Vacuum gauges II
Karl Jousten, PTB, Berlin

1. Introduction and history
2. Ion gauges with emitting cathodes
3. Limits, accuracy and problems of ion gauges
4. Quadrupole mass spectrometers – work principle
5. Limitations of quadrupole mass spectrometers
6. Summary
Measurement principle

Ionisation probability of different gas species for electrons between 10 eV and 10 keV
History

The historical vacuum triode gauge originating from the electrical amplifier, the triode, a vacuum electronic device, called the „tube“.

Baeyer 1909
Buckley 1916 useful gauge
Measurement principle

Ionization gauges for different vacuum ranges
Measurement principle

Ionisation gauges: today’s electrode design

Development of burn-out-proof oxide cathodes allowed medium vacuum IG
Trick: Make the ion path as short as possible

Courtesy of INFICON AG
Measurement principle

Combined gauges

INFICON BCG 450 courtesy of INFICON AG
Ion gauges with emitting cathodes

In the first half of the 19th century pumps improved, but the measured pressures with the triode gauges did not, - the readings were “nailed” to about $10^{-7}$ mbar.

Why?

IVC-1, 1947, Nottingham: X-rays?

The original Bayard-Alpert gauge reduced the lower limit by 100
Bayard-Alpert design

Today no glass envelope in Europe:
Nude gauge or metal envelope.

Emission current
0.1 mA … 10 mA

Electron path length
typical 1.5 \( D \)
$$I^+ = SI_e p$$

$$S = \frac{I^+ - I_0}{I_e p}$$

Dropping due to:
$$I_{ion} = 0.002 I_e @ 0.01 \text{ Pa}$$
Mean free path
Disturbing effects in Bayard-Alpert gauges

1: The desired ionisation, but secondary electron

2: Electron stimulated desorption

3: X-ray effect

4: Inverse X-ray effect

Other: e.g. Dissociation of molecules at hot cathode

Cross section of BA gauge
How does the number of adsorbate molecules compare with the ones in the gas phase in XHV?

Anode surface of typically 10 cm²: $10^{16}$ molecules at one monolayer.

To be compared with density at $10^{-8}$ Pa: $10^6$ molecules/cm³. In the gauge about 10 cm³: $10^7$ molecules.

$10^9$ more molecules on the surface: A huge reservoir to annoy you !!!

Degassing feature of IG

Not too small $I_e$ (self-cleaning)
Towards XHV measurements

\[ I^+ = I_e (n + n_{\text{gen}}) \sigma_{e,\text{gas}} \Delta l c_{\text{ion,C}} + I_{\text{x-ray}}^{-} - I_{\text{to collector}}^{-} + I_{\text{ESD}} \]

Strategies to decrease low pressure limit of emissive cathode ion gauges

1. Increase \( \Delta l \) and
2. Increase ion collection efficiency \( c_{\text{ion,C}} \)
3. Reduce X-ray effect \( I_{\text{x-ray}} \)
4. Cancel forward and inverse X-ray effect
5. Reduce ESD ions and separate them from gas phase ions \( I_{\text{ESD}} \)
6. Reduce outgassing of the gauge \( n_{\text{gen}} \)
7. Amplify ion current / Reduce noise
2. Increase ion collection efficiency

• Close the cylindrical grid anode

Closed cylindrical grids have higher sensitivities than open grid structures.
2. Increase ion collection efficiency

• **Close the cylindrical grid anode**

Closed cylindrical grids have higher sensitivities than open grid structures.

The smaller the collector diameter, the larger the effect. e.g. Benvenuti, 1977: 25 mm diameter and closed grid, 40 mm length, 80 µm collector by a factor of 3 higher sensitivity compared to open one.

Benvenuti, Hauer, Nucl. Instr. and Meth. 140 (1977), 453…460.
2. Increase ion collection efficiency

Even closed grids let ions escape.

- Optimize filament potential $V_f$ and grid-to-filament voltage $V_{gf}$.

Collector diameter 50 µm

Collector diameter 10 µm

3. Reduce X-ray effect

**In the BAG:** Reduce the collector diameter

\[ I_{x\text{-ray}} \propto D \]

**Drawback:** Due to conservation of angular momentum ions may spiral around the collector wire until leaving the grid.

**Solution:** *Closed grids are a necessity, but sensitivity decreases anyhow.*

3. Reduce X-ray effect

**In the BAG:** Measure and subtract the X-ray current.

**Modulator** (Redhead) switches between grid (collects no ions) and collector potential (collects ions).

**Drawback:** Also X-ray current is modulated due to the change of electron trajectories.

**Result:** Higher complexity, commercially used, today rarely.
3. Reduce X-ray effect

**Idea of construction:** Decouple ion collection from ionisation region, so that there is no line of sight for X-rays* from grid to collector.

- The extractor gauge
- The Helmer gauge
- The Ion Spectroscopy gauge
- The Oshima gauge
- The AxTran gauge

* X-rays have high reflection coefficient from most metals!
3. Reduce X-ray effect

F. Watanabe, JVST A 9 (1991), 2744…2746.
3. Reduce X-ray effect

Tubulation changes the sensitivity by 5%, but also the X-ray limit by 70%, probably due to the inverse X-ray effect.

4. Cancel forward and inverse X-ray effect

Unstable because of changing SEY with surface changes. Same material of tube and collector for the sake of stability, or vary tube potential.

F. Watanabe, JVST A 9 (1991), 2744…2746.
5. Reduce ESD ions and separate them from gas phase ions

**Effect:** ESD ions have an higher energy than ions generated in the gas phase for two reasons.

Reason 1: Higher initial energy

Reason 2: Electrostatic potential inside the grid.

**Idea:** Analyze the energy of the ions before reaching the collector.

- The extractor gauge
- The Helmer gauge
- The Ion Spectroscopy gauge
- The AxTran gauge

5. Reduce ESD ions and separate them from gas phase ions

The extractor gauge

Ionisation from gas phase

Ionisation from surface

SIMION applied by O.F. Kieler, University of Magdeburg
5. Reduce ESD ions and separate them from gas phase ions

The Helmer gauge


90° for reduction of X-rays, later also to reduce ESD ions.

Measured $7 \times 10^{-11}$ Pa, but capable of $3 \times 10^{-12}$ Pa.
5. Reduce ESD ions and separate them from gas phase ions

The Ion Spectroscopy Gauge by Fumio Watanabe

Spherical grid and ring filament for enhanced space charge

Heated grid

3D-Hemispherical analyser for full collection efficiency
5. Reduce ESD ions and separate them from gas phase ions

The AxTran gauge by ULVAC

X-ray limit $4 \times 10^{-11}$ Pa nitrogen equivalent

Lowest detectable pressure $3 \times 10^{-12}$ Pa
5. Reduce ESD ions and separate them from gas phase ions

**Idea:** Reduce the number of ESD ions by

- Cleaning the grid (electron bombardment)
- Choosing suitable material (platinum-iridium alloy, platinum clad molybdenum, gold. Less good experience with W-re alloy or Au-plated SS)
- Using a clean vacuum system (no hydrocarbons)
5. Reduce ESD ions and separate them from gas phase ions

But there are limits !!!

Desorbed by electrons hitting the grid surface are:

1. Ionized molecules \textit{Can be separated}
2. Ionized fragmented molecules \textit{Can be separated}
3. Neutral molecules* \textbf{Cannot be separated}!!!
4. Fragmented neutral molecules \textit{Difficult to separate (by cracking pattern)}

* Neutrals from a Pt-Ir grid dominate after bake-out!
6. Reduce outgassing of the gauge

**What outgasses thermally?**

- The filament itself
- Material heated by the hot filament
- Anode material heated by electrons (10 mA, 100 V generates 1 Watt !!)
- All other materials in the gauge (that are more or less at “room” temperature)
6. Reduce outgassing of the gauge

Strategies:

a. Choose suitable filament material
b. Reduce heating power of filament
c. Reduce emission current
d. Use cold cathodes
e. Use materials inside gauge and for its tubulation of low emissivity, high thermal conductivity and high heat capacity
f. Degas materials before operating the gauge
7. Amplify ion current / Reduce noise

Secondary electron amplifiers were extensively used by many investigators, even ion counting techniques.

Problems: Outgassing, stability.

Also Lock-in technique was used (Watanabe, 1989).
Comparison of the types of ionisation gauges

Max. deviations in % from 1 st calibration within 6 months

<table>
<thead>
<tr>
<th></th>
<th>EXG</th>
<th>BAG1</th>
<th>BAG2</th>
<th>IMG1</th>
<th>IMG2</th>
</tr>
</thead>
<tbody>
<tr>
<td>N₂</td>
<td>-2.5</td>
<td>-4.3</td>
<td>-3.2</td>
<td>-6.2</td>
<td>+5.9</td>
</tr>
<tr>
<td>Ar</td>
<td>-1.9</td>
<td>-3.8</td>
<td>+3.8</td>
<td>-2.4</td>
<td>+3.1</td>
</tr>
<tr>
<td>He</td>
<td>-5.9</td>
<td>-4.4</td>
<td>-3.6</td>
<td>+8.4</td>
<td>-5.0</td>
</tr>
<tr>
<td>H₂</td>
<td>+9.4</td>
<td>-1.9</td>
<td>-3.6</td>
<td>-1.0</td>
<td>-1.3</td>
</tr>
</tbody>
</table>

From D.Li, K. Jousten, Vacuum 70 (2003), 531...541.
Comparison of the types of ionisation gauges

Outgassing rates in Pa L/s

<table>
<thead>
<tr>
<th></th>
<th>EXG at 1.5mA</th>
<th>BAG1 at 4mA</th>
<th>BAG2 at 1mA</th>
<th>IMG1/IMG2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outgassing rate</td>
<td>2.4$\times$10^{-8}</td>
<td>8.1$\times$10^{-8}</td>
<td>3.0$\times$10^{-8}</td>
<td>none</td>
</tr>
</tbody>
</table>

From D.Li, K. Jousten, Vacuum 70 (2003), 531...541.
## Comparison of the types of ionisation gauges

**Pumping speeds in L/s**

<table>
<thead>
<tr>
<th>Gas</th>
<th>IMG1</th>
<th>IMG2</th>
<th>BAG1 at 4mA</th>
<th>BAG2 at 1mA</th>
<th>BAG2 at 10mA</th>
</tr>
</thead>
<tbody>
<tr>
<td>N₂</td>
<td>0.045</td>
<td>0.065</td>
<td>0.019</td>
<td>-</td>
<td>0.045</td>
</tr>
<tr>
<td>Ar</td>
<td>0.2</td>
<td>0.21</td>
<td>0.067</td>
<td>0.037</td>
<td>0.23</td>
</tr>
</tbody>
</table>

From D.Li, K. Jousten, Vacuum 70 (2003), 531...541.
Problems special to accelerators:

- Radiation (Example: IG close to photon absorber)
- Strong magnetic fields (shielding necessary)
- RF radiation (Example: RF cavity -> shield)
Applications

Problem: IG measures density, not pressure

Sealed-off chamber, cool it down.

\[ \frac{p_2}{p_1} = \frac{T_2}{T_1} = \frac{77}{300} = 0.257 \]

But IG will have same reading!
Applications

The effects of tubulation, conductance, pumping speed, and thermal transpiration
Applications

Orientation of a gauge

![Diagram showing orientation of a gauge with gas flow and pressure indications.](image-url)
Advantages of mass spectrometers:

• Residual gas is automatically analyzed
• X-ray effect is intrinsically reduced
• High sensitivity (multiplier 1E-10 Pa, off-axis multiplier for XHV 1E-12 Pa)

Disadvantages of mass spectrometers:

• Producing significantly residual gases
• Changes gas composition
• Still ESD effect (QMS with energy analyzer before quad, e.g. Bessel-box)
• Higher outgassing than total pressure gauge
• Difficult to calibrate
Quadrupole mass spectrometers

Ion source

Cathode

100 V  30 V

Ion source slit

Quadrupole rods

Resonant ion

Non-resonant ion

90 V (field axis potential) + DC + RF

Detector

Resonant ion

Non-resonant ion
Quadrupole mass spectrometers

Extraction and transmission probability:

Sensitivity of QMS: $10^{-6}$ A/Pa compared to ionization gauge of about $10^{-4}$ A/Pa

Ideally, oscillations are independent of the energy of the ion (energy spread is not critical, simple ion source feasible). But fringe fields have the higher effect the longer the flight time through them. → Ion energy does matter!

Courtesy: Inficon
Quadrupole mass spectrometers

\[ V = +[U + u \cos(\omega t)] + FA \quad FA \text{ field axis potential} \]

\[ V = -[U + u \cos(\omega t)] + FA \]

Stability characterized by two parameters (Mathieu’s equations):

\[ a = 0.194 \frac{zU}{mr_0^2 f^2} \]

\[ q = 0.097 \frac{zu}{mr_0^2 f^2} \]

\[ r_{cyl} = 1.147r_0 \]
Quadrupole mass spectrometers

Stability characterized by two parameters (Mathieu’s equations):

\[ a = 0.194 \frac{zU}{mr_0^2 f^2} \]

\[ q = 0.097 \frac{zu}{mr_0^2 f^2} \]

Solved as eigenvalue problem

Stable states for oscillatory motion for special \(a\) and \(q\)

Symmetry between \(x\) and \(y\)
Quadrupole mass spectrometers

Stability characterized by two parameters (Mathieu‘s equations):

\[ a = 0.194 \frac{zU}{mr_0^2 f^2} \]
\[ q = 0.097 \frac{zu}{mr_0^2 f^2} \]
\[ \frac{a}{q} = \frac{2U}{u} \quad \text{All ions!} \]
\[ \frac{2U}{u} < 0.336 \quad \text{Detection possible} \]

All masses/ions lie on common straight line. Line defines min and max of mass filter.
Quadrupole mass spectrometers

Close to the tip of stability diagram the transmission probability is proportional to the resolution:

\[ T \propto R \frac{m}{\Delta m} = \frac{1.5064}{U/V} \frac{U/V}{(U/V)_{\text{max}}} \]

For \( R=100 \) the voltage ratio is 0.9849
For \( R=110 \) this ratio 0.9863
This difference of 0.14\% corresponds to a 10\% peak change!

Similar stability of \( U/V \) is required for peak stability.
\[ T \propto \text{area above line} / \text{below line} \]
Quadrupole mass spectrometers

To detect higher mass \( U(\text{DC}) \) and \( u(\text{RF}) \) must be increased

\[
\frac{m}{\Delta m} T = \text{const} \quad \Delta m = 1 \quad \rightarrow \quad T = \frac{\text{const}}{m}
\]

\( U(\text{DC})/u(\text{RF}) \) constant

\( U(\text{DC})/u(\text{RF}) \) not constant

H not filtered
Quadrupole mass spectrometers

$S(H_2)_{\text{max}}$

$S(\text{He}) \approx S(N_2)$

$T(m) = 28/m$

Relative sensitivity (to N$_2$) vs. Mass (amu)
Quadrupole mass spectrometers

From ion gauges we are used to that

\[ I^+ = SI_e p \]

For QMS →

Reason:

More electrons → negative space charge in ion source → ion extraction efficiency to the quadrupole and/or transmission probability through the quadrupole is decreased

Lieszkovsky, Filipelli, Tilford, JVST A 8 (1990), 3838…3854
Quadrupole mass spectrometers

<table>
<thead>
<tr>
<th>Gas species</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ar</td>
<td>1,96</td>
<td>1,33</td>
<td>1,56</td>
<td>2,08</td>
<td>1,23</td>
</tr>
<tr>
<td>CH₄</td>
<td>1,18</td>
<td>0,96</td>
<td>1,23</td>
<td>0,77</td>
<td>0,86</td>
</tr>
<tr>
<td>CO₂</td>
<td>1,47</td>
<td>0,77</td>
<td>1,39</td>
<td>1,52</td>
<td>0,94</td>
</tr>
<tr>
<td>N₂O</td>
<td>1,27</td>
<td>0,47</td>
<td>0,91</td>
<td>0,90</td>
<td>0,63</td>
</tr>
<tr>
<td>CO</td>
<td>1,04</td>
<td>0,99</td>
<td>1,05</td>
<td>1,00</td>
<td>0,99</td>
</tr>
<tr>
<td>N₂</td>
<td>1,00</td>
<td>1,00</td>
<td>1,00</td>
<td>1,00</td>
<td>1,00</td>
</tr>
<tr>
<td>H₂</td>
<td>0,61</td>
<td>1,09</td>
<td>1,42</td>
<td>0,31</td>
<td>1,54</td>
</tr>
<tr>
<td>He</td>
<td>0,10</td>
<td>0,35</td>
<td>0,31</td>
<td>0,48</td>
<td>0,40</td>
</tr>
</tbody>
</table>

Sensitivity on gas species for different QMS. Huge differences and hardly related to ionization probability!

Lieszkovsky, Filipelli, Tilford, JVST A 8 (1990), 3838…3854
Quadrupole mass spectrometers

Tracer kept constant at 1E-6 Pa. Matrix gas was increased.

Lieszkovsky, Filipelli, Tilford, JVST A 8 (1990), 3838…3854
About 20 relevant gas species (H₂, He, Ne, Ar, Kr, Xe, N₂, O₂, CH₄ + higher hydrocarbons, CO, CO₂, H₂O, NOₓ, SiH₄, …, SF₆, dry air) for total pressure gauges.

More relevant gases for QMS (even proteins etc.), 100?

For 100: $1.27 \times 10^{30}$ possibilities of combination of gas species!

For 30: $1.07 \times 10^{9}$ possibilities of combination of gas species!

For 10: 1023 possibilities of combination of gas species!

Impossible task!!!
Quadrupole mass spectrometers

Vacuum Technology — Vacuum gauges — Characterization of quadrupole mass spectrometers for partial pressure measurement

Élément introductif — Élément central — Élément complémentaire
Quadrupole mass spectrometers

ISO 14291

Mass resolving power: \( R = \frac{m}{\Delta m} \)

Mass number stability: \( \frac{\Delta m_{\text{peak}}}{\Delta t} \)

Minimum detectable partial pressure: \( \text{MDPP} = \frac{3\sigma_I}{S} \)

Minimum detectable concentration: \( \text{MDC}_x = \frac{\text{MDPP}_x}{P_{\text{carrier}}} \)

Fragmentation factor: \( f_i = \frac{I_i}{I_0} \)
Quadrupole mass spectrometers

![Graph showing mass spectrometry results](image)

- CO$_2$
- Rel. Intensity
- Mass [amu]
- Compounds: $^{12}\text{C}^+$, $^{16}\text{O}^+$, $^{12}\text{C}^{16}\text{O}_2^{++}$, $^{12}\text{C}^{16}\text{O}^+$, $^{13}\text{C}^{16}\text{O}_2^+$, $^{12}\text{C}^{16}\text{O}^{18}\text{O}^+$
Vacuum gauges II

We have discussed

Ionization gauges with emissive cathode
Effects in emissive cathode gauges
Low pressure limits of ion gauges
Problems in applications
Work principles of quadrupole mass spectrometers
Limits of QMS
Calibration and standards
Vacuum gauges II

Questions?