Summary

Electron propagation is only possible through vacuum. The vacuum level varies between regions of the electron microscope; the highest vacuum level (<10^{-7} Pa or 10^{-9} mbar) being in the gun where electrons are emitted and accelerated and is required to keep source and accelerator clean. The specimen area also requires a high vacuum level to reduce contamination, which is especially important for chemical analysis that requires the electron beam to rest in the same area for a long time. Hydrocarbon build up (contamination) on the observed area often results from a low system vacuum level in combination with existing surface contaminants. Turbo-molecular and oil-diffusion pumps for high vacuum cannot work against atmospheric pressure and they require a mechanical pre-vacuum pump to back them for proper and efficient operation.

Electron beams are generated either by thermal emission or field emission. Field emission sources provide lower energy spread and high coherence required high resolution spatial resolution imaging and electron spectroscopy.

Electrons are focused by simple round magnetic lenses which properties resemble the optical properties of a wine glass…. Unlike in light optics the wavelength (2pm for 300kV) is not the resolution limiting factor. Lens aberrations however and instabilities of the electronics (lens currents etc.) limit the resolution of even the best and most expensive transmission electron microscopes to about 50pm.

Recording an image means detecting electrons. Depending on their energy electrons can be detected by different detectors. A high detector efficiency and a high signal to noise ratio allows faster recording and reduces the exposure (beam damage) of the sample to the electron beam. A high linearity and high dynamic range permits to quantify images and to record high and low intensities in one image (important for diffraction experiments).
Components of a scanning electron microscope

1. Electron propagation is only possible through vacuum!
2. Need a good vacuum system to reduce contamination!

Outline:

This section of the course describes main components of the scanning electron microscope: pumping systems, electron sources, electron optics and detectors.

1) Pumping Systems
2) Electron sources
3) Electron optics
   A. Basics
   B. Magnetic Lens
   C. Aberrations and Spatial Resolution Limits in a SEM
   D. Modern SEMs
4) Detectors
   A. Secondary electrons
   B. Back scattered electrons
   C. X-ray detectors
1) Pumping system: block diagram

- **Primary vacuum (>0.1 Pa)**
  - Mechanical pump
- **Secondary vacuum (<10^{-4} Pa)**
  - Oil diffusion pump
  - Turbomolecular pump
- **High and ultra-high vacuum**
  - Gun & specimen area (<10^{-6} Pa)
  - Ion getter pump
  - Cold trap

1) Pumping system: Primary vacuum

- **Scroll pumps**
  - No oils or sources of contamination, “Dry”
  - Expensive
  - Lower ultimate vacuum pressure (10^{-2} torr)
  - High maintenance, scrolls need to be yearly

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Rotary vane pump

- Uses oil (wet) which can be a source of contamination
- Noisy but not active all of the time (buffer tank backing)
- Cheap and low maintenance
1) Pumping system: Secondary vacuum

**Oil diffusion pump**
- Vibration free
- "cheap", reliable and low maintenance
- Possible Contamination sources: oil vapor
- High pumping capacity (>500 l/s)
- Best with cold trap (limits oil vapor migration)

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1) Pumping system: Secondary vacuum

**Turbomolecular pumps**
- Rotation speed 20-50,000 rpm
- Magnetic bearings
- Pumping volumes 50-500 l/s
- No oils or sources of contamination
1) Pumping system: High / Ultra-high vacuum

Ion getter pump (IGP)
- no vibrations
- No exit; no “pumping”
- improves vacuum by “trapping” gas molecules in sputtered Ti layers
- Requires High Vacuum via secondary pumping before using and for proper operation

2) Electron Sources

http://www.feibeamtech.com
2) Electron Sources: Comparison of Sources

Some data for electron guns (~20kV)

<table>
<thead>
<tr>
<th>Gun type</th>
<th>Thermoionic</th>
<th>Field emission</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>W filament</td>
<td>LaB₆ cathode</td>
</tr>
<tr>
<td>Temperature (K)</td>
<td>2800</td>
<td>1900</td>
</tr>
<tr>
<td>Cathode diameter (μm)</td>
<td>100</td>
<td>20</td>
</tr>
<tr>
<td>Source diameter (μm)</td>
<td>30</td>
<td>20</td>
</tr>
<tr>
<td>Current density at emitter (A/cm²)</td>
<td>3</td>
<td>30</td>
</tr>
<tr>
<td>Emitted current (total) (μA)</td>
<td>200</td>
<td>80</td>
</tr>
<tr>
<td>Brightness β (≥20 kV/A/cm² sr)</td>
<td>1 x 10⁴</td>
<td>1 x 10⁵</td>
</tr>
<tr>
<td>Max. beam current (nA)</td>
<td>1000</td>
<td>1000</td>
</tr>
<tr>
<td>Energy spread at emitter (eV)</td>
<td>0.6</td>
<td>0.4</td>
</tr>
<tr>
<td>Energy spread at the gun exit (eV)</td>
<td>1.5-2.5</td>
<td>1.5-2.5</td>
</tr>
<tr>
<td>Probe current noise (%)</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>Probe current drift (%/h)</td>
<td>&lt;1</td>
<td>&lt;1</td>
</tr>
<tr>
<td>Operating gun pressure (mbar)</td>
<td>&lt;1.1 x 10⁻⁵</td>
<td>&lt; 5 x 10⁻⁶</td>
</tr>
<tr>
<td>Emitter lifetime (h)</td>
<td>10 - 100</td>
<td>1000</td>
</tr>
<tr>
<td>cathode conditioning</td>
<td>not necessary</td>
<td>not necessary</td>
</tr>
<tr>
<td>gun annealing</td>
<td>not necessary</td>
<td>not necessary</td>
</tr>
<tr>
<td>sensitivity to external magnetic field and vibrations</td>
<td>faible</td>
<td>faible</td>
</tr>
</tbody>
</table>

* virtual cross-over

2) Electron Sources: thermionic gun

- Electron boil from surface….heated to overcome the work function to push electrons in vacuum level
- Tungsten wire heated to ~2800K
- LaB₆ crystal heated to 1900K
- Main advantages: simple, cheap, no high vacuum required and maintenance friendly
- Disadvantages: low brightness, high energy spread and large source size (10-100 μm)
2) Electron Sources: Emission of electrons

- Thermonic emission
- Schottky emission
  - field-enhanced thermionic emission \((10^8 \text{V/m})\)
- Extended Schottky emission
  - thermally assisted field emission
- Cold field emission
  - tunnel effect (quantum tunnelling)

Increasing Temperature
- Increasing electric field

Cathodes (tips)

- **Cold field emission** \((E=10^9 \text{V/m})\)
  - W single crystal with a sharp tip (radius ~25nm)
    - Advantages:
      - Small energy dispersion (<0.1eV)
      - high coherence, high brightness
      - higher resolution at lower energies
    - Disadvantages:
      - expensive, high vacuum necessary
      - cold emission needs flushing (cleaning) after 8 hrs

- **Thermally assisted emission**: Schottky effect
  - Large tip size
  - W/Zr tip at 1700-1800K
  - ZrO complexes lower work function
  - Continuous operation
    - "self-cleaning"
2) Electron Sources: Field emission guns (extraction)

First anode (extractor)
- Some kV
- $5.10^9$ V/m

Second anode
- Final acceleration
- Grounded- to slight voltage for focusing and increasing electron current

Characteristics
- Tip and anodes form an electrostatic condensor
- Cross-over (virtual source) size is $\Omega \sim 5$nm

3) Electron Optics: Basics

Figure 6.1. Image formation by a convex lens. A point object is imaged as a point and the collection semiangle of the lens is defined relative to the object (d) or the image (i).

Figure 6.2. How to draw a ray diagram: First construct ray 1 through the middle of the lens, then ray 2, parallel to the optic axis, to determine the lens strength. Finally, draw line 3 parallel to 2 to define the focal plane where the parallel rays are focused. Thus an asymmetric object is imaged off axis and rotated through 180°.
3) Electron Optics: Basics

Parallel beams are focused to one point in the focal plane

Object plane

Image plane

Focal plane

Figure 6.1. Image formation by a convex lens: A point object is imaged in a point and the collection semiangle of the lens is defined relative to the object (A) or the image (x).

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3) Electron Optics: basics

1. Over-focus
2. Focus
3. Under-focus

Figure 6.5. (a) Ray diagram illustrating the concepts of overfocus, in which a strong lens focuses the rays before the image plane, and (c) underfocus, where a weaker lens focuses after the image plane. It is clear from (c) that at a given underfocus the convergent rays are more parallel than the equivalent divergent rays at overfocus ($\theta_2 < \theta_1$).

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3) Electron Optics: Basics – Apertures

- Angle limiting apertures
- Defines convergence angle
- Used to change depth of field, current, and probe size in combination with condenser lens system

Figure 6.10. (A) Ray diagram illustrating how a diaphragm restricts the angular spread of electrons entering the lens. Only electron paths less than a semiangle $\beta$ subtended by the aperture at the object are allowed through the lens (full ray paths). Electrons from the object scattered at angles $>$ $\beta$ are stopped by the diaphragm (dashed ray paths).

3) Electron Optics: Condenser lens system, SEM

- Low convergence = Large spot size
- High convergence = Small spot size

First Condenser Lens (C1)
- Defines probe size
- Small spot <-> small current
- Large spot <-> high current
- Used to change Probe Current
3) Electron Optics: Condenser lens system, SEM

Double Condenser Lens systems in modern microscopes allow increased flexibility in controlling probe current and convergence angle on the sample.

3) Magnetic Lenses: Lenses for light, lenses for electrons

- **Light: glass lenses**
  - deflection of light through changing refraction index

- **Electrons: (charged particles)**
  - Electrostatic lenses
  - Magnetic lenses: Lorentz Force!
  - Variable focus (no moving parts)
  - Tunable correctors (astigmatism)
3) Magnetic Lens: Electrons in a magnetic field

- Homogeneous field, a small
- Component of \( \mathbf{v} \parallel \mathbf{B} \) almost unchanged
- Component of \( \mathbf{v} \perp \mathbf{B} \): \( v_r \ll |v| \)
- Spiral with radius \( r = m v_r/eB \)
- All electrons crossing the axis in one point are focused into the same point, \( a, v_r \)
- Focal length depends on \( B \) increasing \( B \) lowers \( f \)

Image rotation!

3) Magnetic lens

- Electron optics: no sharp interface at lens « surface »
- "Pole piece" metal cone that confines the magnetic field
- Image rotation!
- No (simple) divergent lens! « multi-poles » lenses
- Correction of aberrations
3) Magnetic lens

- Field with rotational symmetry
- Lorenz Force: \( \mathbf{F} = -e \mathbf{v} \times \mathbf{B} \)
  - Electrons on optical axis: \( \mathbf{F} = 0 \)
  - Electrons not on optical axis: deviated
- Optical axis is the symmetry axis

**Scherzer 1936:**
Magnetic lens with rotational symmetry:
Aberration coefficients:
- \( C_s \): spherical
- \( C_c \): chromatical
Always positive!!

\[
C_s = \frac{1}{16} \left[ \frac{e^2}{\lambda^2} \right] \left[ \frac{e^2}{\lambda^2} + 2(\lambda^2 + \lambda^2) \frac{e^2}{\lambda^2} \right] \left[ \frac{e^2}{\lambda^2} + 2(\lambda^2 + \lambda^2) \frac{e^2}{\lambda^2} \right] \]  
\[
C_c = \frac{1}{4} \left[ \frac{e^2}{\lambda^2} \right] \left[ \frac{e^2}{\lambda^2} + 2(\lambda^2 + \lambda^2) \frac{e^2}{\lambda^2} \right]
\]

Resolution limit: \( D_{\text{res}} = 0.66 \lambda^{3/4} C_s^{1/4} \)

Example:
\( \lambda = 0.00197 \text{nm}, C_s = 1 \text{ mm} \)
\( D_{\text{res}} = 1.8 \times 10^{-10} = 1.8 \text{ Å} \)

3) Electron Optics: aberrations:

Lens aberrations
- Focus
- Astigmatism
- Spherical and chromatical aberrations

- Can be corrected or minimized

Physical limits
- Diffraction limited resolution
2) Electron Optics: Chromatic Aberration

Focal length varies with energy critical for non-monochromatic beams (advantage for FE guns)

3) Electron Optics: Spherical Aberration

- Focal length varies with distance from optical axis, i.e., rays from the center to edge of the lens have different focal points
- Image of the object is dispersed (or blurred) along the optical axis
- Circle of least confusion $d_s = \frac{1}{2} C_s \alpha^3$

* Paraxial ray means a ray on the optic axis or very close to it, which the ray in the diagram is not. It is drawn further out to illustrate the idea of the circle of confusion.
3) Electron Optics: Aberrations-astigmatism

Astigmatism: focal length varies in different planes.

This cause the image to be direction

Astigmatism Aberration

3) Electron Optics: Aberration Correctors

Astigmatism:
Light optics:
- Correction with cylindrical lenses

Electron optics:
- Correction with quadrupole lenses,
  2 quadrupole lenses under 45 degree allow to control strength and direction of correction

Spherical Aberration:
Light optics:
- correction with combination of convergent and divergent lenses

Electron optics:
- Correction with hexapole or quadrupole and octopole lenses
3) Electron Optics: Resolution

- Spatial resolution depends on the size of the interaction volume
- Interaction volume differs material, accelerating voltage, spot size
- Different particles have different interaction volumes sizes and thus different resolution for the same microscope settings

2) Electron Optics: Resolution

Raleigh’s Criterion: Diffraction Limited Resolution

Green light: \( \lambda \approx 532 \text{ nm} \),
\( \beta \) (objective collection angle)~1 rad
n= 1.7 for oil immersion lens
** d=190nm
Electrons 10 keV: \( \lambda \approx 0.0122 \text{ nm} \)
n=1 for vacuum
\( \beta=0.1 \) rad given SEM geometry
**d=0.075 nm

Airy Diffraction Disks

Lens aberrations limit spatial resolution to \(~1 \text{ nm}\)

Thermionic SEM – limited by source brightness
FEG SEM – limited by lens aberrations
3) Electron Optics: Resolution

Limits for a modern SEM

- Spherical aberration
  \[ d_{sp} = C_s \cdot \alpha^3 \]

- Chromatic aberration
  \[ d_{ch} = C_{ch} \left( \frac{N}{E} + 2 \frac{N}{I} \right) \alpha \]

- Diffraction (Airy, Rayleigh)
  \[ d_d = 0.61 \frac{\lambda}{n \cdot \sin \alpha} \]

- Brightness \( \beta \) conservation
  \[ d_{\beta} = \frac{4T \cdot 1}{\pi^2 \beta \cdot \alpha} \]

- Combined
  \[ d_{ch} = \sqrt{d_{sp}^2 + d_{ch}^2 + d_{\beta}^2 + d_d^2} \]

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3) Electron Optics: Resolution – at Low kV

Low voltage, high resolution
Observation of the real surface
Uncoated samples
Very little beam damage
High voltage, high resolution
Edge effects, fine details not resolved
Beam damage

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thomas.lagrange@epfl.ch • www.epfl.ch • cime.epfl.ch • +41 (0)21 6934430
3) Electron Optics: FEG Microscopes

Specifications of CIME FEI XL-30

- 1-30 kV Schottky FEG
- SE imaging resolution: 2nm at 30 kV; 8nm at 1 kV
- Equipped with: Everhart-Thornley secondary-electron (SE) detector; backscattered electron (BE) detector; EDAX Si(Li) EDX detector with ultra-thin window for light element analysis

Installed and operating since 1996

3) Electron Optics: Modern Microscopes

- Electron beam resolution
  - (site survey required to determine attainable resolution)
  - Resolution @ optimum WD
    - 0.8 nm at 15 kV
    - 0.8 nm at 2 kV
    - 0.9 nm at 1 kV
    - 1.5 nm at 200 V
  - Resolution @ coincident point
    - 0.8 nm at 15 kV
    - 0.9 nm at 5 kV
    - 1.2 nm at 1 kV
- Maximum horizontal field width
  - E-beam: 1.5 mm at beam coincident point (WD 4 mm)
- Landing energy range
  - 50 V – 30 kV
- Probe current
  - E-beam: 1 pA to 22 nA
3) Electron Optics: Modern Microscopes

3) Electron Optics: Modern Microscopes - Conventional Mode

magnetic field inside pole-piece
3) Electron Optics: Immersion lens – Modern Low kV SEMs

specimen inside “lens”

- TLD (SE & BSE)
- Solid State BSE
- STEM

- A small specimen sits inside the lens gap
- Has very short focal distance, range of 2 to 5 mm
- Short focal distance means low aberrations, small probe sizes, and high resolution

Secondary electrons spiral upward in the strong magnetic field of the lens and are collected by the detector positioned above the lens (**In-Lens Detectors**)

3) Electron Optics: Modern Microscopes

BSE at 2kV: ~1nm resolution

BSE at 500V: ~5 nm resolution
3) Electron Optics: Modern Microscopes

MERLIN™ – Analytical power for the sub-nanometer world –

Gemini® II column
Low current configuration (Max. probe current 40 nA):
For high resolution investigations:
- 0.6 nm at 30 kV (STEM mode)
- 0.8 nm at 15 kV at optimal WD
- 1.4 nm at 1 kV at optimal WD
- 2.4 nm at 0.2 kV at optimal WD
- 3.0 nm at 20 kV at 10 nA @ WD = 8.5 mm

- Double condenser lens
- Aperture independent probe current adjustment
- Beam Booster
- Brightness of the electron probe maintained for low landing energies

3) Electron Optics: Modern Microscopes

MERLIN™ – Analytical power for the sub-nanometer world

- High stability field emitter cathode
- Maximum probe current 300 nA
- Beam Booster
- Brightness of the electron probe maintained for low landing energies
- Energy selective Backscatter detector (EsB)
- In-lens Secondary Electron detector
- GEMINI® II final lens

- Proven GEMINI® final lens design
- New double condenser lens for highest probe current possibilities (300 nA)
- Beam booster technology maintains brightness of all electron probes including low landing energies
- True on-axis in-lens SE and BSE detectors

thomas.lagrange@epfl.ch • www.epfl.ch • cime.epfl.ch • +41 (0)21 6934430
Combined Deceleration of high voltage electrons and the filtering of aberrated electrons provides improved resolution at Low kV imaging.

3) Electron Optics: Modern Microscopes
3) Electron Optics: Modern Microscopes

Specifications

SEI Resolution
- 1.0\(\mu\)m guaranteed at 15\,kV
- 1.5\(\mu\)m guaranteed at 1\,kV, in GB mode
- 2.3\(\mu\)m guaranteed at 1\,kV, in SEM mode
- 0.8\(\mu\)m guaranteed at 30\,kV, in STEM mode
- 3.0\(\mu\)m guaranteed at 15\,kV, 5\,nA, 8\,mm WD

3) Electron Optics: Modern Microscopes
3) Electron Optics: Modern Microscopes

4) Detectors: SEM, signals

- Secondary electrons (~0-30eV), SE
- Backscattered electrons (~eVo), BSE
- Photons: visible, UV, IR, X-rays
- Auger electrons
- Phonons, Heating
- Absorption of incident electrons (EBIC-Current)
4) Detectors: SEM imaging with electrons

Energy spectrum of electrons leaving the sample

- SE: secondary electrons 0-50eV
- BSE: backscattered electrons E>50eV

Electrons with low energy (0-50eV) leaving the sample surface
- Intensity depends on inclination of the surface
- Topography
4) Electron detectors: Secondary Electrons

Photomultiplier Everhart-Thornley detector

Collects and detects lower energy (<100eV) electrons: The positive collector voltage (= +200 à +400V) attracts the SE toward the detector, the 10kV post acceleration give them enough energy to create a bunch of photons for each SE.

4) Electron detectors: In-lens SE

Topographical information with on-axis in-lens SE detector

On axis In-Lens SE detector

- Small collection angle
- Topographical information
- High resolution (low working distances, less spherical aberration and collection geometry limits SE2 and SE3 electrons)
4) Electron detectors: backscattered electrons

- Electrons with high energy (~Eo) backscattered from below the surface
- Intensity depends on density (atomic weight)
- ~composition contrast (Z)

![Nb₃Sn in Cu matrix](image)

5) Electron detectors: Semiconductor BSE

BSE semiconductor detector: a silicon diode with a p-n junction close to its surface collects the BSE (3.8eV per electron hole pair)

- large collection angle
- slow (poor at TV frequency)
- some diodes are split in 2 or 4 quadrants to bring spatial BSE distribution info

Detects higher energy (>5kV) electrons: SEM backscattered electrons
4) Electron detectors: In-lens BSE (EsB)

Compositional contrast with on-axis in column EsB detector

On axis In-Lens EsB detector
- Small collection angle
- Loss BSE – more Z-contrast information
- Bias control for improved selectivity and contrast

GEMINI® II design
- Complete detection system
- Unique double in-lens detection
- Acquisition of pure secondary and backscatter electron signals
- Separation of compositional, topographical and crystalline surface information

Electron matter interactions in a thin sample: Inelastic Scattering

Characteristic X-rays

Energy Dispersive Spectroscopy

Light Elements
- Light Elements are difficult to measure as valence electrons are involved in characteristic X-ray production and chemistry. For heavy elements (>1 ha) the Kα x-ray is not associated with chemical bonds.

Heavy Elements e.g. Fe-Kα (metal) - Fe-Kα (oxide) - No bond shift

Light Elements e.g. C-Kα (diamond) – C-Kα (Graphite) – there is a bond shift
4) X-ray Detectors: Silicon Lithium

- X-Ray energy converted to electrical charges:
  - 3.8eV / electron-hole pair in average
  - Must operate an optimum working distance for highest solid angle (known take-off angle)
  - electronic noise+ imperfect charge collection: 150 eV resolution / Mn Ka line
  - Detector acts like a diode: at room temperature the leak current for 1000V would be too high!
  - The FET produces less noise if cooled and Li migrates at room temperature, which is also why the detector is cooled using LN2

4) X-ray Detectors: Silicon Drift Detector (SDD)

- Still 3.8eV / electron-hole pair in average
- electronic noise+ imperfect charge collection: 129 eV resolution / Mn Ka line
- The optimized FET design has lower noise than old generation SiLi detectors, so it can be cooled with thermoelectric cooler (PTEC)
- Improved light element sensitivity
- Windowless systems
QUESTIONS?