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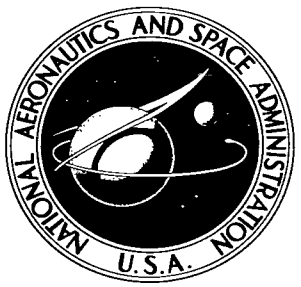

A SMALL VOLUME, ULTRAHIGH VACUUM SYSTEM FOR IONIZATION GAGE STUDIES

by R. A. Outlaw and James M. Bradford, Jr.

Langley Research Center
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SUMMARY

A small ultrahigh vacuum system has been developed for use in the research and development of ionization gages capable of ultrahigh vacuum measurements. The system operates at estimated pressures of less than 5×10^{-13} torr (66.5 pN/m^2). These pressures are obtained by a combination of gage and cryogenic pumping and a rigorous baking procedure. The system was specifically designed for study of magnetron ionization gage characteristics, but can be used additionally to study any low outgassing device small enough to fit in the system.

A presentation is made of the details of the system and of the techniques used for obtaining the low pressure. A discussion of the pressure measurement techniques is given and calculations are shown which estimate the ultimate pressure obtained by the system.

INTRODUCTION

The study of ionization gages for use on current ultrahigh vacuum facilities requires a vacuum system with a working pressure range below 10^{-10} torr (13.3 nN/m^2). Other investigators have developed systems which are of glass construction with the gages under study being fused onto the test manifold (refs. 1 to 4). Even though these systems are suitable for many studies, glass blowing in the initial construction and for each change of configuration and also the possibility of changing gage characteristics during mounting and demounting procedures, are definite disadvantages.

This report presents a system which was developed to overcome these disadvantages. The approach was to use a small metal manifold and to attach the gages under study by using demountable, commercially available, metal ultrahigh vacuum flanges. The test gages can be readily interchanged simply by bolting or unbolting the mounting flange. To achieve ultrahigh vacuum, the system utilizes the pumping of the test gages in conjunction with the cryogenic pumping of a pyrex finger immersed in liquid helium. The system design and a description of the techniques used to achieve ultrahigh vacuum are presented.

SYMBOLS

Values given in parentheses are in the International System of Units (SI).

A	cross-sectional area, square centimeters
A_s	surface area, square centimeters
C	conductance (subscripts denote sections; see fig. 10), liters/second (cubic meters/second)
d	thickness of material, millimeters
I_1	collector current with the modulator at grid potential, amperes
I_2	collector current with the modulator at ground potential, amperes
i	positive ion current, amperes
i_r	residual current, amperes
K	permeability or permeation velocity, torr liters per second for 1 millimeter thick per square centimeter area per centimeter mercury (gas pressure difference)
M	molecular weight, atomic mass units
p	pressure, torr (newtons/square meter)
Q	mass flow rate of gas, torr liters/second
S	pumping speed, liters/second (cubic meters/second)
T	temperature, degrees Kelvin
α	modulation factor
γ	Clausing's factor

DESCRIPTION OF SYSTEM

The system block diagram is shown in figure 1. It is essentially a manifold (with the gages attached) evacuated through a valve by an ion pump and a mechanical roughing pump. Figure 2 shows the manifold with its attachments and the manifold valve. The manifold itself is made of two stainless-steel 90° elbows 2 inches (5.1 cm) in diameter that are welded together. Seven 1.5 inch (3.8 cm) stainless-steel flanges using copper gaskets are welded to the manifold and connect the gages, the liquid helium-cooled pyrex finger, the bleed valve, and the manifold valve to the manifold. The manifold with its attachments is hereinafter referred to as the UHV system.

Three of the gages located on the manifold are Redhead cold-cathode magnetron gages (hereinafter called magnetron gages) and the other is a modulated Nottingham gage (hereinafter, modulated gage). The manifold valve is a 1.5 inch (3.8 cm) bakable valve and is located between the manifold and a 50 liter/sec (0.05 m^3/sec) ion pump.

The UHV system is baked in a box-shaped oven which is raised and lowered over the UHV system by a manual lift apparatus. The oven, which can attain temperatures up to 450° C, encloses the UHV system during the baking procedure and rests on an asbestos-covered table. Temperatures of the various components of the UHV system are measured and recorded during the baking procedure with thermocouples and the appropriate instrumentation.

The magnetron gages on the UHV system are the commercially available cold-cathode vacuum gages (ref. 5) which for this system are operated at a voltage of 4800 volts and a magnetic field of 1050 gauss (0.105 tesla). The modulated Nottingham gage is described in reference 6 and is employed on the UHV system as a secondary standard for comparison with the pressure indications from the magnetron gages. The ion currents from the modulated gage and one of the magnetron gages are measured by an electrometer capable of sensing currents as low as 2×10^{-15} ampere although the lowest currents encountered in these tests were about 2×10^{-13} ampere. The other two magnetron gages were monitored by the electrometers in their respective commercial controllers (fig. 3). All of the gages were constructed of 7740 pyrex glass. The elements of the modulated and the magnetron gages are shown in figure 4.

A photograph of the entire vacuum system is shown in figure 5. The electron bombardment control shown in figure 5 was used in the cleaning procedure for the modulated gage. A schematic of this control is shown in figure 6.

The liquid helium which cools the pyrex finger to 4.2° K is transferred into a small container constructed from two liquid nitrogen glass dewars and expanded polystyrene (fig. 7). The liquid helium transfer into the small dewar is accomplished by pressurizing

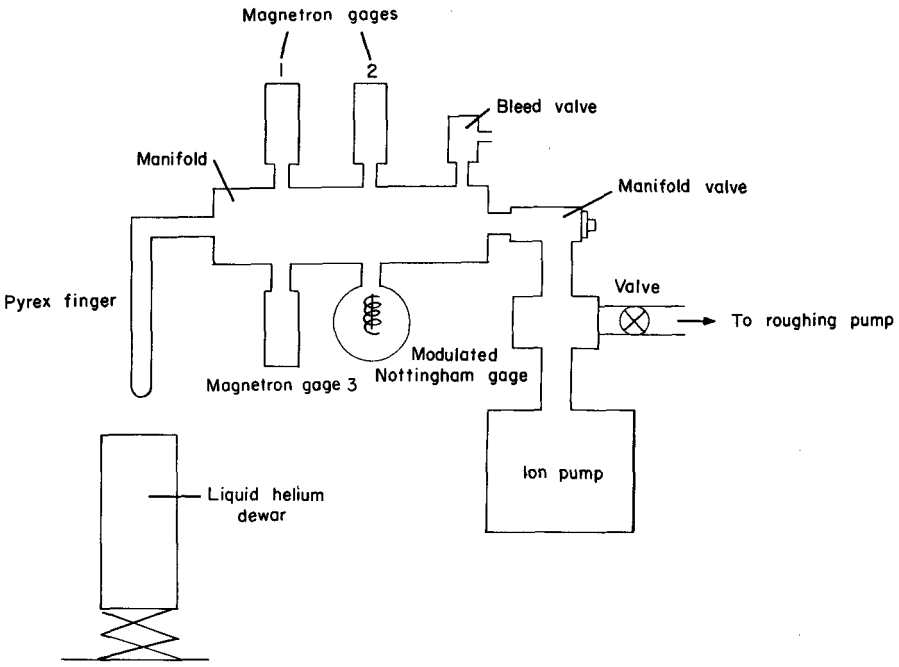


Figure 1.- System block diagram.

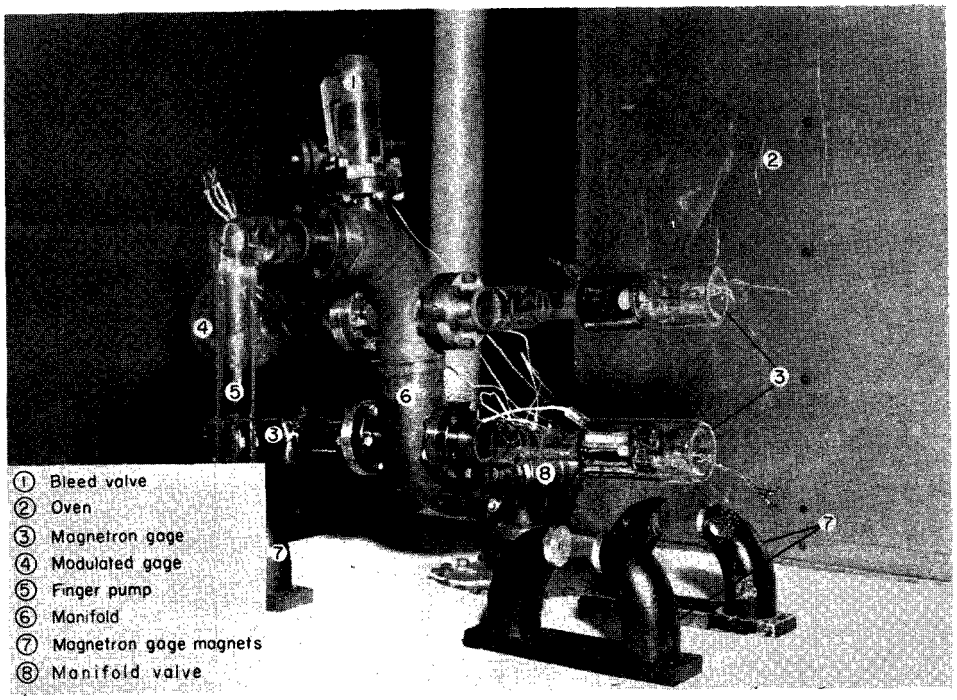


Figure 2.- UHV system. L-65-3567.1

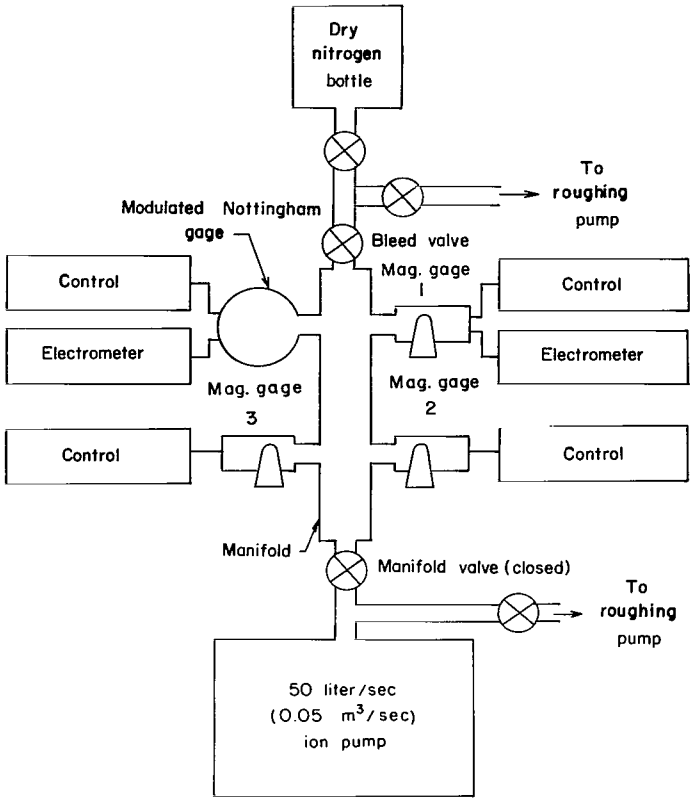


Figure 3.- Block diagram of gage comparison assembly.

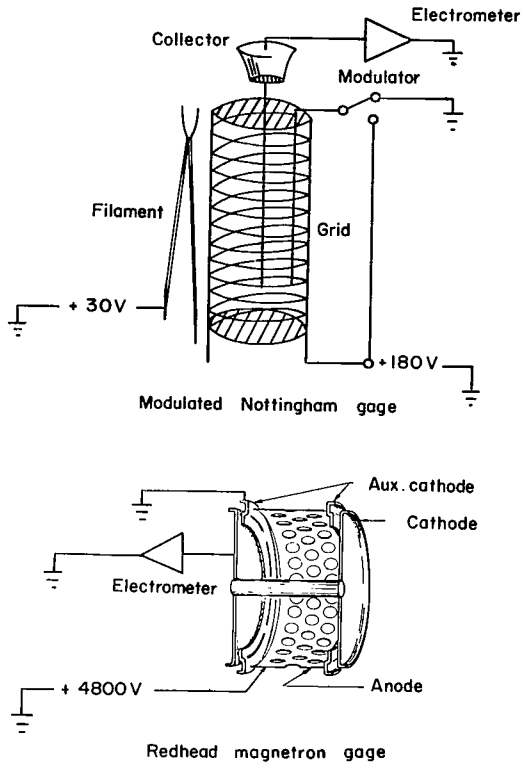


Figure 4.- Elements of gage types employed.

a 100 liter (0.1 m^3) commercially made cryogenic container which forces the liquid helium through a vacuum-jacketed transfer line into the dewar.

PROCEDURE

In general, to produce low pressures the system must have a high pumping speed or a low gas load or both. This system produces low pressures by combining the low pumping speeds obtained from a pyrex finger immersed in liquid helium and the low pumping speeds of the magnetron gages with the low outgassing rate of the system. The low outgassing rate was obtained by a stringent baking procedure.

Initially, the UHV system was pumped to about 10^{-7} torr (N_2) by the roughing pump and the ion pump. The UHV system was then baked at 410° C for 100 hours. The oven was then removed, the controls were connected to the gages, and the gages were turned on. The modulated gage was then cleaned by electron bombardment with 800 volts between the filament and grid and with 300 milliampere emission current. The modulated gage was bombarded for 45 minutes every 2 hours for about 8 hours. After bombardment, the

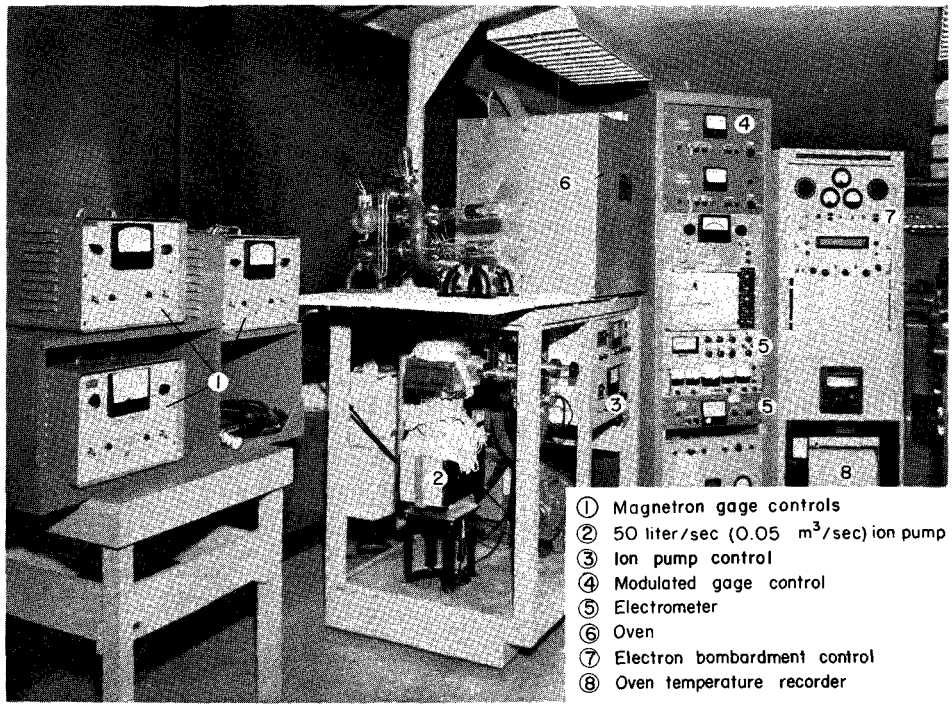


Figure 5.- Integral system. L-65-3568.2

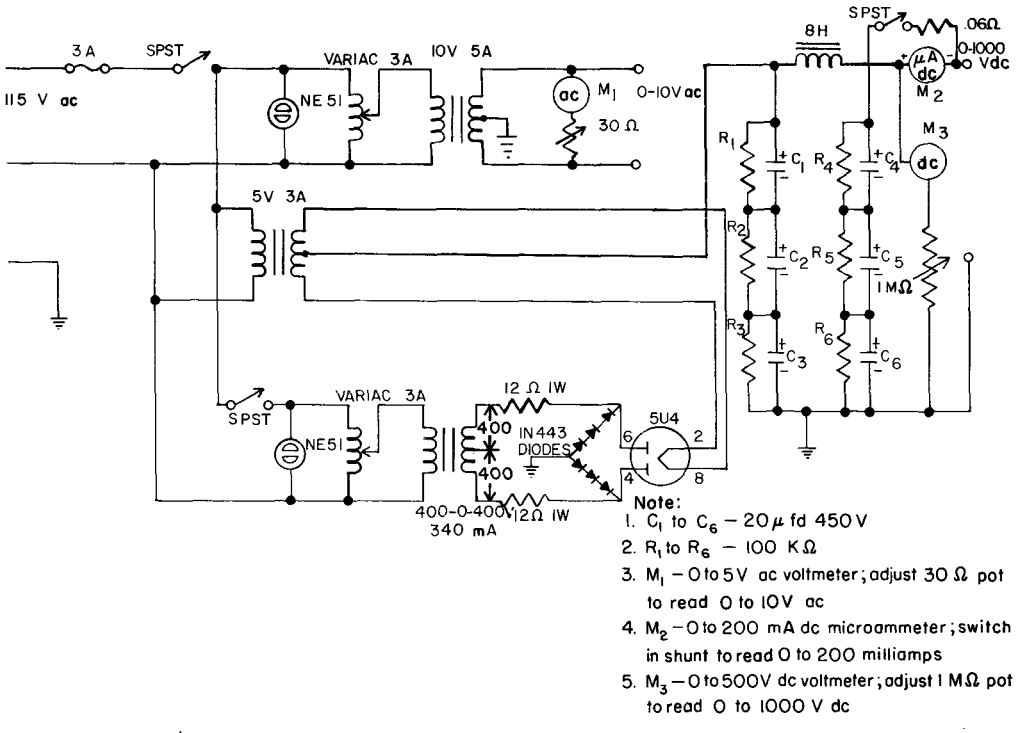


Figure 6.- Schematic of electron bombardment power supply.

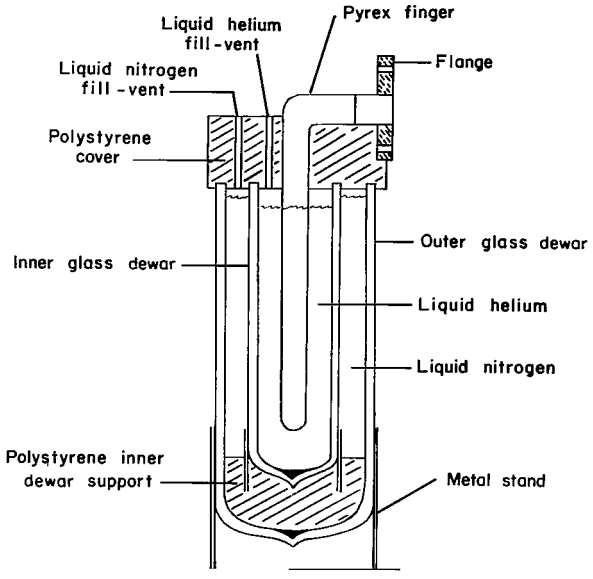


Figure 7.- Cutaway of liquid helium finger pump assembly.

entire UHV system was wrapped with heater tapes so that a temperature gradient could be maintained over the UHV system for more efficient removal of the evolved gases. Then, the gages were baked at 425° C, the pyrex finger at 450° C, the manifold at 400° C, and the manifold valve at 350° C. After baking for 75 hours, the system temperatures were reduced to room temperature, the relative temperature differences being maintained until the manifold valve reached room temperature.

During the bake with the tapes, the modulated gage was degassed by electron bombardment every 2 hours in the manner previously described. A thermocouple was placed on the glass near the filament of the modulated gage to insure that the glass did not develop a hot spot and melt during the degassing period. When the temperature of the manifold reached 50° C the manifold valve was torqued closed. Closing the valve isolated the UHV system from the ion pump which would only pump down to about 10^{-8} torr (N_2). At this point in the procedure the pumping was done only by the magnetron gages. After the heater tapes were removed, the oven was again placed over the UHV system and the UHV system was put through a bake cycle at an oven temperature of 100° C with the gages operating. The UHV system was then baked for 200 hours. At the end of this period the pressure was about 2×10^{-9} torr (N_2) and had not significantly decreased in the last 24 hours of the period. The temperature was reduced to room temperature and the oven again removed. Two days after the oven was removed, the magnetron gages indicated a pressure of 2×10^{-12} torr (N_2) (which, as will be shown later, is an actual nitrogen equivalent pressure of about 4.5×10^{-11} torr). At that point in the pumpdown procedure the pyrex finger was immersed in liquid nitrogen and no significant reduction in the pressure was observed. This indicated that the residual gases remaining in the UHV system would not adsorb on glass at 77° K. The liquid nitrogen was removed from the pyrex finger and replaced by liquid helium. Immediately the magnetron gages indicated a decrease in pressure which continued steadily until all the gages indicated pressures less than 5×10^{-14} torr (N_2). At this pressure the gage discharge appeared to have been extinguished. A plot of pressure as a function of time of pumpdown is shown in figure 8.

To verify that the magnetron gages were extinguished, the bleed valve was opened and dry nitrogen was admitted into the UHV system at a rate that caused the indicated

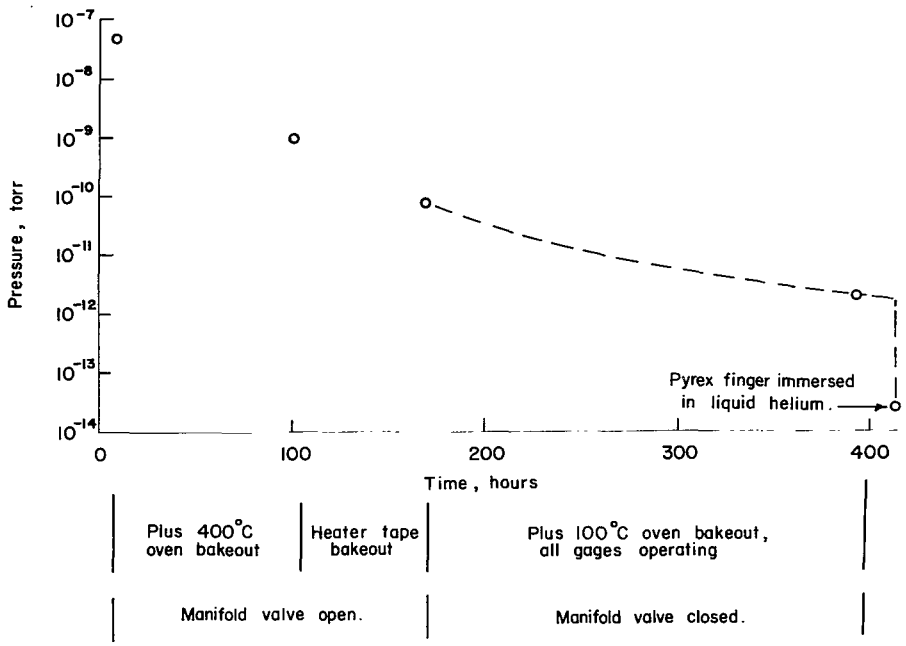


Figure 8.- Operation cycle of system.

pressure to increase to 10^{-11} torr (N_2). The magnetron gages did not indicate the increase in pressure until about 30 seconds after the modulated gage indicated the pressure rise. This time element indicates that the discharge in the magnetron gages had been extinguished at the lowest pressures. The bleed valve was then closed and the magnetron gage indications again decreased to less than 5×10^{-14} torr (N_2). The decrease in pressure, after the bleed valve was closed, was uniformly indicated by all of the magnetron gages.

During a later test series, when the pyrex finger was not cooled by liquid helium, a comparison was made between the magnetron gages and the modulated gage. This comparison was made in order to measure the nonlinear ion current-pressure relationship of the magnetron gage below 2×10^{-10} torr (N_2). (See ref. 5.) The pressure indicated by the magnetron gages at the beginning of the comparison was about 2×10^{-12} torr (N_2). At this pressure nitrogen was admitted into the UHV system at a rate sufficient to cause the pressure to increase about 5×10^{-12} torr (N_2) indicated magnetron pressure.

The true nitrogen equivalent pressure was determined from the modulated gage indications. The procedure for obtaining true nitrogen equivalent pressures from the modulated gage is given in the appendix. The bleed valve was then opened further, the pressure increased, and the pressure indications from the gages again compared. The result of a series of these comparisons of the modulated gage and the magnetron gage that had the electrometer measuring the ion current is shown in figure 9. It is noted that

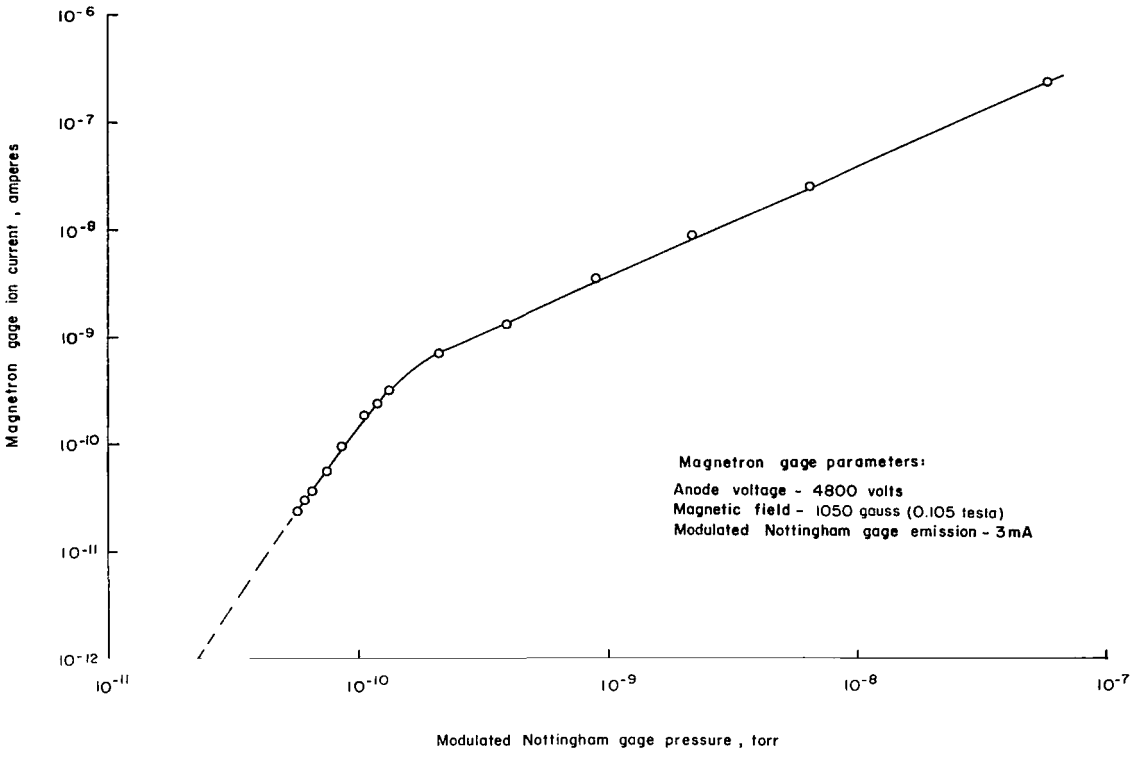


Figure 9.- Nonlinear response of Redhead magnetron gage.

there was a significant amount of residual gas, probably He, present at the lower pressures of figure 9. It has been shown in reference 7, however, that the change in slope of the magnetron gage for He is almost identical to the change in slope for N_2. The fact that the residual gas was present at the lower pressures should not have influenced significantly the ion current-pressure relation shown in figure 9.

CALCULATIONS

The ultimate pressure in the system obtained by the pumping of the magnetron gages alone (manifold valve closed) was 2×10^{-12} torr (N_2) indicated or 4.5×10^{-11} torr (N_2) corrected for the nonlinearity of the gage (fig. 9). The determination of the true pressure obtained when the system was cryogenically pumped by the liquid helium could only be approximated because the magnetron gages were extinguished. In reference 8 it is stated that these magnetron gages were extinguished at about 3×10^{-12} torr and since the gages in this investigation were extinguished, the pressure was probably less than 3×10^{-12} torr. To approximate the lowest pressure two separate approaches were taken to calculate the gas load in the system. The first approach was to assume that the only significant gas load in the system was the helium that permeates from the atmosphere

through the system pyrex glass. By calculating this permeation rate, the system gas load was determined.

A second approach was to calculate the system gas load before cryogenic pumping. Since the magnetron gages were still indicating at this point, the pressure within the system was known. By calculating the pumping speeds of the gages at this pressure, the gas load was then ascertained.

Finally, after the cryogenic pumping speed of the system was calculated, the lowest pressure in the system was obtained.

The procedure for the first approach is as follows: The total gas load Q is a combination of leak rate, outgassing, hydrogen permeation through the metal, and helium permeation through the pyrex glass of the system. When there is negligible leak rate, outgassing, and hydrogen permeation as compared with the helium permeation, then Q can be determined by

$$Q = 0.76\frac{KA_S p}{d} \tag{1}$$

where the constant 0.76 converts K from cm^3 at STP to torr liters. The value of K is a function of temperature and from reference 2 at 25° C for permeation of helium through pyrex 7740 glass

$$K_{He} = 10^{-10.8}$$

The total surface area of the glass is

$$A_S = 1.32 \times 10^3 \text{ cm}^2$$

and the wall thickness d is 1.5 mm. The total pressure difference of the helium will be the partial pressure of helium at atmospheric pressure and from reference 9 this is

$$p = 4 \times 10^{-4} \text{ cm Hg}$$

Substituting the values of K, A_S, p, and d into equation (1) gives

$$Q_{He} = 4.3 \times 10^{-12} \text{ torr liter/sec}$$

This value is that of the gas load from the permeation of helium through the walls of the pyrex apparatus on the system.

The second method of approach to determine the system gas load can be made if the proportionality is assumed between sensitivity and pumping speed of the magnetron gage. The pumping speed for helium has been reported to be 0.15 liter/sec (150 cm^3/sec) in the linear region of the gage characteristic curve (ref. 5). At the point where the magnetron

gage indicates 2.4×10^{-12} torr (N_2) (9×10^{-12} amp), the sensitivity can be determined from figure 9 to be approximately 0.20 amp/torr (N_2) as compared with an average sensitivity of 3.8 amps/torr (N_2) in the linear region. If it is assumed that the pumping speed is directly proportional to the sensitivity, then the pumping speed per gage for helium would be 0.008 liter/sec (8 cm^3/sec). Now the true pressure can be found. By extrapolating the curve of figure 9, 2.4×10^{-12} torr corrected for linearity is 4.5×10^{-11} torr (N_2) and corrected for gas composition, 3×10^{-10} torr (He). The relative gage sensitivity for the gas correction was determined from reference 5. With the helium pumping speed and the pressure, the gas load can be found from

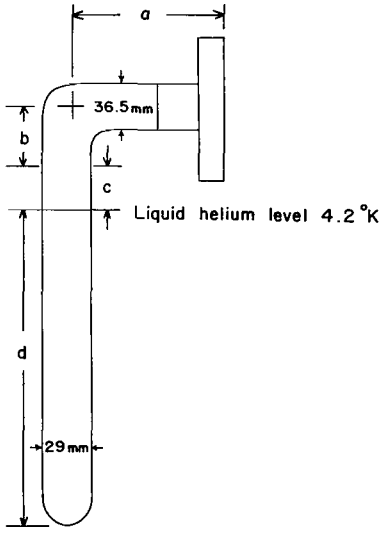
$$Q = Sp \tag{2}$$

where the sum of the pumping speeds for the three gages is 0.024 liter/sec (24 cm^3/sec) therefore

$$Q_{He} = 7 \times 10^{-12} \text{ torr liter/sec}$$

so that Q_{He} is about the same using the two approaches for its estimation. For an average value of the gas load Q, 5×10^{-12} torr liter/sec will be used hereinafter.

The pumping speed of the pyrex finger when immersed in liquid helium is obtained by considering the conductance of each section of the finger at its approximate temperature. As in reference 1, the helium finger is divided into four sections determined by approximated temperatures. (See fig. 10.) The length of the upper part of the finger at 270° K is 9.5 cm (section denoted by subscript a). The length at 190° K is 5.0 cm (subscript b), and at 40° K is 3.0 cm (subscript c). The temperature of the remainder was 4.2° K.



a - 9.5 cm at 270° K
b - 5.0 cm at 190° K
c - 3.0 cm at 40° K
d - 2.0 cm at 4.2° K

Figure 10.- Temperature sections of finger pump.

To determine the conductance, the relation in reference 10 gives

$$C = 3.638\gamma A(T/M)^{1/2} \tag{3}$$

therefore

$$C_a = 97 \text{ liters/sec}$$

$$C_b = 63 \text{ liters/sec}$$

$$C_c = 38 \text{ liters/sec}$$

If every molecule that migrates into the section cooled by liquid helium is assumed to adsorb to the glass walls,

then the pumping speed of that section of the finger cooled by liquid helium is

$$S = 3.638A(T/M)^{1/2} = 24.2 \text{ liters/sec}$$

Now the pumping speed for the finger can be found by using the equation

$$\frac{1}{S_{net}} = \frac{1}{S} + \frac{1}{C_a} + \frac{1}{C_b} + \frac{1}{C_c} \tag{4}$$

therefore

$$S_{net} = 10.65 \text{ liters/sec}$$

The pumping speed of the magnetron gages is neglected since it is much smaller than the pumping speed of the finger.

The total surface area of the glass subject to permeation is now reduced to 1.07×10^3 cm^2 since the reduction in temperature by the liquid helium makes the permeation of gases through the pyrex finger negligible. It is assumed that, since the room in which the system was located was well ventilated, the helium concentration in the room did not increase significantly during the time that the pyrex finger was immersed in liquid helium. The gas load determined when the pyrex finger was cooled must now be reduced by the amount that the glass area subject to permeation is reduced. This value of the gas load when the finger is immersed in liquid helium is 4×10^{-12} torr liter/sec. When this value is divided by the pumping speed of the finger the ultimate pressure in the system is 4×10^{-13} torr.

The manifold is made of stainless steel (304L), and the permeation rate of helium through stainless steel is negligible. However, since hydrogen does permeate stainless steel it is prudent to investigate the permeation of hydrogen through the manifold. The partial pressure of hydrogen in the atmosphere is 3.8×10^{-4} torr, therefore the total permeation should be quite low. The manifold is composed of approximately 20-percent chromium. The permeation rate K_{H_2} of 1 mm thick 27-percent chrome steel at 22° C is reported to be (see ref. 2)

$$K_{H_2} = 1.3 \times 10^{-12}$$

The total manifold surface area is

$$A_s = 597 \text{ cm}^2$$

and the wall thickness is

$$d = 3.2 \text{ mm}$$

Replacing p by $p^{1/2}$ in equation (1) then yields

$$Q_{H_2} = 1.3 \times 10^{-13} \text{ torr liters/sec}$$

Since this H_2 gas load is over a decade less than that calculated for helium and the system pumping speed for hydrogen is greater than for helium, the effect of hydrogen permeation will not be considered.

DISCUSSION AND RESULTS

One of the most difficult problems in achieving these very low pressures is the proper degassing of the components. Many different baking cycles and temperatures were tried before finding the correct procedure to properly degas the components. The modulated Nottingham gage, in particular, was very difficult to clean. One of the advantages of this type of system, however, is that once the gage is clean it will remain so for a considerable period of time.

The magnetron gages on the system were extinguished at the lowest pressures preventing a direct measure of these pressures. Reference 8 states that there is a threshold pressure for these magnetron gages at 2.7×10^{-12} torr (N_2) below which the gage discharge was extinguished. Below this threshold pressure the only currents coming from the gage are from leakage currents and amount to about 5×10^{-14} torr indicated. In reference 7, however, data are presented to show that the magnetron gage discharge has not been extinguished at pressures as low as 3×10^{-13} torr (N_2) which is about 2×10^{-12} torr, true helium pressure. Since the predicted ultimate pressure in the system presented here is about 4×10^{-13} torr, true pressure, the pressure is too low to obtain additional information regarding the pressure at which the gage discharge was extinguished. The gage discharge, however, was extinguished at the lowest pressure.

It was noted that when the pyrex finger was immersed in liquid nitrogen no significant decrease in pressure occurred. The effect of this immersion limits the residual gases in the system to gases which are not adsorbed in significant amounts on pyrex at 77° K. The residual gas most likely to permeate the glass walls of a vacuum system would be helium. The permeation of helium can be greatly reduced by choosing appropriate types of glass. In reference 3 a system was constructed of 1720 aluminosilicate glass. The permeation rate of helium through aluminosilicate glass is a factor of 10^4 less than that of the pyrex glass. The major disadvantage of using aluminosilicate glass is the construction of the system. Special techniques and experience are required to work with this material.

It was not possible to evaluate the problem caused by the decreasing level of the liquid helium in the dewar. The pressure in the UHV system is influenced by the fact that the level of liquid helium falls, which in turn releases adsorbed molecules from the glass as the temperature of the glass increases. This problem could not be studied because the pressure could not be measured as long as there was any substantial amount

of liquid helium in the dewar. The helium level decreased at a rate of approximately 15 cm/hour and the magnetron gages did not indicate pressure until the helium level was about 2 cm from the bottom of the finger.

CONCLUDING REMARKS

It has been shown that it is possible to achieve very low pressures in a metal system where the gages are connected by demountable metal seals. The system is small and inexpensive, but requires liquid helium for its cryogenic pumping. The ultimate pressure is estimated to be lower than 5×10^{-13} torr.

The success of such an assembly depends on the extent and technique of the system bakeout and gage cleanup operations. The ultimate pressure appeared to be limited by the helium permeation through the pyrex glass gage envelopes and by the small conductance of the liquid helium pump.

It has been shown that the system can be readily used for vacuum gage studies using any appropriate test gas at pressures less than 5×10^{-11} torr. Under these conditions the system would contain less than 1 percent residual gases. If helium is used as a test gas, the usable range can be extended below 5×10^{-12} torr since the system residual gas is apparently helium.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., March 29, 1967,
125-24-03-03-23.

APPENDIX

MODULATED NOTTINGHAM GAGE OPERATING THEORY

This appendix describes the way in which the modulated Nottingham gage is used in order to read true pressures in the pressure range where the residual current from photoemission from the ion collector is large compared with the ion current from the positive ions collected.

A more complete discussion of the modulated gage is given in the original presentation on the gage by Redhead (ref. 6). As shown in figure 4 the modulated gage has an additional electrode called the modulator. When held at the same potential (ground) as the ion collector, the modulator collects a constant fraction $(1-\alpha)$ of the ion current and when held at grid potential (180 volts) it collects no ion current. Thus, when the modulator is at 180 volts the ion collector current is

$$I_1 = i + i_r \tag{A1}$$

where i is the positive ion current and i_r is the residual current. When the modulator is held at ground potential the ion collector current is

$$I_2 = \alpha i + i_r \tag{A2}$$

where α is the modulation factor and is independent of pressure. Now α can be found by measurements at high pressures where $i >> i_r$ so that

$$I_2 = \alpha i \tag{A3}$$

and the true ion current can then be found from

$$i = \frac{I_1 - I_2}{1 - \alpha} \tag{A4}$$

The residual current i_r can be found from the collector currents measured at two different pressures p_a and p_b $(\text{where } p_a > p_b)$.

Now

$$I_{2a} = \alpha i_a + i_r \tag{A5}$$

and

$$I_{2b} = \alpha i_b + i_r \tag{A6}$$

thus

$$I_{2a} - I_{2b} = \alpha(i_a - i_b) \tag{A7}$$

but

$$i_a = I_{1a} - i_r \tag{A8}$$

and

$$i_b = I_{1b} - i_r \tag{A9}$$

Then from equations (A6), (A7), and (A8)

$$\alpha = \frac{I_{2a} - i_r}{I_{1a} - i_r}$$

Substituting for i_a, i_b and α in equation (A7) and solving for i_r yield

$$i_r = \frac{I_{1a}I_{2b} - I_{1b}I_{2a}}{(I_{1a} - I_{2a}) - (I_{1b} - I_{2b})} \tag{A10}$$

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