Hot Cathode Gauges for UHV Pressures

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The most common gauges for UHV pressure measurements are Hot Cathode Gauges. These ionization gauges are known to be limited in the lower range due to x-ray and ESD (Electron Stimulated Desorption) effects. I will give a short review of the function of Bayard-Alpert and Extractor gauges as well as the influence of the limiting effects and ways to optimize the gauge design. Some information regarding calibration and accuracy are added.
Hot Cathode Gauges for UHV pressures

Indirect Vacuum Measurement

Ionization Gauges use the probability of gas ionization to determine the particle number density.

The particle number density is related to the pressure.
Hot Cathode Gauges for UHV pressures

Principle of Ionization

- Gas molecule
- Ion +
- Electron -
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Principle of Hot Cathode Ionization

Emission current

Ion current

Cathode
Electron Source
50 Volt

Anode
Electron Drain
200 Volt

Ion Collector
Electron Source
0 Volt

Gas

Ion +

Electron -
Hot Cathode Gauges for UHV pressures

Bayard-Alpert

Hot cathode outside
Anode in spiral form
Centric Ion Collector
Measuring Range app.
$1 \times 10^{-2}$ bis $1 \times 10^{-11}$ mbar
Hot Cathode Gauges for UHV pressures

Hot Cathode Ionization Gauges

The hot cathode ionization gauges initiate a constant electron flow from the glowing cathode (electron source) to the anode (electron drain). These electrons hit a pressure dependent quantity of gas molecules. The gas molecules become positive ions, which cause a pressure related current on the ion collector.

Formula: \( p = \frac{i^+}{(i^- \cdot C)} \)

\( C = \text{System-Sensitivity} \)
Hot Cathode Gauges for UHV pressures

Relation Ion Current to Pressure

![Graph showing the relation between pressure (mbar) and ion current (amps). The graph indicates a linear increase in ion current with decreasing pressure, up to a peak, after which the current decreases.]
Hot Cathode Gauges for UHV pressures

Upper Limit

The measuring range of hot cathode ionization gauges is limited, when the particle number density becomes so high, that the positive ions cannot reach the ion collector in a pressure dependent quantity. The ion current is reduced even when the pressure increases.

To extend the lifetime of the cathodes, a pressure dependent emission start is requested.
The lower limit of hot cathode ionization gauges is caused by the X-ray effect. Electrons emitted from the cathode impinge on the anode and release photons (soft X-rays) there. These X-rays trigger electrons from ion collectors surface and causing an offset current.
Hot Cathode Gauges for UHV pressures

X-Ray Limit

![Diagram of electron flow and pressure graph]
Hot Cathode Gauges for UHV pressures

Improved Ionization Gauge

For measuring pressure in extreme high vacuum was a system developed which reduces the X-ray limited by shielding the ion collector. To keep the ion current on an acceptable level, the positive ions are focussed by an electrical system.

This Extractor system extends the measuring range down to $2 \times 10^{-12}$ mbar. The upper limit is about $1 \times 10^{-4}$ mbar.
Hot Cathode Gauges for UHV pressures

Extractor System: Sectional Drawing

C- Cathode
A- Anode
I- Ion Collector
R- Reflector

C= 100 V
A= 220 V
R= 205 V
I= 0 V
Another effect of hot cathode ionization gauges is called Electron Stimulated Desorption (ESD). Molecules on the anodes surface are desorped by electron bombardment. Some are emitted as neutral molecules others as positive ions.
Hot Cathode Gauges for UHV pressures

Electron Stimulated Desorption

Particles leave the surface as molecules and cause an increasing pressure.

Others become ions and will cause a non pressure related ion current.

Both influence the pressure indication.
Electron Stimulated Desorption

Hot ion gauges are equipped with a “degas” function.

The anode is switched to a higher electrical potential and the emission current is increased. The anodes temperature is increased and particles are desorped.
Hot Cathode Gauges for UHV pressures

Electron Stimulated Desorption

The quantity of particles on the surface is reduced after degassing, but adsorption and desorption is an ongoing process. The ESD effect still remains, but the value can be influenced by the gauge design: size of anode, material of anode and emission current.
Hot Cathode Gauges for UHV pressures

Degas of BA and Extractor

Pressure Indication

Extractor

Degas

BA

Time

p = constant
Hot Cathode Gauges for UHV pressures

Outgassing Effects

The hot cathode produces outgassing effects:
• from the hot filament itself
• from heated surfaces of the gauge head
• from surrounding walls, heated by radiation
Hot Cathode Gauges for UHV pressures

Reduced Outgassing

The outgassing can be reduced by:

• low temperature filament (Yttriumoxid coated Iridium)
• material of electrodes (low gas adsorption)
• material of flange and walls (high heat conductance and low emissivity)
Hot Cathode Gauges for UHV pressures

Summary

An extractor gauge is a better choice for UHV measurement compared to a BA gauge not only because the lower x-ray limit. Also less ESD effects and the lowest thermal outgassing of all available hot cathode gauges recommend it for measurements lower $10^{-10}$ mbar.
Hot Cathode Gauges for UHV pressures

Accuracy

During the final inspection the system sensitivity of each gauge head is measured and documented.

This also called calibration factor at a specific emission current is measured in HV. The reference is, depending on ISO 9000, traceable to national standards.

Together with the knowledge about the electronics a specification can be given:

Uncertainty 10% in a range from x to y.
Hot Cathode Gauges for UHV pressures

Relation Ion Current to Pressure

![Graph showing the relation between pressure and ion current for hot cathode gauges in UHV pressures. The graph plots pressure in mbar on the x-axis and ion current in amp on the y-axis. The curve indicates a linear increase in ion current with decreasing pressure until a peak is reached, after which the current decreases.](image-url)
Hot Cathode Gauges for UHV pressures

Calibration

The calibration in a certified calibration laboratory allows to improve the information regarding traceability to national standards. The complete gauge is compared to qualified references over a wide pressure range.

Calibrations down to 1E-9 mbar are possible.
Quadrupole Mass Spectrometers under UHV/XHV Conditions

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Quadrupole mass spectrometers are widely used as Residual Gas Analysis in various pressure regimes to determine the partial pressures of the gases present. For gas analysis in UHV / XHV special care has to be taken to obtain high sensitivity and low degassing rates. Specific ion source, and detector designs have to be used to analyze partial pressures at these low total pressures, and special vacuum treatment has to be applied to reduce the outgassing rate, especially that of Hydrogen.

A special design of the quadrupole mass spectrometer allows for high resolution measurements in the low mass range. For this the mass spectrometer is operated in the so-called "second stability region", where a higher DC / RF ratio is applied to the rod system. Mass resolution in the order of 400 at mass 4 m/e can be achieved, which allows for the separation of Helium and Deuterium. Due to technical reasons the mass range of such instrument is limited to <20 m/e.
Quadrupole Mass Spectrometers under UHV / XHV conditions

Norbert Müller, INFICON AG, LI-9496 Balzers

*International Workshop on Extreme High Vacuum - Application and Technology (X-VAT)*
Requirement for KATRIN:

- Partial pressure measurements from E-3 mbar to E-11 mbar with determination of Hydrogen isotopy

- Quadrupole Mass Spectrometry under UHV conditions
- High resolution Quadrupole Mass Spectrometry
- Alternatives (?)
Specific analyzer design for UHV applications

- High conductance to the vacuum chamber
  - Open structure ion source
- Low outgassing rate
  - Selection of material
  - Pretreatment of all components
  - Degas function of the ion source for conditioning
  - Minimization of stray ions
- High sensitivity
  - Use of Secondary Electron Multiplier
- Minimum sensitivity to background effects
  - Special detector geometry (90° off axis)
  - Ion counting
RGA under UHV conditions

Grid ion source

- Open design
- Two filaments (W)
- Low degassing rate
  - Minimum amount of material
  - Pt-Ir wires for formation chamber
  - Molybdenum filament holders
- Easy to degas via electron bombardment
- Filaments on positive potential
commonly used technique

Electrons can impact onto the chamber walls --> electron impact desorption

Emission current about 1 mA

advantage of a biased system

Electrons only can impact onto the anode, if there is electron impact desorption, then only from well defined surfaces

Voltages
RGA under UHV conditions

In Situ Analysis

Cathode always positive with respect to ground
Grid Ionsource

Flight paths of electrons

Quadrupole rod system

Entrance aperture

Filament

Formation chamber
Grid Ionsource

Flight paths of electrons
Grid Ionsource

Flight paths of positive ions

- Quadrupole rod system
- Entrance aperture
- Filament
- Formation chamber
Grid Ionsource

Flight paths of positive ions
RGA under UHV conditions

90° off axis arrangement

efficient suppression of
- photons
- fast neutral particles
- stray ions

![Diagram of a mass filter and Faraday cup with labels: mass filter, Faraday cup, mounting flange, deflection unit, housing, SEM 217]
**RGA under UHV conditions**

- **Ion Detection**
  - Discrete Dynode SEM
  - Bakeable to 400°C
  - for analog amplification and for pulse counting
  - Low noise (< 0.1 cps)
RGA under UHV conditions

- Vacuum annealed analyzers
  - Material: Stainless Steel 1.4429 ESU
  - Degassing under vacuum
    - \( p < 1 \times 10^{-5} \) mbar
    - \( T : 900 - 925 \) °C
    - \( t > 3h \)
  - Weld in of feedthroughs
  - Bakeout
    - Total pressure: \( < 1 \times 10^{-7} \) mbar
    - PP Hydrocarbons: \( < 1 \times 10^{-11} \) mbar
    - \( T = 400 \) °C
    - \( t > 10h \)

- Resulting outgassing rate: \( < 1 \times 10^{-10} \) mbar*l/s (with filament on)
RGA under UHV conditions

In Situ Analysis

Typical UHV spectrum

\[ p = 2 \times 10^{-12} \text{ mbar} \]

RGA under UHV conditions

Conditions as in previous figure, more sensitive amplifier setting
⇒ Detection limits in the E-15 mbar- range

P = 2E-12 mbar
RGA under UHV conditions

Most frequently observed Surface Ion Peaks

<table>
<thead>
<tr>
<th>Mass (m/e)</th>
<th>Component</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>H+</td>
</tr>
<tr>
<td>16</td>
<td>O+</td>
</tr>
<tr>
<td>19</td>
<td>F+</td>
</tr>
<tr>
<td>23</td>
<td>Na+</td>
</tr>
<tr>
<td>28</td>
<td>CO+</td>
</tr>
<tr>
<td>35,37</td>
<td>Cl+</td>
</tr>
<tr>
<td>39</td>
<td>K+</td>
</tr>
</tbody>
</table>

Grid Ionsource

Flight paths of positive ions desorbed from the surface of the ion formation chamber
Specially designed Quadrupole Mass Spectrometers are capable of reliably analyzing gas compositions in the UHV / XHV conditions.
High Resolution MS

Specific requirements for KATRIN

⇒ Separation of Hydrogen components

<table>
<thead>
<tr>
<th>Mass</th>
<th>Component</th>
<th>Exact mass value [u]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>H+</td>
<td>1.0078252</td>
</tr>
<tr>
<td>2</td>
<td>D+</td>
<td>2.014102</td>
</tr>
<tr>
<td></td>
<td>H2+</td>
<td>2.01565</td>
</tr>
<tr>
<td>3</td>
<td>3He+</td>
<td>3.016030</td>
</tr>
<tr>
<td></td>
<td>T+</td>
<td>3.016050</td>
</tr>
<tr>
<td></td>
<td>HD+</td>
<td>3.021825</td>
</tr>
<tr>
<td></td>
<td>H3+</td>
<td>3.023475</td>
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<tr>
<td>4</td>
<td>4He+</td>
<td>4.002600</td>
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<tr>
<td></td>
<td>HT+</td>
<td>4.023875</td>
</tr>
<tr>
<td></td>
<td>D2+</td>
<td>4.028204</td>
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<tr>
<td></td>
<td>H2D+</td>
<td>4.029650</td>
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<tr>
<td>5</td>
<td>DT+</td>
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<td>H2T+</td>
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<td></td>
<td>D2H+</td>
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<td>T2+</td>
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<tr>
<td></td>
<td>D3+</td>
<td>6.042</td>
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<tr>
<td></td>
<td>12C++</td>
<td>5.999</td>
</tr>
</tbody>
</table>
High Resolution MS

In Situ Analysis

QMA 410 with Cross Beam ion source and 90° off axis SEM

30 cm ruler for comparison
High Resolution MS

16 mm rod system for highest resolution, stability and transmission (e.g. He/D₂ separation)

8 mm rod system for High-End RGA and analytical applications

6 mm rod system for common RGA
High Resolution MS

Cross Beam ion source with magnets

- Two filaments
- Easy to degas
- Good ion focussing
- Bakeable to 300°C
Cross Beam Ionsource

Flight paths of electrons

- Quadrupole rod system
- Entrance aperture
- Focus
- Grounded electrode
- Extraction
- Formation chamber
- Filament
- Wehnelt
Cross Beam Ionsource

Flight paths of positive ions
Cross Beam Ionsource

Flight paths of positive ions
High Resolution MS

In Situ Analysis

Contribution He to D2 approx. 100 ppm
Sensitivity approx. 1E-4 A/mbar

Attainable resolution using „standard operation parameters“
**High Resolution MS**

In Situ Analysis

\[
a = \frac{8eU}{mr_0^2 \omega^2}
\]

\[
q = \frac{4eV}{mr_0^2 \omega^2}
\]
High Resolution MS

In Situ Analysis

Abscissa: Mass * 100
High Resolution MS

In Situ Analysis

Contribution D2 to He < 1ppm

Sensitivity approx. 5E-6 A/mbar
High Resolution MS

Attainable resolution at mass 3 m/e
High Resolution MS

In Situ Analysis

Resolution (barely) sufficient to resolve all possible components on mass 3 m/e
Specially designed Quadrupole Mass Spectrometers are capable of resolving overlapping components in the low mass range, but show reduced sensitivity ⇒ still sufficient for KATRIN?
Alternative (?)

Proposal from R. Dobrozemsky and G.W. Schwarzinger

Use of different Electron Energies and different Ion Energies to create a multidimensional matrix

R. Dobrozemsky and G.W. Schwarzinger
Ionization cross sections of different species
Alternative (?) | In Situ Analysis

Deuterium 2 m/e : 1.5 %
        4 m/e : 100 %

from: Wiley library of Mass Spectral Data

Ratio strongly depends on electron energy
⇒ Literature data are taken at electron energies of 70 eV

NIST Chemistry WebBook (http://webbook.nist.gov/chemistry)
⇒ Double ionization / fragmentation dependence on electron energy

R. Dobrozemsky and G.W. Schwarzinger
Alternative (?)

⇒ Fragment ions get additional starting energy

R. Dobrozemsky and G.W. Schwarzinger
**Alternative (?)**

Variation of Electron Energy by potential V2 (Cathode potential)

Definition of Ion Energy by potential V4 (Field Axis potential)
Modern Quadrupole Mass Spectrometers allow for computer-controlled, fast adjustment of all mass spectrometer voltages, matrix calculations can be done easily

⇒ how stable are calibration factors?
⇒ how to perform in-situ calibrations?
Summary

Specially designed Quadrupole Mass Spectrometers:

- are capable of reliably analyzing gas compositions in the UHV / XHV
- have sufficient resolving power to separate overlapping gas components
- special setting / variation of parameters could resolve sensitivity problem in high resolution mode