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The Development Of Vacuum Measurements Down To Extremely High Vacuum –XHV

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Abstract

The available measuring devices in the ultra high and extreme high vacuum range with their limitations are presented on the basis of the ultimate attainable pressure. New miniature gauges and calibration devices up now available are shortly reviewed. A complete bibliography is presented.

Key words: vacuum, gauge, ionization, desorption, ESD, photocurrent.

1. Short Historical Introduction

The existence of vacuum and atmospheric pressure became evident with Galileo and his school [1,2,3,4,5] even if some hypotheses and experiments related to those subjects were considered in ancient times [Democritus from Abdera (460–360 b. C), Erone from Alexandria (I e II century b. C)]. In the second half of the seventeenth century, that was crucial for the vacuum science as for many other scientific fields, many researchers performed their experiments which represent real milestones. Among them Evangelista Torricelli (608, 1647), Blaise Pascal (1623, 1662), Otto von Guericke (1602, 1686), Robert Boyle (1627-1691) etc.. Their highly innovative and complex experiments were the starting point for many other experiments and theoretical speculations bearing to kinetic theory and to the great technological realizations of the following centuries. After many disputes between plenists and vacuumists, vacuum existence was accepted and for two centuries (1660-1860) a pressure of several hundreds of pascal, as measured with the Torricelli device [4], was considered sufficient; in fact the experts did not require lower pressures and the pressure measuring system (mercury manometer) did not detect easily lower pressure. From 1850 to 1900, under the pressure of important emerging applications (e.g. electric lamps, cathode-ray tubes), many improvements took place in vacuum technology due to several researchers as Geissler (glass mercury vacuum pump), Sprengel (mercury drops pumps) [6]. In 1870 W. Crookes [7] used a Sprengel pump to reach what he called “perfect vacuum” and measured the pressure with a Torricelli column that was the only available vacuum gauge, but, in 1874, McLeod described his famous gauge [8]. The last 1800 decades may be considered as the beginning of industrial applications on wide scale; in fact Edison started the production of incandescent

lamps [9,10] requiring vacuum conditions realized with Sprengel and Geissler pumps; a 10^{-1} Pa pressure was measured with a McLeod.

In few years around 1900 there was a great improvement of the solid piston oil lubricated pumps, valves, motors etc and pressures even as low as (10^{-3} - 10^{-4}) Pa were reached, as measured with special McLeod gauges [11]. In fifty years the pressure was lowered by six magnitude orders.

At the beginning of 20th century, M. H. C. Knudsen (1871-1895) [12,13] and M. Pirani (1880-1968) [14] developed new gauges: thermo-molecular gauge and thermal conductivity gauge that became so spread and famous to be known simply as the Pirani.

Since the ultimate pressure [10] is related the pumping speed and to several gas flows generated inside the system to be evacuated or from outside (leaks, thermal outgassing, electron or photon stimulated desorption) the great improvement of the lowest attainable pressure in the first half of the twentieth century was due to reduction of leaks, reduction of outgassing by means of heat treatment of vacuum systems and improvement of the pumps.

This last improvement was mainly due to W. Gaede [15] in Germany and I. Langmuir [16] in USA with their realizations in the field of pumps (mercury rotary pump and molecular pump reaching pressure in the range of 10^{-5} Pa). Both the researchers (in 1915-1916) separately invented the mercury diffusion pump and in 1928 C. R. Burch invented the oil diffusion pump. In addition, 1916 is considered the birth date of the ionization gauge that is attributed to Buckley [17]¹ who described the so-called traditional triode as follow: "The manometer consists of three electrodes sealed in a glass bulb which serve as cathode, anode, and collector of positive ions..."; he realized the gauge and compared it with Knudsen and McLeod gauges. This type of ion gauge was universally used for pressure measurements down to 10^{-6} Pa until 1950, but this is a more recent and interesting story [18].

Mechanical gauges started around the half of 19th century with the Bourdon type manometer [19] and, in the first half of 20th century, with the diaphragm sensors. With the development of the electronics of both the control/measuring unit and the sensing heads the well known the CDGs (Capacitance Diaphragm Gauges) [20] became the most diffused high precision gauges covering a wide pressure range from the atmosphere down to 10^{-2} Pa with various sensor heads. The Knudsen original idea of using the momentum transfer between the molecules of the residual gas and a rotating (or capable of rotating) special body was developed into the original commercially available Spinning Rotor Gauge (SRG) [21,22,23] through several realizations at laboratory level (e.g. viscosity gauges, levitation gauges) [24]. At the beginning of 19th century the first pressure balances were available [25].

¹ Even if the first idea of an ionization gauge is attributed to Adolf Baeyer [18]

Table 1 Various steps towards UHV measurements

Gauge type	Pressure range (Pa)	Year/period
Liquid columns		
Mercury column Column Hg or oil manometer (interferometric devices)	$10^5 - 100$ $10^5 - 10^{-1}$	Torricelli, Pascal, Boyle, half 1600 [18] Several authors [28,.....,34]
Mechanical gauges		
Bourdon, Several diaphragms, Capacitance diaphragm gauges Several realizations Commercially available	$10^5 - 10$ $10^5 - 10^{-2}$ $10^5 - 10^{-1}$ $10^5 - 10^{-1}$	Bourdon 1849 [19] Olson e Hurst 1929 and Several other authors) [21] At present
Compression		
McLeod	$1 - 10^{-5}$	McLeod 1874 [8]
Viscosity/ momentum transfer		
Torsion vacuum gauge	$100 - 10^{-2}$	Sutherland 1897[35], H. W. Drewing 1965 [36]
Oscillating fiber Molecular damping force	$100 - 10^{-2}$ $10^{-4} - 10^{-2}$	Langmuir 1913 [37] R. G. Christian and J. H. Leck (1966) [38]
Knudsen radiometer Levitation Spinning rotor gauge	$1000 - 10^{-1}$ $10^{-3} - 1$	Knudsen 1910 [11,12] R. Evrard and G. A. Boutry (1969) [24] Fremery (after 1970) [21,22,23]
Commercially available	$10^{-3} - 1$	At present
Thermal conductivity		
Pirani	$10^5 - 10^{-1}$	Pirani 1906 [14]
Thermocouple	$10^5 - 10^{-1}$	Voegé 1906 [18]
Commercially available	$10^5 - 10^{-1}$	At present
Ionization		
Hot cathode (Traditional triode B-A gauge Modulated Bayard - Alpert Extractor Cold cathode (Penning) Magnetron/inverted magnetron Commercially available	$10^{-1} - 10^{-6}$ $10^{-1} - 10^{-9}$ $10^{-2} - 10^{-11}$ $10^{-2} - 10^{-10}$ $10^{-1} - 10^{-4}$ $10^{-3} - 10^{-11}$ $10^{-1} - 10^{-9}$	Von Baeyer 1909 [18], Buclay [17] Bayard-Alpert 1950 [51] Redhead 1960 [26] Redhead 1966 Penning 1937 [39] Beck e Brisbane 1952 Redhead 1959 [5,41,50] At present

Table 1 summarizes various steps along the never ended and fascinating story of vacuum measurement.

The beginning of some researches on accelerators (around 1924), plasma vacuum fusion devices (1953) and, more recently, on gravity wave observatories [26, 27] requiring very good vacuum conditions and the related experimental realizations pushed towards new very low pressure measurement devices or improvements of the already existing vacuum gauges. To which surface science and the thin film production training realization of electronic, micro and nano-electronic devices must be added so causing flowering of researches and realizations in UHV (pressure lower than 10^{-6} Pa) and, later, XHV (pressure lower than 10^{-10} Pa).

2. Measurements Of Ultra-High Vacuum ($\approx 10^{-6}$ Pa $> p > 10^{-10}$ Pa)

The ultra-high vacuum story belong to the '50 years [40] and it is strongly related to the development of suitable ion gauges from the initial triodes and to the knowledge acquired on the physical phenomena occurring in them and restricting their use to $\approx 10^{-6}$ Pa. For long time the 10^{-6} Pa limit was considered as related to the pumps (diffusion pumps) until W. B. Nottingham at the Westinghouse laboratories (1947) [18] presented as possible explanation the existence, on the ion collector, of a residual current completely independent of the pressure and due to the emission of electrons under the impact of X-rays emitted by the grid under electron bombardment [41]. Basically the to-day's hot cathode ion gauges are made as described by Buckley in 1916. Electrons emitted from the hot cathode are accelerated to the anode and collide with the gas molecules producing positive ions; a fraction of these ions are collected on the collector giving rise to a positive current proportional to the pressure.

There are several components of the positive current as detected by the ion collector which are independent of the pressure, originating a residual current that is due to three main processes (figure 1) [42]:

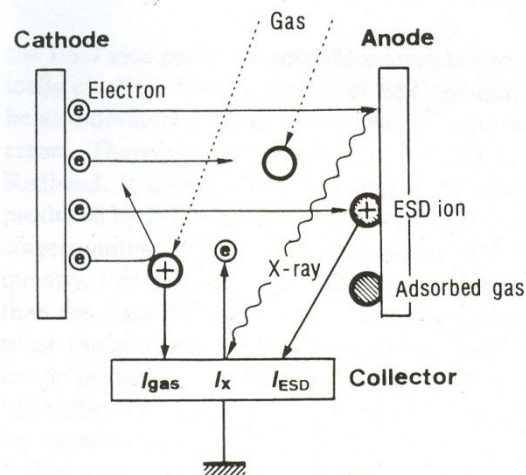


Figure 1 Physical phenomena involved in ionization gauge behaviour (from figure 1 ref [42], reproduced with J. Vac Sci. Technol. permission)

- 1) Emission of electrons by the collector: under bombardment of X-rays generated on the grid by electrons coming from the cathode electrons are emitted by the ion collector resulting on the generation of a positive current not related to the gas phase ions [10, 41, 43]
- 2) Electron stimulated desorption (discovered in 1963); positive ions [from chemically active gases (O₂, H₂, H₂O, CO)] or neutrals adsorbed on the grid may be desorbed under direct electron bombardment of the grid itself [11,44,45,46]
- 3) Outgassing connected with the operation of the hot cathode gauge: there may be local increase of gas density due to evaporation from the cathode and/or thermal outgassing from the gauge electrodes and from the walls; consequently the appropriate choice of the materials, mostly for the cathode, became subject of many research works [47, 40, 49]

All those effects may alter the composition of the gas inside UHV systems and produce positive current not distinguishable from the positive current related to the gas phase. Consequently the possibility of lowering the pressure limit for what concerns both measurements and production is strongly connected to the improvements of the knowledge of the interaction of the gauge with the quantity to be measured. Several solutions to this problem were proposed as it is, tentatively, summarized below:

- 1) ion collector not aligned with the grid
- 2) insertion of electron multiplier to lower the electron current from the cathode without reducing the sensitivity of the gauge
- 3) use of a suppressor electrode to reduce photoelectrons emitted by the collector
- 4) the surface of the ion collector decreased and so the solid angle between the collector and the X ray source
- 5) insertion of an additional electrode (modulator) modulating the ion current without modulating the current produced by the X rays
- 6) introduction of energy analyzer to evaluate the ESD current, once evaluated or considerably reduced the X-ray residual current
- 7) use of magnetic field to increase the mean free path of the electrons to have several impacts with neutral to get an ion current high in comparison with the positive current due to the X-rays, even at low pressure.

The last method brought, in 1937, to the cold cathode ion gauges or Penning vacuum gauge [39], later commercialized. Penning is, in general, unable to maintain the discharge at pressure lower than 10⁻⁴ Pa. Other gauges equipped with magnetic field have been developed generally at research laboratory level as e.g cold cathode magnetron and inverted magnetron gauges [5, 41, 50] .

All the other methods have brought to several hot cathode ion gauges.

The most spread and useful solution was proposed by Bayard and Alpert [51] in 1950 and is based on geometrical and dimensional fundamental changes, by decreasing substantially the diameter of the collector and changing its position as regards the grid and filament (figure 2 a and b). Their gauge became simply known as the Bayard-Alpert (or B-A gauge); it represented a real milestone with the lowest measurable pressure of the order of 10⁻⁹ Pa.

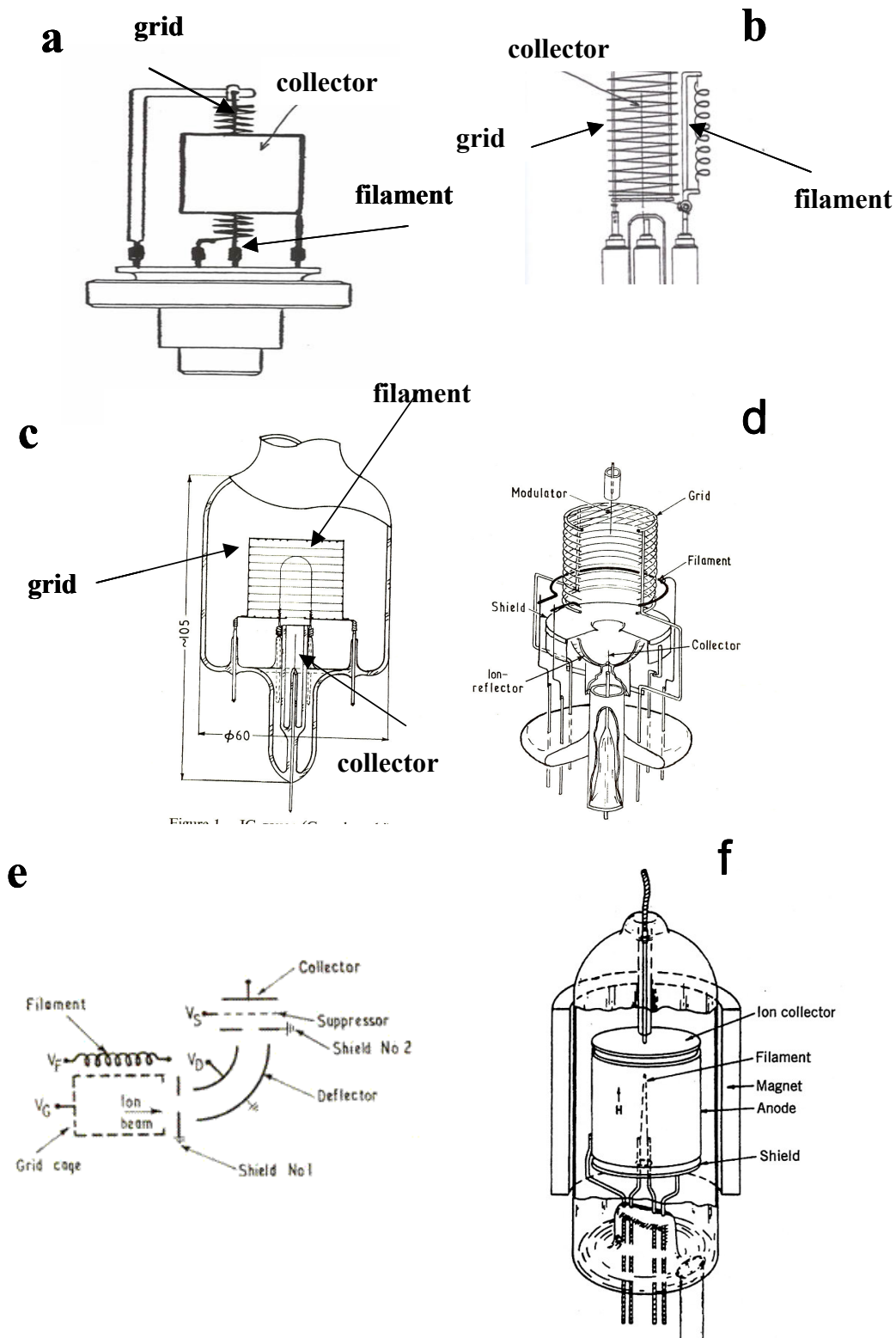


Figure 2 Comparison of several geometries of hot cathode ionization gauges: a traditional triode; b B-A gauge; c Groszkowsky gauge [65]; d Redhead extractor gauge (from figure 1 ref [75], reproduced with J. Vac. Sci. Technol. permission); e Helmer gauge (from figure 1 ref [65]; hot cathode Lafferty gauge (from figure 1 ref [77])

This new measurement possibility carried out many realizations of systems, components, pumps based on new physical principles from the improvements of turbo-pumps to the realization of new ion, getter and cryogenic pumps [6,52]. New types of metal flanges [52, 53, 54,55] were realized and the UHV systems started to be made in metal (mainly stainless steel) instead of glass. However, the hot cathode ion gauges did not solve entirely the problem under several aspects since the gas composition may be altered by chemical interactions of the residual gas on hot surfaces, emission of neutral and positive ions from the cathode and thermal effects due to heating of the walls by radiation from hot cathode [41]. Finally, even in the B-A, the X-ray limit could not be completely eliminated: it was only lowered. From 1950 a lot of studies were performed on the behavior of the ionization gauges as e.g. on their pumping action, thermal desorption as well as on electron stimulated desorption, interaction of molecules with hot filament [41,56], and on lowering again the X-ray limit under the stimulus of the need of ultra-high vacuum conditions for industrial and research applications.

Two main research directions were followed: 1) measurements of the photocurrent by introducing an additional electrode similar to the collector inside the grid (P. A. Redhead has the first studied this configuration [41, 57,58,59]); the modulating electrode can be kept at different potentials), 2) lowering that current by changing the position of the ion collector towards the X-ray source (the grid). Frequently both ways were followed together as it will be mentioned in the next paragraph. In addition several studies were performed with the aim of discriminating the ESD ions from the gas-phase ions [60,61].

Other researches have also been performed on the high and low pressure limits aiming at having linear behaviour over a wide pressure range [62, 63] and on the perturbations to the signal in hot cathode B-A gauges which contribute to errors of gauge measurement. With a B-A gauge pressures as low as 10^{-9} Pa may be detected.

3. Measurement Of Extreme-High Vacuum XHV (10^{-10} Pa $> p > 10^{-13}$ Pa)

XHV measurement story belongs mostly to the need of realization vacuum conditions corresponding to continuously decreasing pressure in the particle accelerators, storage rings and fusion systems [47] with continuously improved vacuum pumps until the chambers themselves became the pumps (getters strips). Two ways were followed related to hot or cold cathode.

Hot cathode ion gauges

On a typical Bayard-Alpert gauge 10^{-8} Pa may be the lowest measurable pressure if $\approx 10\%$ error can be accepted. Since one of the main physical factors, among many others, affecting the ion gauges is related the photoemission of electrons from the ion collector one goal was to measure the X-ray photocurrent or to reduce its value by making the collector less and less accessible to the X rays from the grid. But, as indicated in par. 2., the residual current is not due only to X-ray since it may be defined as the collector current resulting from all processes

except ionization/excitation of gas phase and has the three previously mentioned main components [58]. In 1960 P. A. Redhead first described a modulated B-A gauge [57] in which, as in all other types of hot cathode ion gauges, an electrode similar to the collector is added in the grid space as modulator to measure the residual current and to evaluate the true ion current, with appropriate choices of the potential of this electrode. Many researches have been performed on the way of modulating B-A ionization gauges as well as gauges having different geometry [64,65,66,67] with the aim of separating the ions from gas phase from the positive current due to other sources.

An ion gauge for pressure below 1×10^{-8} Pa was described by Helmer and Hayard in 1966 [68] in which (figure 2e). It was a commercial nude Bayard-Alpert in which the ion collector was moved to outside the grid and the ions driven by means of electrostatic fields; the end of the grid cut off to permits the ions to reach the collector located at the end of 90° electrostatic energy analyzer and with suppressor grid installed between the collector and the collector slit. With a negative voltage applied to this slit the electron current from the collector is suppressed. In a stainless steel system pumped down by sputtering and titanium sublimation pump, baked for 12 h at 400°C pressure as low as 5×10^{-11} Pa was measured. The performances of commercially available [69] Helmer gauges were studied at PTB in 1988 [70] and a X-ray limit $< 4 \times 10^{-11}$ Pa was defined and the limit due to filament evaporation to about 6×10^{-11} Pa. The Helmer gauge as produced by Varian was improved by C. Benvenuti and M. Hauer [71] by using thoria coated tungsten cathode instead of the simple tungsten cathode and by enlarging the grid diameter and several other geometrical improvements. The authors reported that the modified Helmer gauge was able to measure pressure in the 10-12 Pa range (with an electron current of 3 mA an ion current of 10-15 A could be detected). The C. Benvenuti conclusion was "IHG (Improved Helmer Gauge) provides accurate measurements in the 10-14 torr range, but it perturbs the pressure to be measured" probably measuring its own outgassing [72]. This kind of gauge is still used at CERN for XHV measurements.

Pressure as low as 3×10^{-11} Pa with emission current $< 0,1\text{mA}$ was measured with a large-angle ion deflection ($254,6^\circ$) [73, 74].

Another solution to lower the pressure range is represented by the extractor gauge (figure 2 d and e) whose first description was made by P. A. Redhead in 1966; this gauge showed a $< 3 \times 10^{-11}$ Pa X-ray limit and was capable of measuring pressures lower than 10^{-13} Pa with channeltron electron multiplier [75]. The ring shaped filament was thoria coated tungsten, the top of the grid was closed while the bottom was open and operated as a shield. Below the shield an hemispherical electrode (ion reflector) at the same potential as the grid was located; the collector was a short wire projecting through a small hole in the center of the ion-reflector. A modulator electrode as a fine wire penetrated in the top of the grid. The useful range was considered between 10-2 Pa and $\approx 7 \times 10^{-11}$ Pa.

To gain even few percent in the very low pressure a lot of work was required and several gauge modifications, as for example with the axial symmetric transmission gauge [76] with which the estimated lower limit was about 10^{-12} Pa or lower.

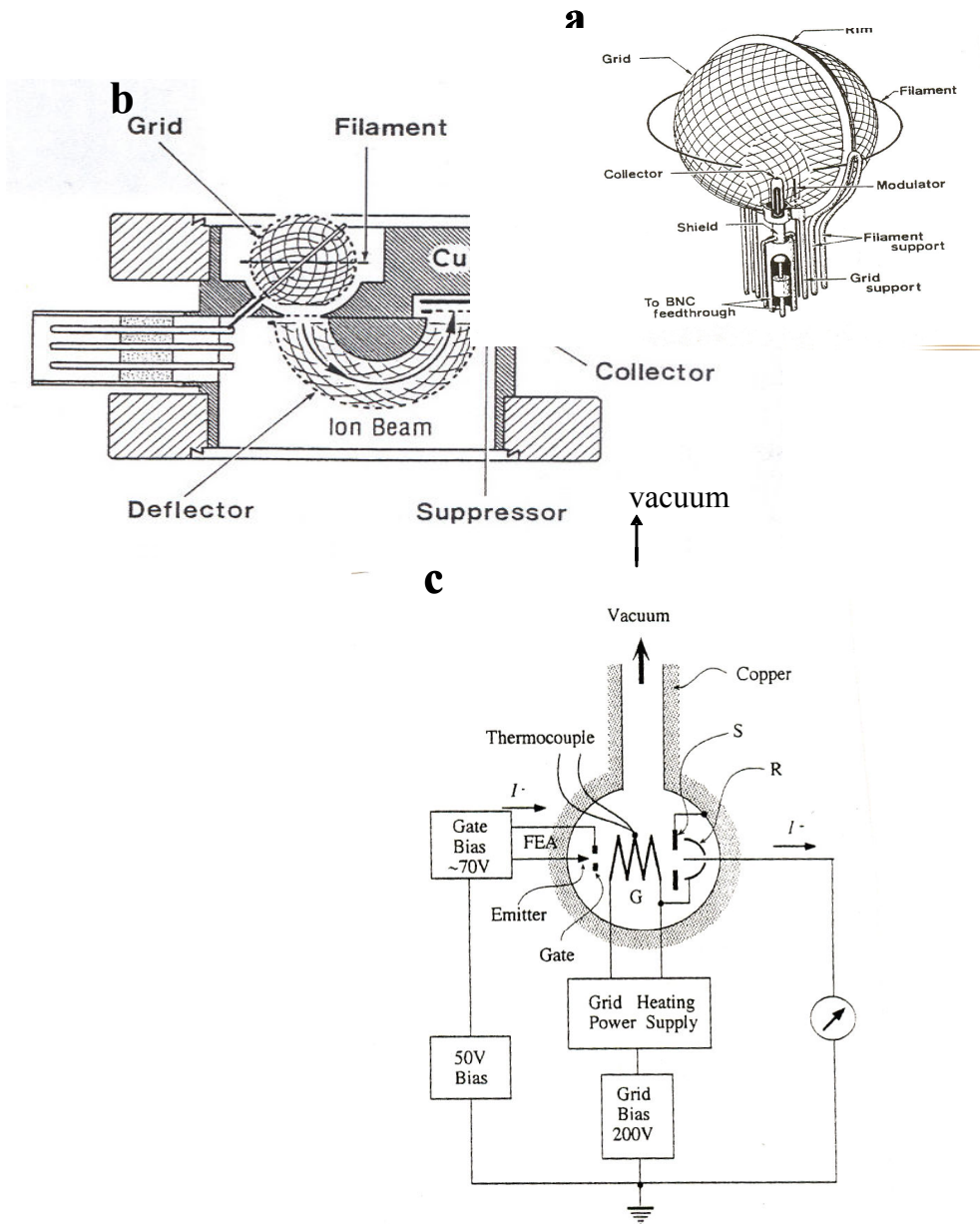


Figure 3 Schematic of some Watanabe hot cathode ionization gauges: a point collector and spherical grid (from figure 1 of ref [79] reproduced with J. Vac. Sci. Technol permission); b spectroscopy gauge (from figure 2 of ref [80] reproduced with J. Vac. Sc.Technol permission); c heated-grid extractor (from figure 7 ref [64] reproduced with Vacuum permission)

J. M. Lafferty [77] proposed a hot cathode magnetron gauge (figure 2f) whose geometry was made in such a way that if the magnetic field is not applied the gauge operates as a conventional triode; if a suitable magnetic field is applied the

electrons may travel on a longer path increasing the number of ion-electron pair production and so the emission current can be lowered. It has in common with other ionization gauges instability problems and linearity range. An axial-emission magnetron suppressor gauge [78] was claimed to be capable of detecting pressure as low as 6×10^{-13} Pa with thoria-coated carbonized tungsten filament and up to $1,33 \times 10^{-2}$ Pa.

By considering that the X-ray limit of the B-A gauge was decreased of 500 times by reducing the collector dimensions, a further reduction could be obtained by reducing the collector dimension to “zero”. Since it is impossible to realize a zero dimension collector F. Watanabe [79] adopted the virtual point collector (figure 3a) that consists on a wire (tungsten) surrounded with a very thin sleeve projected into a spherical grid (26 mm diameter) with a ring filament (30 mm diameter from 0,15 mm thoriated tungsten wire); a modulator electrode can be switched from grid potential to ground and ion current to the point collector can be modulated by 95 %. The X-ray limit resulted to be $2,5 \times 10^{-11}$ Pa. For an appropriate choice of gauge material and with the aim of reducing outgassing and EDS effect F. Watanabe compared the desorption rate of several materials (stainless steel, silver, gold, aluminum and copper) and he found that Cu was the most suitable; so that in 1992, he made another ion gauge [80], the Ion Spectroscopy Gauge (figure 3b), to separate gas phase ions from ESD ions. It has spherical ionizer immersed in a copper block and thermally separated from the other electrodes. The estimated X-ray limit is claimed to be $2,5 \times 10^{-13}$ Pa with emission current of 5 mA and a measured pressure of 10^{-12} Pa. Finally a heated-grid extractor gauge [64] was realized aiming at suppression of ESD species (figure 3c); the grid is heated by passing the current through it to degas. It is made from platinum-clad molybdenum wire; it is mounted in a small copper chamber. From his long experience in XHV measurements F. Watanabe [64] went to the conclusion that “the X-ray limit can be minimized to below 10^{-13} Pa because it is stable error, but, as for the ESD and the outgassing errors, these two still remain un-eliminated, though they have been reduced by two orders of magnitude in the past 20 years. The imperfect elimination is due mostly to their very unstable nature, as compared to the X-ray error”. Consequently, very reliable XHV pressure measurement devices do not seem yet available [81].

Cold cathode ion gauges

As mentioned in par 2. several cold cathode magnetron gauges have been developed [50] and are also commercially available. They have only two un-heated electrodes (a cathode and an anode) between which the so-called cold discharge occurs and is maintained even at very low pressure. They are also named “crossed field” gauges due to the presence of electric and magnetic fields perpendicular each to the other (up to 6 kV and more than 1×10^{-4} T). Since the path of the electrons is increased by a magnetic field one electron may collide with several atoms with consequent production of positive ions and other electrons. The ions produced in the discharge are accelerated to the cathode where they are partly retained and partly cause sputtering of the cathode material. In 1959 Redhead

described a magnetron gauge (figure 4b) and its behaviour [50]; different position of the electrodes is also possible (inverted magnetron gauge) in which anode and cathode are exchanged. In the magnetron gauges for pressure lower than about 10^{-7} Pa the ion current is a $i = Kpn$ function of the pressure, with n ($0,01 \leq n \leq 1,2$) depending on the magnetic field. Those gauges have a sensitivity higher than in the B-A- gauges. Several studies have been performed even recently on the instabilities of the gauge output [82,83,84] and errors caused by external electron source [85]. The drawback represented by spurious currents due a single feedthrough was overcome by N. T. Peacock and R. N. Peacock [86] by connecting an inverted magnetron gauge tube with two feedthroughs to separate the anode high voltage and the cathode current. In addition, if the power is interrupted, cold cathode gauges may need several hours to re-establish the discharge and so an external source may be necessary [87, 88].

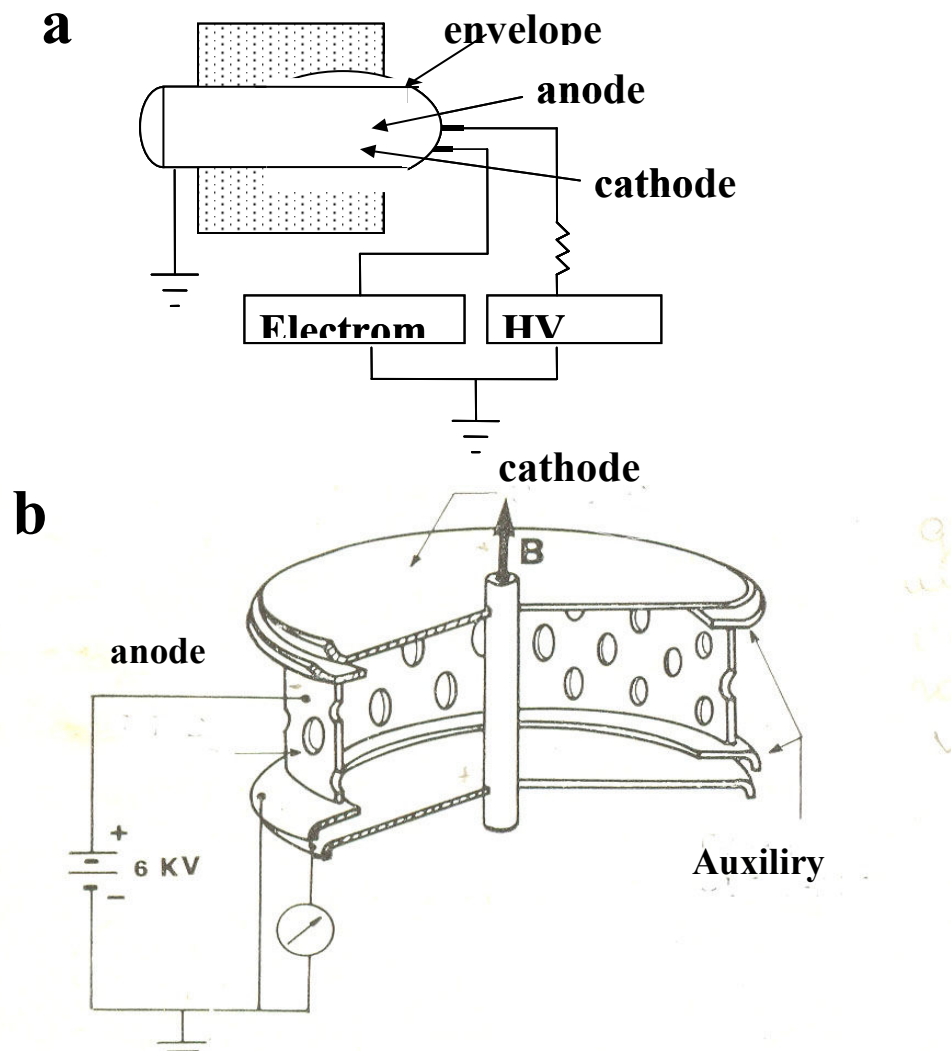


Figure 4 magnetic field cold cathode ionization gauges: a Penning gauge; b Redhead magnetron (from figure 1 ref [50])

4. Miniaturization And New Methods

With the advancements of microsystems miniaturization of gauges to millimeter or even micrometric dimensions has been realized, even if in some cases fundamental physics principles do not allow drastic miniaturization. As described by St. Wilfert and Chr. Edelman [89] two reasons may be considered for reducing drastically the dimensions of the gauges:

Table 2 Pressure ranges of some miniature ionization gauges
(table IV from reference [89])

Pressure range/Pa	Measuring principle/device
$>10^{-8}$ -4	Miniature B.A gauge
10^{-3} -1	Ionization gauge with a SiC junction electron emitter
6×10^{-10} - 10^{-3}	Miniature extractor gauge of Redhead type
10^{-10} - 10^{-3}	Mini-axial-emission ionization gauge with ring anode
10^{-8} - 10^{-1}	HPS mini-ion gauge (MKS instrum)
7×10^{-7} -7	Micro-ion gauge (Granville-Philips)
10^{-4} - 10^{-1}	Miniature cold cathode ion gauge of magnetron type
10^{-1} -6	Miniature cold cathode ion gauge of Penning type Inverted magnetron microelectronic vacuum gauge

- to have active sensors heads with the controller mounted onto the gauge itself, to reduce the influence of the cables;
- to lower the volumes of the gauges to mount several sensors in the same system and to produce large number of these gauges with the modern microelectronic technologies at low price.

It is relatively easy to produce such gauges for pressure down to 1 Pa (diaphragm gauges), 10^{-3} (friction gauges) or even 10^{-5} Pa (thermal thin film sensors), but it seems less practicable to miniaturize the ion gauges without drastic reduction of their sensitivity. Some papers describe pure theoretical suggestions. Table 2 is a summary of published information on the miniature ionization gauge. At present the main possible advantage of such gauges is represented by the price, but great reduction of volume of ionization gauges is not required by many applications at industrial or research level.

Frequently miniaturization is represented by the inclusion of the control unit into the sensor head to considerably reduce the cable length. The only possibility of reducing the dimension of the ion gauges is represented by the electron source that has been substituted by a microtip [90], by cascade static lens [91] or by field emission nanotubes [92]. In this last case the whole structure of the gauges is miniaturized [the gap between the grid and the collector is 0,7 mm (figure 5a)], but the range is considerably restricted (to about 10^{-3} Pa). For the moment the miniaturization techniques do not seem applicable to the very low pressure gauges and do not represent an advantage for what concerns metrological characteristics.

Some researchers tried to improve the gauges for what concerns their greatest drawbacks represented by::

- hot electron source (hot cathode ionization gauges)
- high electric and magnetic field (cold cathode ionization gauges).

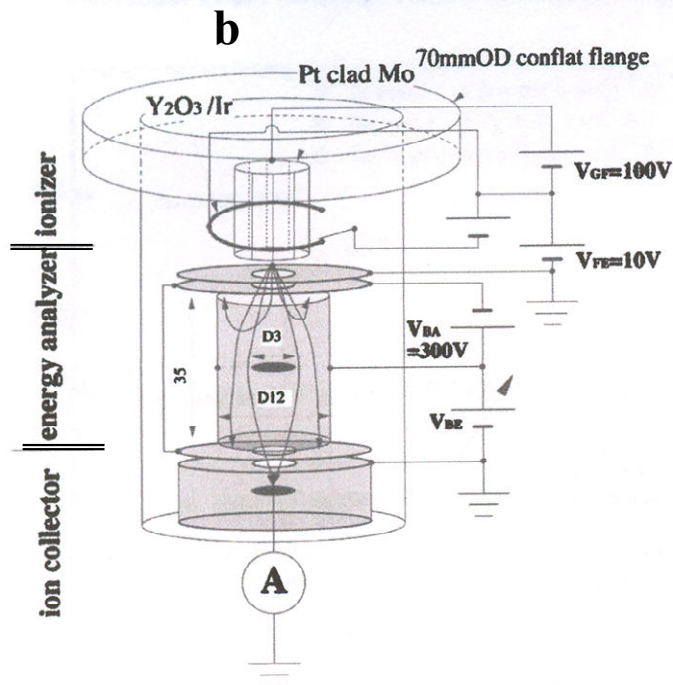
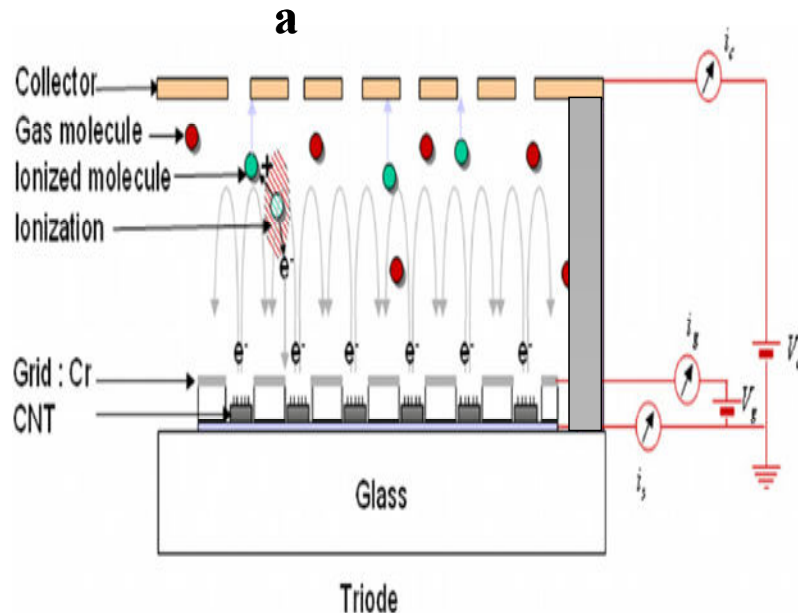


Figure 5 New gauges: a miniature ionization gauges with field emission nanotubes (from figure 2 ref [92] reproduced with Metrologia permission); b axial transmission gauge equipped with Bessel-box energy analyzer (from figure 1 ref [93] reproduced with J. Vac. Sci.Technol. permission)

Therefore, the novelties are related to the production of the electrons with consequent lowering of the emission current as by using cascade static lens [92] for (8×10^{-8} – $1,5 \times 10^{-5}$) Pa range or to the use of a Bessel box energy analyzer [93] in B-A gauge structure to separate the ESD ions from the gas phase ions for pressure measurements down to 5×10^{-10} Pa (figure 5b). Ignition of inverted magnetron gauges has been enhanced by using radioactive isotopes (94): the ignition time resulted to be 10 min at a pressure of 5×10^{-9} Pa with americium source.

5. Needs Of Calibrations

Knudsen calibrated his thermo-molecular gauge by comparing its output with a generated/measured pressure in the so called static expansion system. From that time primary systems were built at industrial or research laboratory level but, at present, they are almost available in the National Metrology Institutes (NMI). By developing instrumentation in which the pressure is measured directly as a force on a surface or systems in which the pressure is calculated by the gas laws as function SI base quantities [95].

For pressure lower than 103 Pa down to $\approx 10^{-4}$ Pa, several systems at NMI (Germany, India, Italy, Korea, UK, etc) are available based on static expansion or Knudsen method by single or multiple expansion. With multiple expansion the pressure ranges from few pascal down to (10^{-5} - 10^{-6}) Pa [96] with relative uncertainty of few parts in 10^{-4} or 10^{-3} , whose value increases with number of the volumes involved. For pressure lower than 10^{-1} Pa down to 10^{-7} Pa, systems are available based on the continuous or dynamic expansion first described by Normand [97]. Such devices are, generally, made of a calibration chamber connected to the pumping chamber through a conductance. The pumping system is, frequently, a combination of mechanical and turbo molecular pumps and the conductance is as near as possible to a hole in a thin wall to be able to calculate its value by analytical [98, 99] or by Monte Carlo methods [100, 101]. Those systems need to be equipped with a gas flow generating and measuring unit that is frequently based on the variable-volume and constant- pressure method [102, 103]. In the simple realization the lower limit may be between 10^{-6} and 10^{-7} Pa. For lower pressure, on the historical point of view it should be mentioned the PTB molecular beam system realized several years ago by G. Messer and G. Grosse [104, 95]. The system could reach (10^{-10} - 10^{-11}) Pa but, due to problems of outgassing, the calibration pressure had to be limited to 10^{-9} Pa. The expanded uncertainty was 7 % in the (10^{-10} - 10^{-7}) Pa range. It must be noted that this system needed to be equipped with calibrated ionization gauges.

For the XHV calibrations for the moment the only possibility is connected with the use of the continuous expansion method with gas flow divider [95, 105]. But even with flow divider the primary systems do not cover the full measuring range of all available ionization gauges. To study the ion gauge behavior several small systems have been built reaching [106] lower than 10^{-11} Pa and new gauge have been developed. One of the main drawback in reaching very low pressure is represented by the outgassing from the walls of the chamber so that studies have

been performed with a TiN coated vacuum chamber pumped by a TMP in which the ultimate pressure was 1×10^{-9} Pa.

6. Conclusion

On macroscopic scale vacuum seems quiet but looking in it more deeply it may appear as a stormy sea boiling of many types of strange displays; as a consequence the gas composition is changeable and to measure the pressure is at least questionable in the range where ionization phenomena depending on the pressure must be considered. From decades reaching and measuring pressure in the XHV has been a challenge and some researcher succeeded by constructing stainless steel vacuum systems covered with non evaporable getter material which may offer the possibility of moving from UHV to XHV and ionization gauges having various geometry. Once the gauge is realized its appropriate calibration in the full working range is not yet possible.

To realize XHV calibration systems is time and money consuming, therefore it should be evaluated if we really need to have many calibration devices around the world in this range or if it could be better to decide all together how many of such systems are really necessary and where they should be realized. In this way it could be possible to apply all our efforts on their realization and on the study of good transfer gauges and handling procedures. In fact, as it is well known from the results of international comparisons up now realized and published, reliable accepted transfer gauges are available in the low [(10^5-10^3) Pa, pressure balances, quartz elices, resonant structures], medium [(10^3-10^{-1}) Pa, capacitance diaphragm gauges], high [$10^{-1}-10^{-3}$ Pa, spinning rotor gauges] vacuum range. For pressure lower than 10^{-3} Pa few studies have been published and in the XHV range few calibrations are available and based on comparisons of new design gauges with B-A and extractor gauges.

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