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Effects of Electron-Surface Interaction in Ionization Gauges*

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Abstract

Studies have been carried out to compare the readings of various gauges in the presence of molecular gases, particularly O_2 and CO. Large anomalous effects originally reported by Ackley, Wheeler and Lothrop and by Redhead are interpreted in terms of ionization by electron impact at the grid surface. A comparison of such effects in various gauges is given. Other effects on pressure measurement due to electron bombardment of the grid are described.

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During the past year, several experimenters have reported an anomalous behavior of ionization gauges which is characterized by a departure from proportionality between the ion collector current and the electron current to the grid. Since this behavior may result in serious errors in pressure measurement, particularly when gauges are operated at low grid currents, the discovery and interpretation of the effect is of major significance in the field of ultrahigh vacuum. The effect was reported by Ackley, Lothrop and Wheeler[1] in a paper given at the last Vacuum Symposium. They demonstrated that the anomalous readings were related to the condition of the grid surface and that the effect could be greatly reduced either by electron bombardment of the grid or by operating the gauge at relatively high electron currents ($I_g > 3$ ma). Redhead[2] independently reported a similar effect and showed that it was unusually large after oxygen was introduced into the system. Other workers have reported related observations.[3,4]

Early conjectures as to the physical processes responsible for the anomalous effect have been replaced by a new explanation put forth independently by the present authors[5] and by P. A. Redhead.[6] In this picture the observed discrepancies are attributed to electron impact ionization of gases adsorbed or chemically bound at the grid surface of the gauge. The present paper describes experiments which indicate the nature and magnitude of the anomalous effects, gives evidence for the impact ionization hypothesis, and describes auxiliary effects of significance in pressure measurement.

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II. Experiments with the Redhead Modulator Gauge and the Schuemann Suppressor Gauge

In his original investigation[2] of the anomalous effect, Redhead used the modulator method.[7] After oxygen was introduced into the system, he found a very large increase in the so-called "residual current", i_r. Such a large increase in i_r could be interpreted either as an increase in the positive ion current to the collector or an increase in the "x-ray" or electron current from the collector. In our first attempts to reproduce these effects we utilized a modulator gauge of somewhat different design but similar principle to that of Redhead. As was true for all gauges used in our experiments, the grid material was molybdenum. With oxygen present and under conditions in which we expected to observe a substantial increase in i_r we actually found a decrease. In fact, a negative "residual current" was observed. That is, when the modulator electrode was made more negative in voltage, the current to the collector actually increased! Furthermore, it was observed that the sum of the currents to the collector and to the modulator increased when the potential of the modulator was made more negative.

These unexpected results with the modulator gauge have not been pursued in detail. They are reported here because they led to some interesting conclusions. First, the modulator method itself is not fully understood under such conditions and may give misleading results. Second, these results suggested that in the presence of oxygen, the collection efficiency of the Bayard-Alpert gauge might vary in an unexpected way as a function of the potential of the ion collector. Third, it seemed desirable to study the effect with a Schuemann suppressor gauge,[8] which unambiguously distinguishes between ions arriving at the collector and electrons leaving it.

The results with the Schuemann gauge are shown in Figure 1. Shortly after oxygen was introduced and maintained at approximately 10^{-8} Torr in a previously outgassed system, the suppressor gauge gave indications of anomalous readings comparable to those in a Bayard-Alpert gauge when it was operated at low emission currents. It was observed that both the ion and electron components of the current rose sharply, but that the ion component was at least 10 to 20 times higher than the electron component, and approximately 10 times higher than that given by a monitor gauge on the system. Since most of the electrons could be accounted for as due to secondary ejection[9] by the impinging ions, it was evident that the primary component in the enhanced or anomalous reading was due to ions arriving at the collector. These results immediately suggested the new interpretation of the anomalous effect based on the production of ions by electron impact at the surface of the grid.

III. Evidence for the Surface Impact Ionization Hypothesis

To gain further insight into the nature of the anomalous behavior, an experiment was carried out to measure the collection efficiency of a single collector Bayard-Alpert gauge as a function of collector voltage. For gases which do not exhibit the effect (noble gases, nitrogen, etc.), the ion current to the collector, I_c, is attributable to ions formed within the grid structure of the gauge. I_c is expected to be a slowly varying function of voltage applied to the collector, V_c, on the basis of the following argument. In view of the electrical potential well formed by the negative collector within the positively charged grid, there is a high probability that a given ion will ultimately strike the collector even if it misses the collector many times in the course of an oscillatory trajectory. Hence the curve of collector current versus the collector voltage normally saturates at a value of approximately 150 to 200 volts. I_c falls off at somewhat more negative values of collector voltage due to the increasing repulsion of the ionizing electrons and a corresponding diminution of the ionization volume.

Experimental data for nitrogen are shown in the dashed curves of Figure 2; the lower curve for I_g = 10 ma, the upper for I_g = 0.1 ma. The potentials of the filament and grid were maintained at their normal operating values of +45 v and +180 v. The collector potential was varied as shown to negative values. The abscissa gives the potential difference between collector and grid. It is seen that the experimental results verify the expected behavior.

In the presence of oxygen, a substantially different dependence of ion collector current on collector voltage was observed. As shown in the solid curves, the dependence of ion current on collector voltage differs markedly for different values of electron current: for I_g = 10 ma, the shape of the curve is similar to those for nitrogen, whereas for I_g = 0.1 ma there is a sharp increase of the collector current with collector voltage. Our interpretation of the above behavior is as follows. When oxygen is present, many ions are formed by electron impact[10] of the chemisorbed gas on the grid of the ionization gauge. Such ions are formed at the very edge of the potential well. If one of these ions misses the collector on its first trajectory through the gauge it has a very high probability of emerging on the opposite side and then being captured at the negatively charged envelope of the gauge. If the ions are ejected from the surface with an initial kinetic energy,[6] the probability of such escape is further enhanced. Thus the collection efficiency for ions formed at the grid is lower than that for ions formed in the volume at normal operating voltages. Under these circumstances the probability of capturing an ion is increased if the potential difference between collector and grid is increased, and I_c goes up rapidly with V_c.

If the Bayard-Alpert gauge structure is surrounded by a metallic cylinder at or near collector potential, the currents to the inner and outer collector can be measured and the above picture verified. Such a gauge has been constructed and the following results obtained. In the presence of nitrogen, the inner collector captured approximately 12 percent of the total number of ions produced; the remainder were collected at the outer cylinder. In the presence of oxygen a similar ratio was observed with the emission current to the grid held at 10 ma. When the grid current was reduced to 0.1 ma, the total number of ions per electron increased, but the fraction captured at the inner collector was reduced to approximately 2½ percent of the total current. Thus the anomalous reduction in collector efficiency is seen to be correlated with the enhanced rate of production of ions. Both effects are consistent with the picture of ion production at the grid surface.

Further support for the hypothesis of surface ionization by electron impact is provided by observation of the ions produced in a Davis and Vanderslice magnetic deflection mass spectrometer. In the presence of oxygen, a large double peak is observed with one component displaced from emerging on the opposite side and then being captured at the negatively charged envelope of the gauge. If the ions are ejected from the surface with an initial kinetic energy,[6] the probability of such escape is further enhanced. Thus the collection efficiency for ions formed at the grid is lower than that for ions formed in the volume at normal operating voltages. Under these circumstances the probability of capturing an ion is increased if the potential difference between collector and grid is increased, and I_c goes up rapidly with V_c.

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Further support for the hypothesis of surface ionization by electron impact is provided by observation of the ions produced in a Davis and Vanderslice magnetic deflection mass spectrometer. In the presence of oxygen, a large double peak is observed with one component displaced from the expected mass 16 position. The magnitude and separation of the peaks indicate a high probability of the dissociative ionization of O^+ at the surface of the ionization enclosure.[11,12]

IV. Comparison of Ionization Gauge Readings in the Presence of O_2 and CO

Although the above results together with those of other experimenters give good evidence for the interpretation of the effect and a qualitative estimate of the possible errors in gauge readings, we undertook a systematic series of runs under controlled conditions to verify the magnitude of the effect and its dependence on the grid current of the gauge.

The system used was a standard glass oil-pumped, zeolite-trapped system with provision for introducing various gases. Three gauges were utilized and compared: (1) a 5966 Bayard-Alpert gauge, (2) a photocurrent suppressor gauge, and (3) a Philips omegatron.[13] The conductance between the gauges was approximately 1.5 l/s. The omegatron was used as a monitor of the pressure; it exhibited no anomalous effects and gave a reliable indication of the partial pressures of the gases in the system.

The results of a series of runs is shown in Figure 3. After a bakeout of the system and outgassing of the gauge (background gas, mainly CO at 7×10^{-11} Torr) oxygen was introduced into the system at time $t = 0$. The oxygen was continuously flowed through the system for 35 minutes and maintained at a pressure of 10^{-7} Torr as read on the omegatron. Then the O_2 valve was closed. In the case shown, one of the two ionization gauges, in this case the suppressor gauge (SIG), was maintained at 10 ma while the other, the Bayard-Alpert gauge (BAG) was run at an emission current of 0.1 ma. As will be shown in the next series of runs, interchange of the currents in the two gauges showed that they behaved in a similar manner; hence the important parameter to be observed here is the electron emission current. The following significant features were noted:

(1) Although pure O_2 was being introduced into the system, there was a very large rate of production of CO in the presence of oxygen. During the initial period both ion gauges were in error, apparently due to the large pumping speed of the gauges for O_2 and CO, as well as to the surface effect. The pumping speeds for these gases was evidently large compared to the conductance between the gauges.

(2) When the O_2 valve was closed, the oxygen peak as read on the omegatron fell to less than 10^{-10} Torr in a few seconds. The predominant residual gas remaining thereafter was CO, which was gradually removed but at a rate far slower than the characteristic pumping time for the system.

(3) In the period following the removal of O_2 the gauge operating at 10 ma approached the omegatron reading within a few minutes and read pressure correctly thereafter.

(4) In the same period, the gauge operating at 0.1 ma departed significantly from the "true" pressure as read on the omegatron and the SIG. The maximum error observed was at least an order of magnitude on the high side.

Figure 4 shows the results of a similar series of runs for five different values of emission current. In these runs the BAG was operated at 10 ma and the SIG at values of 10, 3, 1, 0.1 and 0.01 ma. To simplify the drawing, only the curves for I_g = 0.1 ma are shown for the initial interval during which oxygen was present.

From these curves the following observations can be made:

(1) There is a monotonic increase in the size of the anomalous effect with decreasing electron grid current. For $I_g = 10$ ma, the error in pressure reading is small and disappears within a few minutes; for $I_g \leq 0.1$ ma, the error is large.

(2) The size of the effect decreases with time for all values of I_g although the change is not perceptible for the smallest value of I_g. The time constant for the decay of the effect varies approximately inversely with the electron current.

Figure 5 shows results for CO similar to those above for oxygen. Note that the effect is significantly smaller. For $I_g = 0.1$ ma the maximum error in the reading of the gauge is less than a factor of four. This result is rather surprising in view of the large cross-section for ionization reported by Moore, and further study of CO is called for. Another feature of these curves is the large departure of the readings of both gauges from the omegatron readings in the early part of the runs. This is attributable to a large pumping speed of each gauge for CO and explains in part the similar curves in Figure 3.

While significant qualitative conclusions from these results can be made, a quantitative analysis of the dependence of the time constants awaits further experimentation. Such an analysis is difficult in the case of oxygen because within a short period of time a very large fraction of this gas was converted into CO and the effects of the two gases were not readily separated. This result is in itself worthy of careful note. Further observations which are not reported here showed that while the hot tungsten filament is responsible for a sizeable fraction of the conversion of oxygen to carbon monoxide, other physico-chemical processes may be even more significant. A preliminary experiment demonstrated that oxygen is also converted into CO in a process which involves the electron bombardment of adsorbed gas at the surface of the grid. This could result from the chemical recombination with carbon of atomic oxygen released at the grid.

V. Mechanism of Surface Ionization and Gas Removal

A detailed quantitative picture for the electron impact ionization process is not easy to arrive at since it must vary with the composition of the gas and with the grid surface of the gauge. The observation of a large O^+ peak in the presence of oxygen is suggestive of a process of dissociative ionization of the adsorbed gases as studied by Moore for CO in molybdenum. In fact, Moore observed a large O^+ peak in the presence of O_2. He measured a cross-section for the process as high as 10^{-4} ions per incident electron for oxygen-covered molybdenum. Redhead[6] has measured a maximum cross-section of about 10^{-5} ions/electrons for O_2 on molybdenum and has proposed that in addition to the release of O^+ ions a large quantity of neutral oxygen atoms is also desorbed by electron bombardment. In typical vacuum systems, however, molecular oxygen is one of the least likely components of the residual gas. It is therefore likely that other adsorbed or chemically bound gases play a contributing role. For example, the metallic oxides formed on the surface of the grid may also be dissociated and ionized by electron bombardment to give a major effect. Young[14] measured maximum ionization efficiencies of 10^{-5} ions/electrons (also O^+) for 90 eV electrons impinging on oxidized surfaces of copper, nickel, molybdenum and tungsten. This process may therefore be a significant factor in the high readings observed in an ionization gauge immediately following bakeout of the vacuum system but before outgassing the gauge.

As was indicated in Figures 3-5, the anomalous production of ions at the surface is decreased in the process of measuring it, due to the electron impact removal of the adsorbed or chemically bound gases. The effect may be more rapidly reduced to a negligible value by thoroughly outgassing the grid by electron bombardment. Thereafter, the onset of erroneous readings can be prevented by operating the gauge at sufficiently high electron currents, the minimum current depending on the amount and composition of the background gas in the system. For typical ultrahigh vacuum conditions at 10^{-9} Torr or lower, no anomalous effects have been reported when gauges are operated at values of I_g of 5 ma or greater.

VI. Summary and Conclusions

1. A systematic study of an anomalous non-linearity in ionization gauges has been made, indicating the magnitude of possible errors in ionization gauge readings. The errors are particularly enhanced after oxygen has been introduced into the system.

2. Evidence has been provided for the interpretation of the effect as due to dissociative ionization by electron impact at the grid surface. At low values of grid current, there is an enhanced rate of production of such ions accompanied by a reduced rate of collection in a Bayard-Alpert gauge.

3. The anomalous readings in ionization gauges can be greatly reduced by thorough outgassing of the grid by electron bombardment. Thereafter,

operation at electron currents of 5 ma or greater gives reliable readings.

4. In the presence of O_2 a number of other effects may be present to give erroneous ionization gauge readings; in particular, the composition of the gas may be altered, both by chemical reactions at the hot filament and by electron bombardment of adsorbed gases.

5. Studies of these effects have brought valuable insight into very interesting physical and chemical processes which take place not only in ionization gauges and mass spectrometers, but wherever electrons are incident on solid surfaces.

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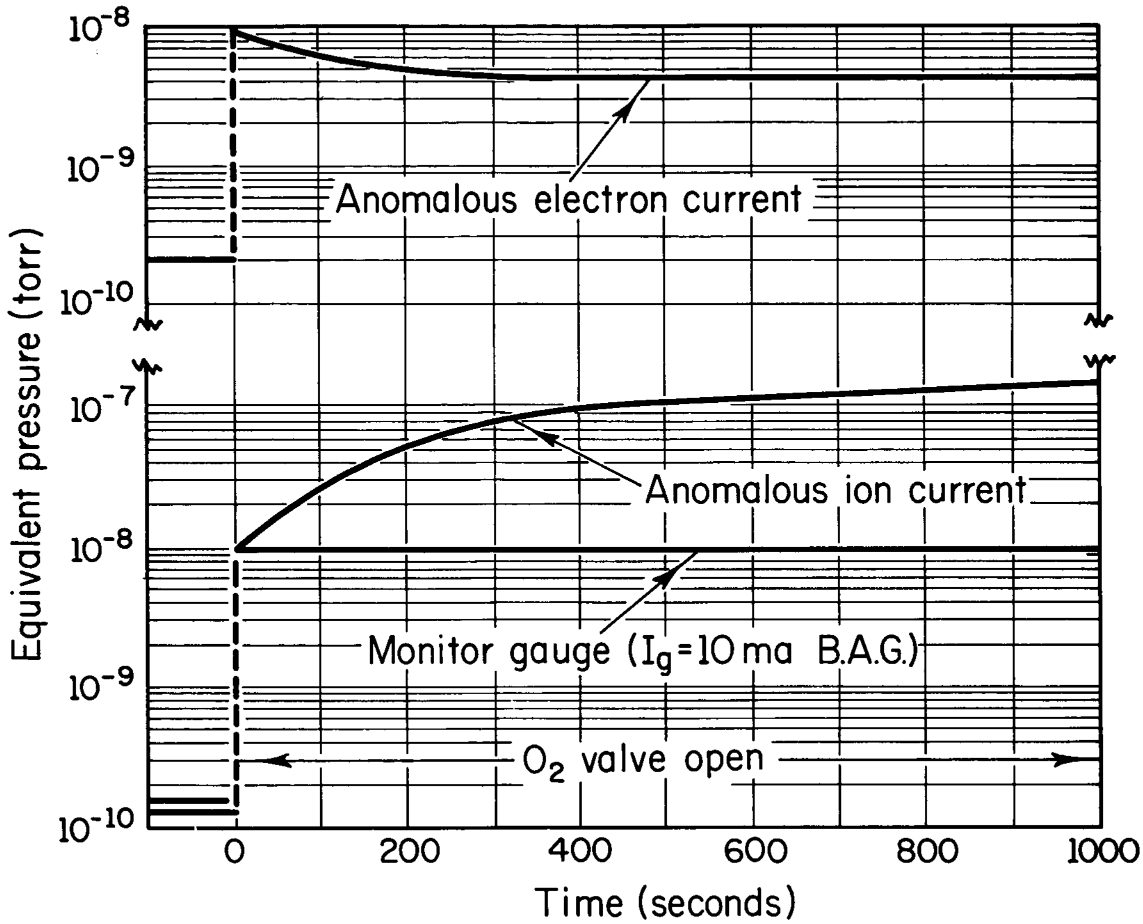
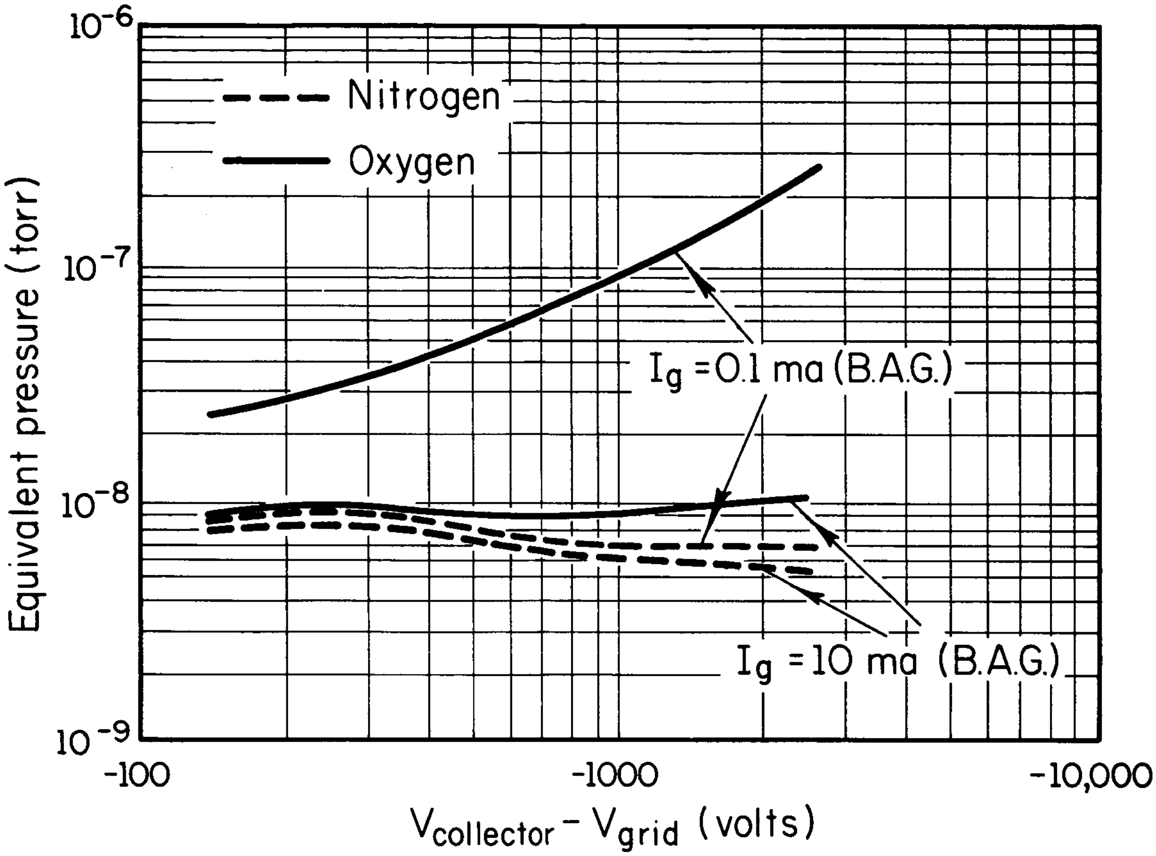
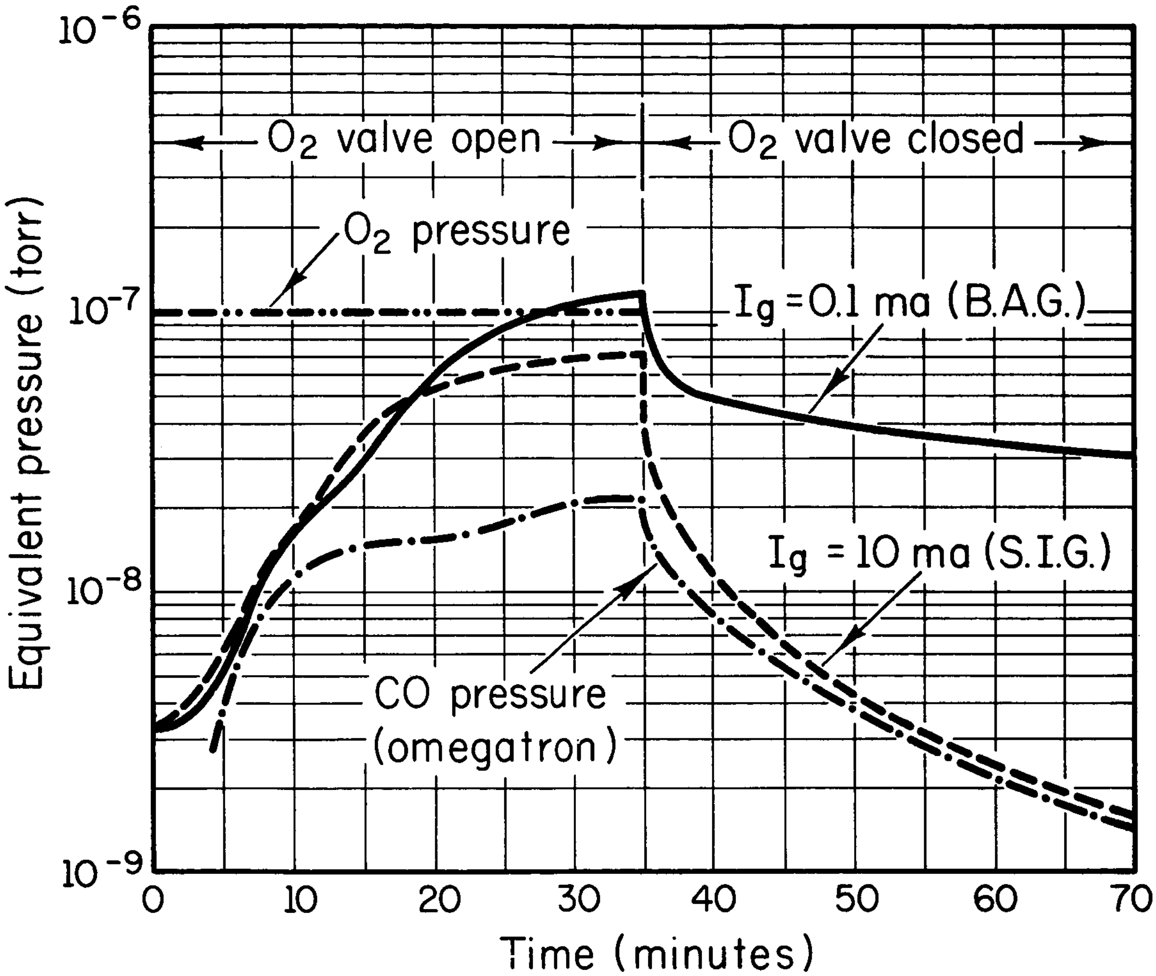


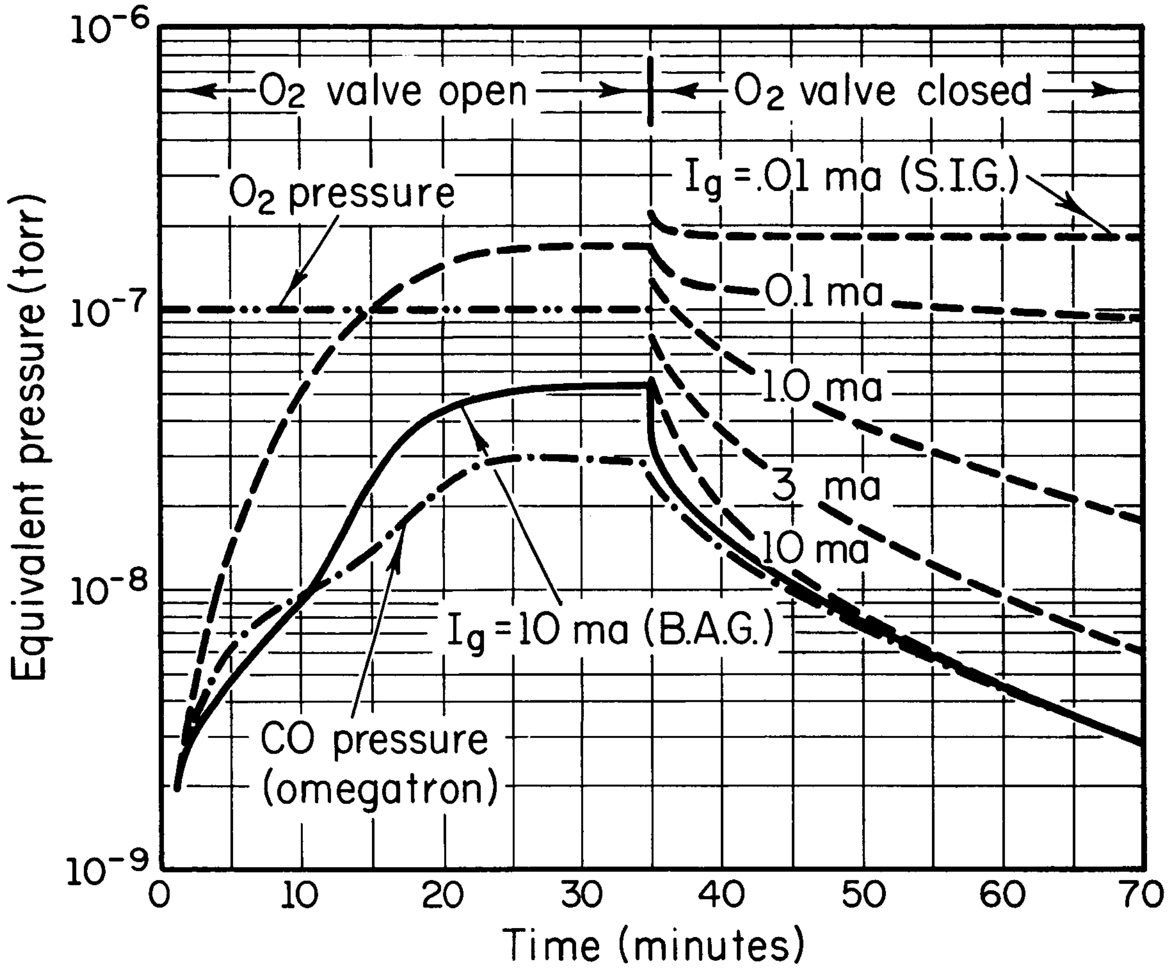
10^{-8}
10^{-9}
10^{-10}
10^{-7}
10^{-8}
10^{-9}
10^{-10}
Equivalent pressure (torr)
Anomalous electron current
Anomalous ion current
Monitor gauge (I_g=10 ma B.A.G.)
O_2 valve open
0
200
400
600
800
1000
Time (seconds)



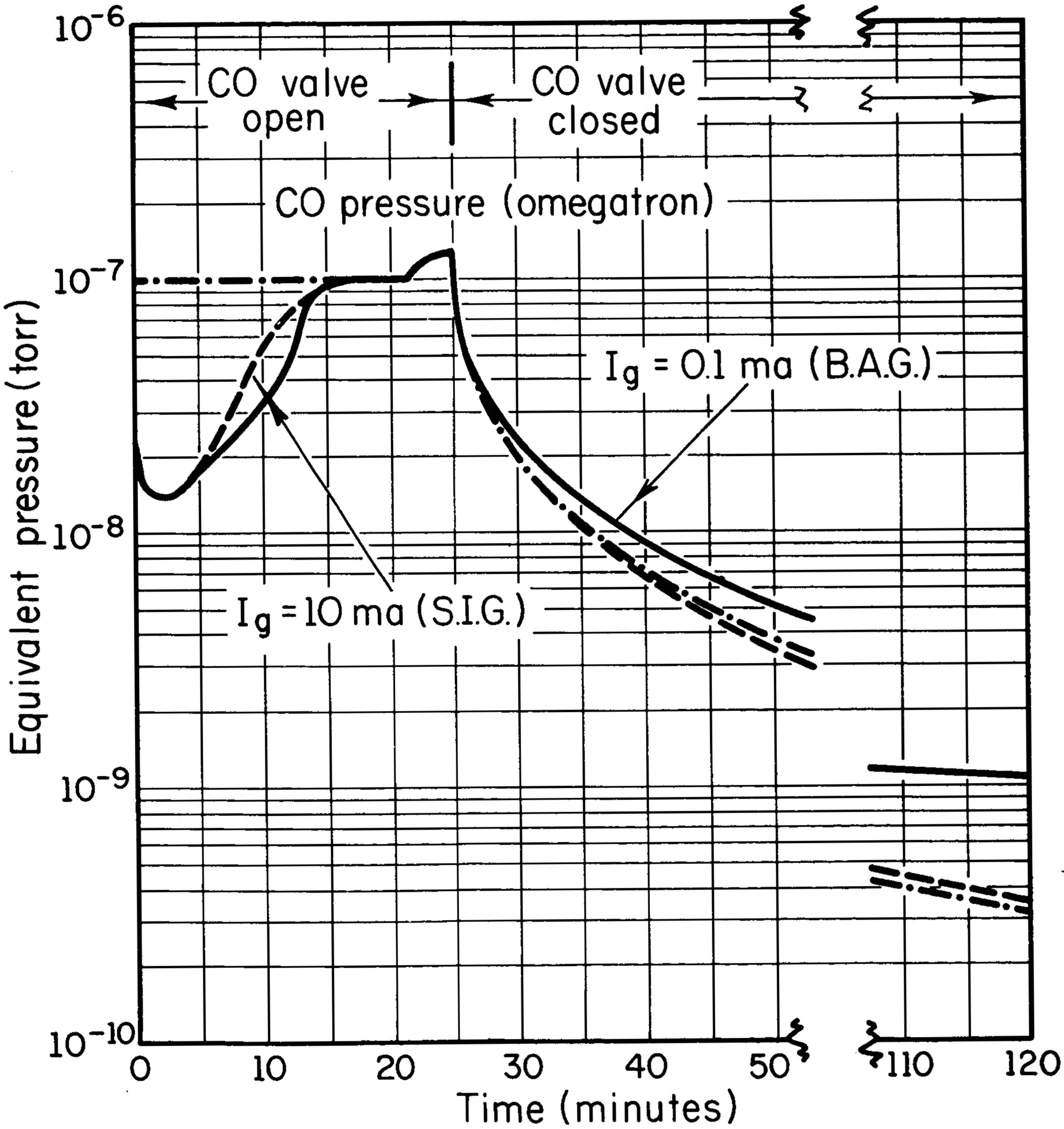
10^-6
10^-7
10^-8
10^-9
Nitrogen
Oxygen
Equivalent pressure (torr)
Ig =0.1 ma (B.A.G.)
Ig =10 ma (B.A.G.)
-100
-1000
-10,000
Vcollector - Vgrid (volts)



O_2 valve open
O_2 valve closed
O_2 pressure
I_g = 0.1 ma (B.A.G.)
I_g = 10 ma (S.I.G.)
CO pressure (omegatron)
Equivalent pressure (torr)
Time (minutes)
10^{-6}
10^{-7}
10^{-8}
10^{-9}
0
10
20
30
40
50
60
70



O2 valve open
O2 valve closed
Ig = .01 ma (S.I.G.)
O2 pressure
0.1 ma
1.0 ma
3 ma
10 ma
Ig = 10 ma (B.A.G.)
CO pressure
(omegatron)
10-6
10-7
10-8
10-9
Equivalent pressure (torr)
0
10
20
30
40
50
60
70
Time (minutes)



CO valve open
CO valve closed
CO pressure (omegatron)
I_g = 0.1 ma (B.A.G.)
I_g = 10 ma (S.I.G.)
Equivalent pressure (torr)
Time (minutes)
10^{-6}
10^{-7}
10^{-8}
10^{-9}
10^{-10}
0
10
20
30
40
50
110
120