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

C.M. Bliven
J.V. Florio

T.G. Polanyi
Scientist-in-Charge

GENERAL TELEPHONE & ELECTRONICS LABORATORIES INC.
Bayside, New York

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ABSTRACT

The pumping speed of an omegatron mass spectrometer was determined both in the presence and absence of the ionizing electron beam; when the electron beam is absent the pumping is attributed to chemical effects taking place at the hot tungsten filament. This chemical pumping speed is 7.15×10^{-6} liter/sec. The difference between the two pumping speeds (2×10^{-6} liter/sec) represents the contribution of electronic pumping to nitrogen removal. These experiments were performed on sealed-off omegatrons; however similar pumping phenomena are present in any system using hot tungsten filaments, and their relevance increases as the operating pressures in the experiments decrease.

Preliminary data were obtained on the interaction between oxygen and the carbon contained in the tungsten filament of the omegatron.

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1. INTRODUCTION

The gas adsorption characteristics of a solid are a measure of the force field at its surface; these characteristics are affected by crystallographic orientation, surface defects, pre-adsorbed gases and surface compound formation. For both solid-state and vacuum electron device applications, the understanding of surface phenomena is becoming progressively more important. The objectives of this investigation are to determine experimentally (1) the laws governing the adsorption of gases on clean surfaces in the very-low-pressure range, and (2) the effects of the adsorption of gases on the work function of surfaces.

Theoretical investigations of surface phenomena, e.g., physical adsorption, chemisorption, photoelectric and thermionic work functions, and reflection of electrons at surfaces are receiving new impetus owing to the progressive development of more refined quantum mechanical models of the surface. Experimental investigations of surfaces are very exacting; ultra-high vacuum techniques must be used and the surface condition of the samples must be determined. It is only very recently that advances in experimental techniques have reached a point where meaningful, i.e. reproducible, experiments on surfaces can be obtained.

In the field of physical adsorption and chemisorption of gases on surfaces, the majority of the experimental data has been obtained at fairly high pressures (about 10^{-5} Torr and above), and the conditions of the surfaces have not been well defined. In many cases, powders or amorphous materials were used. Measurements at very low-pressures were limited by uncertainties in the gas composition.

In these Laboratories high-vacuum instrumentation and techniques have been developed to permit dynamic and continuous measurements of the gas composition in high-vacuum ambients in the pressure

range 10^{-12} to 10^{-5} Torr. These techniques are based on the study of the omegatron mass spectrometer as a tool for measuring partial pressures of gases in vacuum systems. The operating characteristics of omegatrons have been investigated extensively, and the capability of this instrument to measure in detail the composition of the vacuum ambient in the range of 10^{-5} to 10^{-12} Torr has been demonstrated.[1]

Any pressure-measuring instrument, including the omegatron, will change the vacuum ambient; however, the omegatron will affect the vacuum ambient less than any ionization gauge owing to its inherently lower ionizing current requirements -- only 1 μa for full sensitivity. The importance of this characteristic cannot be overemphasized. This small ionizing current entails a small pumping speed, and the very low power required for the filament reduces outgassing and gas reaction phenomena. Additional important characteristics of the omegatron are simplicity of construction, the few materials used, and ease of high-temperature processing.

The ultra-high-vacuum system constructed for the surface adsorption measurements and the omegatron mass spectrometer has been described in Scientific Report No. 2. In the present phase of this investigation, the interaction of gases of interest with the omegatron is being determined to ensure that the measurements of adsorption are not affected by the measurement technique. In a parallel study the variation of the work function of molybdenum as a function of adsorbed gases is being determined.

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1. "Operational Data on the Omegatron as a Vacuum Analyzer," W. R. Watson, R. A. Wallace and J. M. Lech, Seventh Symposium on Vacuum Technology Transactions, 1960.

2. REMOVAL OF NITROGEN IN AN OMEGATRON

Initial experiments on the adsorption of nitrogen on tungsten at very low pressures were made using flash filament techniques in a continuous flow system. In the course of this work some anomalous results were obtained. These anomalies, which were discussed in the Third Scientific Report, consisted of a change of the equilibrium partial pressure of nitrogen for filament temperatures greater than approximately 1800°K. This effect is attributed to chemical pumping at the tungsten filament.

Chemical pumping can also occur at the tungsten filament of the omegatron used to measure the partial pressures of the ambient gases in the high-vacuum system. During this report period, therefore, an investigation was made of the interaction of gases with the omegatron to determine their effect on the partial pressure measurements. This information will also be of general interest in the field of gas surface reactions.

2.1 EXPERIMENTAL RESULTS

An omegatron, designated M-84, was processed and sealed off, and an analysis of its gaseous composition and the perturbation of the nitrogen present in the volume due to the operation of the omegatron was performed fourteen days later. The initial state of the gaseous ambient is shown in Table I, along with pertinent operating parameters. The partial pressure of nitrogen is perturbed by operation of the omegatron, as is evident from Fig. 1 where the data are presented on a semi-logarithmic plot. The linear decrease in pressure over the five-hour period can be characterized by assigning a constant speed for nitrogen removal of 1×10^{-5} liter/sec.

TABLE I

OMEGATRON CHARACTERISTICS

(Envelope Material Nonex 7720 Glass)

	M-84	M-85
Seal-off pressure (Torr)	5.2×10^{-9}	1.3×10^{-8}
Volume of omegatron and tubulation	0.073 liter	0.073 liter
Filament temperature corrected for tungsten emissivity	$1904^{\circ}K$	$1950^{\circ}K$

Gas Ambient During Operation	Partial Pressure (Torr)	
H_2	ND*	5×10^{-10}
He	3.2×10^{-7}	7.85×10^{-7}
CH_4	ND*	1.5×10^{-9}
H_2O	ND*	6.0×10^{-10}
N_2	1.14×10^{-7}	1.47×10^{-7}
A	2.0×10^{-9}	3.9×10^{-9}
Total Pressure	4.36×10^{-7}	9.4×10^{-7}

Operating Data for M-84 and M-85 Tubes:

V_{trap}	1.0 volts
V_{rf}	1.7 volts
V_f	1.2 volts
I_f	1.2 amperes
V_{acc}	70 volts
V_{coll}	22.5 volts
I_{beam}	10^{-6} amperes
Magnetic field strength	~ 5000 gauss

Filament area = 2.8×10^{-2} cm^2

* Not detected.

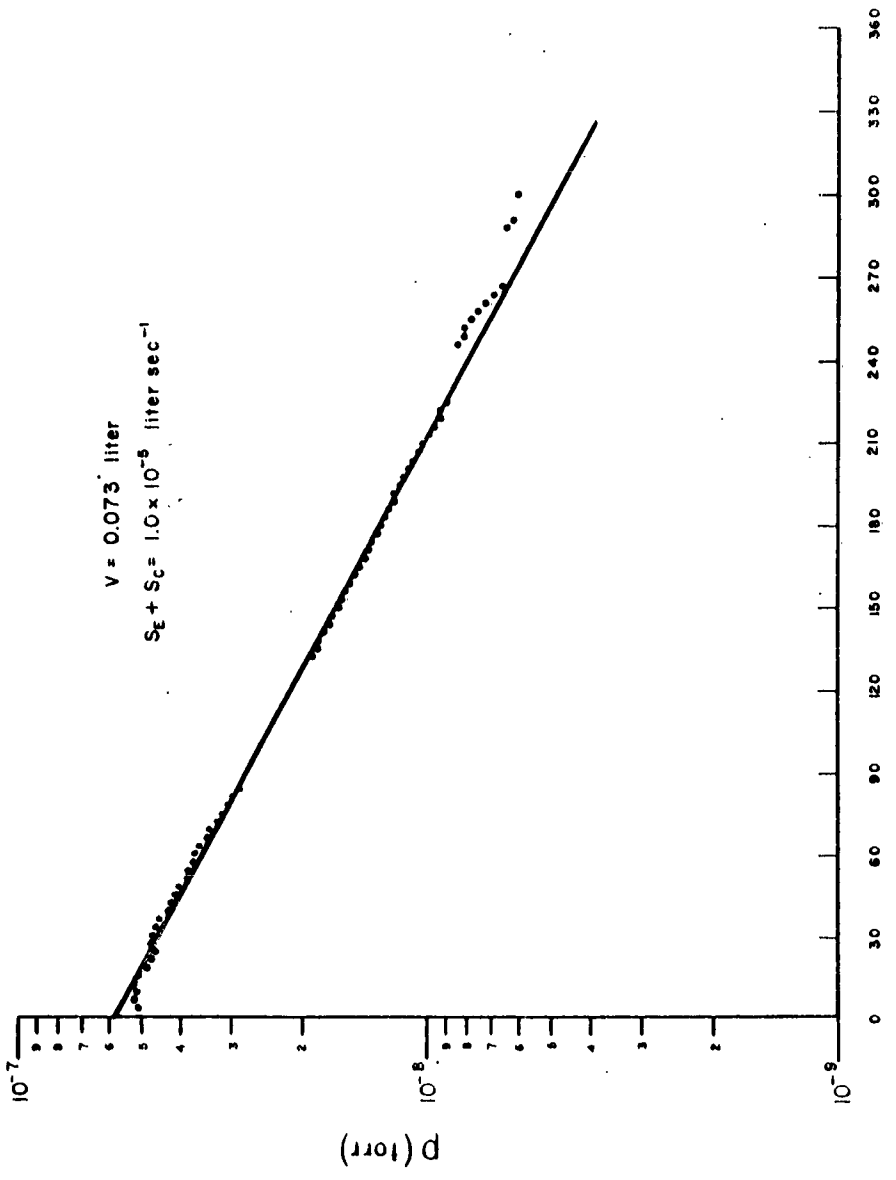

Fig. 1. Partial pressure of nitrogen in omegatron M-84 as a function of time.

After the data in Fig. 1 were obtained, the filament was maintained at normal operating temperature and all other operating voltages removed. Sixteen hours later the operating voltages were restored. At this time the partial pressure of nitrogen was less than 10^{-11} Torr. This is evidence for the assignment of a pumping effect due to the hot tungsten filament, although no quantitative value of this effect was determined.

A second omegatron, M-85, was processed and sealed off and an analysis similar to that performed on omegatron M-84 was started twenty days later. Table I again shows the initial state of the gaseous ambient and the pertinent operating parameters. The major difference between the two tubes is in the amount of helium which has permeated through the glass envelope.

Figure 2 shows the data on the partial pressure of nitrogen for omegatron M-85 under the various imposed experimental conditions. Region AB shows an exponential decrease over a three-hour period which can be characterized by assigning a constant pumping speed for nitrogen removal of 9.15×10^{-6} liter/sec. Region BC represents a time of sixteen hours during which the filament was at room temperature, and all voltages were removed from the omegatron. It is apparent that there is no re-emission of the nitrogen removed in region AB, and that the nitrogen present represents a new state of equilibrium with the surfaces. In region CD the partial pressure of nitrogen was monitored occasionally, and at D all omegatron voltages were removed except for the filament which was kept at its normal operating temperature. Data in region DE were obtained with the operating voltages applied long enough to obtain a pressure measurement. Since the times during which normal operating voltages were applied to the omegatron are small compared with the time that the filament was kept at the operating temperature, the cause of the total decrease of the partial pressure of nitrogen in region DE is assigned to

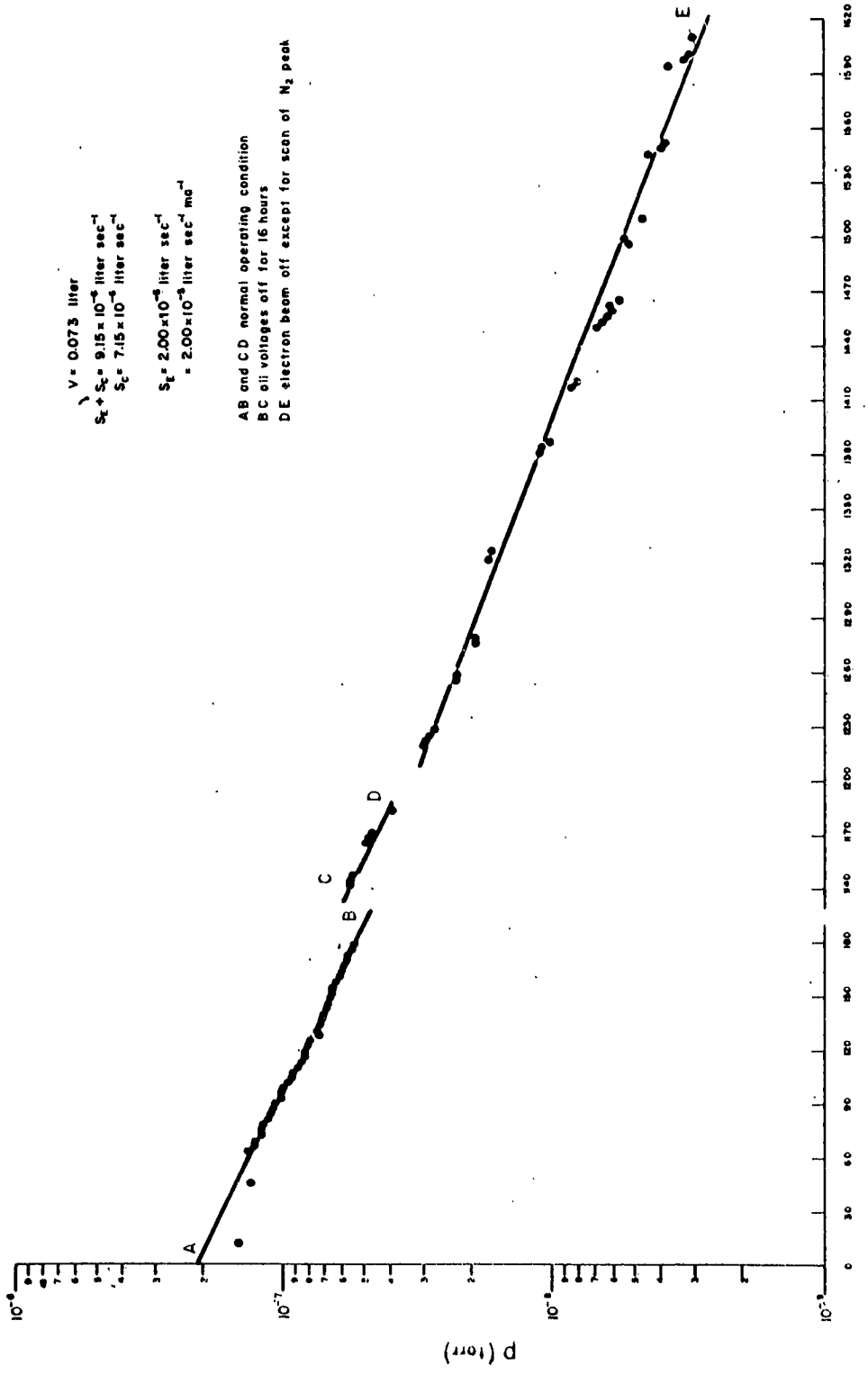

Fig. 2. Partial pressure of nitrogen in omegatron M-85 as a function of time.

the interaction of the hot tungsten filament with the nitrogen in the volume. A straight line has been drawn through the data in region DE, and this characterizes a constant pumping speed for nitrogen removal due to the hot filament of 7.15×10^{-6} liter/sec.

This is called the chemical pumping speed,[2] and that for region AB is, by inference, the sum of the chemical plus electronic pumpings speeds.

The electronic pumping speed for nitrogen in the omegatron, obtained by subtraction, is 2×10^{-6} liter/sec.

2.2 DISCUSSION

In a sealed-off volume such as an omegatron, after a time long enough to insure that the equilibrium state has been attained, the partial pressure of nitrogen is a measure of the excess of nitrogen over that required for complete surface coverage. In this state the rate at which nitrogen is adsorbed at all surfaces is equal to the rate at which it is desorbed from all surfaces.

It is reasonable to assume that for omegatrons M-84 and M-85, where the experiments were started fourteen and twenty days after seal-off respectively, the above-mentioned equilibrium state existed.

The pumping speed of the omegatron for nitrogen has been characterized as the sum of the electronic and the chemical pumping speeds. These pumping actions, under the conditions of this experiment, permanently remove the pumped nitrogen from the volume.

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

We assume the value of 10 per Torr for the gauge constant of the omegatron for nitrogen. That is

$$p = i^+/K\, i^- = i^+ \Big/ \left[\sigma s \left(\frac{T_o}{T} \right) \; i^- \right] \quad \text{Torr} = i^+/10\, i^- \,. \qquad (1)$$ *

The maximum electronic pumping speed S can be calculated on the basis that the rate at which positive ions are formed is equal to the rate at which molecules are removed permanently from the volume:

$$S = \sigma s\, \bar{V}_o\, i^- = K\, \bar{V}\, i^- \quad \text{liter/sec}, \qquad (2)$$

where

$$\bar{V} = kT/q = 1.94 \times 10^{-1} \; \frac{\text{Torr-liter}}{\text{coulomb}} \cdot \quad (T = 300^{\circ}\text{K}) \qquad (3)$$

Thus,

$$\frac{S}{i^-} = 1.94 \text{ liter/sec ampere.} \qquad (4)$$

The experimental electronic pumping speed obtained in these studies is in good agreement with this value.

The chemical pumping speed, S_c, for nitrogen can be accounted for on the assumption that a constant fraction, α, of the nitrogen molecules arriving at the hot filament are removed from the volume. In this case, the equation for mass balance becomes

$$V \frac{dn}{dt} = - n \frac{\bar{v}}{4} a_f \alpha = -n S_c, \qquad (5)$$

or

$$\frac{dn}{n} = - \frac{S_c}{V} \; dt \,. \qquad (6)$$

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* Symbols are defined in the Glossary, p. 11.

Integrating Eq. (6), we obtain

$$n = n_o e^{-(S_c/V)/t} . \qquad (7)$$

The measured value of the chemical pumping speed is, from the data of Fig. 2, 7.15×10^{-3} cm^3/sec. The value of α, at a filament temperature of 1950°K, is

$$= \frac{S_c}{\frac{\bar{v}}{4} a_f} = 2.14 \times 10^{-5} \qquad (8)$$

where

$$\frac{\bar{v}}{4} = 1.19 \times 10^4 \text{ cm/sec, for } N_2 \text{ at } 300^\circ\text{K}$$

and

$$a_f = 2.8 \times 10^{-2} \text{ cm}^2 .$$

Such a small interaction can easily be overlooked in measurements where higher ionizing current devices are used, and where total pressures, rather than partial pressures, are measured.*

This removal mechanism is particularly significant in studies of adsorption processes because it is similar in manifestation to the adsorption process itself. It is therefore essential to minimize or correct for the effect in adsorption measurements.

2.3 FUTURE WORK

The measurement of the adsorption characteristics of nitrogen on a tungsten filament will be continued; for these studies a new flash filament has been assembled in a one-liter flask with a sublimation shield.

* This effect, which has not been described in the literature, has nevertheless been used frequently to reduce the nitrogen pressure in sealed-off devices.

GLOSSARY OF SYMBOLS

n	Molecular number density (molecules/cm^3)
i^+	Ion current (amp)
K	Gauge constant $\left(\frac{\text{ions}}{\text{Torr}}\right)$ $K = \sigma s \frac{T_o}{T}$.
i^-	Electron current (amp)
σ	Differential ionization cross section (ions/cm Torr)
s	Effective electron path length (cm)
T_o	$0^oC = 273.16^o$ K
T	Temperature (^oK)
$\bar{V}_o$	Energy equivalent of temperature = $\frac{kT_o}{q}$, 1.765×10^{-1} $\frac{\text{Torr liter}}{\text{coulomb}}$
$\bar{V}$	Energy equivalent of temperature = $\frac{kT}{q}\left(\frac{\text{Torr liter}}{\text{coulomb}}\right)$ at temperature T^o Kelvin.
q	Electronic charge (coulombs)
S	Pumping speed (liter/sec)
$\bar{v}$	Arithmetical average velocity of gas (cm/sec)
N	Total number of gas molecules
V	Total volume of gas (liter) or (cm^3)
a_f	Filament area (cm^2)
α	Fraction of molecules arriving at filament which is lost.
k	Boltzmann's constant, $k = \frac{R_o}{N_A} = 1.0354 \times 10^{-22}$ $\frac{\text{Torr liter}}{^oK}$
R_o	Universal gas constant, $R_o = \frac{p_o V_o}{T_o} = 62.363$ $\frac{\text{Torr liter}}{^oK \text{ gm-mole}}$
p_o	Normal atmosphere, $p_o = 760$ Torr
V_o	Mole volume, $V_o = 22.4146$ liter

3. ADSORPTION OF OXYGEN ON MOLYBDENUM

A retarding-field technique is being used to determine the changes in work function of an initially clean molybdenum surface on which oxygen is being continuously adsorbed. This technique is based on measuring the displacement of the retarding-field characteristics of a planar diode. The anode work function φ_A can be obtained from the retarding-field current-voltage relation,

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

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 the anode surface. This voltage can be measured continuously during the adsorption process.

3.1 RESUMÉ OF EARLIER WORK

Previous results on the effect of adsorbed oxygen on the work function of a clean molybdenum surface were obtained in a system in which a 0.2 liter/sec VacIon pump was used for monitoring the gas pressure, rather than the usual hot-filament ionization gauge, in order to eliminate possible reactions between the gauge and the incoming oxygen. However, in measuring the molybdenum work function, it is necessary to operate an oxide cathode at a temperature capable of supplying enough electrons for the retarding-field method. Because of its low work function, and hence its ability to emit sufficient electrons at very low temperatures, the oxide cathode is most suitable as the electron

source. Nevertheless, there still remains the possibility of reactions between oxygen and the cathode and, even more likely, the alumina-coated tungsten heater used to heat the cathode. The major reaction is the formation of carbon monoxide between oxygen and the carbon impurities in the tungsten filament. Therefore, in order to eliminate any uncertainties regarding the interpretation of the previous results on the effects of oxygen, further studies were undertaken to analyze the gas ambient during the adsorption process, using omegatron mass spectrometer techniques.

3.2 EXPERIMENTAL RESULTS

The experimental set-up is similar to that described in the previous report (3rd Scientific Report) except for the introduction of an omegatron and a Bayard-Alpert gauge as shown in the insert in Fig. 3. During the experiments reported here, the Bayard-Alpert gauge was not operating.

With oxygen entering the system at a fixed leak rate, the following parameters were measured as a function of time: (1) the molybdenum work function (cathode at 915°K); (2) the total pressure, using the VacIon pump as a gauge; and (3) the partial pressures of the major gaseous species with the omegatron. Typical results are shown in Fig. 3. The major gas constituents are carbon monoxide and oxygen, with the former predominating.

Since the omegatron operates with a hot tungsten filament, its influence on the gas ambient composition was ascertained by repeating the previous experiment with the oxide cathode at room temperature. The gas analysis data, plotted in Fig. 4 show that the omegatron converts a considerable portion of the incoming oxygen to carbon monoxide.

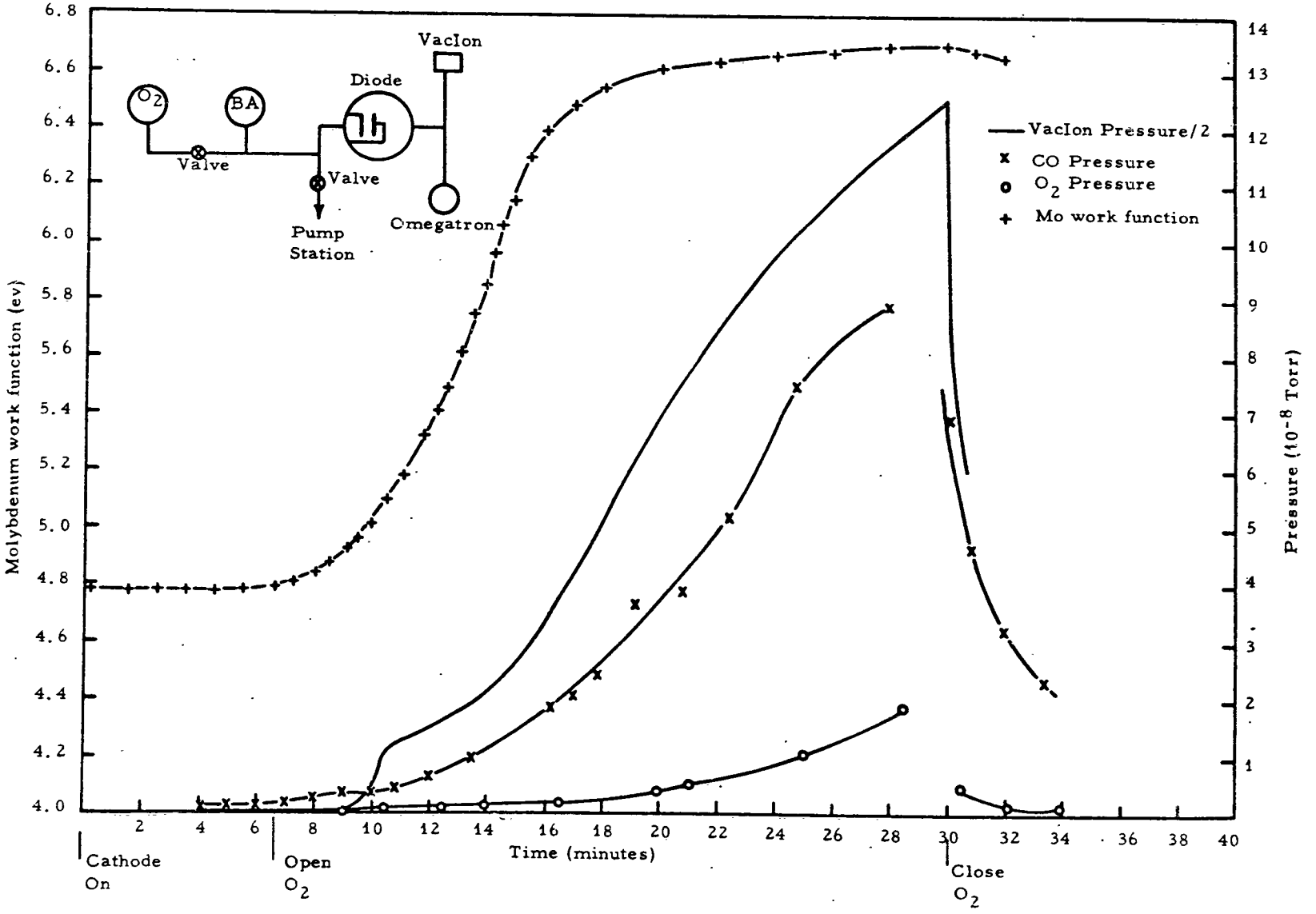

Fig. 3. Experimental set-up and typical results on changes in molybdenum work function and gas ambient during adsorption process.

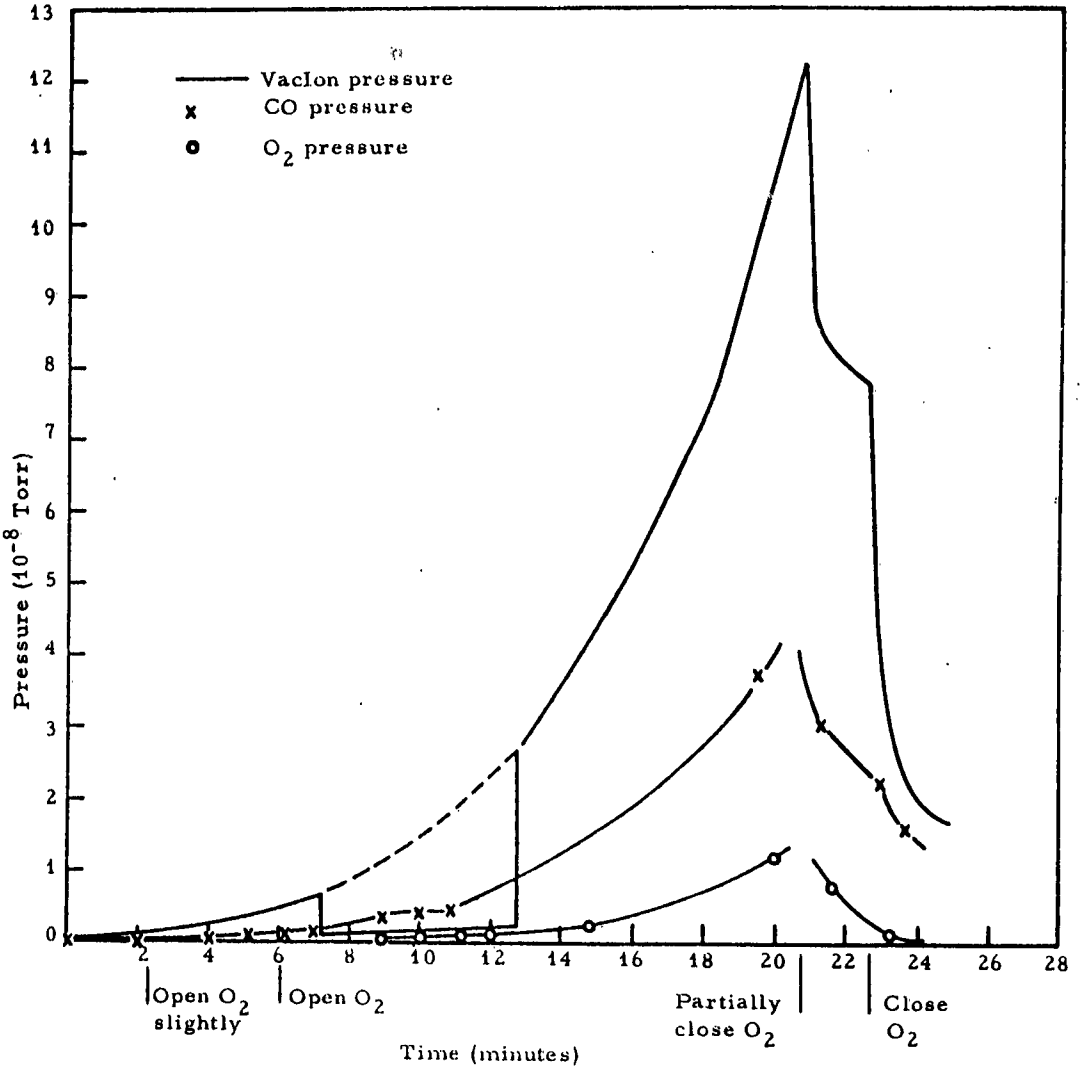

Fig. 4. Composition of gas ambient resulting from reaction between omegatron filament and incoming oxygen.

Further studies (see Fig. 5) with the oxide cathode operating intermittently indicate that it also produces carbon monoxide when the cathode is operating.

3.3 DISCUSSION

Before comparing present results with the earlier data, two differences in the experiment should be noted: (1) the inclusion in the system of the omegatron with its hot tungsten filament and (2) operation of the cathode at 915°K rather than 756°K as in the previous study. Furthermore, as a result of a defective coiled heater (only 1 turn out of six doing the actual heating), the temperature of that portion of the heater necessary to bring the cathode to 915°K is considerably higher than the heater temperature in the earlier studies.

With a fixed oxygen leak rate, the time dependence of the molybdenum work function and total pressure (using the VacIon pump as gauge) does not follow the same characteristic shape as previously observed (Fig. 7, 3rd Quarterly Report). In the present experiments there is no initial plateau in the pressure curve. This appears to be due to the conversion of oxygen into carbon monoxide. With oxygen entering the system the omegatron filament has to be periodically raised in temperature in order to maintain a constant omegatron electron beam current. This increase in temperature results in a rapidly rising carbon monoxide pressure. The omegatron filament acts as a pump for oxygen. The oxygen pressure within the diode portion of the system may be considerably higher than that measured by the omegatron . The sum of the partial pressures of the major gases (carbon monoxide and oxygen) is less, by a factor 2 to 3, than the total pressure indicated by the VacIon pump.

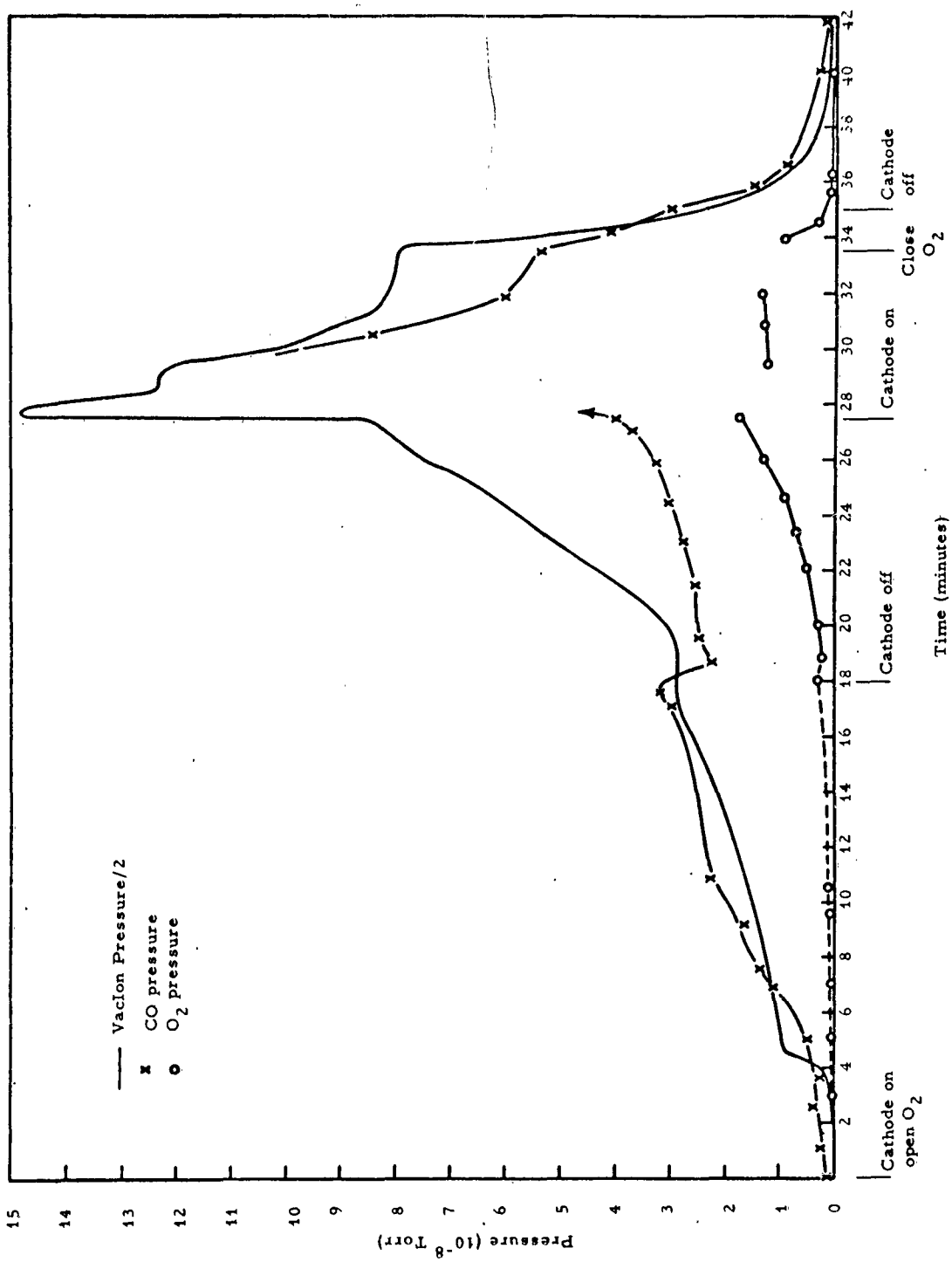

Fig. 5. Effect of intermittent cathode operation on gas ambient composition.

The gas ambient data with a non-operating cathode, shown in Fig. 4, indicate considerable chemical reaction between the omegatron filament and the incoming oxygen. In addition, the use of a 0.2 liter/sec VacIon pump as a pressure indicator at low pressures (below 2×10^{-8} Torr for the pump used in this study) may result in erroneous low pressure values. This is due to the fact that the pump cannot maintain a continuous glow discharge at low pressures. A dc meter in the VacIon pump circuit will measure the average current flow and hence result in an apparent pressure considerably below the true pressure. This was confirmed by placing an oscilloscope across the current measuring device. The total pressure versus time curve, in Fig. 4, shows this effect during the 7-13 minute time interval.

In Fig. 5, the effect of cathode temperature on the gas ambient is shown. When the cathode heater voltage is turned off, the pressure in the system drops momentarily as a result of adsorption on the cooling portions of the heater-cathode assembly. The ratio of oxygen to carbon monoxide increases during the time the cathode is cold. Upon bringing the cathode to operating temperature again, there is a very large burst of carbon monoxide and a reduction in the oxygen to carbon monoxide ratio.

These results show how the gas ambient can be altered by the various operating components of the vacuum system and indicate the need to take extreme precautions in interpreting gas pressure data where there is no mass spectrometric analysis of the gases. In addition, it is also necessary to determine and minimize the influence of the mass analyzing device upon the system.

3.4 FUTURE WORK

In order to utilize the omegatron for gas analysis of a system in which oxygen is being introduced, the carbon content of the omegatron filament must be reduced. Various methods to achieve this will be investigated. Subsequent studies should present conclusive evidence on the adsorption properties of oxygen on molybdenum.

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Preliminary data were obtained on the interaction between oxygen and the carbon contained in the tungsten filament of the omegatron.

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