits the wall before it sticks to the wall.
- π = the probability of capture, or the probability that a gas molecule will stick to the wall upon striking it; that is, $\pi = \frac{1}{n_g}$.
- C = the combination ratio or the number of molecules of a gas which are pumped per titanium atom evaporated.

Now let

- n = number of molecules per cm^3 ,
- v_T = average velocity of a gas molecule at a temperature T ,
- A = the estimated area of the pump wall that is effective in the pumping action,
 $\approx 4700 \text{ cm}^2$ for pump No. 2.

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The number of molecules hitting the pump wall per second is $\frac{A n v_T}{4}$. 9

9 Saul Dushman, Scientific Foundations of Vacuum Technique (John Wiley and Sons, Inc., New York, 1949) p. 91.

If only one impact with the wall were required to trap a molecule, the pumping speed would be

$$S_0 = \frac{A v_T}{4000} \text{ liters/second.}$$

If n_s impacts are required, the pumping speed is

$$S' = \frac{S_0}{n_s} = \frac{A v_T}{4000 n_s} \quad (8)$$

Or in terms of probability of capture

$$S' = \pi S_0 = \frac{\pi A v_T}{4000} \quad (9)$$

If L , the leak rate, is given in cm^3/hr at N.T.P. and R = rate of evaporation of titanium in mg/min , the combination ratio C , is given by

$$\begin{aligned} C &= \frac{\text{number of molecules of gas pumped in a time } t}{\text{number of molecules of Titanium fed in a time } t} \\ &= 0.036 \frac{L}{R} \end{aligned} \quad (10)$$

Pumping speeds for various gases, measured under the standard conditions shown in table II, are listed in table IV along with related quantities.

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Table IV

Pumping Speed for Various Gases Achieved by Pump No. 2

Temperature of gettering surface, T = 20°C

Rate of evaporation of titanium, R = 7 mg/min

= 8 mm of wire/min

Gas	Pumping Speed S liters/sec	Ion Gauge Pressure P_i mm of Hg	Real Pressure P mm of Hg	Average Leak Rate L cc/hr at NTP	Average No. of Hits per Stick n_s	Average Probability of Capture	Average Combination Ratio C
H ₂	7000-8000	1.5×10^{-6}	3×10^{-6}	110	27	3.7×10^{-2}	0.56
N ₂	6500-7500	"	1.5×10^{-6}	50	8	1.3×10^{-1}	0.26
O ₂	6500-7500	"	"	50	7	1.4×10^{-1}	0.26
Air	1000	"	"	7.2	55	1.8×10^{-2}	0.037
A	9	5×10^{-6}	4×10^{-6}	0.18	5200	1.9×10^{-4}	0.0009
He	4	5×10^{-6}	3×10^{-6}	0.57	37000	2.7×10^{-5}	0.0029

The dependence of pumping speeds on the temperature of the gettering surface, the corrected pressure, and the rate of evaporation R was investigated for H_2 , N_2 and O_2 . This behavior and the corresponding curves π - T and π - C are given in Figs. 4a to 7c. Dependence of pumping speeds on the temperature of the gettering surface was investigated for the inert gases argon and helium somewhat qualitatively. It appears that they are pumped slightly better at lower temperatures. Since the immediate objective of these experiments has been to develop a pump suitable for use on an accelerating tube of a high voltage electrostatic accelerator for protons, attention has been concentrated on investigating the pumping of hydrogen.

The spread of values obtained when pumping speeds were measured is indicated in Fig. 4a. A number of factors are responsible for this spread. One of these factors is the "history" of the pumping surface with regard to the amount and type of gas already occluded and the conditions under which occlusion occurred. Pumping speeds tend to be higher if a large amount of titanium has been evaporated onto the wall. In order to preserve uniformity of conditions, pumping speed measurements were taken in as short a time interval as possible. As seen in Fig. 4a, the wall temperature is quite critical for maintaining the constancy of pumping speeds. Fluctuations in wall temperature result from variation in filament power, post heating power, grid power, and frequency of discharge. Because of the strong temperature dependence of the gettering action, the fluctuations in wall temperature cause changes in the pumping speeds.

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Non-uniformity of evaporation may also be partly responsible for the variation in pumping speeds for any one gas. The upper half of the cylindrical chamber is coated with more titanium than the lower half because more titanium evaporates from the top of the post than from the side. As the titanium feeding progresses, an asymmetric cavity may be formed in the top of the post because of deterioration where the wire contacts, and the evaporation becomes more non-uniform.

Errors in pumping speed measurements for a gas also result from impurities in the tank gas supplies used. In most cases, samples of a gas, taken from different tanks under the standard conditions of table II, gave reasonably consistent results.

The pump, due to its slow pumping action for helium, can be used as its own leak detector. Helium, when squirted on a seal, seeps through into the system if there is a leak, and a pressure rise is immediately observed on the ion gauge.

V. DISCUSSION OF RESULTS

The results of the pumping speed investigations not only prescribe the optimum conditions for the best performance of the apparatus as a pump, but suggest, in a qualitative fashion, the nature of the mechanisms involved in the "sorption"¹⁰ of gases.

¹⁰ See reference 9, p. 387.

"Sorption" is defined as any process by which a gas is taken up by a metal. Titanium may combine with a gas in a number of ways, depending on the type of gas and the conditions of combination.^{11,12}

¹¹ See reference 9, chapters 7, 9, and 10.

¹² Colin J. Smithells, Metals Reference Book (Interscience Publishers, Inc. 1949) pp. 379 to 388.

The simplest pumping mechanism is adsorption and subsequent burial by new metal. Because of the inertness of the noble gases, adsorption should be the only means of trapping such gases at the surface of the titanium layer. Since the pumping speeds achieved with this apparatus for the noble gases are too small to be measured unless they are ionized and driven into the surface, the process of adsorption and burial appears to be an inefficient pumping mechanism for noble gases.

For gases which are chemically active with titanium, there are strong chemical forces at the surface in addition to the forces associated with adsorption. The importance of chemisorption, that is, adsorption involving chemical forces, and burial by titanium, as a pumping mechanism is difficult to assess from the present data.

The pressure dependence predicted by a chemisorption and burial pumping mechanism is in qualitative agreement with that observed as shown in curves 5a, 5b, 6a, and 6b. The temperature dependence (Figs. 4a, 4b, and 4c) is also in agreement for higher values of temperature, but the interpretation is not clear at low temperatures. If the process of chemisorption and burial were the principle pumping mechanism, pumping would cease upon the adsorption of one or at most several layers of gas after the evaporation of titanium stopped, whereas, pumping has been observed long after the evaporation was stopped, and the amount of gas taken up by the titanium before the pressure began to rise was much greater than that which could be contained in several adsorbed layers on the area assumed. The effectiveness of chemisorption and burial as a pumping mechanism should be diminished by the continual bombardment of the surface by positive ions and condensing metal atoms which may knock off

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adsorbed gas before it is buried. An estimate of this effect will require further study. Thus chemisorption and burial may contribute to the pumping action, but the assumption that chemisorption is an intermediate step to chemical combination or the solution of the gases in the metal leads to a pumping mechanism which more accurately predicts the results observed.

Titanium may form simple chemical compounds with a gas. However, if stoichiometric combination were the prime pumping mechanism, the strong inverse temperature dependence at temperatures between about 20°C and 200°C would be difficult to understand. The chemical forces which exist between titanium atoms and gas molecules or atoms, except for the noble gas atoms, appear to participate in the formation of gas-metal systems which are more like solutions than compounds in their dependence on various parameters. The formation of compounds as the ultimate state of gas-metal combination is possible but the compounds may be dissolved in the metal.

Titanium is one of a group of metals in which solutions of some common gases, noble gases excluded, or the compounds of these gases differ in important respects from the solutions formed by other metals.^{13,14} First, the gas content of the solution can be

¹³ See reference 12, pp. 380, 385.

¹⁴ See reference 9, p. 552.

very high compared to solutions in other metals, and in some cases, the ratio of gas atoms to metal atoms is comparable to that of the respective compounds. Second, the process of solution is exothermic, and consequently the solubility decreases with an increase in temperature. However, the pressure dependence of the solubilities

of this special group of metals is qualitatively the same as that of other metals. The solubility increases with pressure. A high solubility in titanium for H_2 , N_2 and O_2 would result in high combination ratios and high pumping speeds, as are shown in Figs. 4a to 7c. The expected dependence of pumping speeds on temperature and pressure is also in qualitative agreement with that observed.

Although little is known about the exact process by which gas enters a state of solution, the sequence of adsorption followed by diffusion suggested by Dushman¹⁵ in connection with high vacuum

¹⁵ See reference 9, p. 655.

clean-up by getters is plausible in the present system. Such a model would explain why the combination ratio for hydrogen may for a limited time exceed one (Fig. 5b) when the equilibrium pressure is increased, since the hydrogen can diffuse back into titanium which is not saturated for the new pressure. This model would also explain the temporary increase in pumping speeds of all gases just after a pressure rise. An adsorption-diffusion model might explain the tapering off of pumping speeds at low temperatures if the decrease in diffusion rate with temperature is sufficient to limit the rate of sorption.

As previously mentioned, the noble gases do not combine with titanium in sufficient quantity to give observable pumping rates, and consequently the noble gases are ionized and driven into the surface. The detailed nature of the entrapment has not been determined, but it is reasonable to assume that the pumping speeds for the noble gases depend on (1) the effectiveness of ionization, (2) the depth of penetration into the titanium, and (3) the ability

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of titanium to hold ions driven into it. The problem of reducing the ultimate pressures obtainable in the pump appears to be closely related to the problem of increasing the pumping speeds for noble gases. Ultimate pressures of about 2×10^{-7} mm of Hg were easily achieved by the pump, but efforts to reduce the pressure significantly below 2×10^{-7} mm were unsuccessful. The ultimate pressure is probably due to an equilibrium between the rate of gas occlusion and the rate at which gases, particularly gases with small affinities for titanium, escape the condensed titanium and outgas from other metal parts or the titanium wire. Thus the three factors which determine the pumping speeds for noble gases may also limit the ultimate pressure.

A number of attempts were made to improve the ionization. Magnetic fields, constant and oscillating, parallel and perpendicular to the axis of the pump container were tried to increase the pathlength of the ionizing electrons, but no improvement in ionization was observed. Rough calculations suggest that this lack of improvement may result from a space charge limitation on the electron current density between the grids. Attempts were also made to improve the ionization by trying various values of electron accelerating voltage, both oscillating and rectified. A temporary, small improvement was observed for rectified voltages higher than the 1000 volts given in Table II.

The penetration of positive ions into the titanium was varied by changing the voltage used to accelerate the ions. One thousand volts appeared to be the optimum value in a possible range of 0 to 1300 volts available in pump No. 2. At voltages above 1000 volts, the pressure rose slightly, perhaps due to outgassing of the condensed titanium or other parts. Below 1000 volts, the rise in pressure is probably due

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to insufficient penetration into the titanium by the positive ions.

As mentioned in Sec. IV, the pumping speeds for the noble gases were not investigated in detail, and consequently little is known about the ability of the titanium layer in this system to hold ions driven into it. However, an improvement in the noble gas pumping speeds and the ultimate pressure might be obtained by prior outgassing of the titanium wire to be evaporated to increase its sorption capacity.

The discharge condition which exists while the titanium evaporates from the post is not well understood, but may be due largely to ionization of the titanium vapor as it comes from the post.

VI. APPLICATIONS AND CONCLUSIONS

To evaluate pump No. 2 as a means of evacuating the accelerating tube of a high voltage electrostatic accelerator, the pump was subjected to a lifetime test under the conditions given in Table V.

Table V

Lifetime Test

Temperature of gettering surface	=	approximately 20°C
Rate of evaporation of titanium	=	1.4 mg/min
or	=	1.6 mm of wire/min
Leak rate of H ₂ into the pump	=	20 cc/hr NTP
Equilibrium pressure while pumping	=	1×10^{-6} mm of Hg
Pumping speed for H ₂	=	4000 - 8000 liters/sec at 1×10^{-6} mm of Hg

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The pump was operated under these conditions for seven days, and it required no attention which could not have been provided by automatically stabilized circuits. At this feed rate, one pound of titanium would last about 200 days.* The

* Titanium wire, 0.020" D., may be ordered from Titanium Metals Corporation of America, 60 East 42nd Street, New York 17, New York, at \$42.60 per pound as of October 30, 1952.

power consumption, as noted in Table II was about 1 kw. After three and one-half additional days of running at reduced titanium feed rates, the filament burned out.

A pump with an evaporation device similar to that in pump No. 1, but with no ionization apparatus has been used for about one year in parallel with a D.P.I. 500-MC diffusion pump for the evacuation of a vacuum furnace. Use of the getter pump reduces the initial pump down time by a factor of about 5.

The evapor-ion pump, in its current state of development, satisfies the need for a high speed vacuum pump which is free of organic vapors and high vapor pressure components in those applications where the limitation on lifetime can be tolerated and only traces of noble gases must be pumped. Further development, particularly for the pumping of noble gases and for achieving long lifetime will be necessary before the pump is ready for general use.

Improved ionization should increase the pumping speeds for the noble gases and lower the ultimate pressures. Outgassing of the titanium wire may also improve the noble gas pumping

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speeds and the ultimate vacuum. A reduction in intensity of the discharge by a current stabilizing circuit and changes in geometry may increase the lifetime of the filament and post. An increase in the area of the active surface should increase the pumping speeds.

A program to investigate in more detail the mechanism of pumping and to exploit the potentialities of the apparatus as a pump is being continued in this laboratory.

VII. ACKNOWLEDGMENTS

We wish to thank Prof. R. G. Herb for suggesting the basic ideas involved in the evapor-ion pump and for valuable guidance during the course of development. We are also indebted to Prof. J. L. Powell for discussions on the space charge problem. We would like to thank Melvin Bina for assistance in assembling the apparatus and A. F. Swenson and L. O. Scharnke for machine work and helpful suggestions on design problems.

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CAPTIONS FOR FIGURES

Fig. 1 Evapor-ion Pump No. 1. Titanium wire *W* is pushed down continuously by a wire feeder shown in detail in Fig. 3, into the resistance heated graphite crucible *C*. Titanium, after evaporating, condenses on the inner surface of the pump wall and produces a pumping action. Components in the upper half of the pump chamber accomplish ion pumping. After the system has started operating, flap valve *FV* is closed to cut off other pumps.

Fig. 2 Evapor-ion Pump No. 2. Titanium wire *W* is pushed down by a wire feeder *R* (details in Fig. 3), onto the graphite post *P*, heated by electron bombardment. Continuously evaporated titanium condenses on the pump wall and a pumping action results. The same filament *F* supplies electrons for electron bombardment and ion pumping. Leads carrying current to the components from outside the pump, by means of lead-thrus like *B*, are not shown in the diagram.

Fig. 3a Cross-sectional view of titanium wire feeder.

Fig. 3b Schematic of operation of titanium wire feeder. Details of operation are given in Section III.

Fig. 4a Pumping speed as a function of the temperature of the gettering surface, for H_2 , N_2 and O_2 . At temperatures $> 150^\circ C$, pumping speeds were measured at relatively

higher pressures, as indicated, because of high ultimate pressures. The spread of values obtained is indicated by the individual points plotted; curves are drawn through average values. Only the average values are plotted in the rest of the curves.

Fig. 4b The probability that a gas molecule will stick to the wall upon striking it, as a function of the temperature of the gettering surface, for H_2 , N_2 and O_2 .

Fig. 4c The number of times a gas molecule hits the wall before it sticks to the wall, as a function of the temperature of the gettering surface, for H_2 , N_2 and O_2 .

Fig. 5a Pumping speed as a function of the pressure in the main pump chamber, for H_2 , for two different rates of evaporation of Ti, indicated on the curves. Dotted lines indicate the pumping speeds corrected for the ultimate pressure, as explained in Sec. IV.

Fig. 5b The probability that a H_2 molecule will stick to the wall upon striking it, as a function of the number of molecules of H_2 pumped per titanium atom evaporated. Rates of evaporation of titanium, which are kept constant, are indicated on the curves, and the pressure at which each pumping speed was measured is listed near the point.

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Fig. 6a Pumping speed as a function of the pressure in the main pump chamber, for N_2 and O_2 . Dotted lines indicate the pumping speeds corrected for the ultimate pressure, as explained in Sec. IV. Rate of evaporation of titanium was held constant at 7 mgm/min.

Fig. 6b The probability that a gas molecule will stick to the wall upon striking it, as a function of the number of molecules of a gas pumped per titanium atom evaporated. The rate of evaporation of titanium was kept constant; the pressure at which each pumping speed was measured is listed near the point.

Fig. 7a Pumping speed as a function of the rate of evaporation of titanium, for H_2 , N_2 and O_2 . Three points for H_2 , at a pressure of 1×10^{-6} mm of Hg, were obtained during the life-time test discussed in Sec. VI, and hence under a different set of conditions. While taking data for O_2 , heavy discharges made conditions somewhat unstable.

Fig. 7b The probability that a H_2 molecule will stick to the wall upon striking it, as a function of the number of molecules of H_2 pumped per titanium atom evaporated. The pressure was kept constant. Three points at a pressure of 1×10^{-6} mm of Hg, were obtained during the life-time test discussed in Sec. VI, and hence under a different set of conditions. Rate of evaporation of titanium is listed near each point.

Captions for Figures

Fig. 7c The probability that a gas molecule will stick to the wall upon striking it, as a function of the number of molecules of a gas pumped per titanium atom evaporated, for N_2 and O_2 . Pressure was kept constant, the rate of evaporation of titanium is listed near each point. While taking data for O_2 , heavy discharges made conditions somewhat unstable.

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FIG. 1

FIG. 2

FIG. 3A

FIG. 3B

PUMPING SPEED S, LITERS / SEC.

 $\times 10^3$
 NITROGEN

HYDROGEN

OXYGEN

 Ti EVAPORATION RATE
 $R = 7 \text{ MGM. / MIN.}$
 H_2 , - $P = 3 \times 10^{-6} \text{ MM. Hg.}$
 $\text{N}_2 \& \text{O}_2$, - $P = 1.5 \times 10^{-6} \text{ MM. Hg.}$

1. $P = 5 \times 10^{-5} \text{ MM. Hg.}$
2. $P = 1 \times 10^{-4} \text{ MM. Hg.}$
3. $P = 5 \times 10^{-5} \text{ MM. Hg.}$
4. $P = 1 \times 10^{-4} \text{ MM. Hg.}$
5. $P = 1.5 \times 10^{-4} \text{ MM. Hg.}$

FIG. 4A

-100

0

100

200

TEMPERATURE, $t^\circ \text{C.}$
 1
 2
 3

 4
 5

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Ti EVAPORATION RATE $R = 7 \text{ MGM. / MIN.}$

H_2 , - $P = 3 \times 10^{-6} \text{ MM. Hg.}$

$\text{N}_2 \text{ \& } \text{O}_2$, - $P = 1.5 \times 10^{-6} \text{ MM. Hg.}$

1. $P = 5 \times 10^{-5} \text{ MM. Hg.}$

2. $P = 1 \times 10^{-4} \text{ " "}$

3. $P = 5 \times 10^{-5} \text{ " "}$

4. $P = 1 \times 10^{-4} \text{ " "}$

5. $P = 1.5 \times 10^{-4} \text{ " "}$

FIG. 4B

PROBABILITY OF CAPTURE, π

0.20
0.18
0.16
0.14
0.12
0.10
0.08
0.06
0.04
0.02
0

NITROGEN

OXYGEN

HYDROGEN

-100

0

100

200

TEMPERATURE, $t^\circ \text{C.}$

FIG. 5A

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HYDROGEN

 $t \approx 20^\circ \text{C.}$

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NITROGEN & OXYGEN

$P = 5 \times 10^{-7}$ MM. Hg.

$t \approx 20^{\circ}\text{C.}$

T_i EVAPORATION RATE,
 $R = 7$ MGM. / MIN.

FIG. 6B

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FIG. 7A

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HYDROGEN

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NITROGEN & OXYGEN

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