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STUDY OF ADSORPTION OF GASES ON SOLIDS IN THE HIGH VACUUM RANGE

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GENERAL TELEPHONE & ELECTRONICS LABORATORIES INC.
Bayside, New York

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OFFICE OF AEROSPACE RESEARCH
UNITED STATES AIR FORCE
BEDFORD, MASSACHUSETTS

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ABSTRACT

An investigation of the interaction of nitrogen with a hot tungsten filament has shown that a chemical pumping effect occurs. The magnitude of this effect and its variation with temperature is discussed.

Preliminary results on the study of the effect of adsorbed oxygen on the work function of molybdenum at room temperature have indicated that there are three stages in the adsorption process: during the first two stages, there is a rapid increase in the work function, whereas in the last stage there is a small increase and very slow rate of rise in work function.

1. THE INTERACTION OF NITROGEN WITH A TUNGSTEN FILAMENT

1.1 INTRODUCTION

To check the operation of the high-vacuum system,[1] a study was initiated of the interaction of nitrogen with a tungsten filament. Similar studies have been made by many other investigators. In this investigation a constant pressure of nitrogen was maintained in an experimental high-vacuum chamber by a flow method. The partial pressure of nitrogen was monitored by an omegatron mass spectrometer. The variation of partial pressure as a function of time and filament temperature is a measure of the interaction rates involved.

In preliminary experiments an anomalous behavior was encountered in that after allowing the filament to return to room temperature, the new equilibrium partial pressure of nitrogen was greater than the original equilibrium pressure established. This effect, which occurred only at initial filament temperatures greater than about 1800°K, is attributed to chemical pumping of nitrogen at the tungsten filament.

The experimental environment, measurements and results of this investigation are discussed below.

1.2 EXPERIMENTAL RESULTS

The ultra-high vacuum system used provides for differential pumping of separate chambers isolated by bakable valves. Three pumps are used; two ion pumps and one three-stage oil diffusion pump, preceded by two bakable molecular sieve adsorption traps. A block diagram of the system is shown in Fig. 1. Bayard-Alpert inverted ionization gauges are

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[1] Scientific Report Nos. 1 and 2 on Contract No. AF19(628)-331.

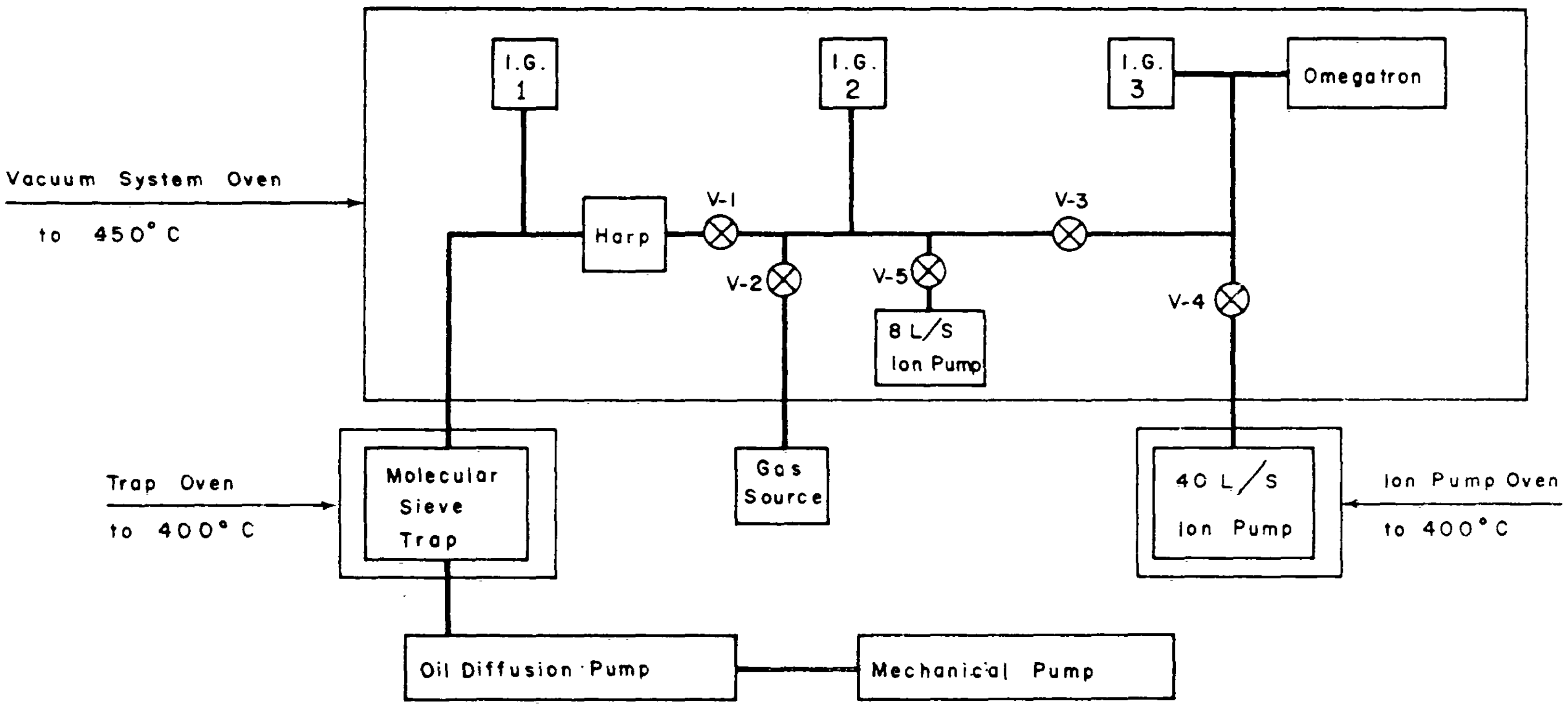

Fig. 1. Block diagram of ultra-high vacuum system for surface adsorption studies.

used to monitor total pressures in each chamber, and an omegatron mass spectrometer is connected to the experimental chamber to measure the partial pressures of the ambient gases. All portions of the system above the diffusion pump can be baked to at least 400°C, and all active components can be outgassed by joule heating, electron bombardment or induction heating.

After initial processing of the system the partial pressures of the residual gases were below the present limit of detection, approximately 10^{-12} Torr. (All pressures reported are based on an assumed gauge constant of 10 Torr^{-1}.)

A tungsten filament of the ionization gauge was selected for initial study of adsorption phenomena, and was outgassed by heating at 2500°K for four hours before admitting nitrogen. With the filament at a temperature of approximately 2000°K, a constant pressure (p_o) of nitrogen was established in the experimental chamber by adjusting valves V3 and V4 to set the leak rate of nitrogen into the chamber equal to the rate at which nitrogen is removed by the ion pump. After steady-state conditions were attained, the filament was cooled to room temperature (~ 300°K), and the change in the partial pressure of nitrogen was continuously monitored by the omegatron.

After the filament adsorption process was essentially completed, the pressure, instead of asymptotically rising to p_o, rose to a substantially higher value, as shown in Fig. 2. This represents an additional pumping effect at the high temperature. To investigate this anomaly, measurements were made of the equilibrium pressures at different filament temperatures. The data are shown in Fig. 3 as the logarithm of the ratio of the low-temperature equilibrium pressure to the equilibrium pressure at temperature, T, as a function of reciprocal temperature. Also shown to the right in Fig. 3 is the electronic pumping effect where the magnitude of the

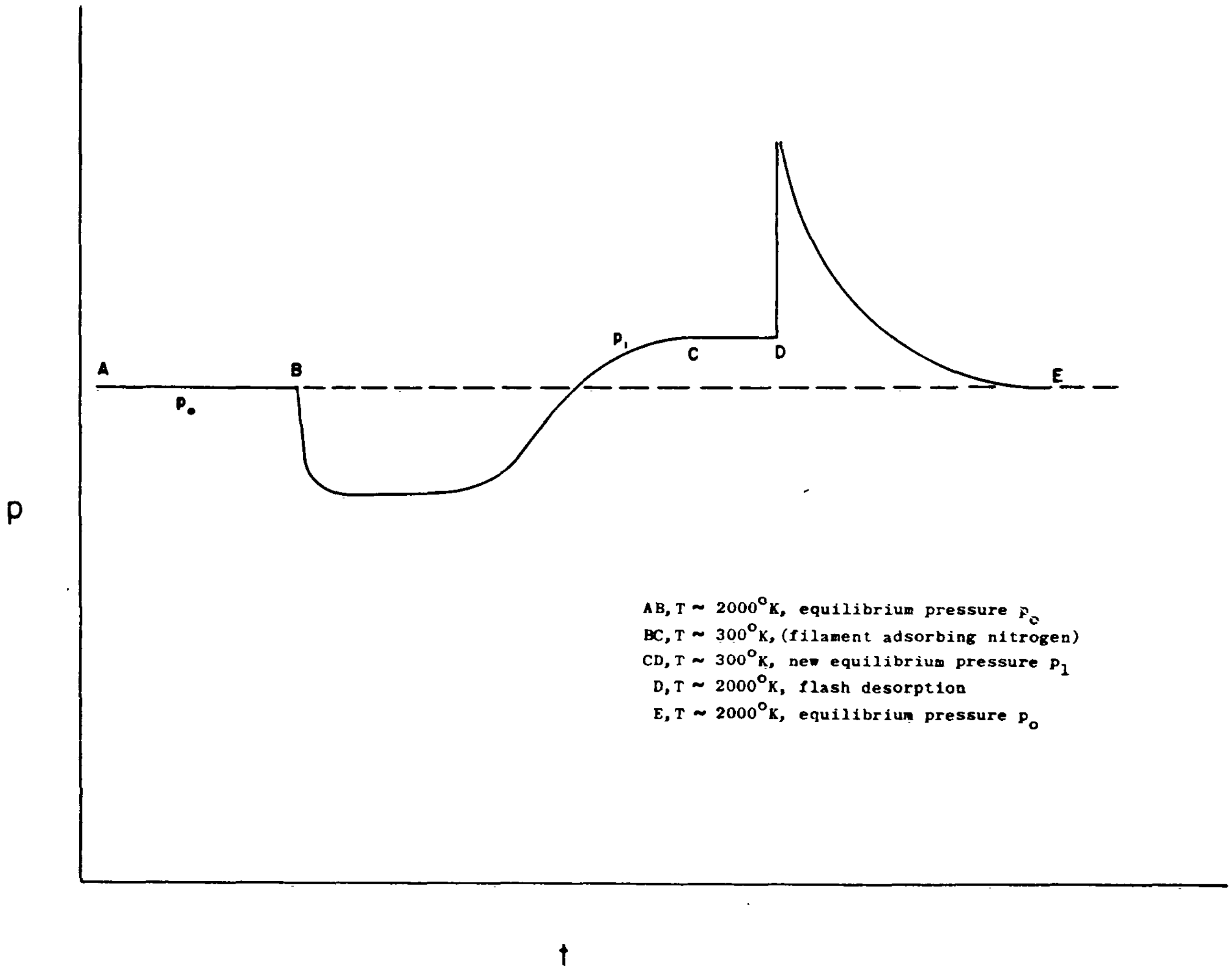

Fig. 2. Nitrogen pressure vs time with the tungsten filament at the temperatures shown.

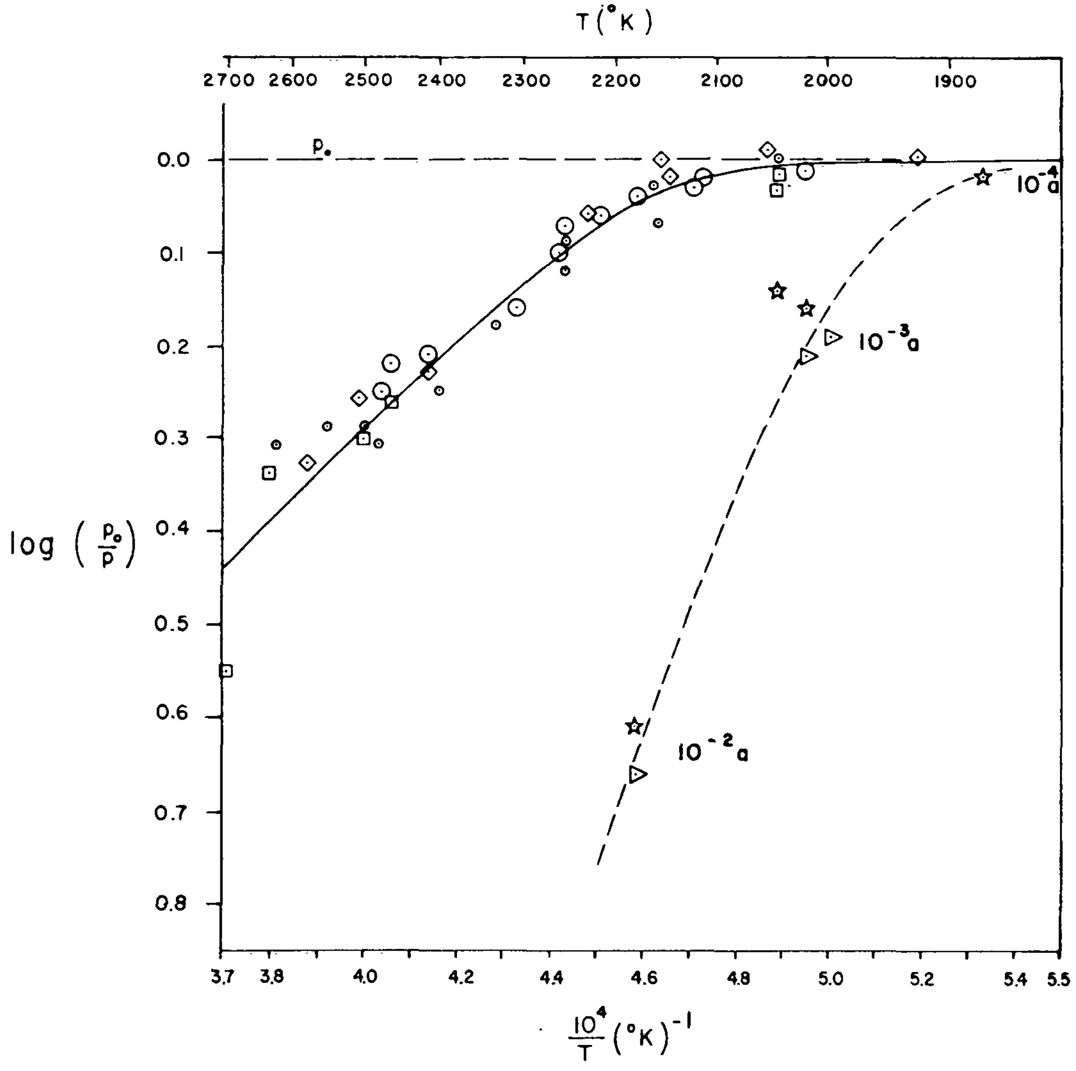

Fig. 3. (Left) Equilibrium pressure as a function of temperature;
(Right) Electronic pumping effect as a function of temperature.

electron ionizing current is indicated on the figure. No difference in the equilibrium pressure was noted with the filament less than approximately 1800°K, for several equilibrium pressures between 1.7×10^{-8} Torr and 2.4×10^{-8} Torr. These data are discussed in Section 1.3 in terms of the equations of mass balance for the vacuum system.

1.3 DISCUSSION

The differential equation satisfying the conditions set forth above is, neglecting omegatron pumping, wall adsorption and desorption effects,

$$V\frac{dn}{dt} - C_1 - n\frac{\bar{v}}{4}a_p - n\frac{\bar{v}}{4}a_f\,\alpha(T) \tag{1}$$

where V = volume

n = molecular density

$\bar{v}$ = average velocity of molecules

a_p = effective pump aperture

a_f = filament area

C_1 = molecular inflow rate

$\alpha(T)$ = fraction of molecules arriving at filament which are lost due to chemical pumping

T = absolute temperature.

At equilibrium, and for $\alpha(T) = 0$

$$C_1 = n_o\frac{\bar{v}}{4}\,a_p. \tag{2}$$

Equation (1) can now be written as

$$\frac{4V}{\bar{v}}\,\frac{dn}{dt} = a_p\,(n_o - n) - a_f\alpha(T)\,n. \tag{3}$$

At equilibrium, and for $\alpha(T) > 0$

$$\alpha(T) = \frac{a_p}{a_f}\left(\frac{n_o}{n} - 1\right) . \tag{4}$$

Figure 4 shows $(n_o/n)-1$ as a function of reciprocal temperature; the curve at the upper right is proportional to the sum of the electronic and chemical pumping speeds. The ionizing electron current is indicated on the curve. The curve at the lower left is proportional to the chemical pumping speed alone. These plots were obtained from the smooth curves drawn through the data shown in Fig. 3.

The determination of $\alpha(T)$ depends on the value of a_p, which requires a measure of C_1, the rate of admission of nitrogen to the system. This quantity was not measured directly owing to premature termination of the experiments caused by valve failure. However, an indirect measure of C_1 (and thus a_p) was obtained by analyzing the transition between the equilibrium concentrations at two temperatures. This transition region is plotted in Fig. 5. The assumption was made that the chemical pumping of nitrogen by the tungsten filament is a function of temperature only. This is justified by the experimental data which indicate that the ratio of the equilibrium pressures is a smooth function of temperature. With this assumption,

$$\alpha(T) = \alpha(T_2), \text{ for } t_1 < t \leqslant t_2 . \tag{5}$$

At $t = t_2$,

$$\alpha(T_2) = \frac{a_p}{a_f}\left(\frac{n_o}{n_2} - 1\right) . \tag{6}$$

Substituting this value in Eq. (3), we have

$$\frac{4V}{\bar{v}}\frac{dn}{dt} = a_p n_o - a_p \frac{n_o}{n_2} n \cdot \tag{7}$$

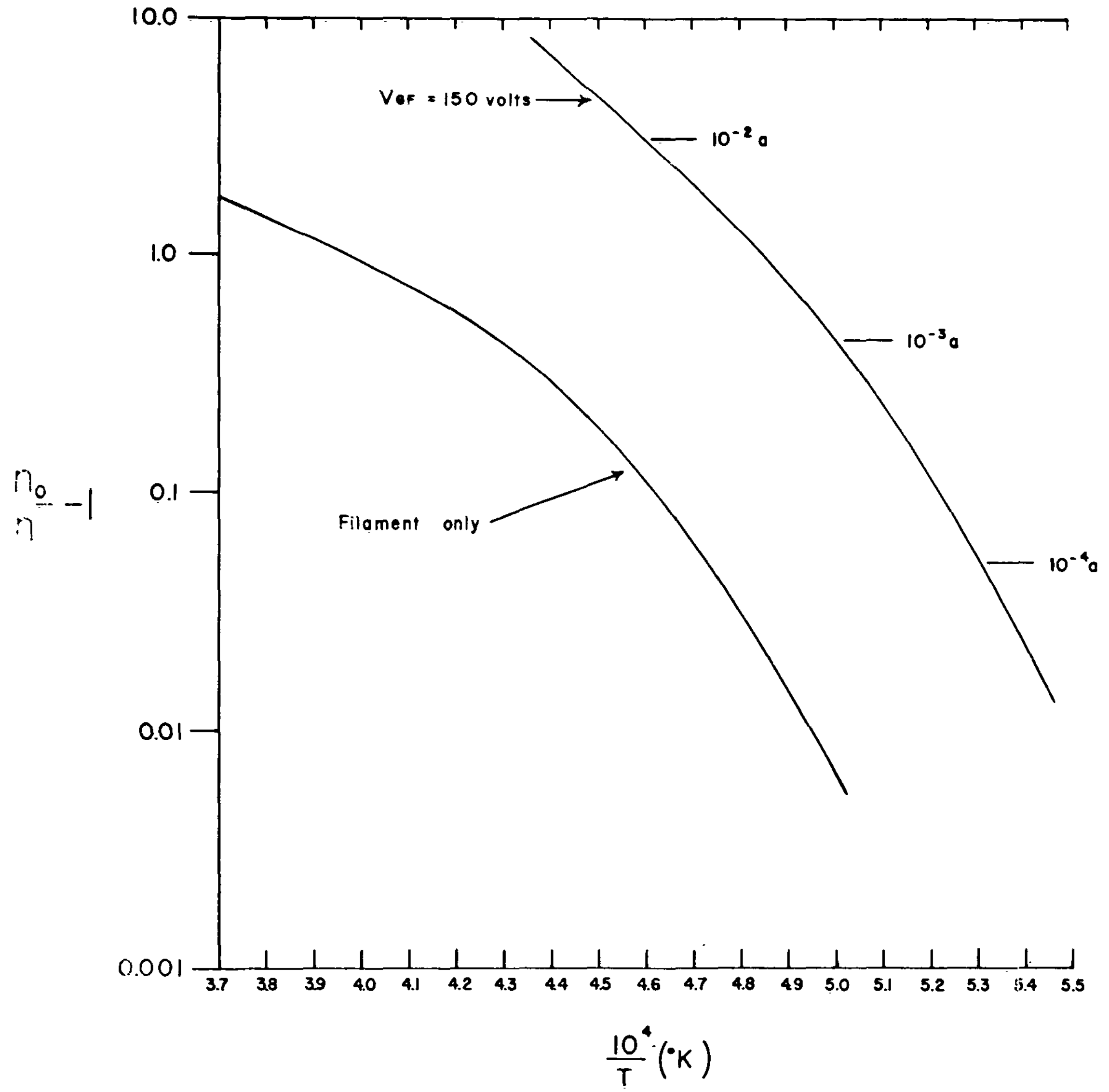

Fig. 4. (Left) Chemical pumping of filament vs temperature; (Right) Electronic and chemical pumping vs temperature.

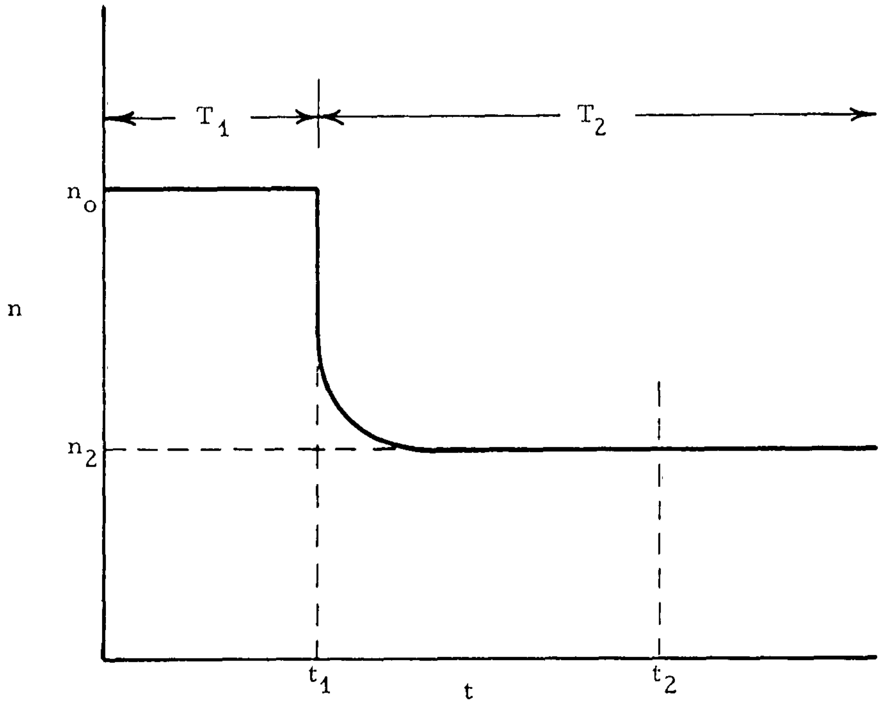

Fig. 5. Transition region between equilibrium concentrations at two temperatures.

Integration from n_o to n_2 gives

$$n = n_2 + (n_o - n_2) \exp\left[-\frac{n_o}{n_2} \cdot \frac{\bar{v}}{4} \frac{t}{V} a_p\right], \tag{8}$$

where the time constant

$$\tau = \frac{V}{S} = \frac{V}{\frac{n_o}{n_2} \cdot \frac{\bar{v}}{4} a_p}, \tag{9}$$

in which S is the pumping speed.

From the data for one transition between ~ 1800°K and 2700°K,

$$p_o = 1.74 \times 10^{-8}\,\text{Torr} \cong n_o, \quad T < 1800^\circ\text{K}$$
$$p_2 = 4.8 \times 10^{-9}\,\text{Torr} \cong n_2, \quad T = 2700^\circ\text{K}$$
$$\tau = 15 \text{ seconds.}$$

Under these conditions the total pumping speed due to the pump and the tungsten filament is, from (9)

$$S = \frac{V}{\tau} = 2.66 \times 10^{-2} \text{ liter/sec.} \tag{10}$$

The effective pump aperture, a_p, is

$$a_p = \frac{S}{\frac{n_o}{n_2} \cdot \frac{\bar{v}}{4}} = 6.2 \times 10^{-4}\,\text{cm}^2. \tag{11}$$

The function $\alpha(T)$ is determined from Eqs. (4) and (11), and the experimentally determined curve of $(n_o/n)-1$ in Fig. 4, as follows:

$$\alpha(T) = \frac{a_p}{a_f}\left(\frac{n_o}{n} - 1\right) = 1.7 \times 10^{-3}\left(\frac{n_o}{n} - 1\right). \tag{12}$$

From Eqs. (1) and (11), the chemical pumping speed of tungsten filament for nitrogen is

$$S = \frac{\bar{v}}{4} a_f \alpha(T) = 7.35 \times 10^{-3} \left(\frac{n_o}{n} - 1 \right) \frac{\text{liter}}{\text{sec}}, \tag{13}$$

and for T = 2185 $^{\circ}$K, $S_c = 8.9 \times 10^{-4}$ liter/sec.

The work described here will be continued in order to establish unambiguously the magnitude of the chemical pumping effect and to determine the effect of this pumping on the adsorption measurements.

The value of S_E calculated at the same temperature of 2185 $^{\circ}$K (10^{-2} ampere electron emission current) is

$$S_E = 2.3 \times 10^{-2} \frac{\text{liter}}{\text{sec}} \quad .$$

1.4 FUTURE WORK

The measurements described above will be continued in order to establish unambiguously the magnitude of the chemical pumping and to determine the effect of this pumping on the adsorption measurements. Separate experiments will be undertaken to determine whether chemical pumping can be observed in sealed-off omegatrons.

2. ADSORPTION OF OXYGEN ON MOLYBDENUM

2.1 INTRODUCTION

As is well known, the work function of a material is a sensitive indicator of foreign matter deposited on its surface. A retarding-field technique has been developed for the rapid and accurate determination of the variation in work function of a surface on which electronically active material is deposited.

The retarding-field method is based on measuring the displacement of the retarding-field characteristic of a planar diode. The anode work function φ_A can be obtained from the retarding-field current-voltage relation,

$$I_r = sAT^2 \exp\left[-e\,(-U_a + \varphi_A)/kT\right], \tag{14}$$

where I_r is the retarding-field current (amps), U_a is the anode voltage (volts), s is the emitter area (cm^2), A is taken to be 120 (amp/cm^2 per deg^2), and e/k is 11,610 (deg/volt). The change in the applied voltage (U_a) necessary to maintain a constant current measures the variation in anode work function resulting from a deposit of active material onto an originally clean anode surface. This voltage can be measured continuously during an evaporation or adsorption process; a critical coverage of the collector surface is indicated by a minimum or maximum in the curve of anode work function versus time. This method used previously to measure sublimation rates of Ba and BaO from different substrates[2] is now being adapted to measure the adsorption of gases on surfaces and the variation of the work functions of surfaces due to gas adsorption.

2.2 EXPERIMENTAL RESULTS

Preliminary results have been obtained on the effect of adsorbed oxygen on the work function of molybdenum at room temperature (~ 300°K). The work function was measured in the planar diode structure shown in Fig. 6 by the retarding-field method using an oxide cathode (BaO on Ta) at 756°K as the electron source and a polycrystalline molybdenum anode as the collector. To minimize the possibility of reactions between the

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[2] J. Florio, J. Appl. Phys., 34, 200 (1963).

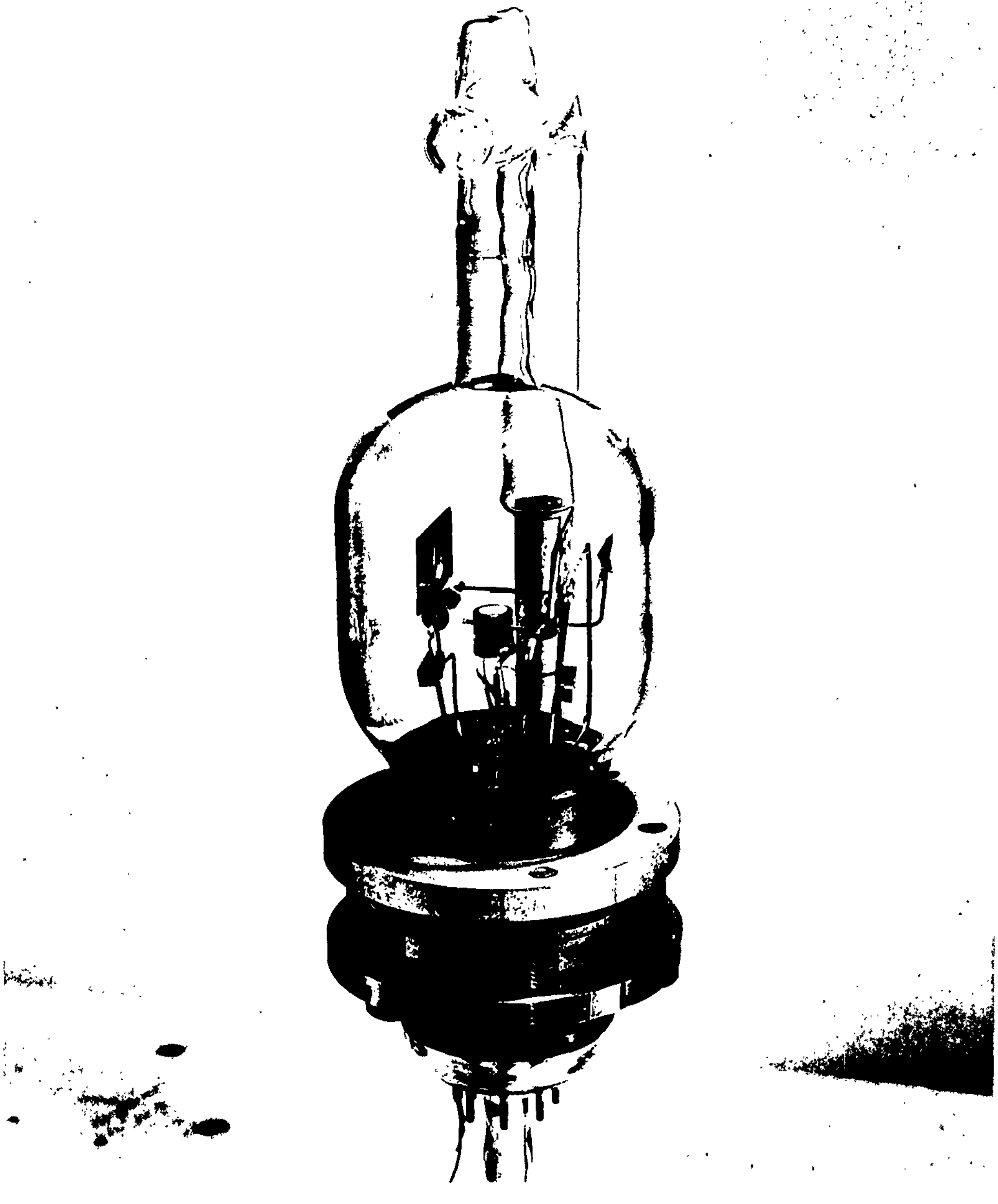

Fig. 6. Planar diode structure for measurments of effect of adsorbed gases on the work function of molybdenum.

the hot-filament ionization gauge and incoming oxygen, a 0.2 liter/sec VacIon pump was appended to the diode envelope for monitoring the gas pressure in the tube. The molybdenum anode was cleaned by electron bombardment. Spectroscopically pure oxygen from Linde Air Products was introduced into the system through a bakable metal valve. After completion of the cathode processing and anode bombardment in a vacuum system employing a 5 liter/sec VacIon pump, the latter was sealed off from the system. The experimental setup is shown at the upper left in Fig. 7.

With oxygen entering the system at a fixed leak rate, the following parameters were measured as a function of time: (1) the pressure in the diode assembly, using the VacIon pump as a gauge; (2) the anode work function from retarding-field data, Eq. (1); and (3) the effective cathode work function from the saturation emission ($I_s = sAT^2 \left[\exp(-e\,\varphi_c/kT) \right]$). Typical results obtained after flushing the system several times with oxygen, are shown in Fig. 7.

Prior to the admission of oxygen to the system (background pressure of 2×10^{-9} Torr), the anode and cathode work functions remained constant at values of 4.66 ev and 2.05 ev, respectively. Upon opening the leak valve, the pressure rises until a quasi-equilibrium state is reached and the pressure holds constant at $\sim 7 \times 10^{-8}$ Torr; the adsorption of oxygen on the molybdenum anode and oxide cathode is indicated by the changes in their work function. There are three phases in the adsorption process.

The rate of change in work function is similar for both the molybdenum and oxide surfaces. The results indicate three characteristic stages: during the first two stages the pressure remains unchanged, indicating a constant overall rate of adsorption of the incoming molecules.

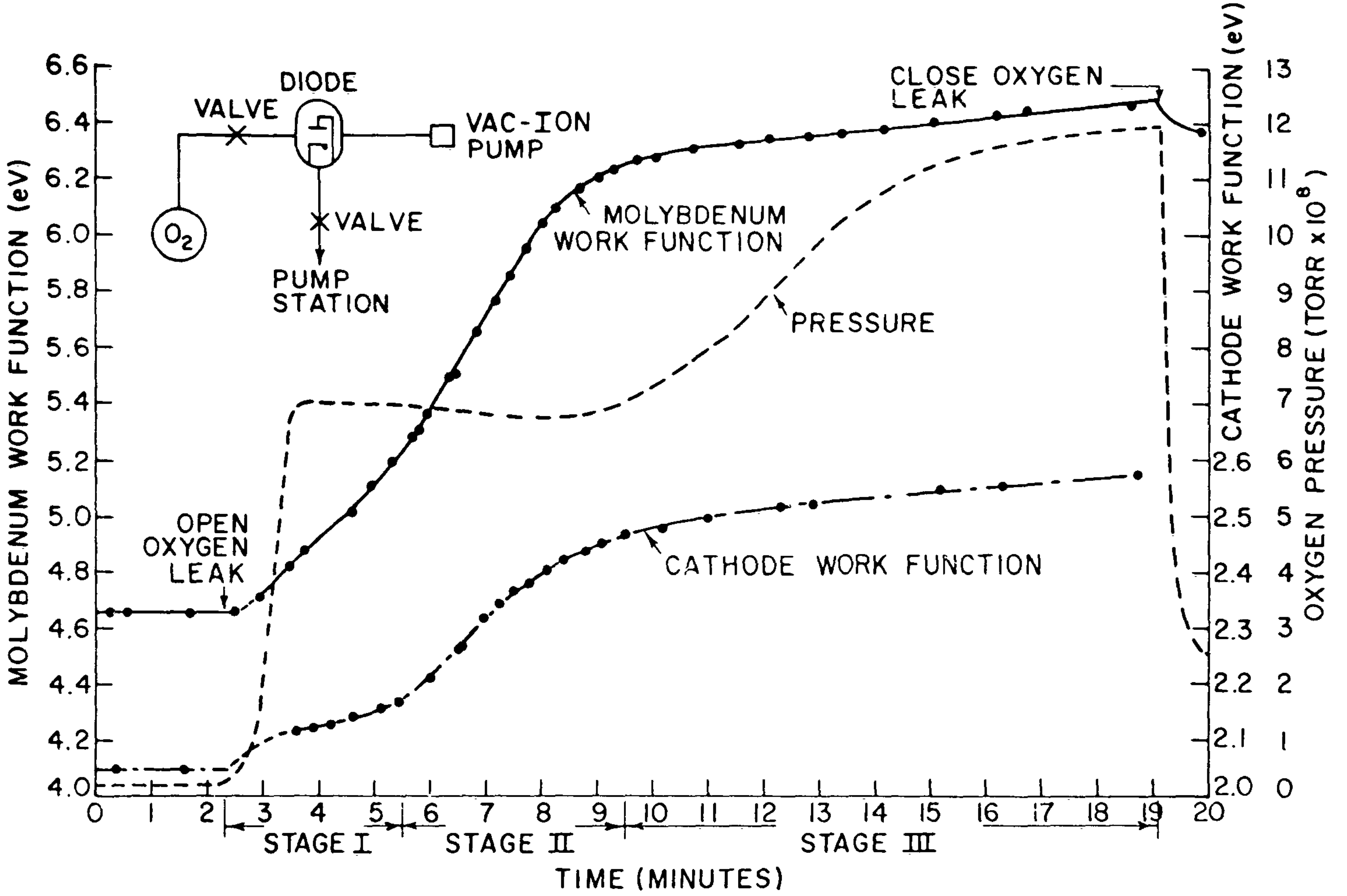

Fig. 7. Experimental setup and typical results on changes in molybdenum and cathode work functions and pressure, with time.

The initial rapid increase in work function tapers off at the end of the first stage of the adsorption process, then the rate again increases rapidly in the second stage (more clearly evident for the oxide cathode than the molybdenum anode). In the last stage of adsorption there is a very slow rate of rise in work function and a considerable increase in pressure, indicating a decrease in sticking coefficient and slowing down of the adsorption process.

2.3 DISCUSSION

The effect of oxygen on the work function of tungsten, reported by Becker,[3] using field-emission data, shows a similar time dependence to that observed for oxygen on molybdenum using the retarding-field technique. Owing to the unavailability of data on the sticking coefficient of oxygen on molybdenum, it is not possible to calculate the quantity of oxygen adsorbed during the various stages of the adsorption process. Nevertheless, considering the similarity in the chemical and electrical behavior of tungsten and molybdenum, an estimate of the oxygen adsorbed can be obtained by using Eisinger's data on the sticking of oxygen on tungsten (0.1 to 0.2).[4] The quantity of oxygen adsorbed on the molybdenum surface at the end of the first two stages is calculated to be $3\text{-}6 \times 10^{14}$ molecules/cm^2 and $9\text{-}18 \times 10^{14}$ molecules/cm^2, respectively. Assuming negligible adsorption of oxygen during the third stage, the latter value agrees reasonably well with the total oxygen adsorbed on tungsten, which was found to be 11×10^{14} molecules/cm^2 by Schlier[5] and 12.3×10^{14} molecules/cm^2 by Eisinger.[4]

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[3] Becker, Solid State Physics 7, 379 (1958).

[4] Eisinger, J. Chem. Phys. 30, 412 (1959).

[5] Schlier, J. Appl. Phys. 29, 1162 (1958).

The adsorption process is believed to occur in three stages: (1) during the first stage chemically adsorbed molecules dissociate, and the atoms are chemically bonded to the metal with an appreciable surface dipole moment; (2) at the end of the first stage the ability of the molecules to dissociate is reduced owing to a lack of available adjacent sites, so that during the second stage a layer of undissociated molecules, chemically bonded to the metal surface with a large dipole moment, results; and (3) during the last stage, the molecules are weakly bound with a negligible surface dipole moment. The results of Ehrlich,[6] indicating that oxygen is chemically bonded to tungsten in two states, is consistent with this hypothesis.

2.4 FUTURE WORK

To investigate the possibility of reactions with oxygen to form carbon monoxide, these studies will be continued in a system containing an omegatron for analysis of the gas ambient.

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[6] Ehrlich, J. Phys. Chem. Solids, 5, 47 (1958).

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2. Gases
3. Electron tubes
4. Electron tube parts

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II. Contract AF19(628)-331

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Scientific Report No. 3, Dec. 31, 1962, 17 pp. incl 7 figures.
Unclassified Report

An investigation of the interaction of nitrogen with a hot tungsten filament has shown that a chemical pumping effect occurs. The magnitude of this effect and its variation with temperature is discussed.

(over)

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1. Adsorption
2. Gases
3. Electron tubes
4. Electron tube parts

I. AFCRL Project No. 4619
Task No. 461901

II. Contract AF19(628)-331

III. General Telephone & Electronics Laboratories, Bayside, NY

UNCLASSIFIED

Preliminary results on the study of the effect of adsorbed oxygen on the work function of molybdenum at room temperature have indicated that there are three stages in the adsorption process: during the first two stages, there is a rapid increase in the work function, whereas in the last stage there is a small increase and very slow rate of rise in work function.

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IV. G. M. Bliven

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