Vacuum systems

Why do we need vacuum?
- Maximize mean free path (≈ average distance between impacts)
- Minimize scattering of electron or X-ray
- Enhance life of electron gun & avoid arcing in the gun

Multiple device can produce vacuum...
- Rough pumping (mechanical, low vacuum)
- Diffusion, turbo and scroll pumps (medium-high vac.)
- Ion pump (high vacuum)

How do we measure vacuum?

Unit for vacuum

<table>
<thead>
<tr>
<th></th>
<th>Pascal (Pa)</th>
<th>Torr</th>
<th>Bar</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Pa</td>
<td>1</td>
<td>7.5 x 10⁻³</td>
<td>1 x 10⁻⁵</td>
</tr>
<tr>
<td>1 Torr</td>
<td>133.32</td>
<td>1</td>
<td>1.332 x 10⁻³</td>
</tr>
<tr>
<td>1 bar (~atmospheric pressure)</td>
<td>1 x 10⁵</td>
<td>750</td>
<td>1</td>
</tr>
</tbody>
</table>

Some vacuum value in different SEM / EMP instruments...
- **Variable pressure SEM:** Low vacuum, typically 1 to 200 Pa
- **Tungsten filament:** ~2 to 4 * 10⁻⁴ Pa (= 1.5 to 3 * 10⁻⁶ Torr)
- **Excellent vacuum for EMP:** ~4 * 10⁻⁵ Pa (= 3 * 10⁻⁷ Torr)
- **LaB₆ cathod:** Low 10⁻⁶ to mid 10⁻⁶ Pa
- **Field-emission gun:** ~10⁻⁷ to 10⁻⁸ Pa
**Electron beam – mean free path (λ):**

Minimize collisions between gun ➔ column ➔ sample

\[ \lambda = \frac{A}{N_0 \rho Q} \]

- **A** = atomic weight
- **N₀** = Avogadro’s number (6.02 x 10²³ atoms/mol)
- **ρ** = density
- **Q** = cross section (probability of an event)

Smaller cross section or lower density ➔ greater mean free path

**Signal detection**

Electron & X-ray collection:
Scattering of emitted electrons and X-rays reduces signal to noise ratio...

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**Pressure ranges in SEM / EMP…**

<table>
<thead>
<tr>
<th>Pressure</th>
<th>Molecules per cm²</th>
<th>Main molecules</th>
<th>Molecule-molecule distance</th>
<th>Mean free path</th>
</tr>
</thead>
<tbody>
<tr>
<td>760 Torr</td>
<td>10¹⁹</td>
<td>N₂, O₂</td>
<td>~5 nm</td>
<td>~0.1 μm</td>
</tr>
<tr>
<td>(10⁵ Pa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10⁻² Torr</td>
<td>10¹⁴</td>
<td>H₂O, ±N₂, O₂</td>
<td>~0.2 μm</td>
<td>~1 cm</td>
</tr>
<tr>
<td>(~1.3 Pa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10⁻⁷ Torr</td>
<td>10⁹</td>
<td>H₂O</td>
<td>~10 μm</td>
<td>~10⁵ cm (~0.6 mile)</td>
</tr>
<tr>
<td>(~10⁻⁸ Pa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10⁻¹⁰ Torr</td>
<td>100,000 (10³)</td>
<td>Mostly H, He (diffuse through wall)</td>
<td>~100 μm</td>
<td>~10⁶ m (~50 miles)</td>
</tr>
<tr>
<td>(~10⁻⁸ Pa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10⁻¹⁸ Torr</td>
<td>1</td>
<td>Interstellar vacuum… Almost reached at the Large Hadron Collider (CERN)!</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(~10⁻¹⁶ Pa)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- **Particle accelerator ≈ 10⁹ to 10¹⁴ Pa**
- **Best vacuum on Earth = LHC / CERN ~10⁻¹⁵ to 10⁻¹⁶ Pa**

To reach that: 2 weeks pumping, use of TiZr getters, several “mobile” pumps, several “bakeout” at 300°C, and 780 ton pumps! Yet, the tube operate at 1.9 K...
Essential system

1. Mechanical pumps
   - Backing high vacuum pump
   - “Rough” pumps entire system when required
     o Rotary direct and indirect drive pumps
     o Dry pumps (scroll, piston, claw, diaphragm)

2. High vacuum pumps
   - Establishes and maintains high vacuum in gun, column, and sample chamber
     o Oil diffusion pump
     o Turbo-molecular pump
     o Ion pump

3. Vacuum measurement devices
   - Required to switch ON/OFF valves (some pumps do not work above a certain pressure)
   - Monitor the vacuum and enable emergency shutdown in case of problem

Complete vacuum system (JEOL)

High-vacuum pump
Ion pump
Gun
Sample chamber
Air inlet valve

Nitrogen tank for pneumatic valve and vacuum release

Mechanical pump
Sample exchange airlock
Air inlet valve

PIG = pirani vacuum gauge
- PIG1 = vacuum in gun
- PIG2 = vacuum in chamber
- PIG3 = vacuum in airlock

AVx = air valve
Vx = valve
**Mechanical pump**

- Typical rotary pump creates low pressure by rotating cam or vane in oil.
- Rotates away from inlet, compressing air on other side and forcing through the outlet port.

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**Indirect drive (belt drive)**

**Direct drive (rotary-vane)**

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**Two stage rotary pump (Edwards RV8)**

Two-stage rotary vane type
Working principle diagram of vacuum pumps
1. Senior exhaust valve
2. Passage
3. Low-level exhaust valve

OUT
**Dry scroll pump**
- Use one fixed and one orbiting scroll to create crescent-shaped gas pockets
- Gas pockets are compressed and air is forced through central exit port
- No oil used for sealing or lubrication
- Completely dry and contamination-free

**High vacuum pumps**

1) **Oil diffusion pumps** (gas transfer)
   \[ 10^{-3} \text{ to } 10^{-10} \text{ Torr} \]

2) **Turbomolecular pumps** (gas transfer)
   \[ 10^{-4} \text{ to } 10^{-10} \text{ Torr} \]

3) **Ion pumps** (gas capture)
   Up to \[ 10^{-11} \text{ Torr} \]
   *Must operate in conjunction with other high vacuum pumps*
Turbomolecular pumps

- Purely mechanical, very clean, fast
- Stack of rotors which deflect incoming gas molecules with rotating-angled blades
- Molecules hit underside of blade and are driven in direction of exhaust
- Work at ~60,000 rpm
- Back with mechanical pump

See also: http://www.lesker.com/newweb/vacuum_pumps/vacumpumps_technicalnotes_1.cfm
Ion pump (gas capture)

Gases are taken up by reaction with fine particles of metal, or by ion implantation

- **Anode**: Parallel array of short stainless steel tubes
- **Cathode**: Plates of Ti or Ta near ends of tubes
- Generally used for *electron gun*.

Generate strong magnetic field parallel to tubes

1. Gas is ionized in tubes by electrons released from cathode
2. Ions strike cathode and sputter Ti
3. Results in chemical reactions and ion burial

Measuring pressure: vacuum gauges

- Bourdon (dial)
- Piezo
- Diaphragm manometer
- McLeod
- Pirani
- Capacitance manometer
- Thermocouple
- Hot cathode (Bayard-Alpert)
- Cold cathode (inverted magnetron, Penning)
- Residual gas analyzer (RGA)

*Pressure (torr)*

$10^3$ $10^2$ $10^1$ $10^2$ $10^3$ $10^4$ $10^5$ $10^6$ $10^7$ $10^8$ $10^9$ $10^{10}$
**Mechanical pump (gas transfer pump)**

1) Rough pumps system from 1 atmosphere
2) Backs high-vacuum pump

*NOTE: Can use one pump for both purposes, or use two pumps*

**Three general types of mechanical pump**

- Indirect drive (Welch)
- Direct drive (Alcatel, Edwards, etc.)
- Dry pumps* (Edwards, Varian, Pfeiffer, Leybold, Anest Iwata)

* Dry pumps works **without** oil, and therefore are preferable to create a “clean” vacuum…

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**Molecular flow vs. viscous flow**

Initial pumping of volumes exposed = **viscous flow** regime. With so many gas molecules, their mean free path (MFP) is so short that they collide more readily with each other than with the walls of the tube. They move as a mass in the general direction of low pressure.

When gas pressure drops MFP becomes greater than internal tube diameter. Individual gas molecules do not encounter other gas molecules necessarily moving in one direction (to low pressure) = **molecular flow** regime. The flow of gas in independent of pressure gradient, and depends mainly on tube dimensions and molecule speed (= temperature). In this case, backstreaming of molecules into the high vacuum chamber is possible.
Backstreaming

Refers to the movement of gases (including pump oil vapor) from pumps into the vacuum chamber. It can be an important issue with diffusion pumps.

Design of diffusion pumps can make some difference. Placement of a continuous operation cold plate over the diffusion would be the best solution to capture oil drops before they reach the sample chamber, but it is rarely included in microprobe design.

Oil diffusion pumps have a long history and are considered by many to be less costly and easier to use in a multiple user facility. However, the alternative is the turbo pump.

Oil diffusion pump

1) Oil heated – boils
2) Vapor streams up and is deflected out & down through baffles
3) Large oil vapor molecules transfer momentum to air molecules that randomly enter pump (3-stage stack at right)
4) Oil re-condenses on side of pump that is actively cooled by circulating chilled water
5) Air molecules build up at base of pump
6) Mechanical backing pump removes air from base (4th stage)

500 - 1000 l/s pump rate
Can’t operate above ~10^{-2} torr
(can “crack” the oil)

Water cooling coils
Foreline
Pump oil
Heater

Kurt J. Lesker Co.
Measuring pressure: **LOW vacuum gauges**

1) Thermocouple gauge (1 to $\sim10^{-3}$ Torr)

Thermocouple welded to filament, filament temperature dependent on thermal loss to gas. Thermocouple voltage responds to gas pressure.

2) Pirani gauge (1000 to $\sim10^{-5}$ torr)

Two filaments, one measurement, one reference.

Filaments heated and the difference in temperature causes change in resistance of Wheatstone bridge (4 resistors, three known value). The current required to rebalance circuit is, therefore a measure of pressure.
Measuring pressure: **HIGH vacuum gauges**

3) Cold cathode (Penning) gauge
\((\sim 10^{-2} \text{ to } \sim 10^{-9} \text{ Torr})\)

*Similar: inverted magnetron gauge*

- Rely on external event to start (cosmic ray, radioactive event)
- Positive ions released by high voltage discharge.
- Bombard metal cathode, releasing secondary electrons, which can, in turn, ionize gas atoms, adding to the discharge.
- Measure ion current and/or electron current, which is proportional to pressure unit (less molecule ionized = lower current).

Measuring pressure: **HIGH vacuum gauges**

4) Hot Filament (Bayard-Alpert) gauge
\((\sim 10^{-3} \text{ to } \sim 10^{-11} \text{ Torr})\)

- Essentially an electron gun.
- Thermionic emission of electrons ionizes gas.
- Read ion current (function of pressure).
RGA: Residual Gas Analyzer

Residual gas analyzers are specialized mass spectrometers, used to detect and quantify gas partial pressures in vacuum chamber. They may be of the magnetic sector design, or quadrupole design (above). Gas molecules are ionized, and then accelerated into an ion detector, separated by their mass-to-charge ratio (m/z).

Snapshot of vacuum in a microprobe

Residual Gas Analyzers are rarely utilized in EMP. They are valuable for...

- Diagnosing vacuum problems
- Giving appreciation that a ‘vacuum’ is full of gas molecules, and which ones.

This example show a nitrogen peak (N₂, mass 28) 10x the water (H₂O, 18). Compared to a records of "good vacuum", an “abnormal” vacuum is identified here, and suggests a minor but significant leak of room air into the chamber.

In normal case, N₂ drops quickly immediately after pumpdown, whereas H₂O is more slowly evacuated ("sticky" gas).