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PRODUCTION AND MEASUREMENT OF EXTREME VACUA

by

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PRODUCTION AND MEASUREMENT OF EXTREME VACUA

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Abstract: The performance of any vacuum system depends on the choice of vacuum chamber materials, pumps and pressure gauges. The first requirement for the achievement of extreme vacua (below 10^{-9} Pa) is the use of low degassing materials. At present, stainless steel and inconel alloys are available which provide (H_2) degassing rates lower than 10^{-10} Pa m s^{-1} ($\simeq 10^{-13}$ torr ℓ s^{-1} cm^{-2}) when properly conditioned. The second requirement is a precise knowledge of the low pressure behaviour of gauges and pumps. The latter is often difficult to meet because of the mutual interference between measuring and pumping limitations. Our approach has been to use cryopumps which provide pressure independent pumping speeds together with materials of low and known degassing rate, thus generating known pressures down to the picopascal range. The low pressure limitations of any measuring device can then be easily obtained. This approach has been followed for the optimisation of a total pressure gauge of the Bayard-Alpert type which is characterised by a low pressure measuring uncertainty of about 2.5×10^{-11} Pa. An external collector gauge of the beam deflected type has also been developed which has already given direct pressure readings in the picopascal range. These new gauges have been used, subsequently, to investigate the low pressure behaviour of other pumping systems such as the combination of sputter ion and titanium sublimation or getter pumps.

INTRODUCTION

The vacuum requirements of particle accelerators, and particularly of machines where high intensity proton beams are stored, have given in the last few years a strong impulsion to the vacuum technology of extremely low pressures. At CERN-ISR the two kilometres long vacuum chamber operates at an average pressure lower than 10^{-9} Pa /1,2/; in two regions about 30 m long pressures in the 10^{-11} Pa range have been obtained /3/. These achievements had required some development in order to meet the ISR's "industrial" criteria, wherein priority has been given to reliability, robustness and simplicity rather than to extreme performance and innovation. Moreover, innovation was often not possible due to the urgency required in the solution of our problems. It was also often not needed due to the existence of a multitude of new instruments and ideas promulgated during the prolific years of the 1960's. Most of these latter innovations were acclaimed suitable for the most strict pressure requirements. A large part of our development activity, as described below, has therefore simply consisted in testing existing instruments and techniques at pressures below 10^{-9} Pa in order to apply them to the ISR.

MATERIALS

Stainless steel is certainly now the most widely used material in UHV systems. The ISR vacuum chamber, made of 316 LN stainless steel, degasses at a rate (H_2) of 2.6×10^{-10} Pa m s^{-1} (2×10^{-13} torr ℓ s^{-1} cm^{-2}) at ambient temperature. This degassing rate is obtained by heating in a vacuum furnace at 950°C and $\simeq 10^{-3}$ Pa for a few hours prior to installation and baking in situ at 300°C under vacuum. It has been shown that a rate of degassing one order of magnitude lower can be obtained by lowering the pressure during the furnace treatment to $\simeq 10^{-4}$ Pa /4/. Rates in the 10^{-11} Pa m s^{-1} range have also been obtained by baking in air at various temperatures /5,6/. Even better results have been obtained with Inconel 600 and 625, which are compatible with stainless steel for flanging and welding. Samples baked at 950°C and 10^{-4} Pa for a few hours have shown a rate of degassing of $\simeq 1.3 \times 10^{-11}$ Pa m s^{-1} /7/, and a large vacuum chamber baked in situ at 550°C and under vacuum has been reported to yield a rate of 6×10^{-12} Pa m s^{-1} /8/.

Any vacuum system built with materials characterised by these degassing rates and pumped with speeds in the range of 10^3 to 10^4 $\ell\ s^{-1}$ per m^2 of wall surface area should consequently reach pressures in the 10^{-11} Pa range or lower. However, such achievements are very seldom quoted in the literature. The reason is that at these extreme vacua the pumping speeds vanish and/or the pressure measuring devices are limited in often unpredictable ways. The discrimination between these two possible limitations is usually difficult as the evolution of vacuum technology has often shown. Recall, as an example, the universally accepted limitation of oil diffusion pumping which was in fact due to a limitation of the ionisation pressure gauge before the Bayard-Alpert innovation.

Our approach to clarify this ambiguity has been the use of cryopumping as a way of obtaining pressure independent pumping speeds. Vacuum systems of known degassing rate, pumped by known pumping speeds have then been used to produce the extreme and known vacua to which to compare the indication of pressure measuring devices. These latter have subsequently been improved and then in turn used to investigate the low pressure behaviour of other pumping systems.

CRYOPUMPING

The ideal pump, best in all respects, would be a hole with "absolute" vacuum behind. This pump would provide known pumping speed and furthermore, pressure independent throughout the range of molecular flow. Condensation cryopumping at about 2 K provides the closest approach to this ideal situation: at this temperature all the gases except He have vapour pressures below 10^{-13} Pa and sticking probabilities close to unity.

However, investigations carried out on condensed H_2 at low pressure have shown an unexpected deviation from the extrapolated vapour pressure curve, resulting in a temperature independent saturation pressure in the 10^{-8} Pa range /9/. This effect was initially attributed to desorption induced by infrared thermal radiation absorbed in the condensed H_2 layer /10/. Later an indirect mechanism was proposed in which the thermal radiation is absorbed by the pumping surface thereby generating phonons which can travel back to the condensed H_2 and produce molecular desorption /11,12/. This indirect mechanism is based on two main experimental observations. The first is that the same radiation load, impinging on H_2 condensed on various substrates, yields desorption rates which are proportional to the substrate's absorptivity. The second is the strong reduction of H_2 desorption which is obtained by condensing, prior to the H_2, a layer of a heavier gas. This layer, characterised by a Debye temperature much lower than that of H_2, acts as a low pass filter, preventing the most energetic phonons produced in the solid substrate from reaching the H_2 film.

The action of precondensed gas layers, as well as the dependence of the H_2 saturation pressure on the temperature of the radiating surface, are shown in Fig. 1. This figure contains practically all the required information on the low pressure behaviour of cryopumping. The pumping surface must be properly shielded from 300 K radiation and liquid N_2 temperature is adequate for the shield. If the pumping surface is only loaded with radiation from 77 K and covered with a condensed N_2 layer, an H_2 pressure of about 10^{-11} Pa at saturation is established, i.e. it is then independent of further H_2 pumping. For subsaturation quantities of H_2, a temperature variation of the baffle (source of thermal radiation) results in a set of curves which are parallel to those shown in Fig.1 but translated

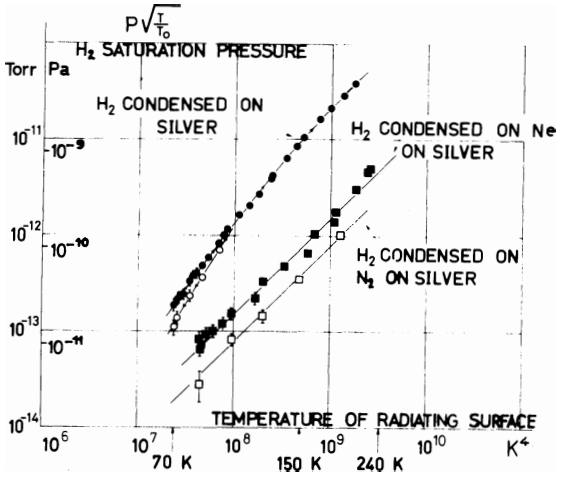

Fig. 1. The dependence of the saturated vapour pressure of H_2 condensed on various substrates on the temperature of the radiating baffle. The pressures are corrected for thermomolecular transpiration. N_2 equivalent pressures measured at room temperature are about three times higher.

towards lower pressures. This implies that, in practical cases, a temperature variation of the baffle permits one to determine the contribution of the H_2 vapour pressure to the measured vacuum. According to our experience, if the pumping surface (Ag plated or bare stainless steel) is baked at 300°C and then cooled with liquid He when the pressure is in the 10^{-8} Pa range, this contribution is below 10^{-12} Pa. In such cases any higher pressure existing inside a vacuum system is simply the ratio of the total degassing rate of the system's walls to the pumping speed of the cryopump. In other words, the pressure is known upon measuring these two quantities independently.

This basic investigation into the behaviour of condensed gases has enabled us to build a first group of cryopumps for use in the ISR. All these pumps, already described in detail elsewhere /13/, are of the same type (see Fig. 2) which, henceforth, we shall call 'first generation'. Their characteristics have been primarily dictated by vacuum considerations, namely:

i) the cryosurfaces are silver-plated to minimise the radiation induced H_2 saturation pressure.

ii) for the same reason, the liquid N_2 cooled baffle has been designed in such a way as to minimise (less than 1 part in 10^3) the transmission of the incident room temperature radiation /14/.

iii) gas desorption from lateral surfaces of the He vessel, resulting from liquid He level variation, is prevented by a second wall which surrounds these surfaces completely. The volume enclosed between these two walls is filled with He gas for cooling during the liquid He transfer and subsequently evacuated to make the temperature of the outer wall independent of the liquid He level.

The pumping speed per cm^2 of baffled pumping surface is about 9 ℓ/s for H_2 and 3 ℓ/s for N_2. The pumps so far built, of two different sizes, provide 4'500 and 27'000 ℓ/s pumping speed for H_2. Their capacities are 10 and 75 ℓ of liquid helium and autonomies, at 4.2 K, are of 40 and 80 days respectively. Autonomies are 20% to 30% lower when operating at 2.3 K in the condensation mode.

The liquid He losses of these pumps are mainly due (more than 95%) to radiation emitted from liquid N_2 cooled surfaces and absorbed by the pumping surface and by the protecting wall in amounts proportional to their surface area. In a liquid He container presenting a diameter equal to the height, for example, the area of the pumping surface is only about 20% of that of the protecting wall, which is therefore responsible for more than 80% of the total losses. It is unfortunate that these losses mainly originate on surfaces which have no pumping function. On the other hand, only a minor fraction of the cooling capacity of the escaping He gas is used for removing the heat conducted along the neck of the He vessel.

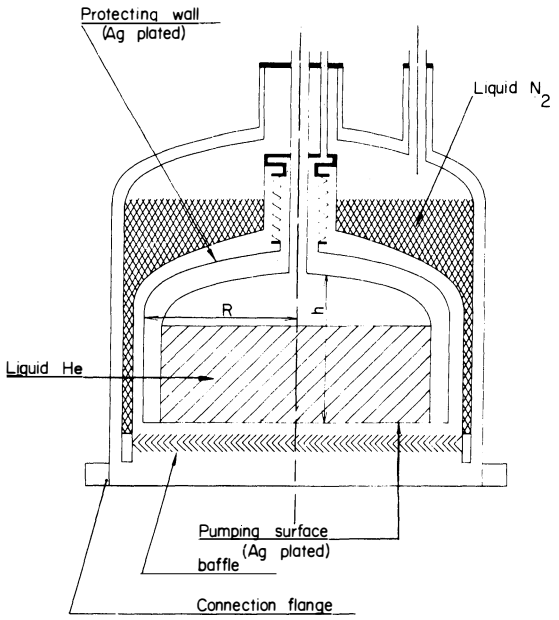

Fig. 2. First generation cryopump

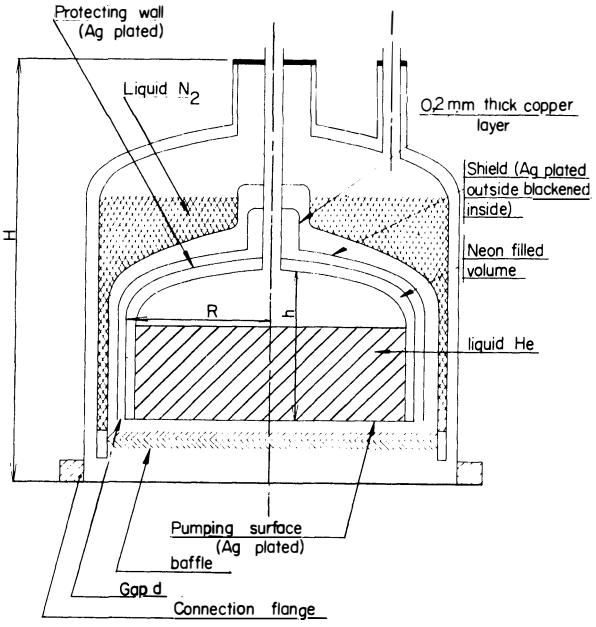

Fig. 3. Second generation cryopump

A straightforward solution to both these inconveniences is to protect the outer cylindrical wall of the He vessel with a shield welded between He and N_2 temperatures and cooled by the escaping gas. However, this could not be done in the first generation pump as is shown in Fig. 1 because the part of the neck where the shield should be connected was not accessible. The structure of the pump was therefore modified as shown in Fig. 3 /15/. The volume enclosed by the two walls of the liquid He container is filled with Ne gas at ½ bar and sealed. The Ne has the function of a thermal switch. Above about 20 K it provides the thermal contact which was previously provided by the gaseous He introduced from the outside. Below this temperature, when, in any case, the thermal capacity of the metals is negligibly low, the Ne condenses leaving a vacuum which provides the desired thermal insulation. The lateral shield, which is now an integral part of the second generation pump, determines with its temperature the He losses originating on the protecting wall. This temperature depends on various parameters, namely the width of the gap 'd', height 'h', radius 'R' (see Fig. 3) and the thermal impedance of the neck to which the shield is welded. However, it can be shown that this temperature does not exceed 30 K if $h < 2R$, $d = 10^{-2}$ R and if the neck is properly designed. Under these circumstances the losses due to the thermal radiation from the shield are negligible with respect to those produced by radiation from the baffle and absorbed by the pumping surface. The liquid He consumption Q (in cm^3 of liquid per day) can then be expressed in the very simple form

$$Q = 0.1S \text{ cm}^3 \text{ day}^{-1}$$

where S is the area of the pumping surface expressed in cm^2.

So far, 8 pumps of this model and of two different sizes have been built. Both models provide an autonomy of about 200 days at 4.2 K. The smaller pumps contain 12 ℓ of He and give ≃ 4'500 ℓ/s pumping speed for H_2. The larger pumps contain 32 ℓ of He and give ≃ 11'000 ℓ/s pumping speed for H_2 /15/.

Two of these pumps (a large one of the 1st generation and a small one of the second generation) have been used extensively for investigating the low pressure limitations of ionisation gauges. The ultimate pressure of these pumps is the ratio of the degassing rate of the outer wall and measuring dome which are at room temperature in normal operating conditions (see Figs. 2 and 3) to their pumping speed for H_2. These pressures are about 2.5×10^{-11} Pa and 1.5×10^{-11} Pa (N_2 equivalent) for the small and the large pump respectively if built with the standard ISR stainless steel. Lower ultimate pressures are achievable with another cryostat which was used for optimising the parameters of the cryopumps /16/. In this case the volume between the liquid N_2 vessel and the outer wall at room temperature is closed and independently pumped. The only degassing is then produced by the measuring dome for which the effective pumping speed (for H_2) is ≃ 4'500 ℓ s^{-1}. Even when using domes of relatively large size (≃ 1000 cm^2 surface area) and standard ISR stainless steel, pressures of about 2.5×10^{-12} Pa are achieved. All these pumps not only provide the required extreme vacua but also the long operating times which are often necessary to reach equilibria at these pressures.

PRESSURE MEASUREMENTS

For different reasons we have investigated the low pressure behaviour of modulated Bayard-Alpert gauges (BAG) and also of external collector gauges, more particularly of bent beam or Helmer type (HG). Our interest in the BAG originates from the need to replace about 400 pressure gauges of this type which were initially installed on the ISR vacuum system /17/. These gauges became obsolete as a result of vacuum improvement (from ≃ 10^{-8} to ≃ 7×10^{-10} Pa, N_2 equivalent) which has been found necessary to inhibit pressure and beam instabilities in presence of intense proton beams /1/. The main specifications for the new gauges are on the one hand to provide pressure measurements in the 10^{-10} Pa range (low pressure uncertainty not higher than 7×10^{-11} Pa) and, on the other hand, be compatible with the existing power supplies, which had been designed for modulated BAGs.

The investigation on the HG had to provide a solution to the problem of measuring pressures in the 10^{-11} Pa range. Such pressures were required in a few intersecting regions of the ISR in order to minimise the background of spurious proton-gas interactions /3/. This very low pressure gauge was also to act as a reference instrument in laboratory work, particularly for the acceptance tests of the new BAGs for the ISR.

Bayard-Alpert Gauge

The main low pressure limitation of the ionisation gauge, since its early days, was due to photo-emission of electrons from the ion collector under the effect of soft x-rays produced by the electron bombardment of the grid. Although the drastic reduction in collector area introduced by Bayard and Alpert (1950) /18/ provided a decrease of this x-ray current (I_x) as well as the corresponding pressure (P_x) by two to three orders of magnitude, Alpert himself remarked some years later (1958) that it might not be possible to reduce P_x indefinitely by reducing the collector wire diameter because "there may be a critical size of the wire below which the probability of collecting ions goes down as rapidly as does the x-ray effect" /19/. This statement was confirmed by experimental results obtained with both open-ended and closed grids /19/. Some years later (1965) Comsa /20/ came theoretically to the conclusion that the low pressure limit of the Bayard-Alpert gauge (BAG) should remain constant when reducing the collector wire diameter below 0.1 mm, because the reduction of I_x might be accompanied by an equal reduction of sensitivity (S). This conclusion was based on the fact that the tangential component of the speed of the ions produced inside the grid would prevent a fraction of them from colliding with the ion collector when first approaching it. These ions could not subsequently be collected, independently of the number of their orbits around the collector, if the radial electrostatic field present in the grid volume is ideally perfect /20/. The number of "non-collectable" ions would increase proportionally to the reciprocal of the collector diameter below about 0.1 mm /20/ - hence the measuring limitation in the low 10^{-9} Pa pressure range.

However, a systematic investigation carried out on a BAG with open-ended grid structure (Groszkowski, 1965 /21/), provided results in contradiction to this statement. In fact, a collector diameter reduction from 100 to 8 μ produced a decrease in sensitivity of only a factor of 5 instead of the expected 12.5. Furthermore, already in 1961, Van Oostrom had shown that S values of about 9×10^{-2} Pa^{-1} ($12\ torr^{-1}$) for N_2 could be achieved with collector diameters as small as 4 μ /22/. The corresponding estimated P_x was below 10^{-10} Pa. This result was obtained with closed grids of 20 mm diameter made of molybdenum and tungsten collectors. It should be noted that the S value quoted by Van Oostrom required grid and filament bias voltages much higher than those normally used for BAGs (about 300 and 220 V instead of 150 and 50 V positive with respect to the collector). Schutze and Stork (1962) /23/ also came to the conclusion that P_x values as low as 7×10^{-10} Pa are possible when using a collector of 40 μ diameter and 10 mm length.

A few years later the BAG was practically abandoned for low pressure applications upon the appearance of gauges with collector external to the grid and providing P_x below 10^{-10} Pa (Clay and Melfi, 1966 /24/; Redhead, 1966 /25/; Groszkowski 1966 /26/; Helmer and Hayward, 1966 /27/.

The first step in our investigation (which has been described in detail in ref. 28) was to check the dependence of the ion collection efficiency on the diameter of the collector for open and closed grid structures. The results are shown in Fig. 4. For open-ended grids (triangles) the reduction of the collector diameter affects catastrophically the sensitivity of the gauge. These results agree very well with those obtained by Groskowski (full line in Fig. 4) for the same bias voltages on the electrodes (+ 220 V on the grid and + 50 V on the filament with respect to ground). In contrast, for closed grid structure the gauge sensitivity remains constant, within our measuring accuracy, for collector diameters down to 25 μ (dots). These latter results have been obtained

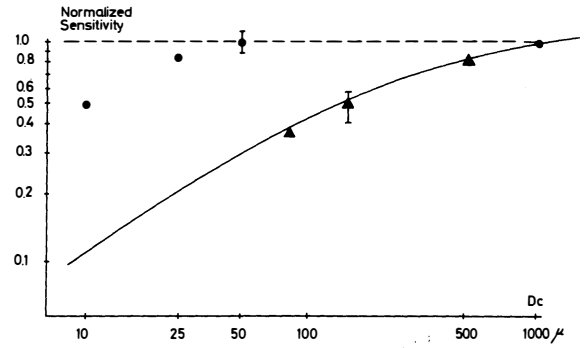

Fig. 4. Collection efficiency as a function of the collector diameter for open-ended (triangles) and closed (dots) grid structures. The error bars represent the spread of the results on about 20 seemingly identical gauges.

in spite of the low bias voltages applied to grid and filament (+ 150 V and + 50 V respectively, as available on the existing ISR power supplies).

The results of Fig. 4 are equally valid for grids of 25 and 35 mm diameters. However, in the latter case the sensitivity S is higher due to increased path length of the electrons inside the grid which results in an enhanced ion production in the grid volume. When mounted in the standard ISR tubular connection with 60 mm inner diameter and standard bias voltages applied to grid and filament, the larger grids have provided S = 0.32 Pa^{-1} (42 $torr^{-1}$) and the smaller ones S = 0.20 Pa^{-1} (27 $torr^{-1}$). Larger grid diameters also provide the additional advantage of decreasing the residual current of the gauge by decreasing the solid angle subtended from the grid to the collector /25/ (see Fig. 5). For the characteristics of the gauge finally chosen for ISR (closed grid with 35 mm diameter and collector with 50 μ diameter, standard bias voltages on the electrodes) the pressure equivalent to the residual current is about 10^{-10} Pa. Values even lower could be obtained by combining differently the results shown in Figs. 4 and 5. An interesting by-product of the use of very thin collectors is the high modulation factor k (Redhead's definition /29/). For the ISR gauges, equipped with two modulators of 0.7 mm diameter, k = 0.85.

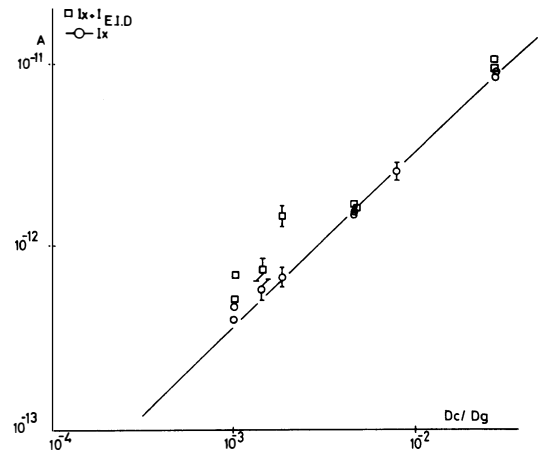

Fig. 5. Variation of residual current due to x-rays (I_X) and measured by modulation ($I_X + I_{EID}$) as a function of the ratio of the collector to grid diameter. I_{EID} represents the residual current due to ions desorbed from the grid under electron bombardment. The error bars have the same meaning as in Fig. 4. The electron emission is 10 mA, the applied voltages + 150 V on the grid, + 50 V on the filament, - 30 V on the collector with respect to ground.

The very low residual currents of these gauges and the possibility of carrying out tests at extremely low pressures have also put in evidence another limitation which appears when working at high electron emission current and low electron energy /28/. This effect, which has been attributed to formation of space charge around the filament, has been found to be more important for straight helix than for hair-pin filaments. When the latter structure is used and the electron emission currents do not exceed 4 mA this limitation is lower than 3 × 10^{-11} Pa /28/.

Helmer Gauge

Among the gauges with collector external to the grid cage the HG is the one which has most extensively been studied in our laboratories. Seven such gauges in its commercial version have originally been purchased. All of them have initially shown a common limit of about 2.5 × 10^{-10} Pa when tested on our various low pressure systems. This limit was later attributed to sublimation of tungsten from the cathode /30/. After thoria coating of the filaments all the HGs have reached a pressure indication of about 2.5 × 10^{-11} Pa at 10 mA emission current. In this operating mode the power dissipation by Joule effect in a newly thoriated cathode is about 4 W. Ageing raises the latter value, and the lowest pressure indication has been found to increase accordingly. The variation of power dissipation in the filament also varies following variation of the electron emission current. By setting the latter at 4 mA, pressures below 10^{-11} Pa have been measured by two different HGs.

However, a reduced electron emission current implies a reduction of the already small ion currents to be measured. To overcome this problem, and also because the production of the Helmer gauge has been discontinued by the manufacturer, we have built two prototypes of an improved HG (IHG), see Fig. 6. The main improvement was expected from the use of the same grid and filament structure successfully used for our standard ISR gauge (high sensitivity). Tests on IHGs are still in progress but a few significant results are already available. The sen-

sitivity has been raised from 7.5×10^{-2} Pa^{-1} ($10\ torr^{-1}$) to $0.34\ Pa^{-1}$ ($45\ torr^{-1}$) and the pressure equivalent to the residual current consequently reduced from 1.5×10^{-11} Pa to $\simeq 3 \times 10^{-12}$ Pa. Ion currents corresponding to pressures of about 10^{-12} Pa have been measured as shown in Fig. 7. This figure reproduces the typical pattern of the collector current originally obtained by Helmer /27/, when varying the bias voltage of the upper deflecting electrode (A in Fig. 6). The region 1, on the left, shows the currents of the electrons extracted by x-ray from the lower plate B of the deflecting system. This current is therefore negative and its absolute value must be added to the positive ion current which is collected in normal measuring position and which is shown, already corrected, in the region 2. The negative currents shown in region 3 on the right hand side are attributed by Helmer to charged particles extracted from the upper inflecting electrode (A).

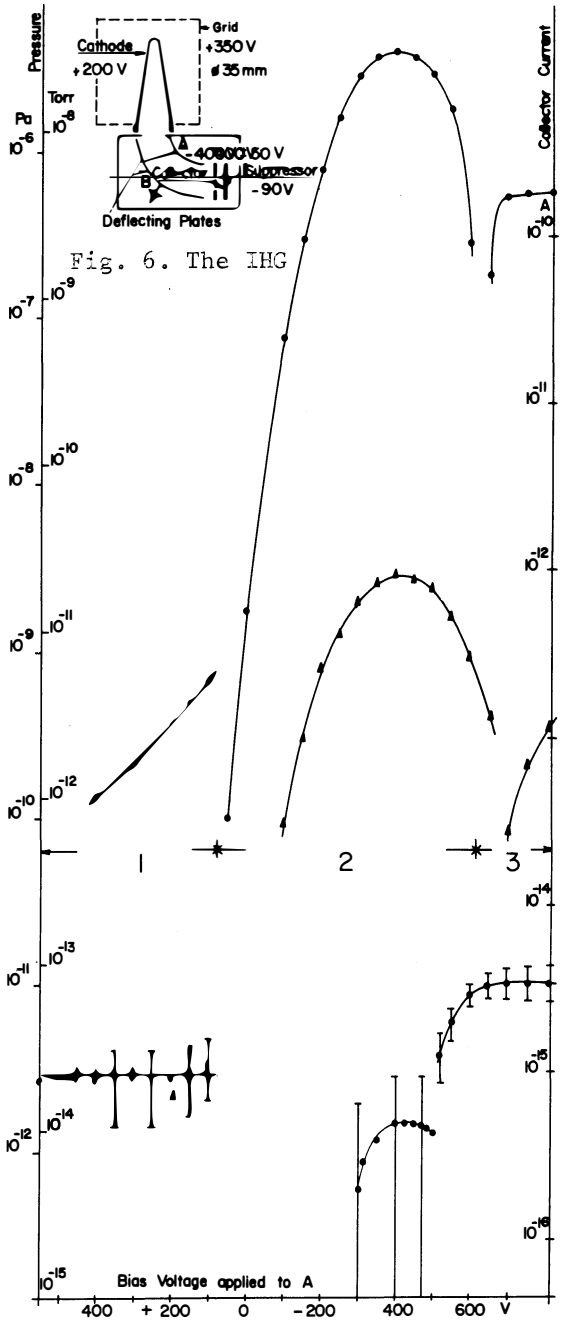

Fig. 6. The IHG

Fig. 7. The dependence of residual currents (regions 1 and 3, right hand scale) and measured pressure (region 2, left hand scale) on the voltage applied to the upper deflecting electrode A. The residual currents are negative. Electron emission current 1 mA.

Unfortunately, when these measurements were being carried out, the thoria coating of the filament was not in good condition and an emission current of 1 mA was required to keep thermal degassing low. With such a value of the emission current the ion current corresponding to 10^{-12} Pa is only about 4×10^{-16} A. This is low but still within the measuring possibilities of the electrometers we have used (of vibrating reed type, with a noise of about 2×10^{-17} A). Significant, in this respect, is the fact that the negative currents due to x-rays are reproducible over a broad pressure range (see region 1 in Fig. 7) and, furthermore, proportional to the emission current between 1 and 10 mA (not shown in the figure). Even when taking into account other possible sources of measuring uncertainty, Fig. 7 shows that pressures in the low 10^{-12} Pa range have been achieved and measured.

OTHER PUMPS

The use of adequate gauges has permitted us to study the low pressure behaviour of other pumps or pump combinations, namely sputter-ion with Ti sublimation pumps or sputter-ion with non evaporable getters. The first combination is used routinely to reach pressures in the 10^{-10} range in the ISR, and even lower pressures (down to the picopascal range) in laboratory systems characterised by a smaller ratio of degassing surface area to pumping speed. Sporadically, a limitation up to the 10^{-10} Pa range has been noticed due to an anomalously high H_2 degassing of the sputter-ion pumps. This effect seems to be caused by pumping of large quantities of water vapour during the baking and has always successfully been cured by baking the sputter-ion pump at 300°C in air.

Less studied is the low pressure behaviour of the non-evaporable getter (Zr-16% Al alloy) on which we report elsewhere in this conference /31/. So far, this getter has not shown any low pressure limitation in the two vacuum systems in which it has been used. The ultimate pressures of these systems have been about 2×10^{-10} Pa and 2×10^{-11} Pa.

CONCLUSIONS

The development work carried out in our laboratory has once more shown that the limitations in the controlled achievement of extreme vacua over the last decade can again be attributed to limitations in pressure measurements rather than by materials or pumps. The HG, with some improvements, seems to be adequate for measuring any vacuum which can now be produced in practical systems operating at ambient temperature.

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