Vybrané kapitoly z elektronové mikroskopie
část 4 – Vakuové systémy pre elektronovou mikroskopii

Marek Talába

Content

- Introduction

- Why we need vacuum in electron microscopes?

- Pumping of vacuum system - Basic vacuum terms and relations

- Vacuum pumps

- Vacuum gauges

- Design of vacuum system in electron microscopes

- Leak checking and Residual Gas Analysis (RGA)

- Summary

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Introduction

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What is vacuum?

- **Vacuum** is space that is devoid of matter.

- An approximation to such vacuum is a region with a gaseous pressure much less than atmospheric pressure.[Wikipedia]

- The quality of a vacuum is measured by the amount of matter remaining in the system.

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Vacuum ranges

- No defined borders, only consensus

- Sometimes in a broader sense

   High Vacuum = any environment < 0.1 Pa

Vacuum range	Pressure in Pa
Low vacuum	$10^3 - 10^2$
Medium vacuum	$10^2 - 0.1$
High vacuum	$0.1 - 10^{-5}$
Ultra high vacuum	$10^{-5} - 10^{-10}$
Extremely high vacuum	$<10^{-10}$

Why we need vacuum?

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Why we need vacuum in electron microscopes?

There are three main reasons why the electron microscope must be operated under vacuum:

- to ensure straight particle movement without collisions

- to protect electron emitter from oxidation and damage by ion sputtering

- to keep clean environment and sample surface

Avoiding Collisions

- Basic parameter describing probability of collision between particles in vacuum is mean free path (MFP).

- The MFP of an electron is the average distance an electron travels between two consecutive scattering events

$$\lambda = (\sqrt{2}\sigma n)^{-1}$$

λ is MFP, σ is Collision cross-section and n is particle density

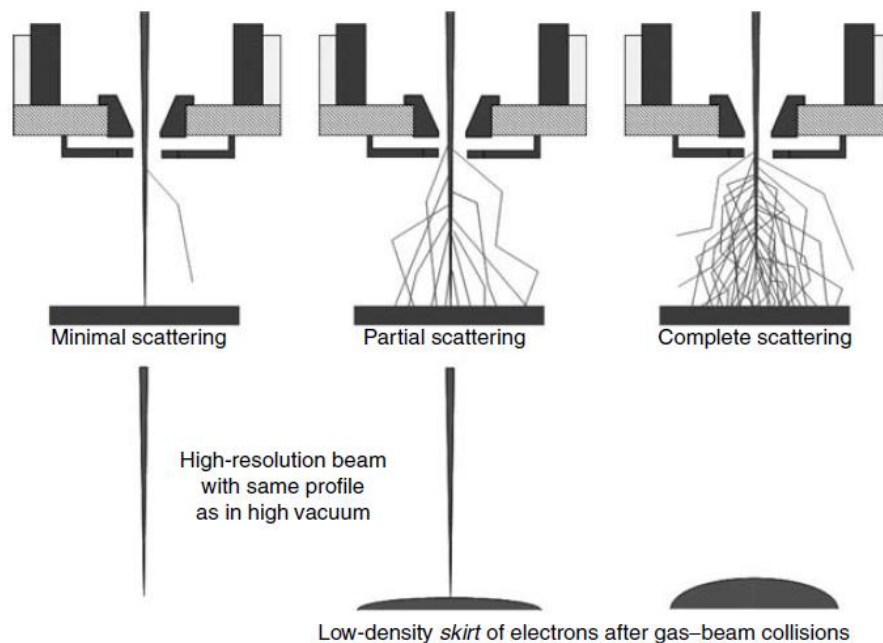- Approximation MFP = $6.4*10^{-3}$ / p   [m; Pa]
- *Example: 1 cm at 0.6 Pa*

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Avoiding Collisions

- Basic parameter describing probability of collision between particles in vacuum is mean free path (MFP).

Vacuum range	Pressure in Pa	Molecules / m3	Mean free path
Ambient pressure	10^5	2.7×10^{25}	~100 nm
Low vacuum	10^3 – 100	10^{25} – 10^{22}	0.1 – 100 µm
Medium vacuum	100 – 0.1	10^{22} – 10^{19}	0.1 – 100 mm
High vacuum	0.1 – 10^{-5}	10^{19} – 10^{15}	10 cm – 1 km
Ultra high vacuum	10^{-5} – 10^{-10}	10^{15} – 10^{10}	1 km – 10^5 km
Extremely high vacuum	$<10^{-10}$	$<10^{10}$	$>10^5$ km

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Electron and ion beam scattering

- Collisions with residual gas deflects particles from straight movement

- Expected beam trajectory (optical path) not valid anymore

Minimal scattering   Partial scattering   Complete scattering

High-resolution beam with same profile as in high vacuum

Low-density *skirt* of electrons after gas–beam collisions

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Emitter Tip Environment

- Oxygen can damage thermionic cathodes

- Suppress ion bombardment of the FEG tip
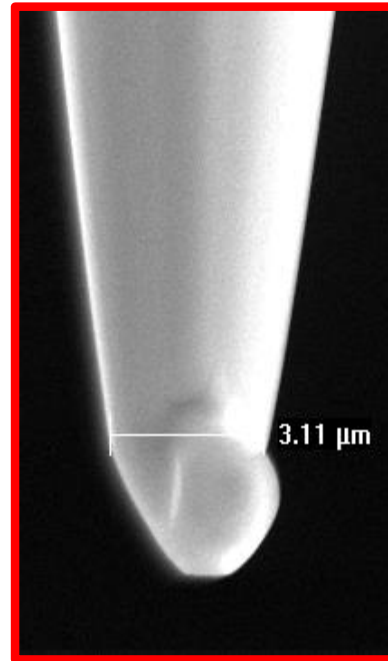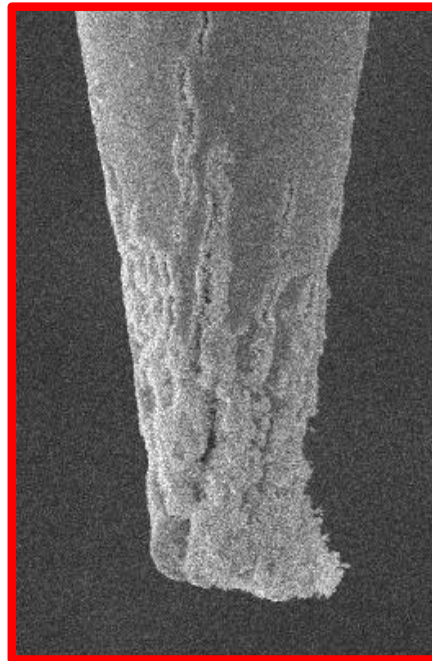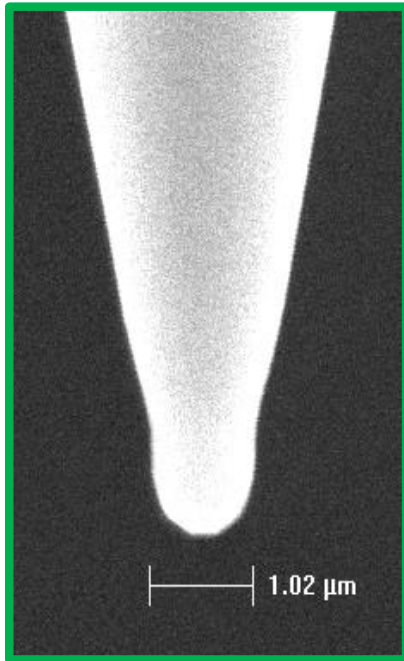
Sample contamination

- Contamination influences high resolution even at high accelerating voltage like on the example below (30 kV)

Contamination grown during single scan!   Clean sample

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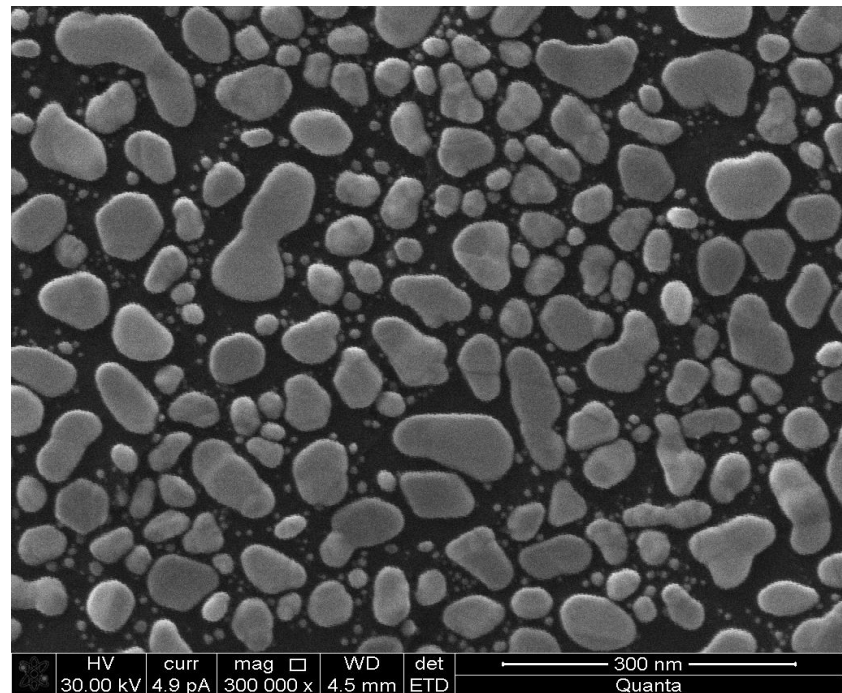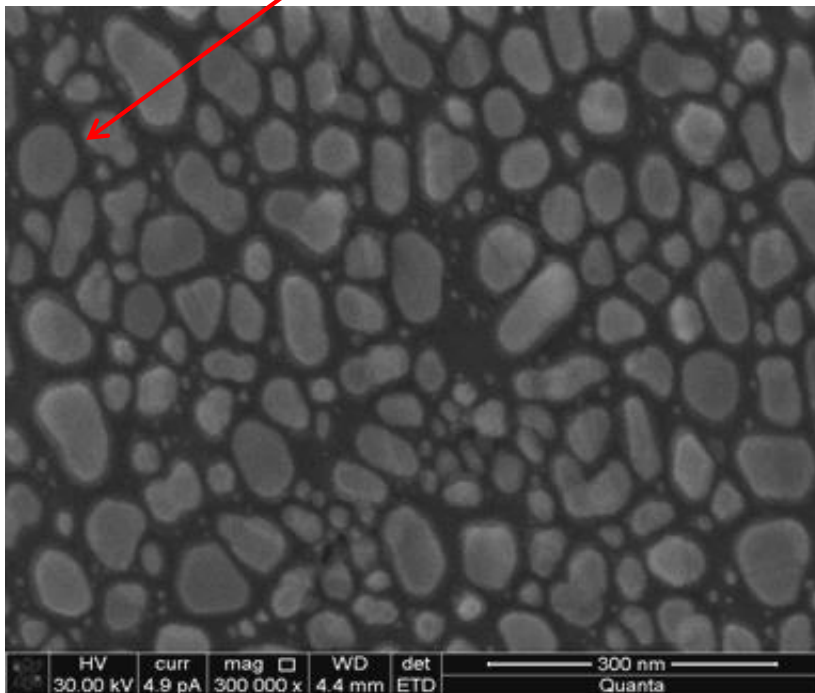
Pumping of vacuum system

Pumping of Vacuum System

- The vacuum achieved at steady state is the result of a dynamic balance between the total gas load Q_{tot} and the ability of the pump to remove gas from the volume – effective pumping speed S_{eff}. The ultimate pressure p_u is then given by:

$$p_u = \frac{Q_{tot}}{S_{eff}}$$

$$Q_G = \sum_i Q_i$$

Gas Load Sources

We can divide these sources into several categories:

- Leaks (real or virtual)
- Outgassing (surface)
- Diffusion (volume)
- Vaporization
- Permeation
- Back-streaming
- Process generated gases
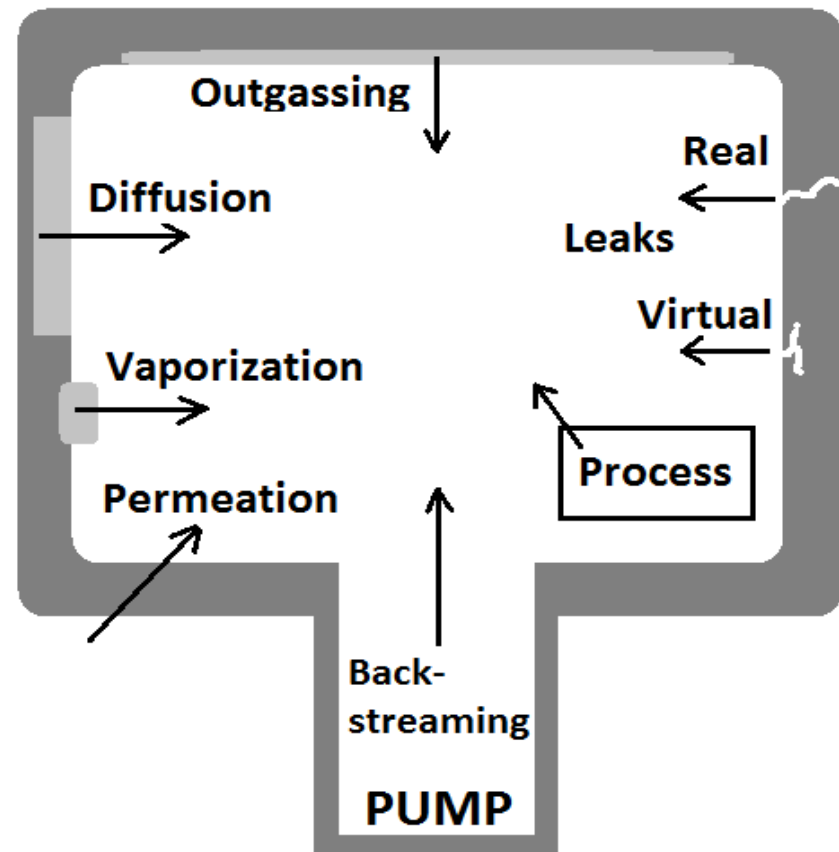
Gas Load Sources

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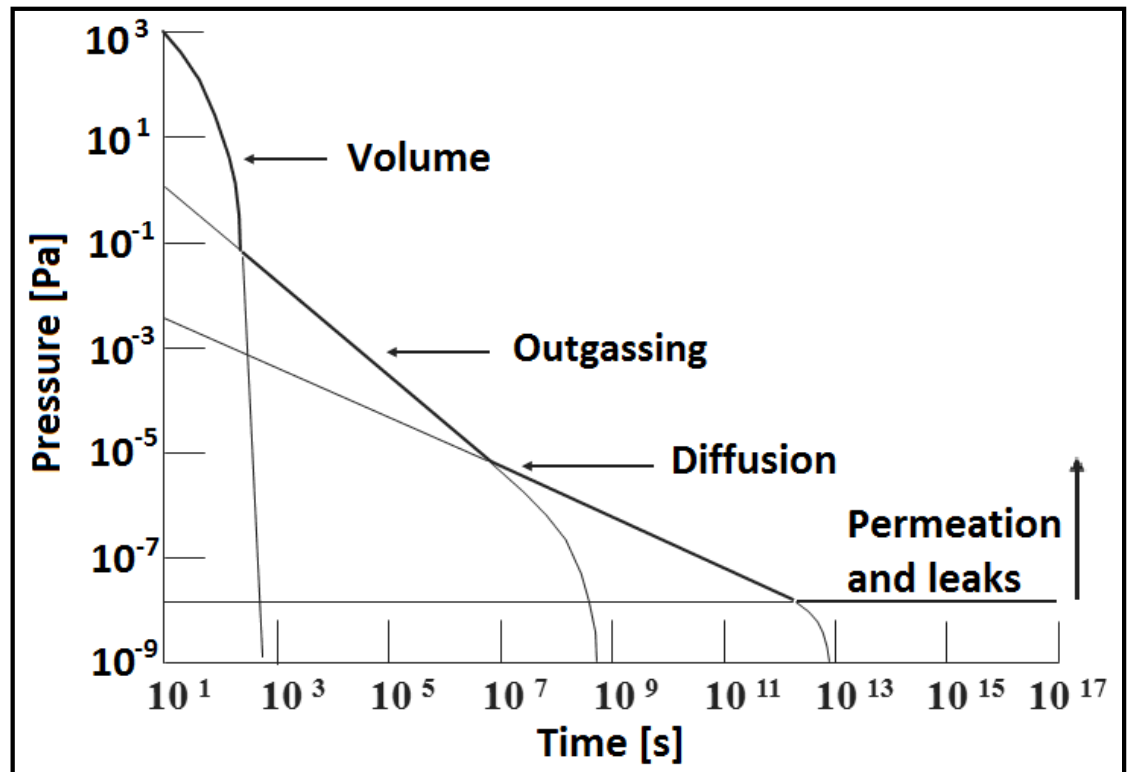
Gas Load Reduction

We can divide these sources into several categories:

- **Leaks (real or virtual)** - no trapped volumes, effective sealing method
- **Outgassing (surface)** - keep system in evacuated state, venting with dry nitrogen or inert gas
- **Diffusion (volume)** - selection of suitable materials for chamber construction
- **Vaporization** - materials (e.g. lubricants) with low vapor pressure
- **Permeation** - suitable materials, sufficient wall thickness
- **Back-streaming** - use low backing pressure
- **Process generated gases** - design system with high enough effective pumping speed

Types of gas flow

- **Viscous Flow** - collisions between molecules dominate, generally **p** > 10 Pa.

- **Molecular Flow -** collisions between molecules and wall dominate, generally **p** < 0.1 Pa.

- **Transition Flow -** region between viscous and molecular flow.
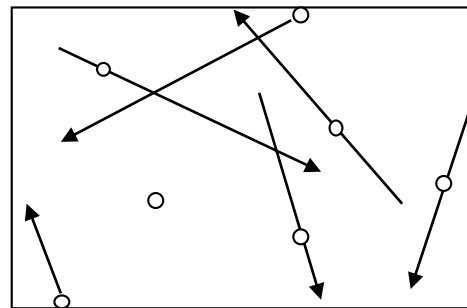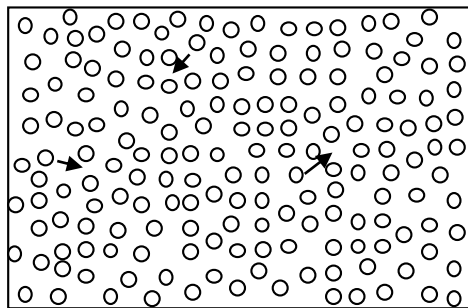
Conductance of Aperture

The conductance for an aperture of surface area A (in cm^2) in the case of laminar flow of air is given by

$$C_L = 20 \cdot A \ \ [l \cdot s^{-1}]$$

The conductance for an aperture of surface area A (in cm^2) in the case of molecular flow of air is given by

$$C_M = 11.6 \cdot A \ \ [l \cdot s^{-1}]$$

Conductance of pipe

The conductance for long pipe (L >> D, $p_2 > p_1$, p = average value)

in the case of laminar flow is given by

$$C_L = 137 \cdot p \cdot \frac{D^4}{l} [l \cdot s^{-1}]$$

The conductance for long pipe (L >> D, $p_2 > p_1$, p = average value)

in the case of molecular flow is given by

$$C_L = 12,1 \cdot \frac{D^3}{l} [l \cdot s^{-1}]$$

Vacuum system calculations

Analogy with electric circuit

- Vacuum resistance $\mathbf{W} = 1 / \mathbf{C}$
- Pressure ~ Voltage
- Flow ~ Current

$$W = W_1 + 1/(1/W_2 + 1/W_3) = W_1 + W_2W_3/(W_2+W_3)$$

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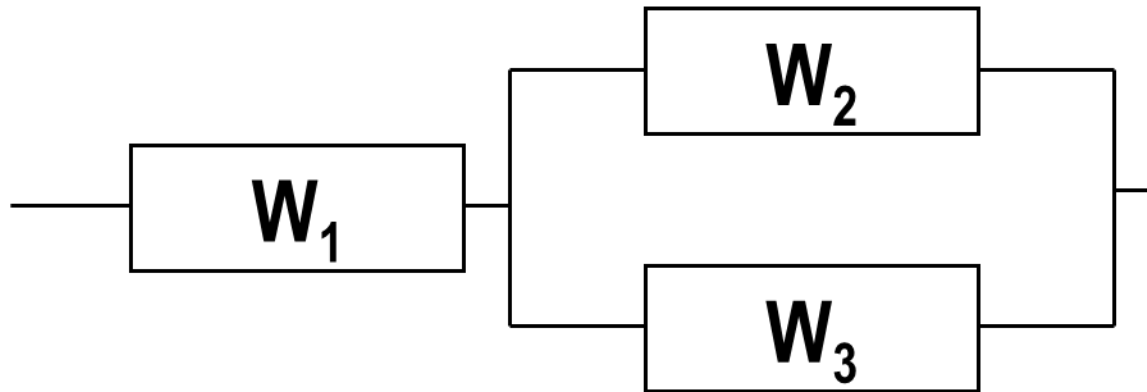
Example 1 - Vacuum oven

- Diaphragm (membrane) pump **1 l/s**, ultimate pressure ~1000 Pa

- Hose length = 3 m

Pumping speed vs. hose diameter?

50 l
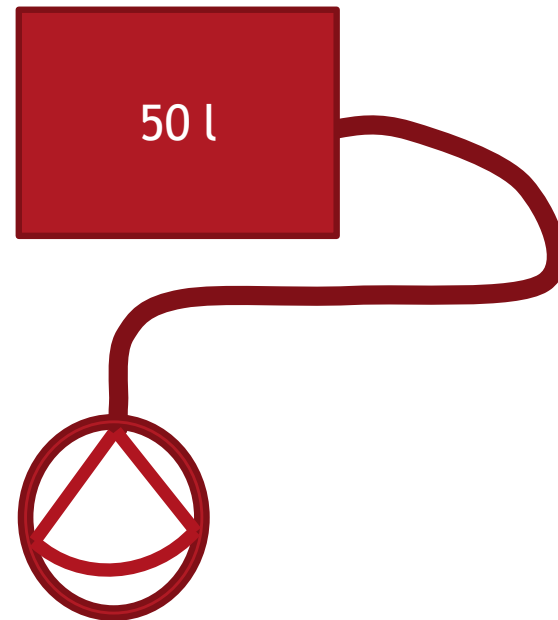
Example 1 - Vacuum oven

Pumping speed vs. hose diameter?

- Conductance Pipe: $G_P = 137*D^4*p/L$ (aperture effect negligible)

50 l
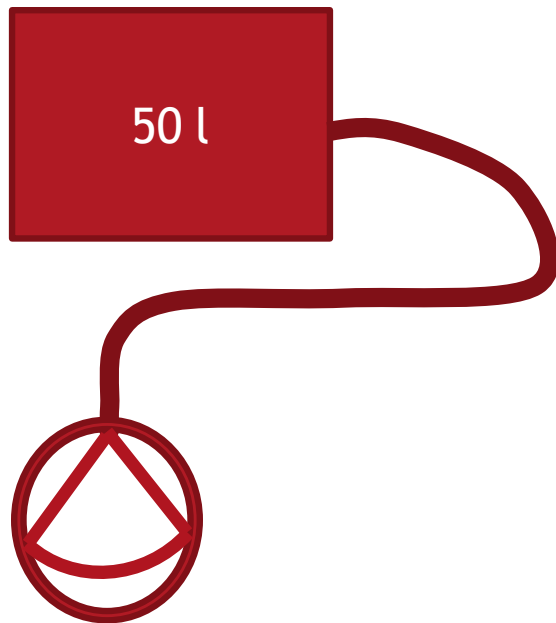
D [mm]	p [kPa]	G_P [l/s]	G total [l/s]
25	100	1.8E+06	1.00
	1	1.8E+04	1.00
10	100	4.6E+05	1.00
	1	46	1.00
5	100	2.9E+04	1.00
	1	29	0.97
2	100	7.3	1.00
	1	0.73	0.42

Example 2 – high vacuum chamber

- Typical HV pressure range $10^{-2} - 10^{-4}$ Pa

- Maximum pumping speed for selected valve ?

- Effective pumping speed ?

  - Valve diameter 10 cm
  - Tube length 0,5 m
  - TMP shield 80 % open
  - TMP pumping speed 300 l/s

50 l
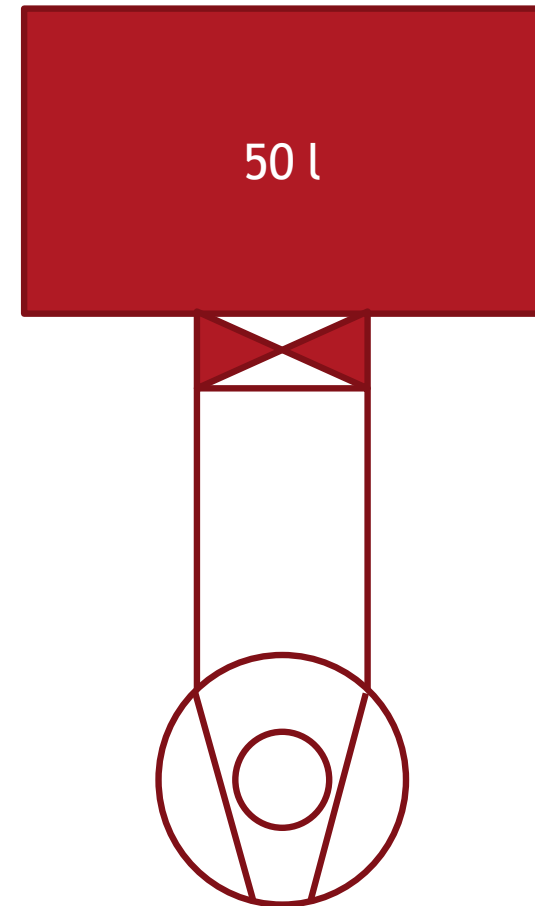
Example 2 – high vacuum chamber

1. Valve (aperture) conductance

G_V = 11.6*3.14*10^2/4 **= 911 l/s**
 = maximum pumping speed for selected port

2. Tube conductance
G_T = 12.1*10^3/50 = **242 l/s**

3. TMP shield = G_V * 0.8 = **730 l/s**

Effective pumping speed:

TMP inlet	= **300 l/s**
TMP + shield	= 213 l/s
TMP + tube	= 113 l/s
At chamber port	= **100 l/s**

50 l
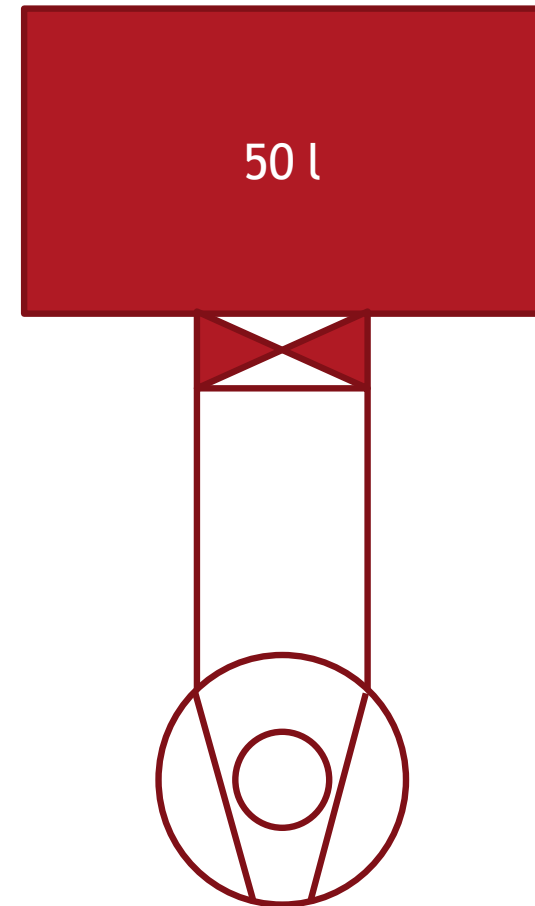
Vacuum pumps

Vacuum pumps

- Gas transfer pumps: compress and move gas out

- Gas capture pumps: entrap gas inside the pump

Gas transfer pumps		Gas capture pumps
Gas displacement pump	Momentum transfer pump	
Membrane pump	Turbomolecular pump	Ion getter pump
Rotary vane pump	Molecular pump	Cryo pump
Scroll pump	Diffusion pump	
Roots pump		
Screw pump		
Piston pump		

Diaphragm pump

- Requires inlet and outlet valve to achieve aligned gas displacement
- Oil free, corrosion resistant
- Only about 7 kPa ultimate pressure for single stage
- Multiple stages – ultimate pressure up to 50 Pa
- Well suited for low pumping speeds up to 10 m^3/h
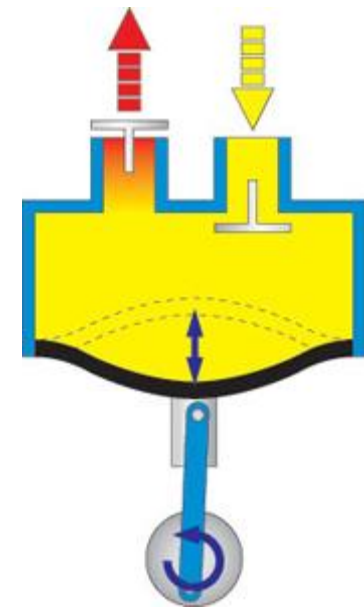
Rotary Vane Pump

- Enclosed gas is compressed until the discharge valve opens against atmosphere

- Pump oil is essential for the function – lubricates and seals

- Single and two stage versions – higher flow x higher compression ratio

- Pumping speed 2 – 200 m^3/h

- Ultimate pressure <1 Pa
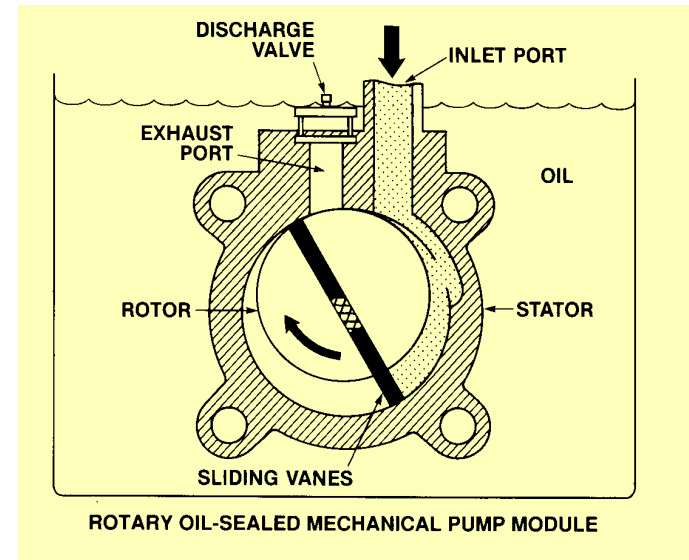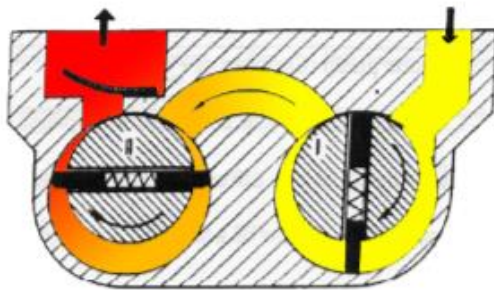
Scroll pump

- Lubricant-free within the vacuum envelope

- No need of shaft seals

- Pumping of corrosive and oxidizing gases

- Pumping speed 5 – 100 m^3 / h

- Ultimate pressure <10 Pa
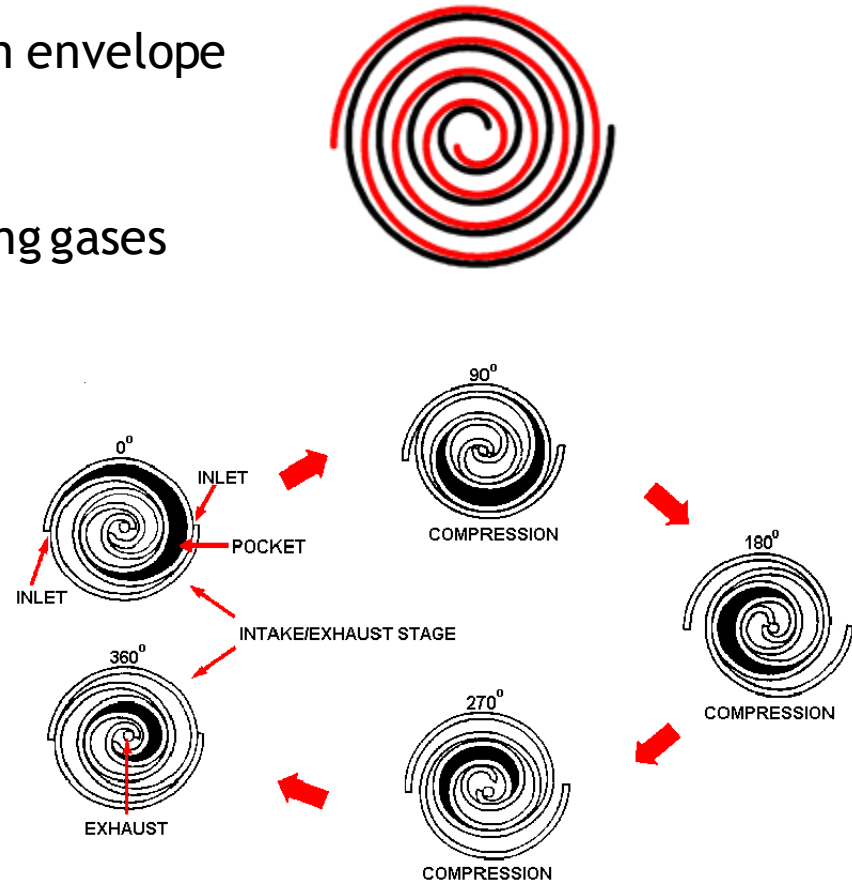
Turbomolecular pump

- High speed moving blades transfer momentum to molecules
- Can not aperate at atmospheric pressure - Requires backing pump
- Backing pressure ~1 kPa
- Ultimate pressure 10^{-6} Pa (ISO-K) or 10^{-8} Pa (CF)
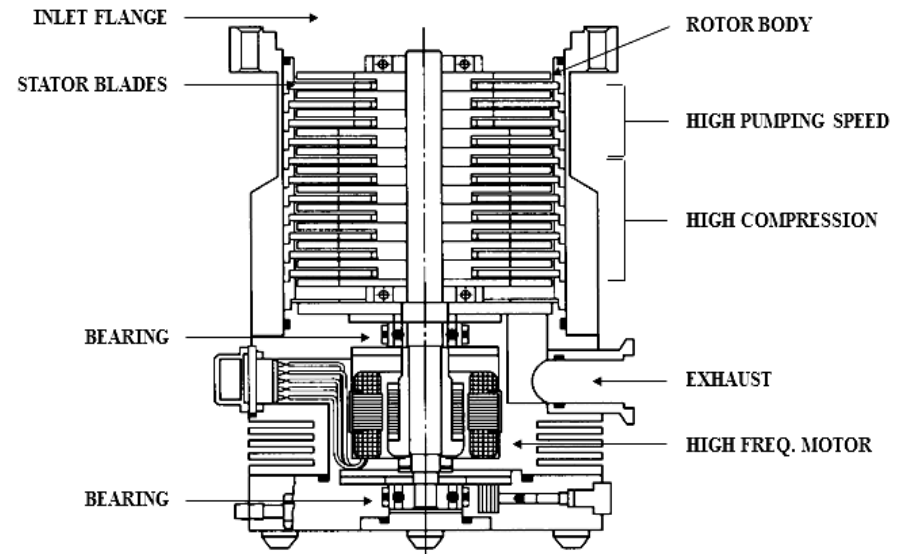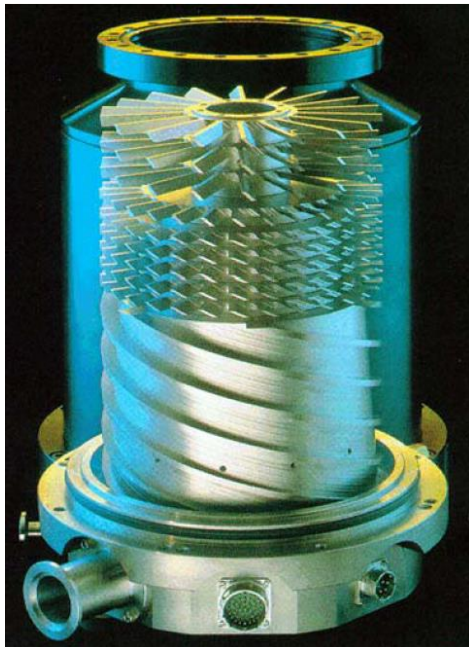
Ion getter pump

- Gas ionization by free accelerated electrons

- Ion accelerates to cathode

- Neutral Ti atoms sputtered

- Gas atoms chemically bounded or "buried"

- For noble gasses
 - Triode version
  - Ta cathode

Vacuum gauges

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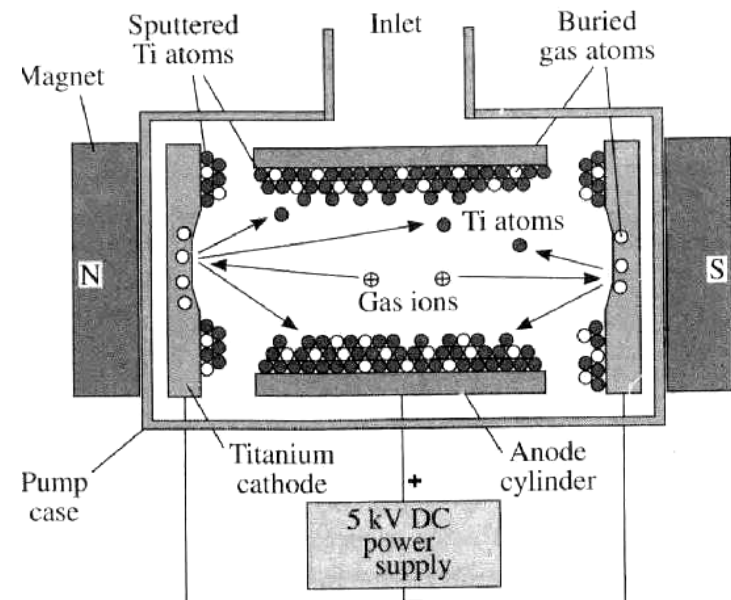
Pressure measurement

Direct measurement of force = gas independent

- Piezo sensors

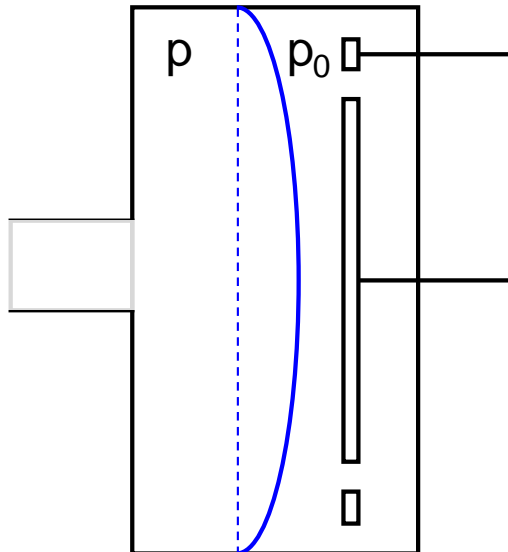- Capacitance sensors

Indirect measurement = gas dependent

- Pirani and thermocouple sensors

- Cold cathode (Inverted magnetron, Penning, Philips)

- Hot cathode (Ionisation, Bayard-Alpert) sensors

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Measurement ranges
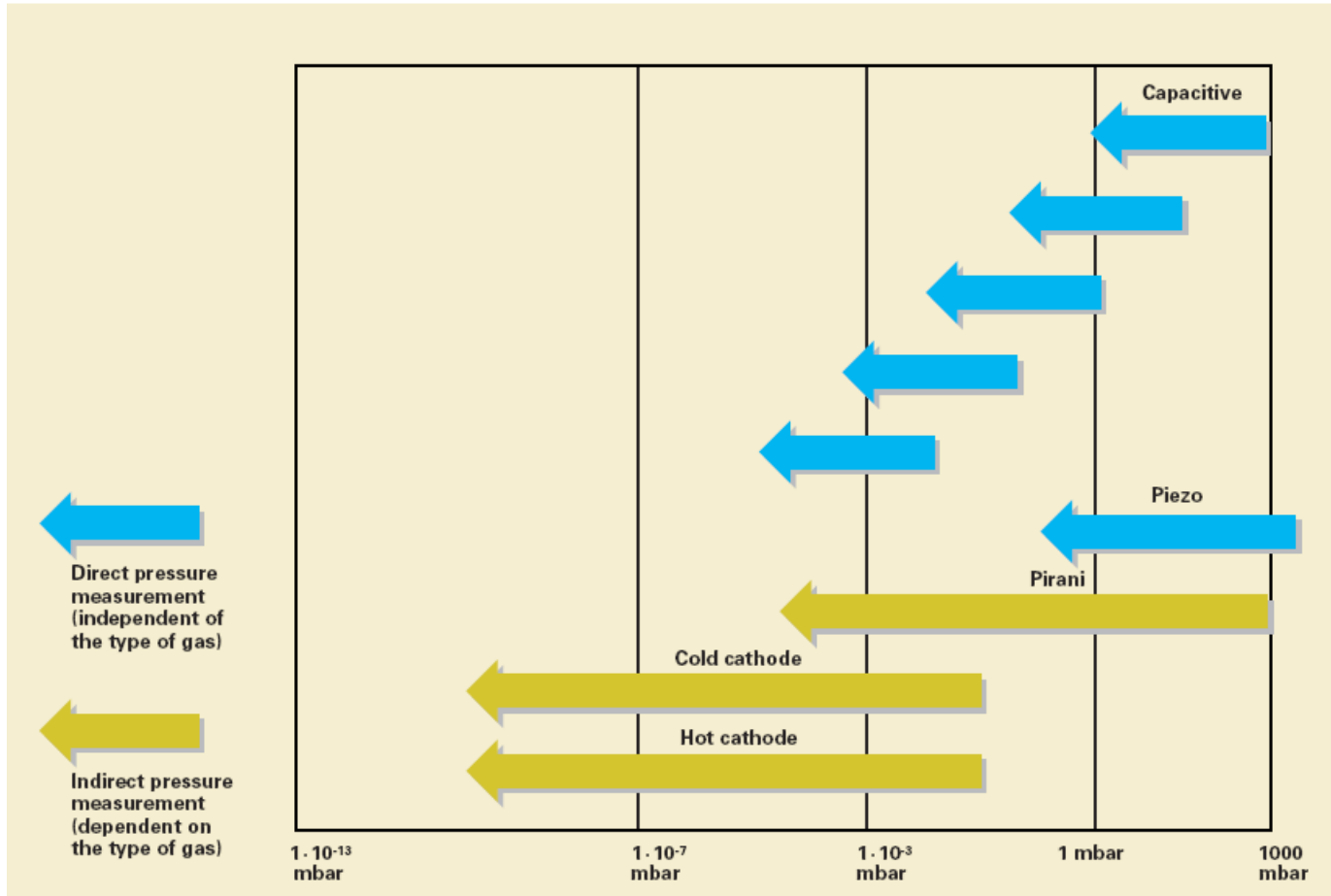
Capacitance diaphragm gauges

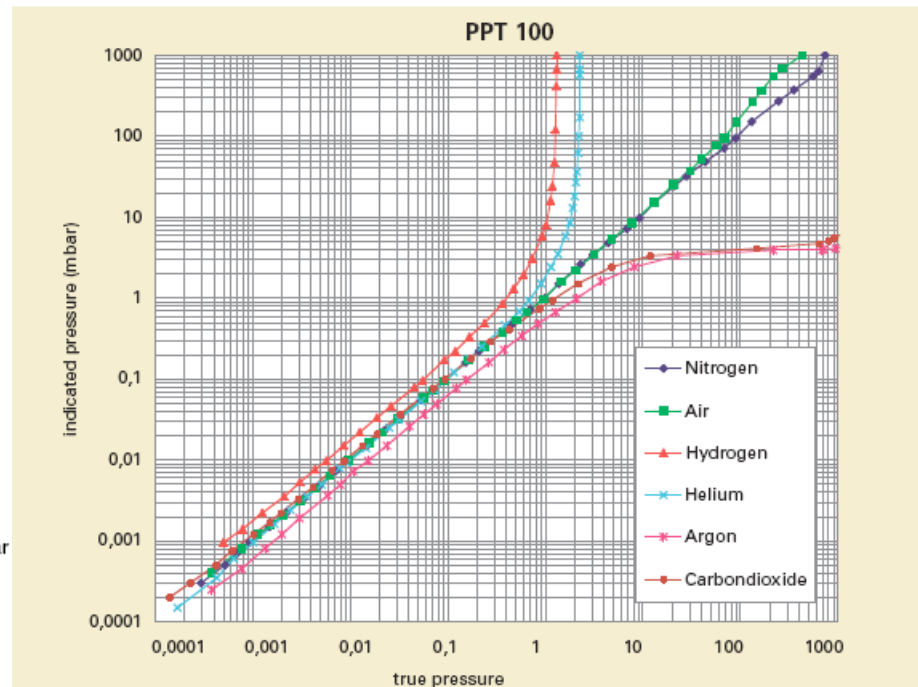- Usually measures over 4 decades, usable 3 decades
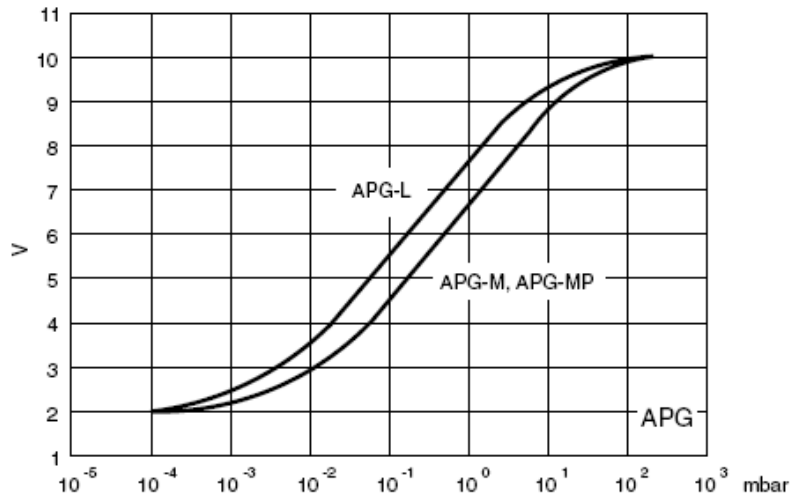
- Very high accuracy 0.2% and stability

$p_0 \ll p$
Condenser capacity $C = \varepsilon \cdot S / d$
-thermal effects
-usually linear output

Pirani gauge

- Measurement of heat los through conduction

- Radiation and convection must be minimized

- Accuracy 10-15% (50% near to atmopshere), repeatability 2%, gas dependent, low price
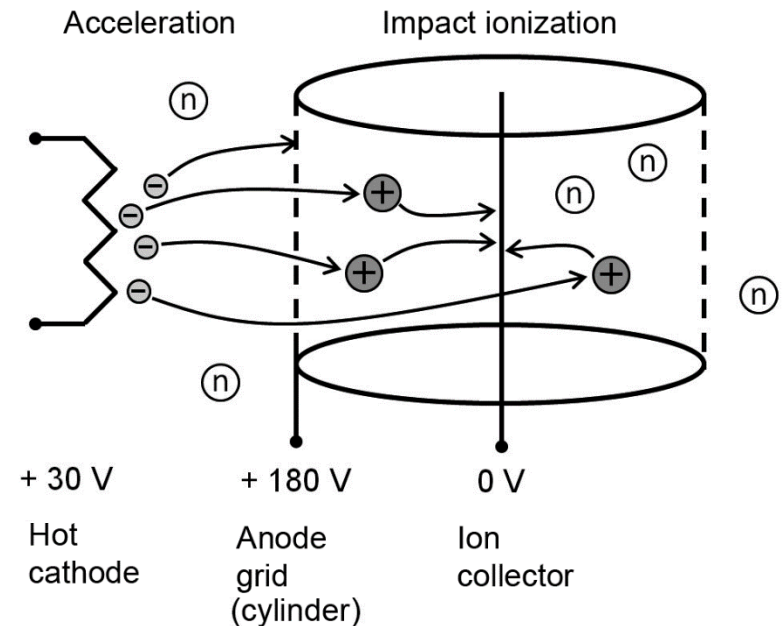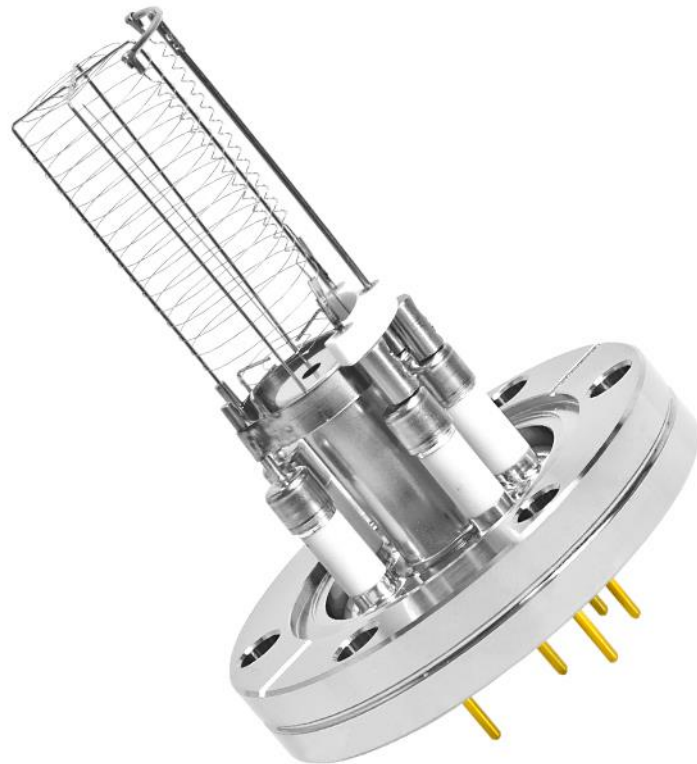
Cold Cathode / Penning Gauges

- Measures current in glowing discharge
- Sputtering effects at higher pressures
- Accuracy 30%, repeatability 5%, gas dependent.
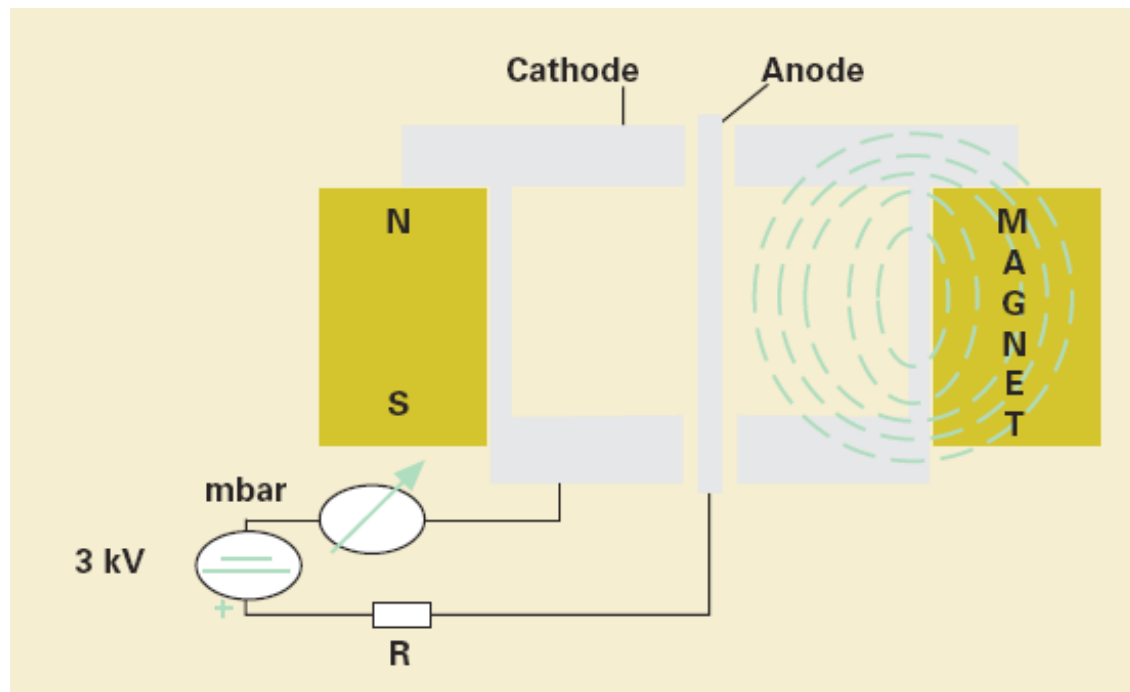
Hot cathode (Bayard-Alpert) gauge

- Heated cathode emits constant electron current

- Accuracy 10%, repeatability 5%

Acceleration | Impact ionization

+ 30 V

Hot cathode

+ 180 V

Anode grid (cylinder)

0 V

Ion collector

Vacuum systems
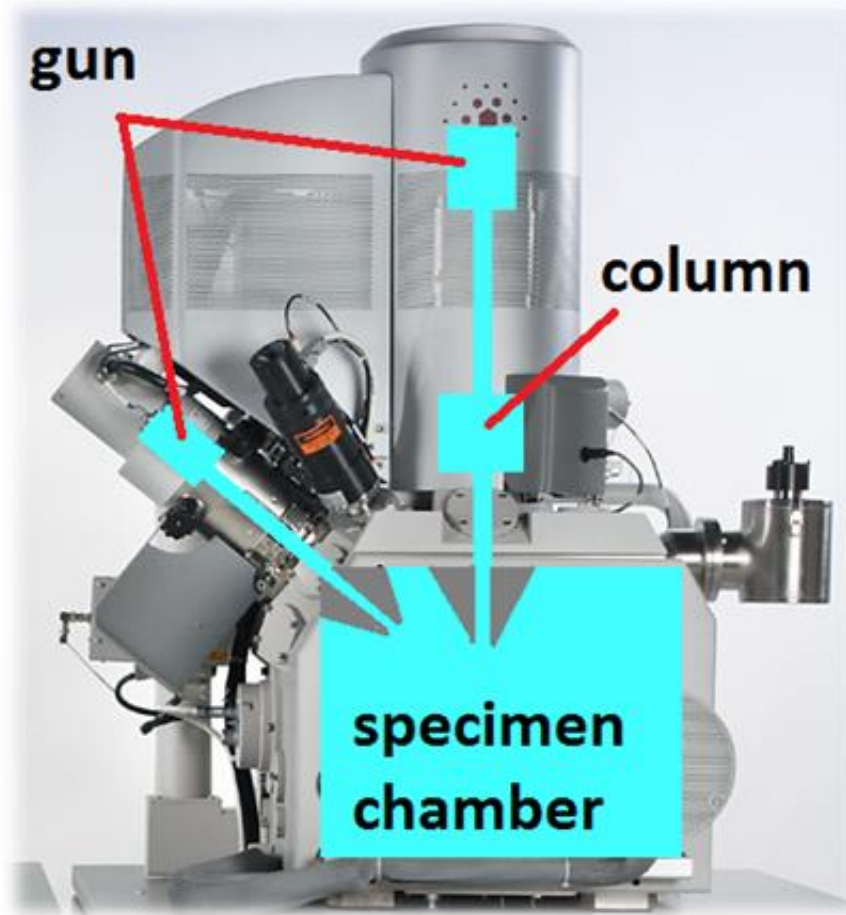
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Pressure ranges

Vacuum systems consists of several stages:

- Low or roughing vacuum – achieved with either rotary vane pump, scroll pump or diaphragm pump

- High vacuum – achieved with turbomolecular pump – vacuum in microscope chamber.

- Ultra high vacuum – achieved ion getter pump – vacuum in electron/ion column.
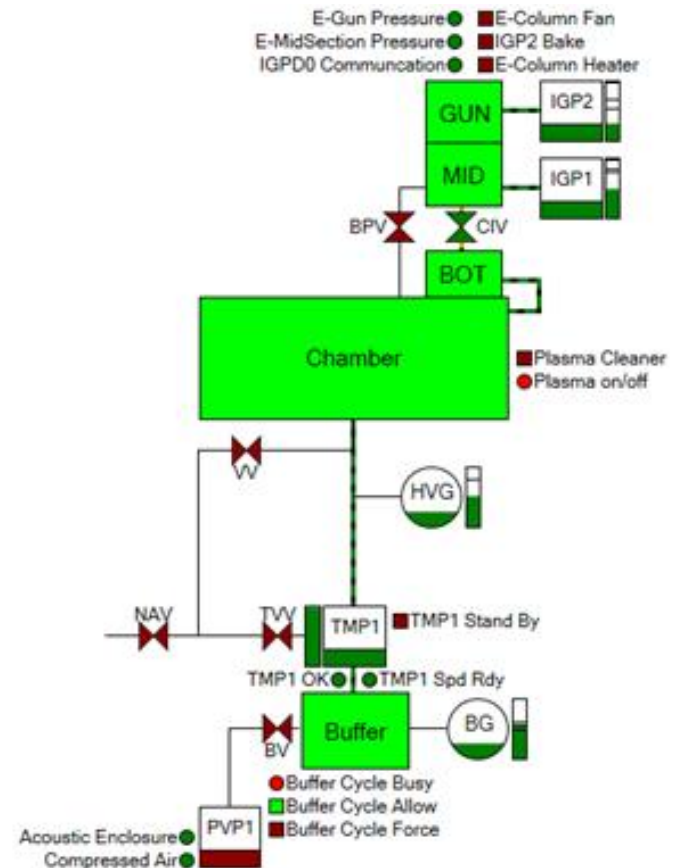
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SEM/SDB vacuum system

Pressures

Gun:	$10^{-7} - 10^{-8}$ Pa
Column:	$10^{-5} - 10^{-7}$ Pa
S. Chamber:	$10^{-2} - 10^{-5}$ Pa

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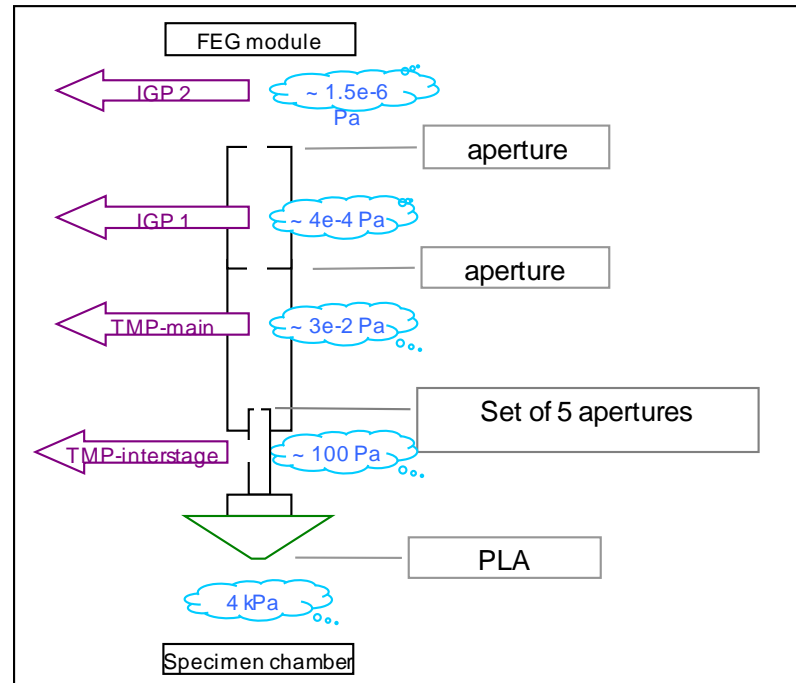
SEM/SDB vacuum system

Pressures
Gun: $10^{-7} - 10^{-8}$ Pa
Column: $10^{-5} - 10^{-7}$ Pa
S. Chamber: $10^{3} - 10^{-5}$ Pa
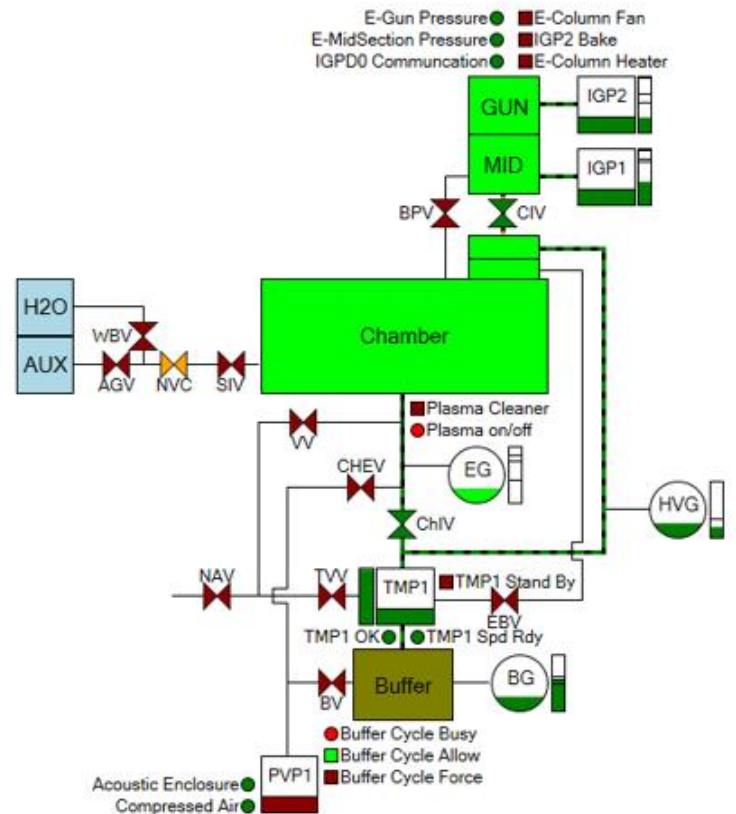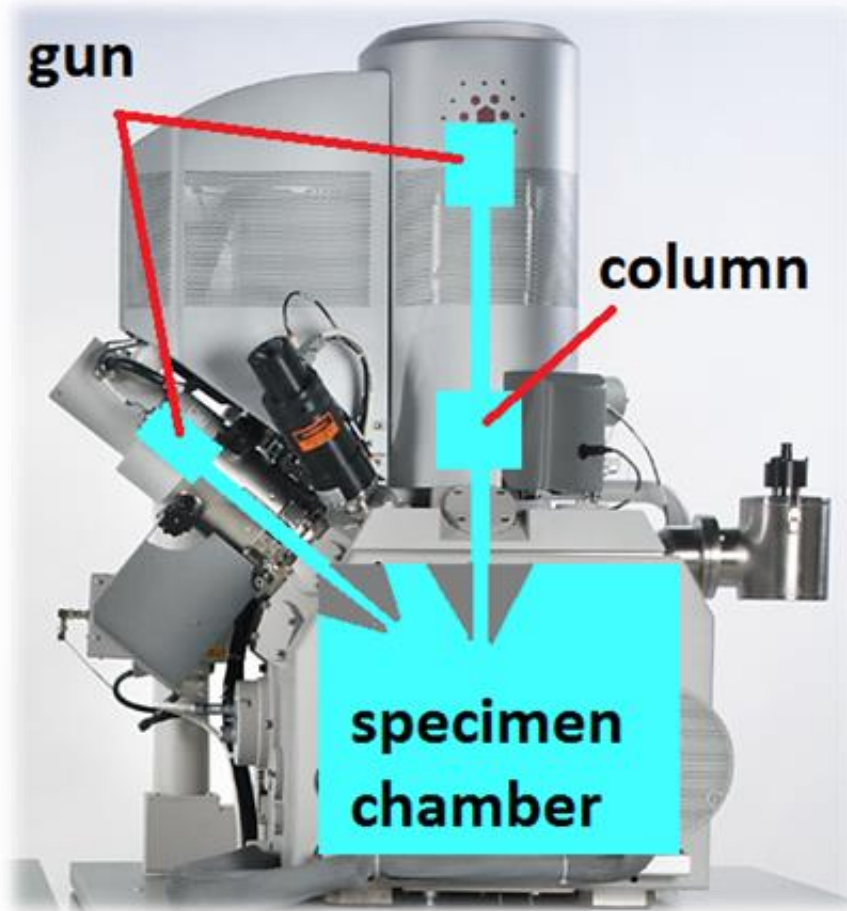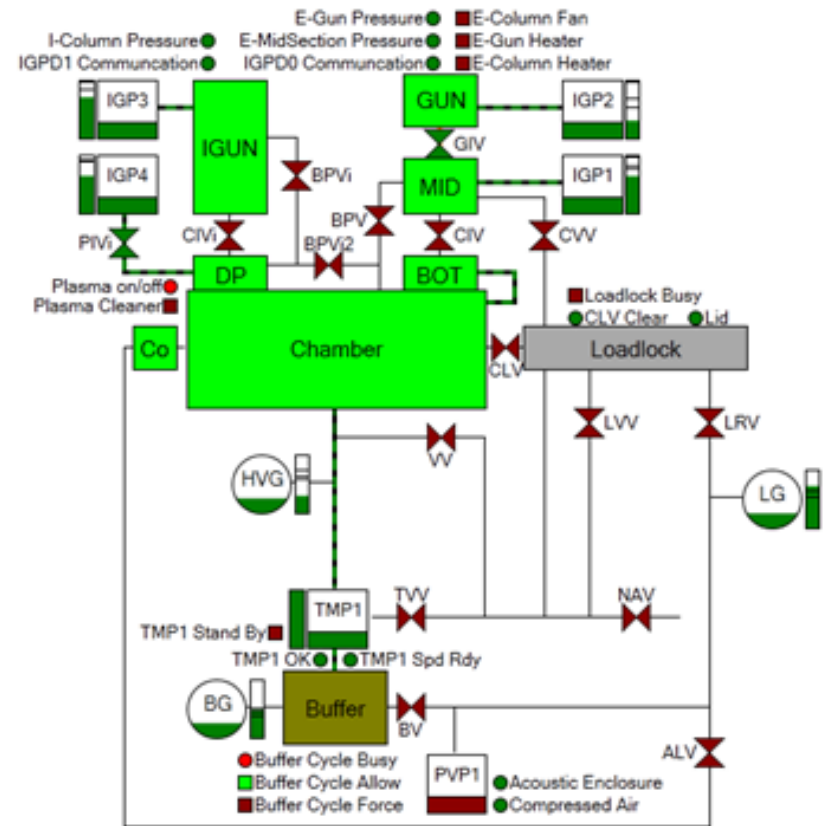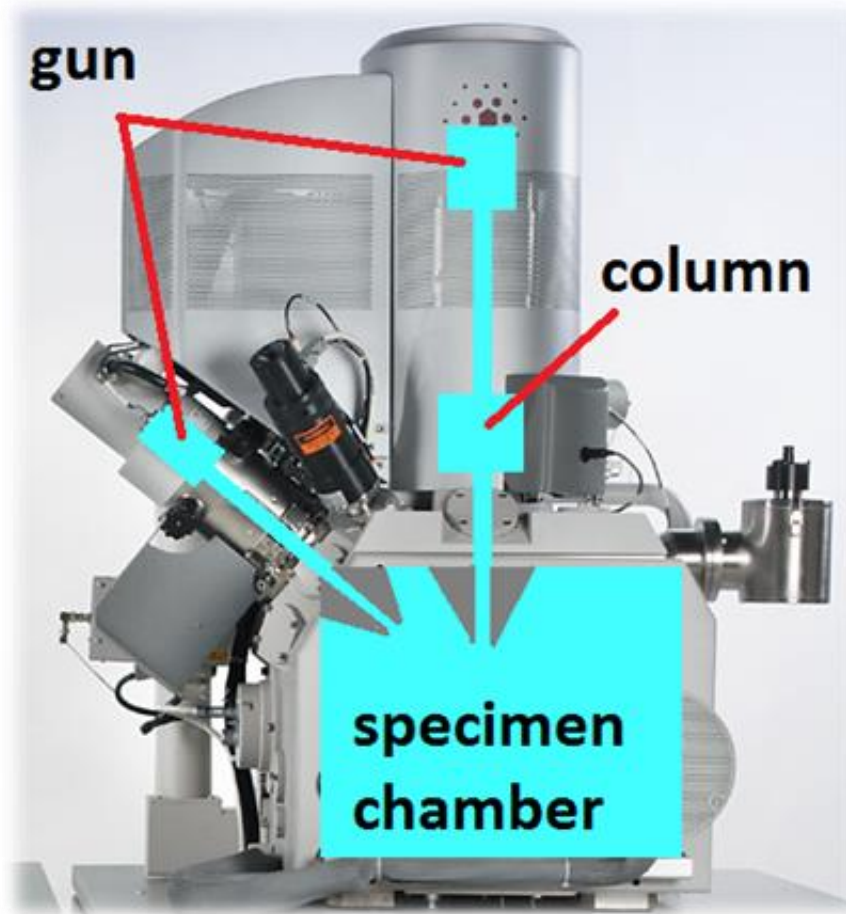
Differential Pumping – ESEM Mode

- Vacuum system of EM has several stages with different vacuum level

- Stages are isolated by valves or differential pumping apertures (DPA)

- DPA prevents diffusion of gas molecules into the higher vacuum area faster than they can be pumped out.
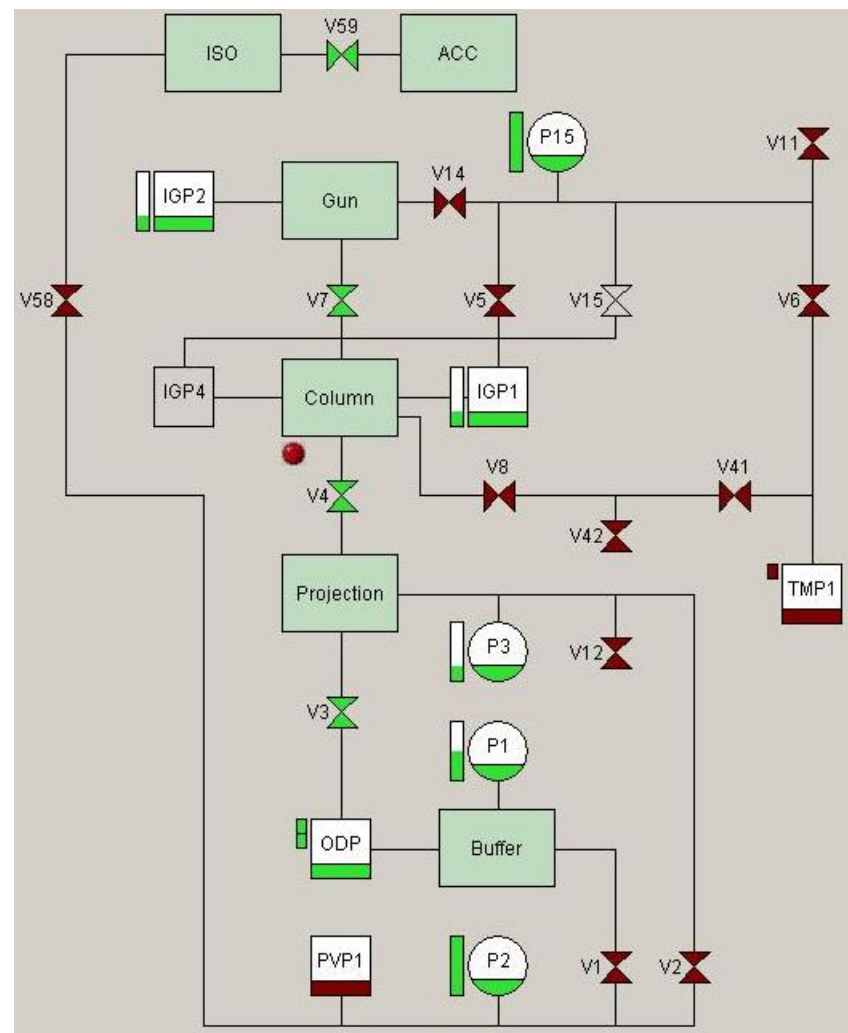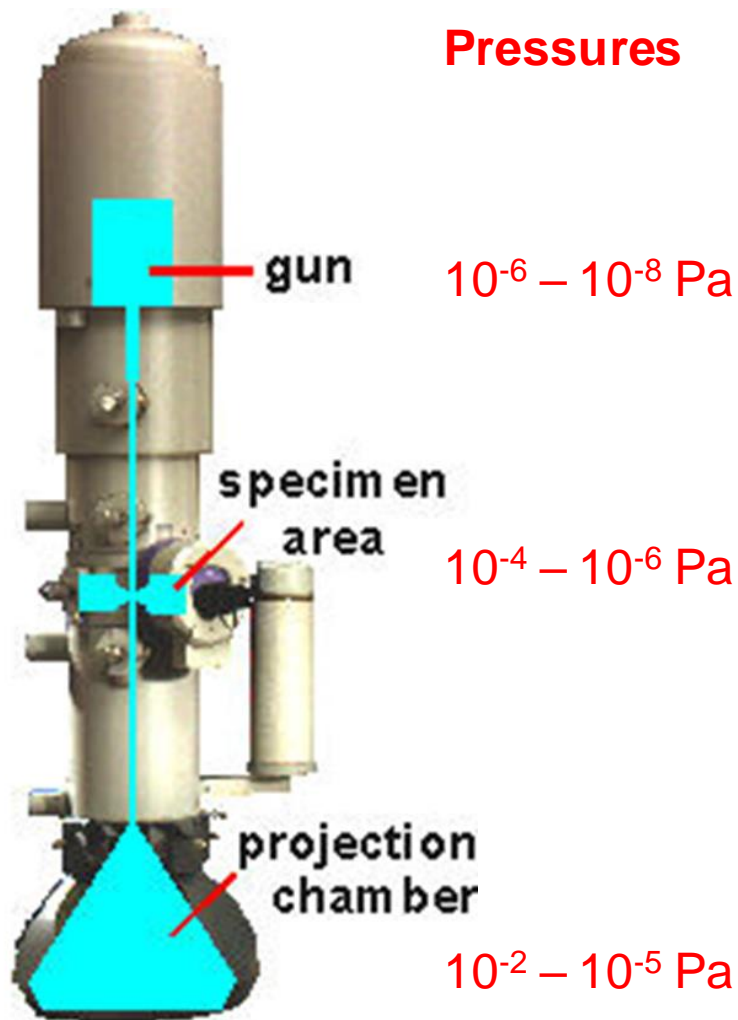
SEM/SDB vacuum system

Pressures
Gun: $10^{-7} - 10^{-8}$ Pa
Column: $10^{-5} - 10^{-7}$ Pa
S. Chamber: $10^{-2} - 10^{-5}$ Pa

TEM vacuum system

Pressures

gun — $10^{-6} - 10^{-8}$ Pa

specimen area — $10^{-4} - 10^{-6}$ Pa

projection chamber — $10^{-2} - 10^{-5}$ Pa

Leak checking and Residual gas analysis

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Leak detection

- Two types leaks – real and virtual

- Large vs. Small leaks – selecting a leak detection method

Leak rate [mbar.l.s^{-1}]

| Test Method	10^0		10^{-2}		10^{-4}		10^{-6}		10^{-8}		10^{-10}		10^{-12}
Acoustic Leak Detection	▓	▓	▓										
Bubble Testing	▓	▓	▓	▓									
Pressure Decay	▓	▓	▓	▓	▓								
Halogen Gas Detection	▓	▓	▓	▓	▓	▓	▓						
Helium Leak Detection	▓	▓	▓	▓	▓	▓	▓	▓	▓	▓	▓	▓	

Helium leak detection

Helium is a superior choice of tracer gas used to find leaks for a multitude of reasons. Helium is:

- Non-toxic
- Inert and non-condensable
- Normally not present in the atmosphere at more than trace amounts
- Relatively inexpensive
- Readily passes through leaks due to its small atomic size
- Non-flammable
- Available in various size cylinders
- Available in purities appropriate for medical usage

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Helium leak detection

- Helium counter-flow leak detectors are designed in accordance with the schematic diagram in figure below.

- A mass spectrometer (MS) is mounted on the intake flange of a Turbomolecular pump.

- A backing pump Sv evacuates the Turbomolecular pump via valve V2.
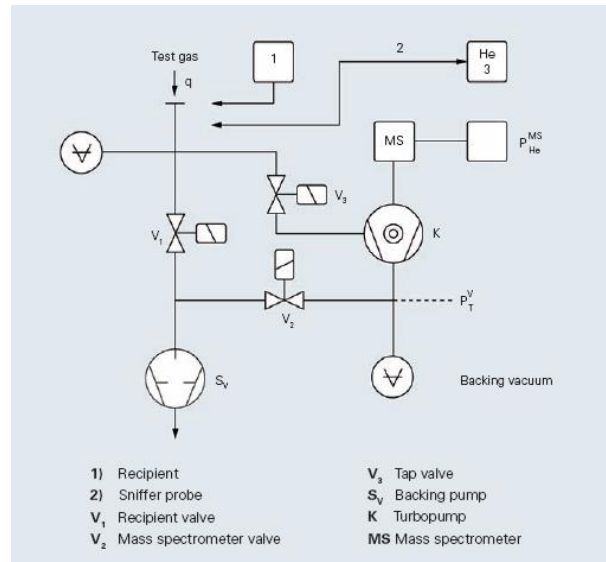
Residual gas composition

- Typpical vacuum system without leaks

- To reach $<10^{-6}$ Pa, bakeout is used

- Metal seals in UHV region

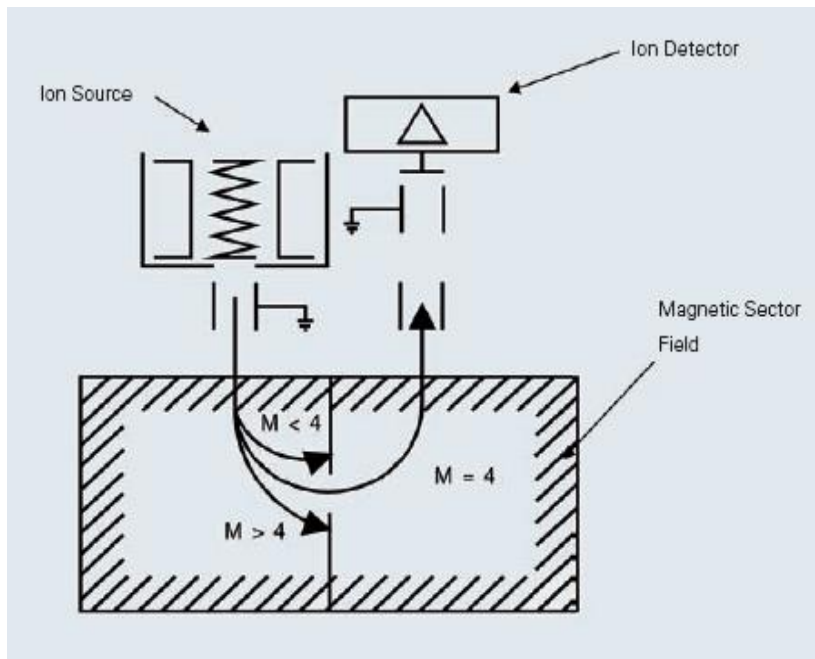| Pressure [Pa]	Major constituents
atm.	wet air
0.1	water vapor
10^{-4}	$H2O$, CO
10^{-7}	CO, $N2$, $H2$
10^{-8}	CO, $H2$
10^{-9}	$H2$

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Mass Spectrometer for RGA

- Most frequent – quadrupole mass spectrometer

- Analysis whole residual gas composition

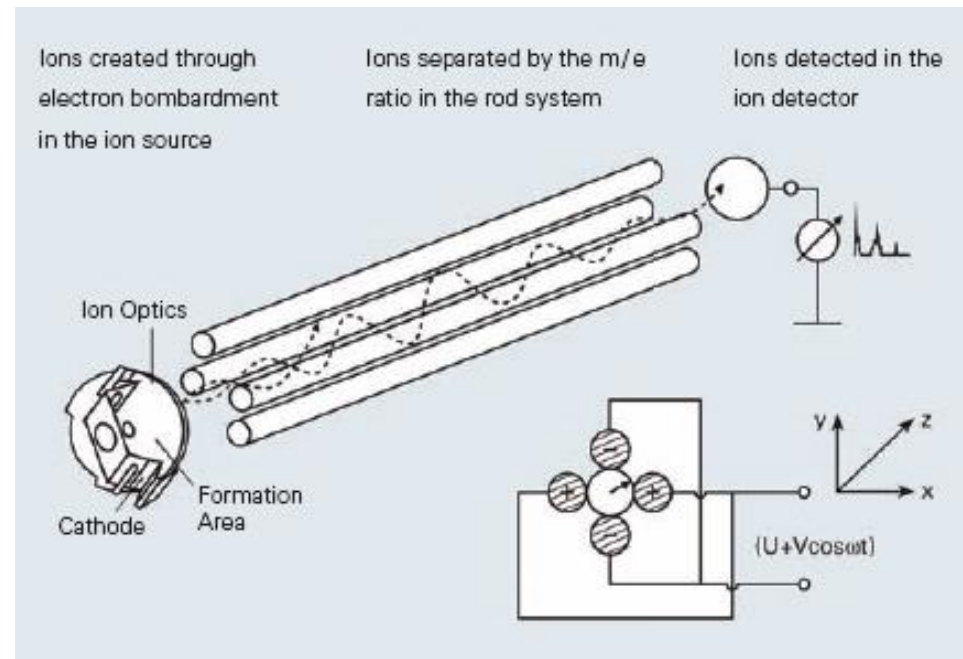- Reveals leaks

- Evaluates contamination
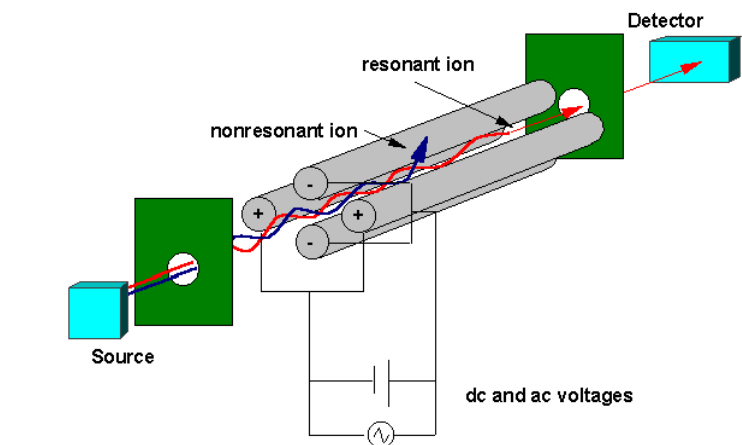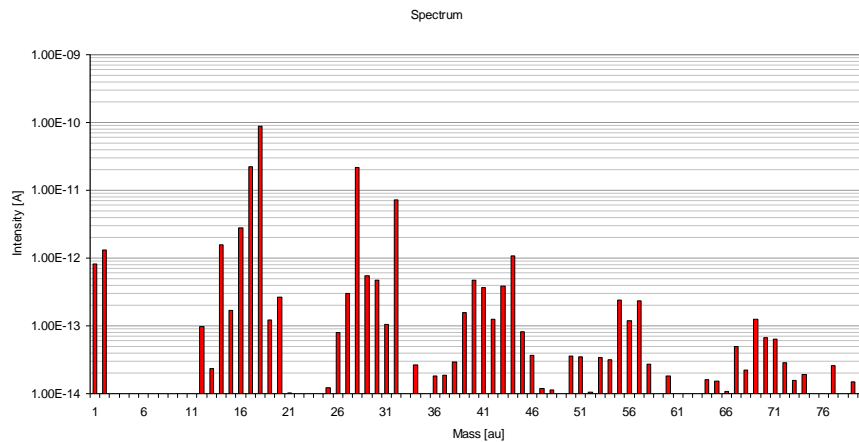
Mass Spectrometers for RGA

1. Sector Field MS

2. Quadrupole MS

From: www.pfeiffer-vacuum.com

Summary

Design of vacuum system in electron microscopes

- Consider basic vacuum rules

- Calculate dimensions of pipes and apertures

- Select suitable sealing for each part (minimize number of joints)

- Choose active components (pumps, gauges) with respect to specific EM requirements

- Verify assembly of whole system

- Measure leak rates and RGA