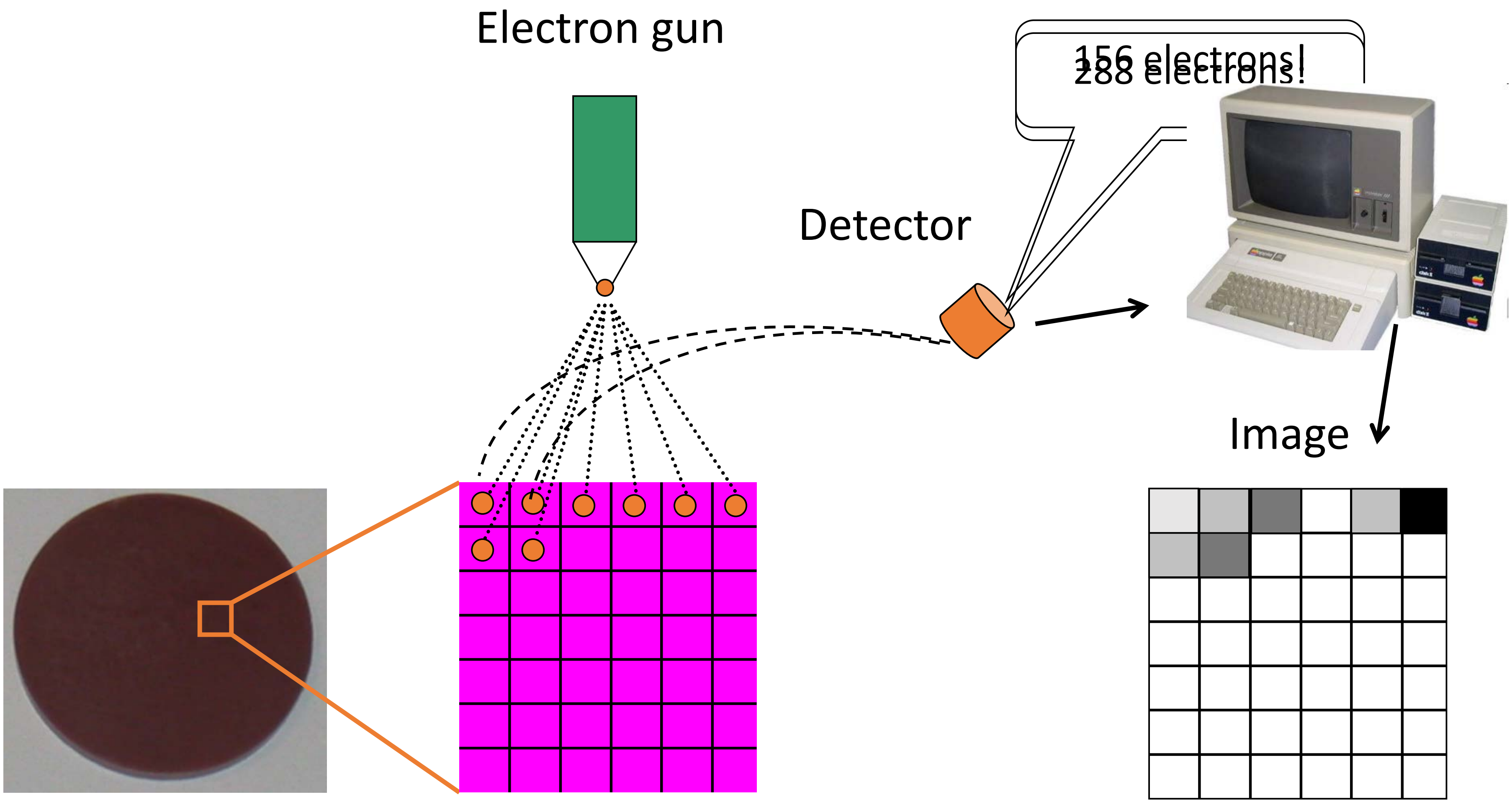
上周课程回顾

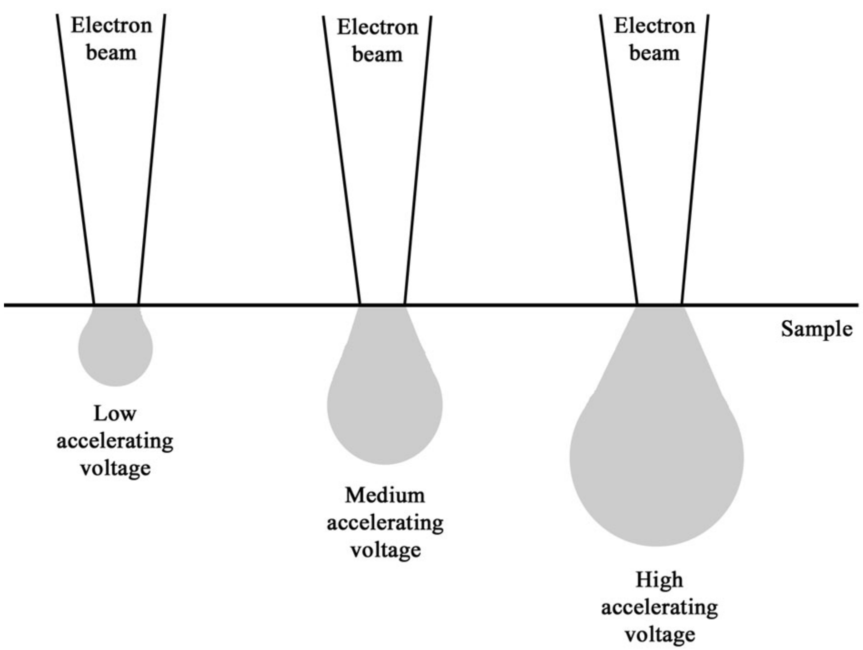
- 表征技术的意义：
  - 解析
  - 理解
  - 鉴定
  - 操控
- 表征技术的三要素
  - Probe (Incident particles)
  - Probe-sample interactions
  - Response (outgoing particles)
- 表征技术的分类
- SEM的用途
  - Topography and morphology
  - Chemistry
  - In situ imaging

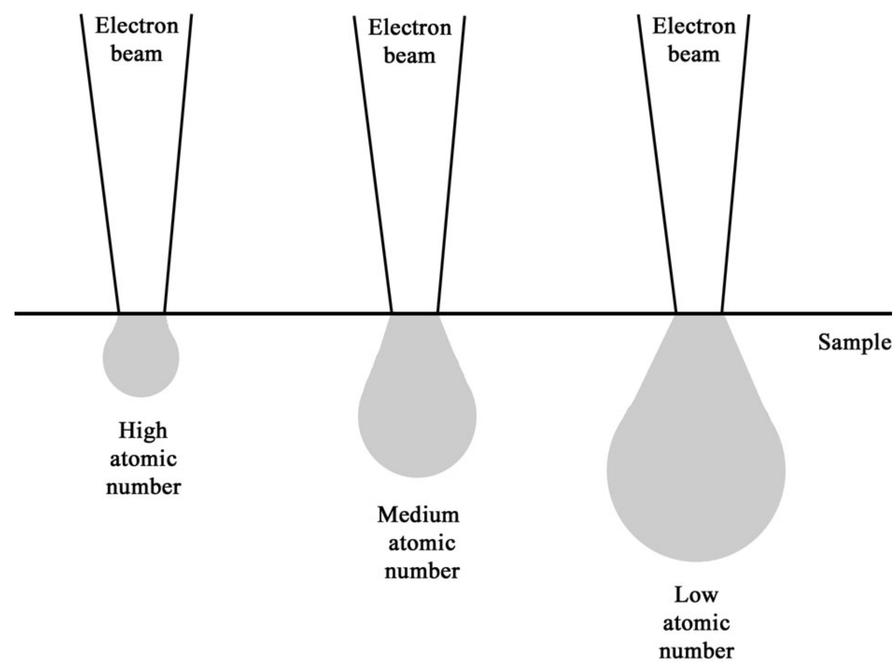
How do we get an image?



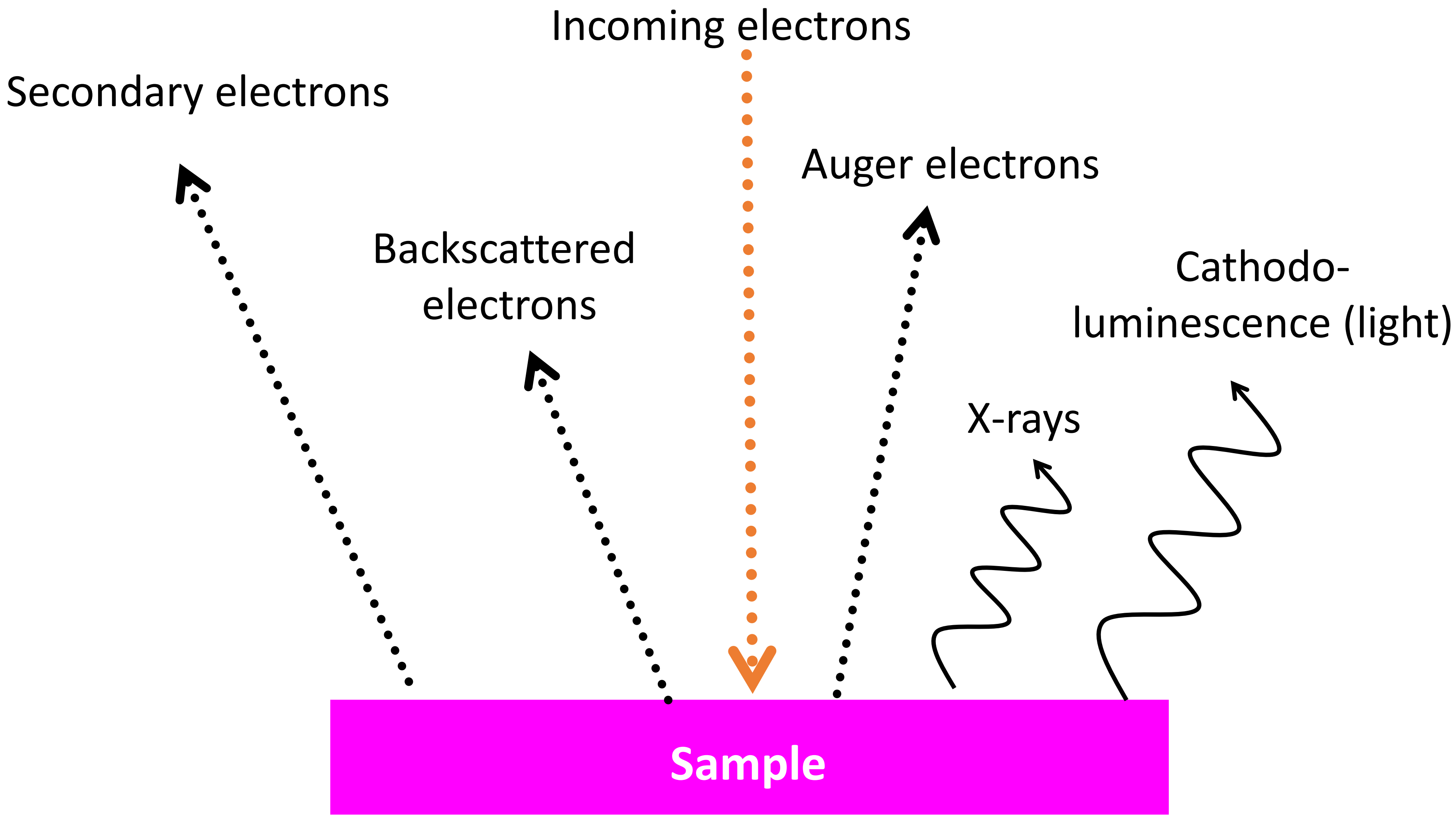
Electron beam-sample interactions

- The incident electron beam is scattered in the sample, both elastically and inelastically
- This gives rise to various signals that we can detect (more on next slide)
- Interaction volume increases with increasing *acceleration voltage* and decreases with increasing *atomic number*

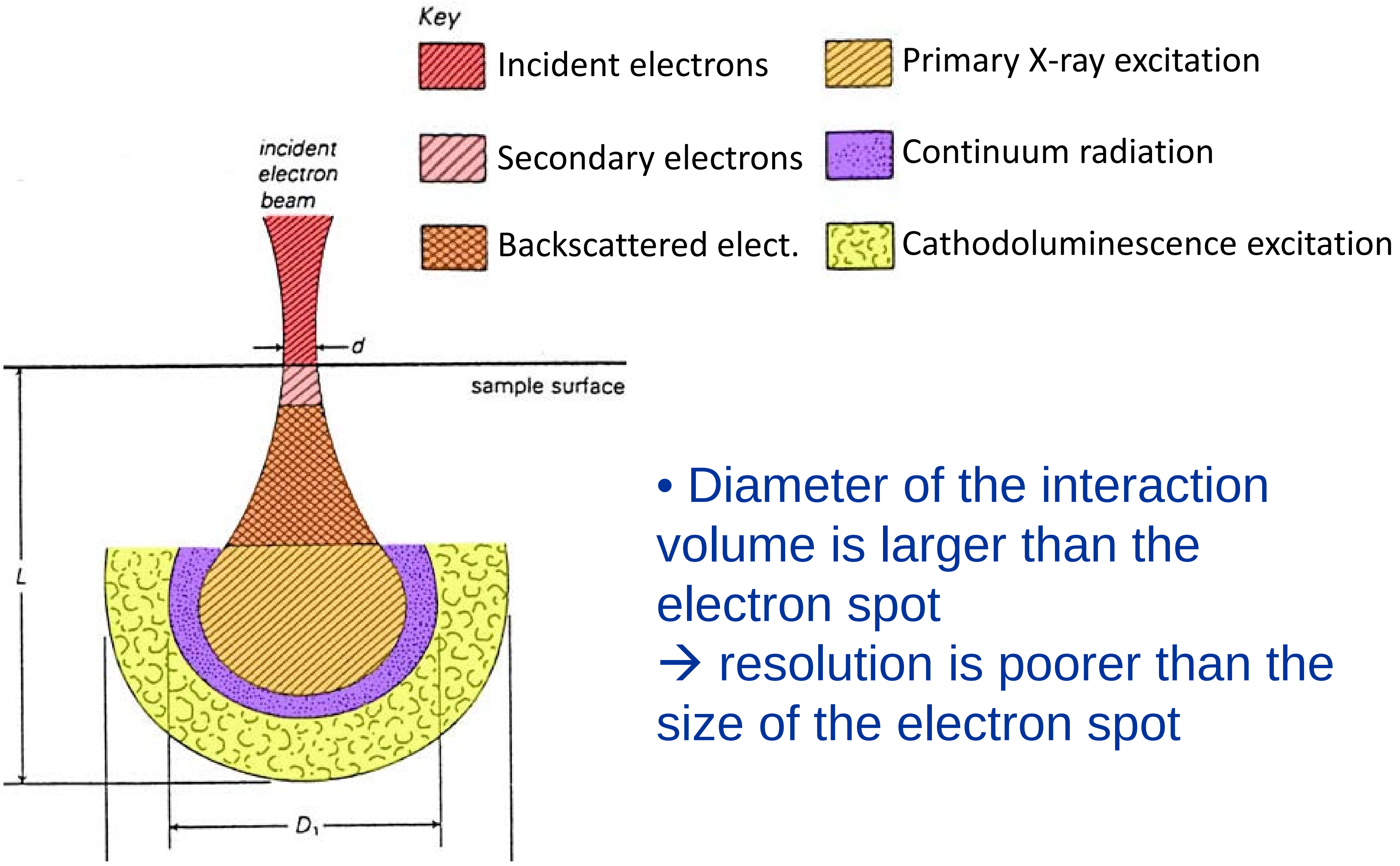




Signals from the sample



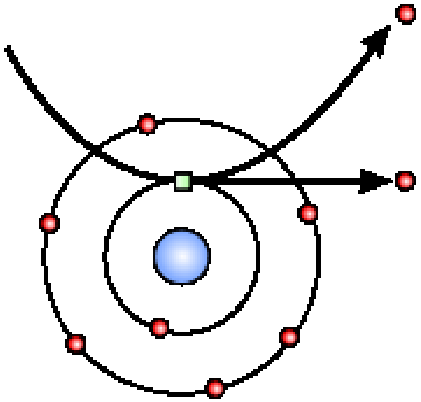
Where do the signals come from?

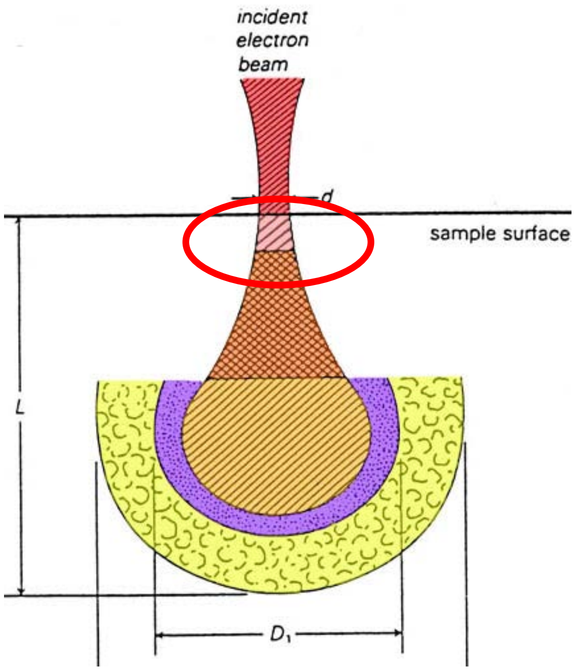


- Diameter of the interaction volume is larger than the electron spot
→ resolution is poorer than the size of the electron spot

Secondary electrons (SE)

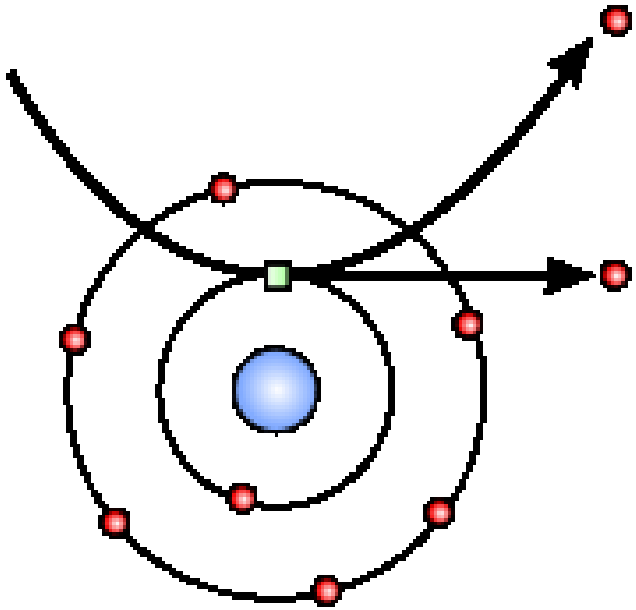
- Generated from the collision between the incoming electrons and the loosely bonded outer electrons
- Low energy electrons (~10-50 eV)
- Only SE generated close to surface escape (topographic information is obtained)
- Number of SE is greater than the number of incoming electrons
- We differentiate between SE1 and SE2





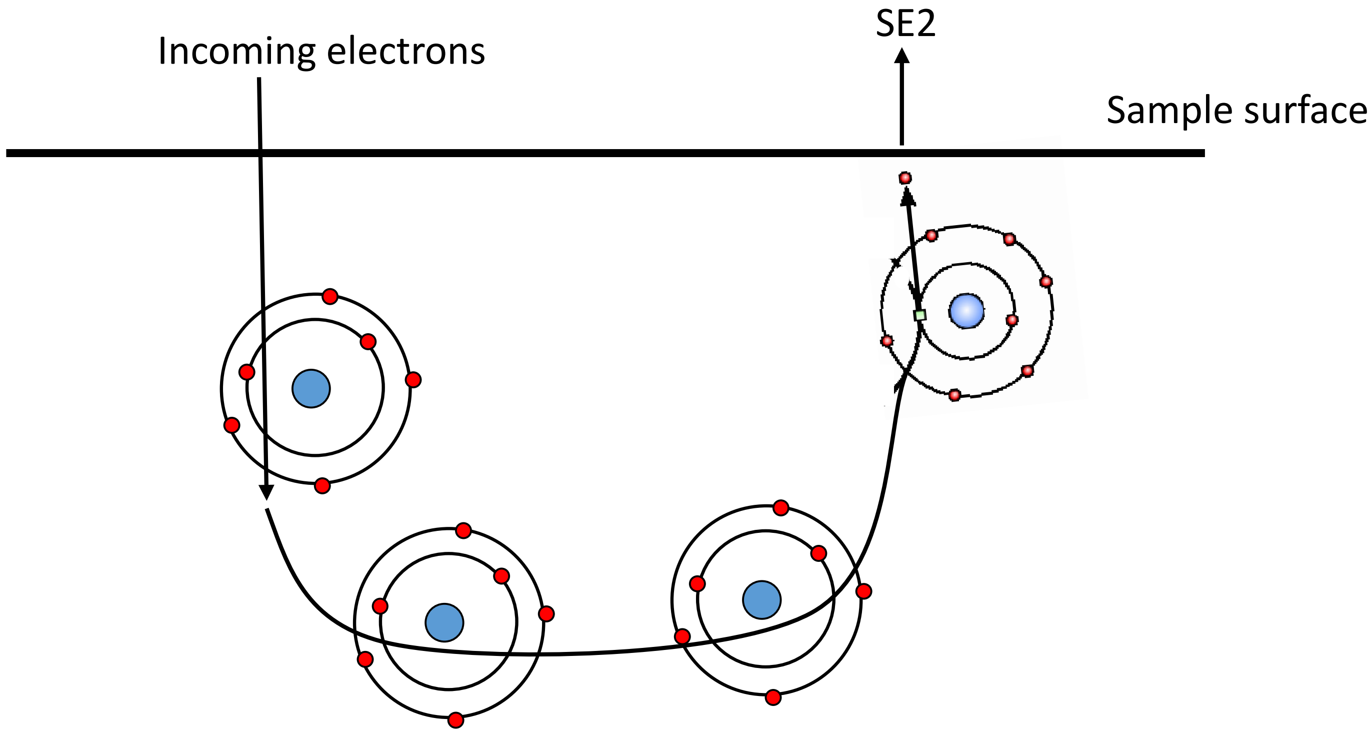
SE1

- The secondary electrons that are generated by the incoming electron beam as they enter the surface
- High resolution signal with a resolution which is only limited by the electron beam diameter



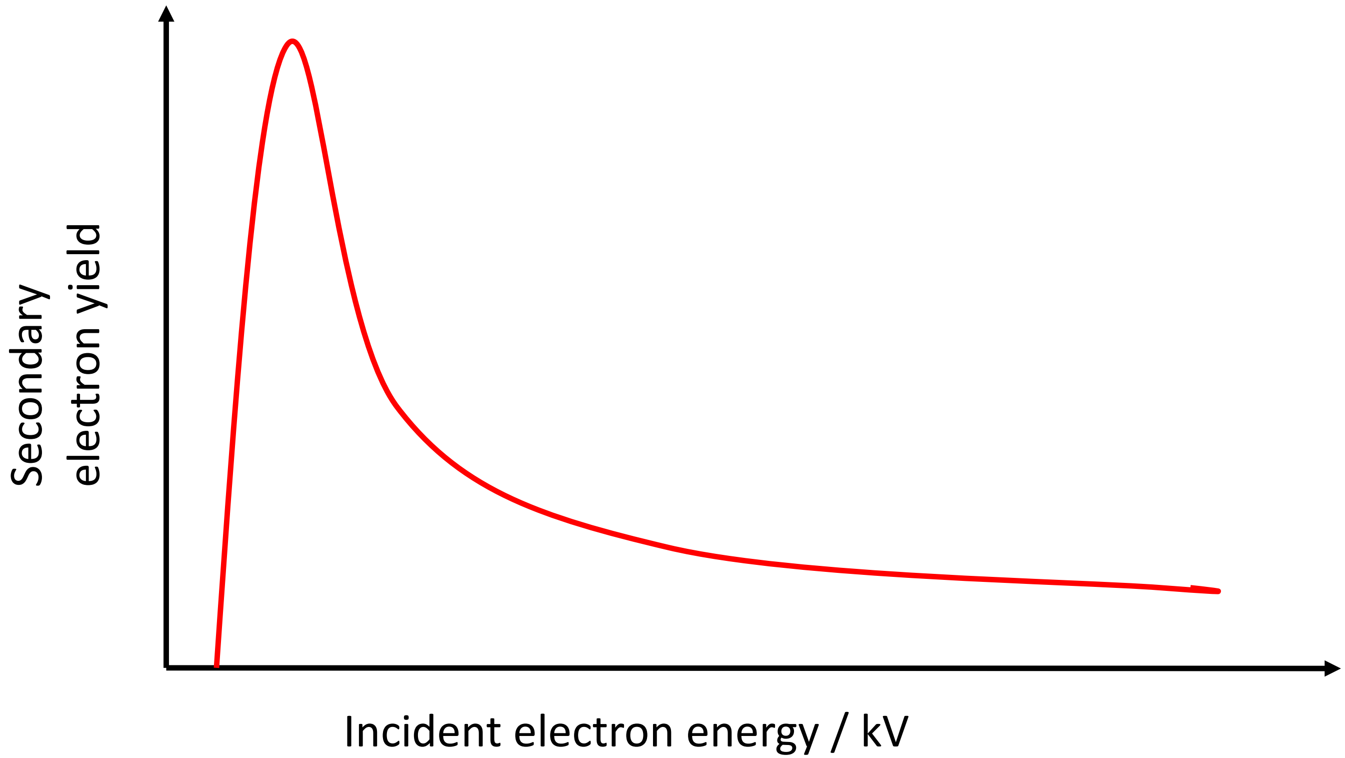
SE2

- The secondary electrons that are generated by the backscattered electrons that have returned to the surface after several inelastic scattering events
- SE2 come from a surface area that is bigger than the spot from the incoming electrons → resolution is poorer than for SE1 exclusively



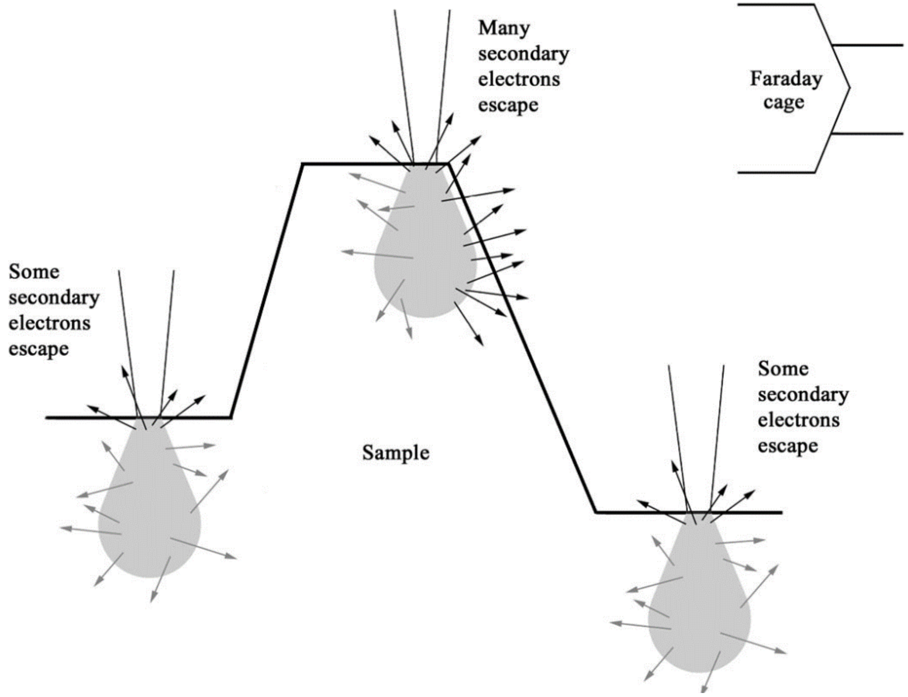
Factors that affect SE emission

- Work function of the surface
- Beam energy and beam current
  - Electron yield goes through a maximum at low acc. voltage, then decreases with increasing acc. voltage



Factors that affect SE emission

- Atomic number (Z)
  - More SE2 are created with increasing Z
  - The Z-dependence is more pronounced at lower beam energies
- The local curvature of the surface (the most important factor – morphology contrast)

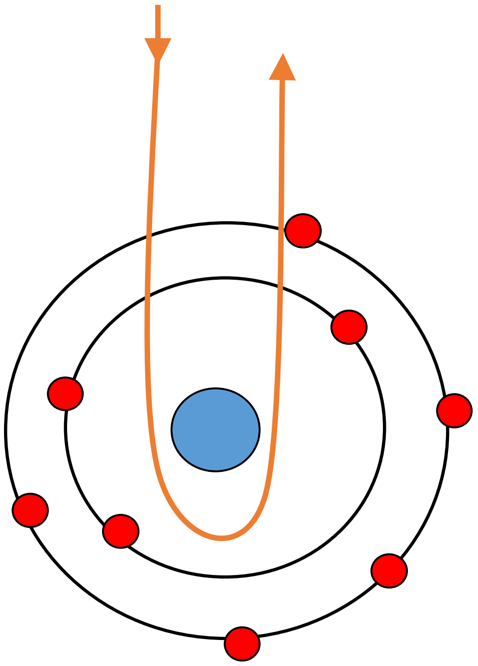


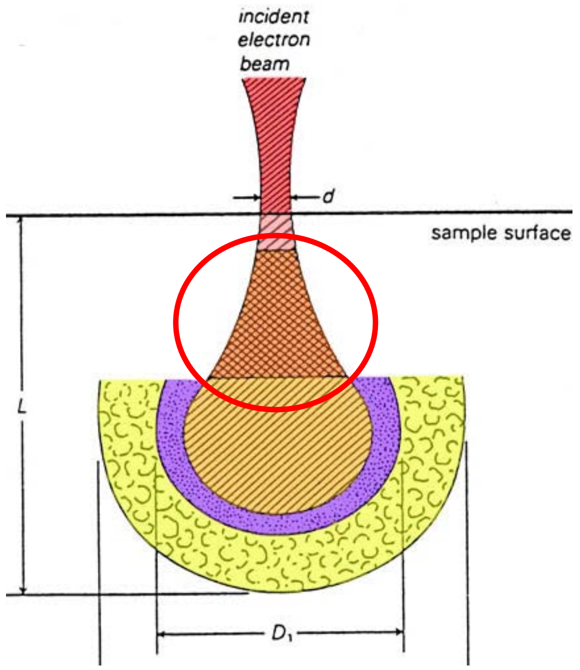
High resolution image setup

- By placing the secondary electron detector inside the lens, mainly SE1 are detected
  - Resolution of 1 - 2 nm is possible

Backscattered electrons (BSE)

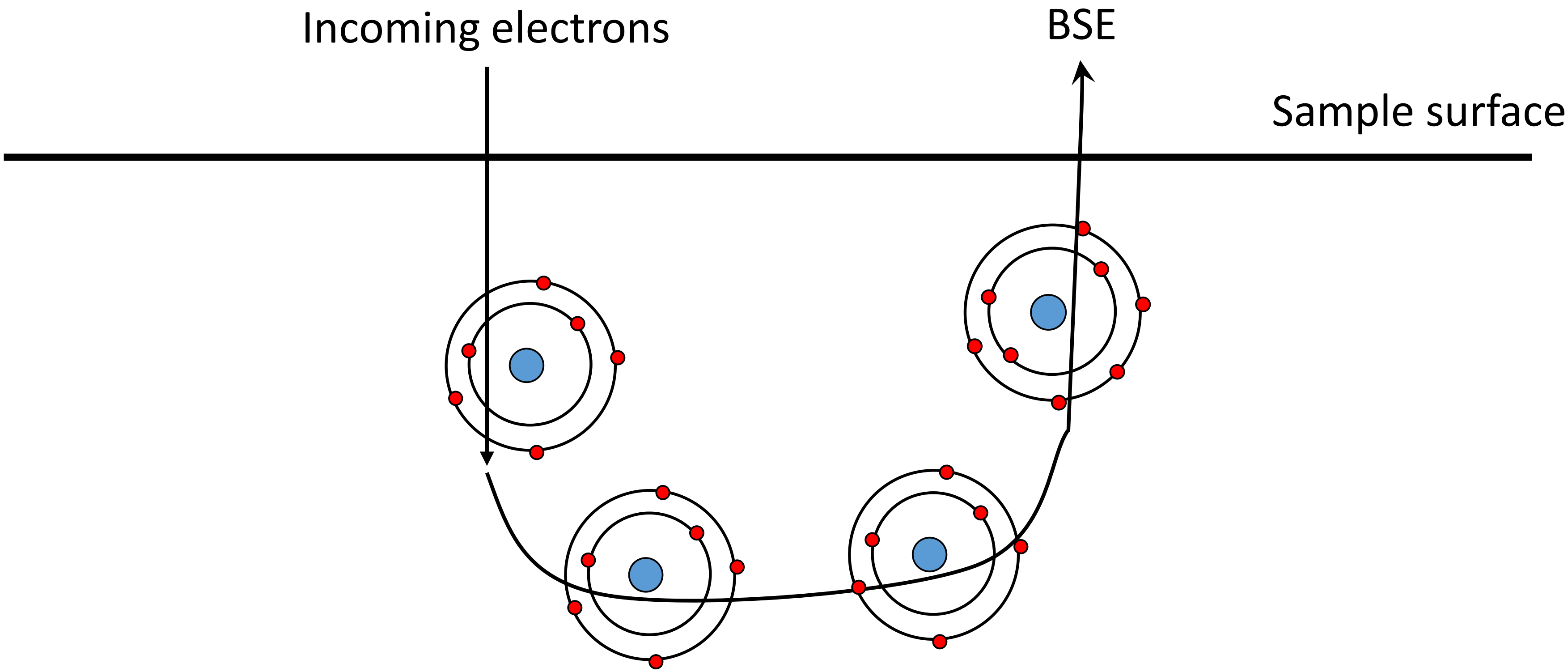
- A fraction of the incident electrons is retarded by the electro-magnetic field of the nucleus and if the scattering angle is greater than 180° the electron can escape from the surface





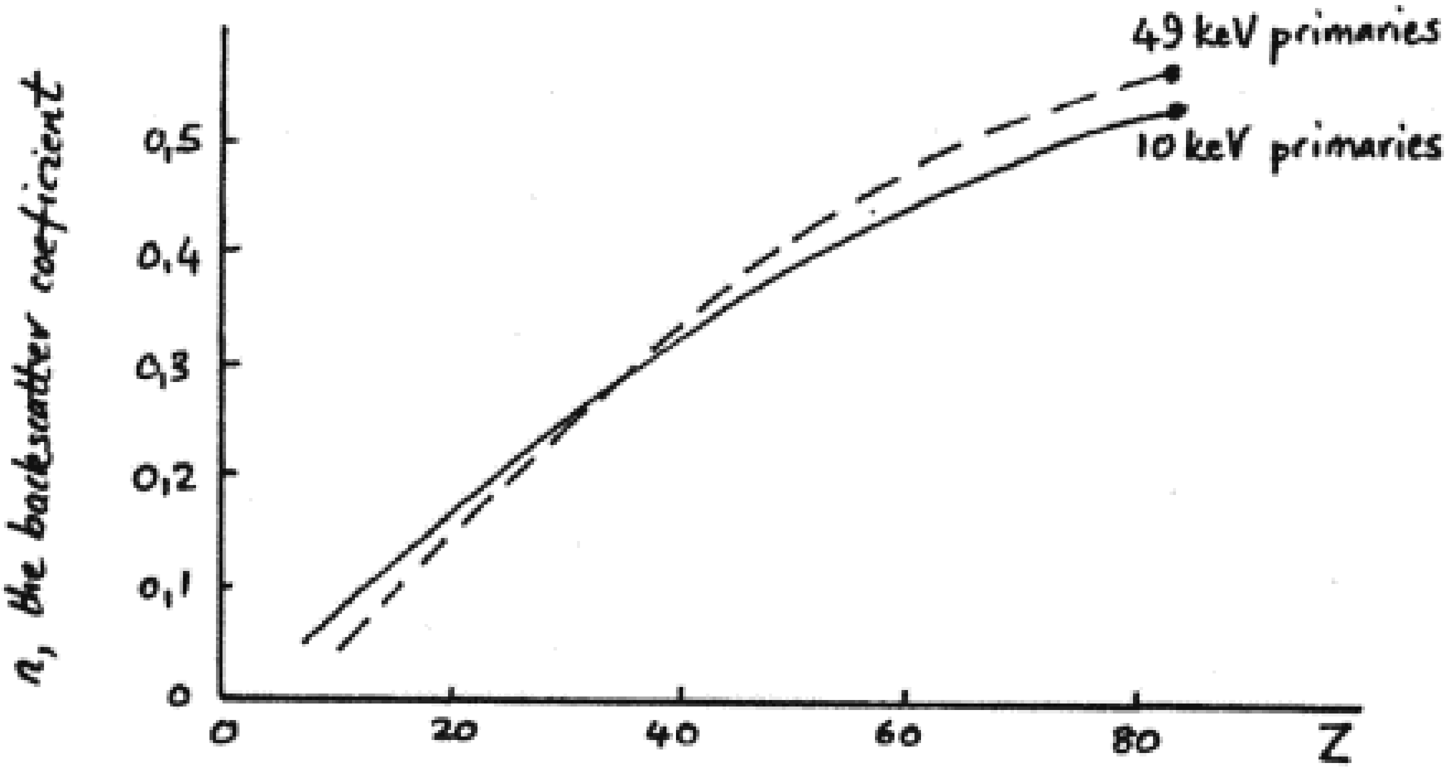
Backscattered electrons (BSE)

- High energy electrons (elastic scattering)
- Fewer BSE than SE



BSE as a function of atomic number

- For phases containing more than one element, it is the average atomic number that determines the backscatter coefficient



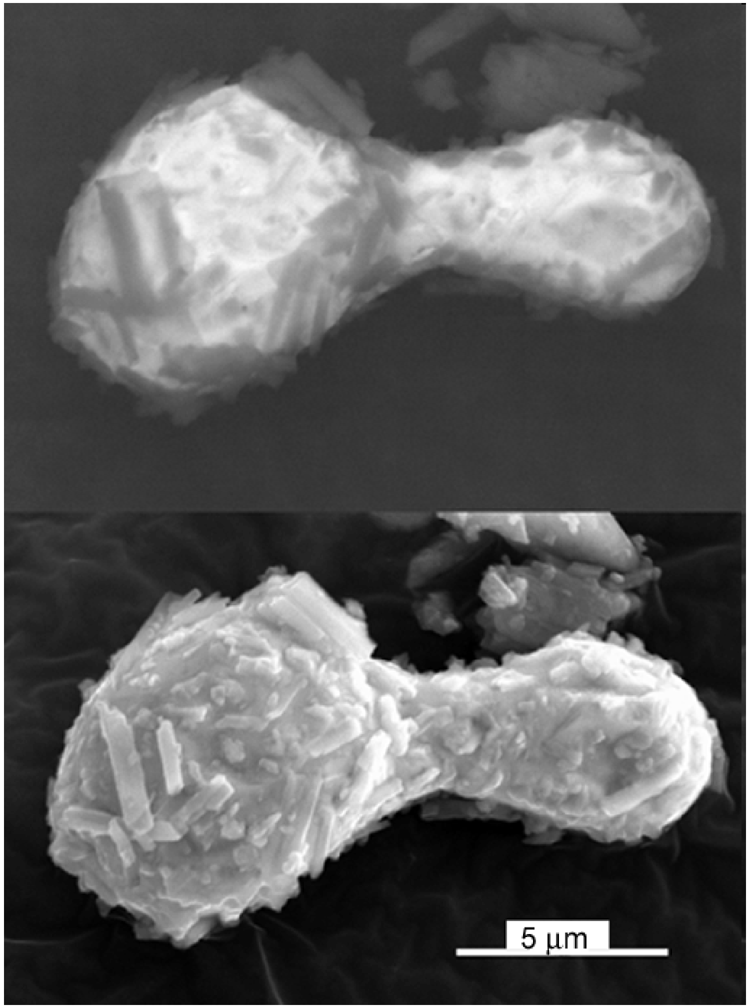
Factors that affect BSE emission

- Direction of the irritated surface
  - more electrons will hit the BSE detector when the surface is aligned towards the BSE detector

- Average atomic number

- When you want to study differences in atomic numbers, the sample should be as levelled as possible (sample preparation is an issue!)

BSE vs SE

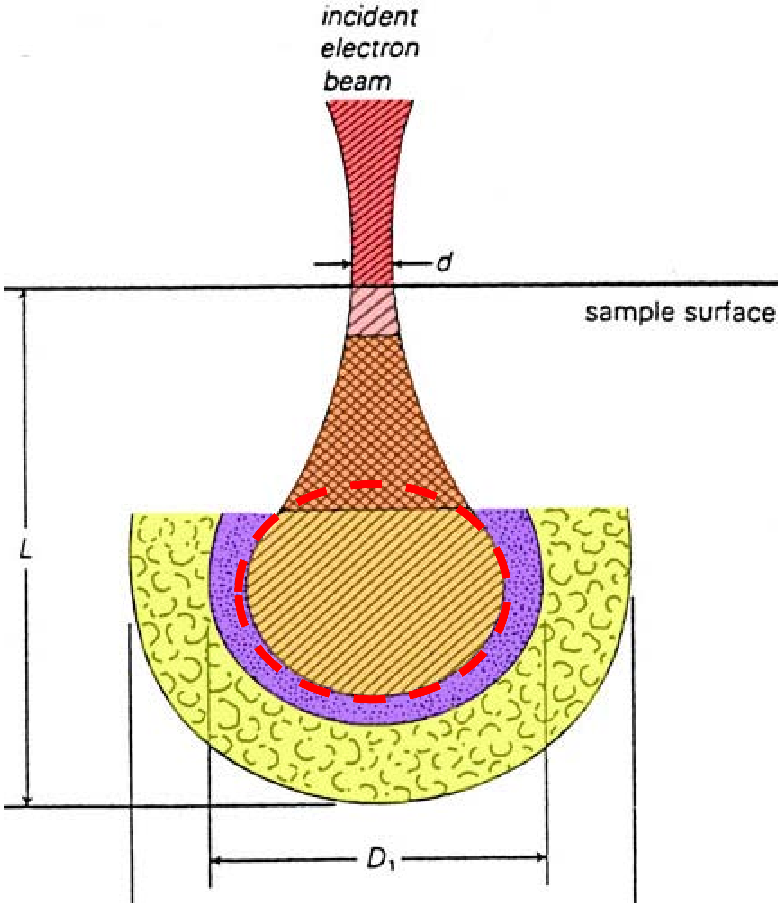


BSE

SE

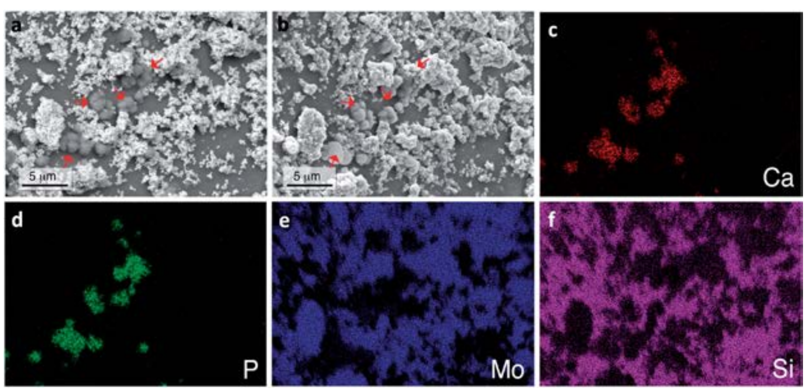
X-rays

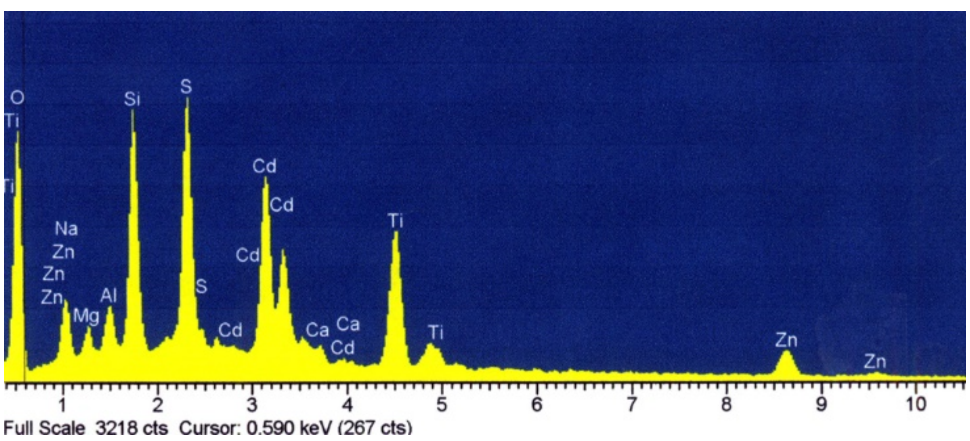
- Photons not electrons
- Each element has a fingerprint X-ray signal
- Poorer spatial resolution than BSE and SE
- Relatively few X-ray signals are emitted and the detector is inefficient
  - relatively long signal collecting times are needed



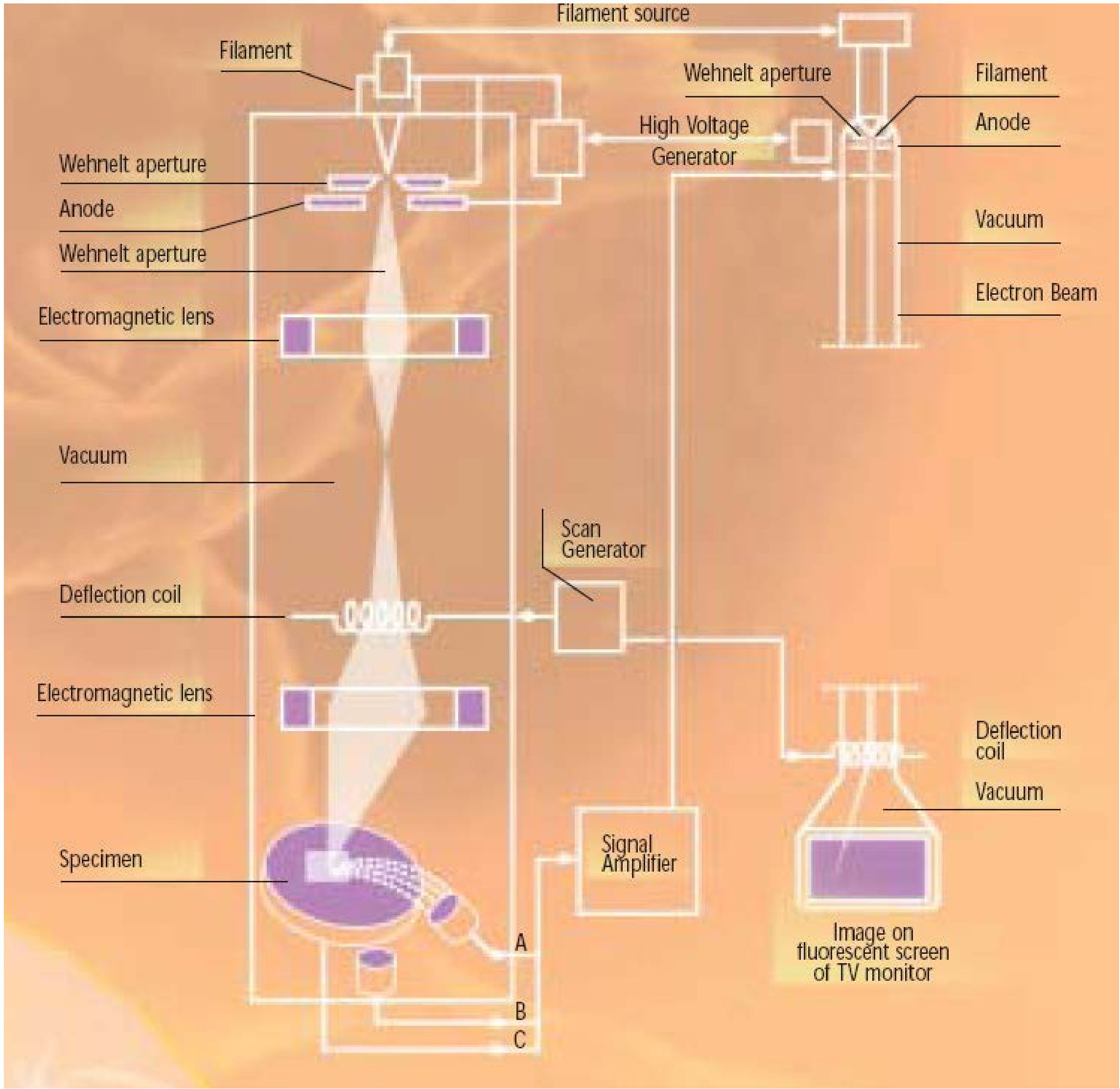
X-rays

- Most common spectrometer: EDS (energy-dispersive spectrometer)
- Signal overlap can be a problem
- We can analyze our sample in different modes
  - spot analysis
  - line scan
  - chemical concentration map (elemental mapping)
- Statistics
  - Signal-to-noise ratio
- Drift in electron beam with time
- Build-up of a carbonaceous contamination film after extended periods of electron probe irradiation





Components of the instrument



- electron gun (filament)
- electromagnetic optics
- scan coils
- sample stage
- detectors
- vacuum system
- computer hardware and software (not trivial!!)

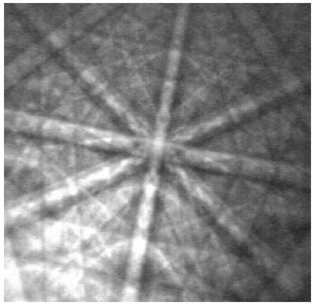
Accessories

Accessories on Quanta 200:

- GAD – Gaseous Analytical Detector
  → for X-ray analysis in gaseous environments
- GSED – Gaseous Secondary Electron Detector
  → 500 µm aperture, allowing 20 Torr chamber pressure
- Hot stage GSED
  → Must be used at temperatures above 500° C
- EBSD – Electron Backscatter Diffraction
  → Grain orientation, grain and subgrain structures, phase identification, micro textures
- Hot stages – 1000° C and 1500° C

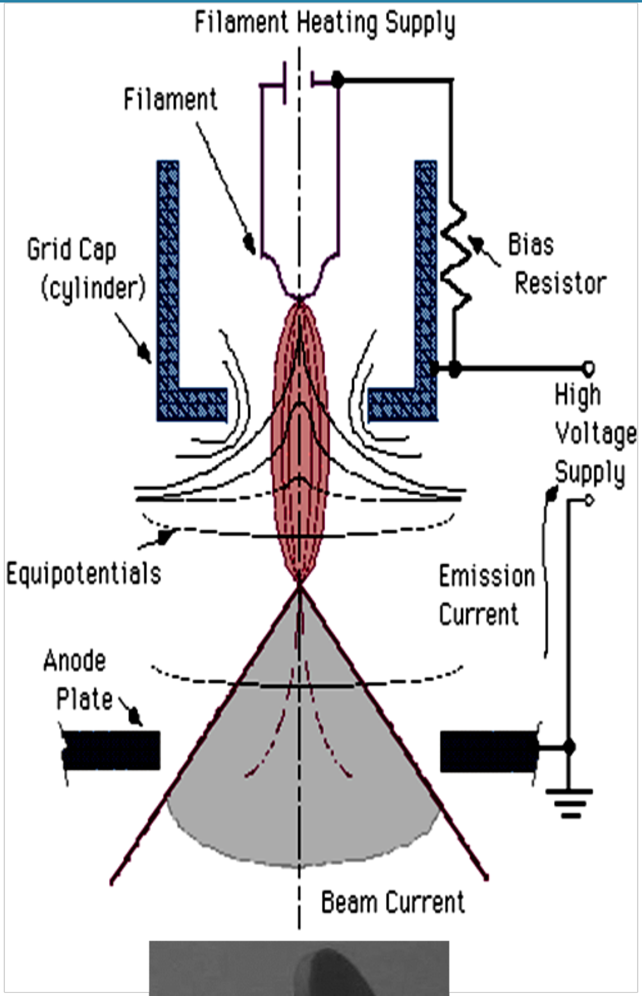
- ETD – Everhart-Thornley Detector
  → Secondary electron detector
- LFD – Large Field Detector
  → used in low vacuum and ESEM mode (SE)
- SSD-BSD – Solid State Backscattered Detector
  → Backscatter electrons
- EDS – Energy dispersive spectroscopy
  → X-ray analysis

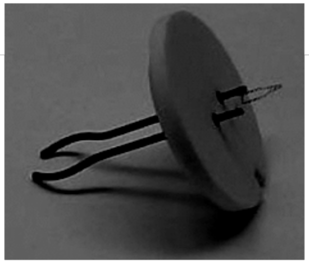



Electron guns

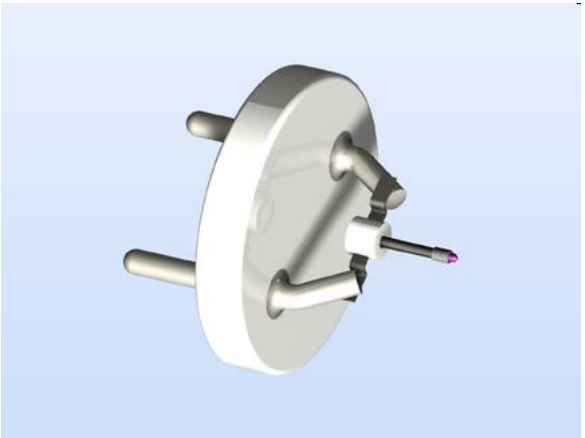
- We want many electrons per time unit per area (high current density) and as small electron spot as possible
- Traditional guns: thermionic electron gun (electrons are emitted when a solid is heated)
  - W-wire, LaB_6-crystal
- Modern: field emission guns (FEG) (cold guns, a strong electric field is used to extract electrons)
  - Single crystal of W, etched to a thin tip



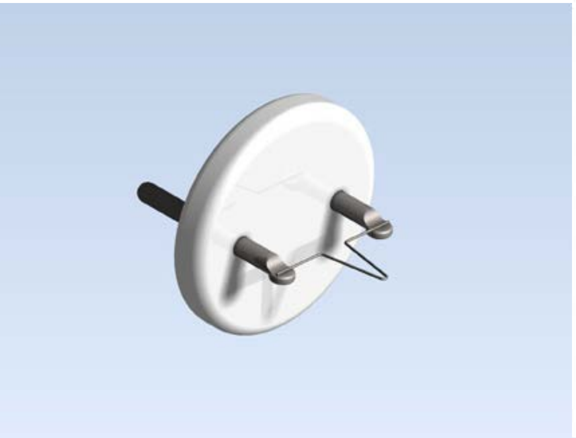


Electron guns

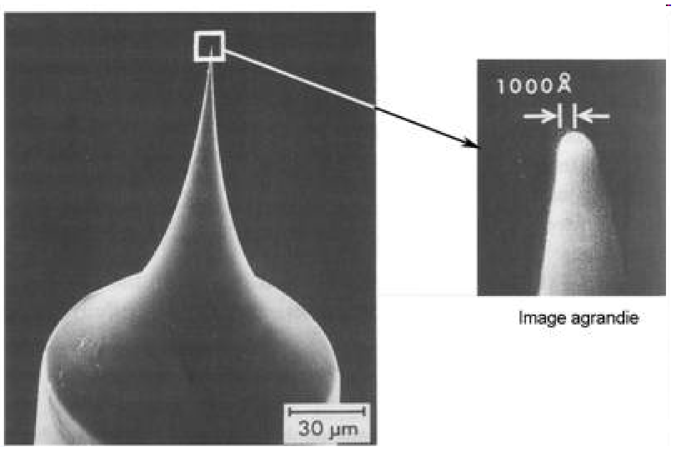
- With field emission guns we get a smaller spot and higher current densities compared to thermionic guns
- Vacuum requirements are tougher for a field emission guns



Single crystal of LaB_6



Tungsten wire



Field emission tip

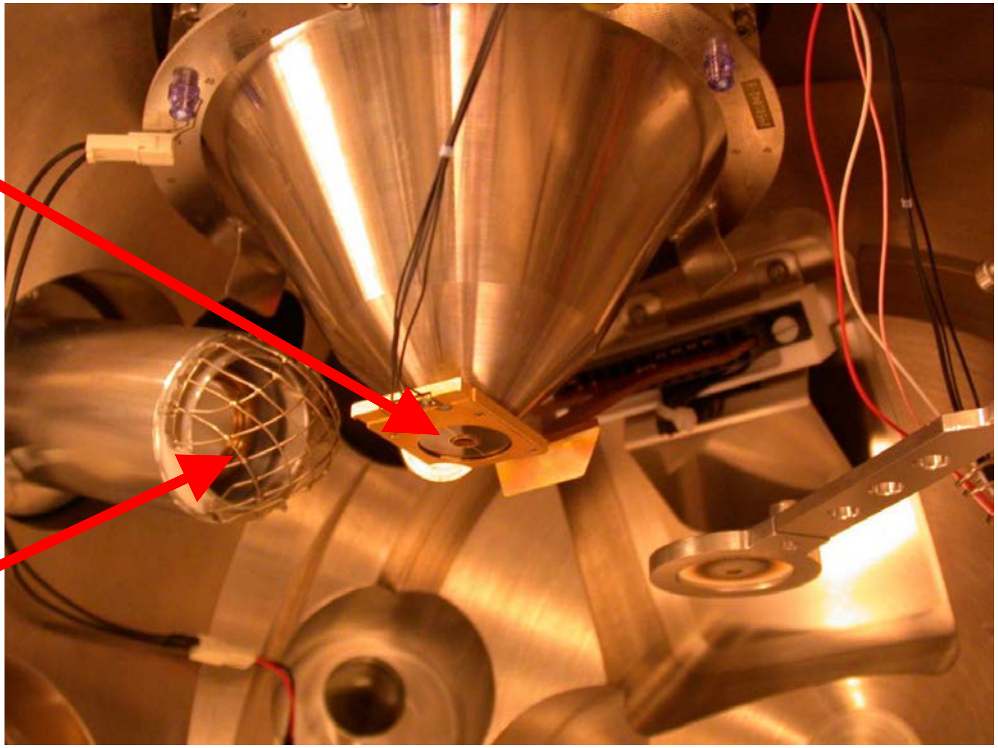
Why do we need vacuum?

- Chemical (corrosion!!) and thermal stability is necessary for a well-functioning filament (gun pressure)
  - A field emission gun requires ~ 10^{-10} Torr
  - LaB_6: ~ 10^{-6} Torr

- The signal electrons must travel from the sample to the detector (chamber pressure)
  - Vacuum requirements is dependant on the type of detector

Detectors

Backscattered electron detector:
(Solid-State Detector)

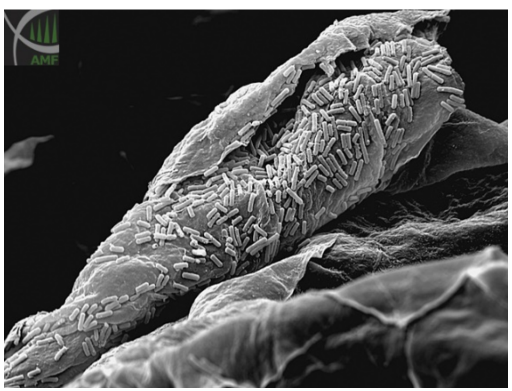


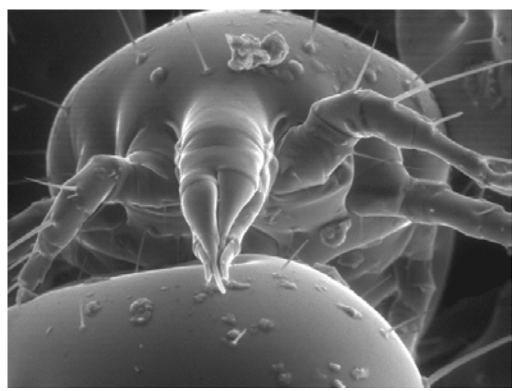
Secondary electron detector:
(Everhart-Thornley)

- Secondary electrons: Everhart-Thornley Detector
- Backscattered electrons: Solid State Detector
- X-rays: Energy dispersive spectrometer (EDS)

Environmental SEM: ESEM

- Traditional SEM chamber pressure: ~10^{-6} Torr
- ESEM: 0.08 – 30 Torr
- Various gases can be used
- Requires different SE detector





Why ESEM?

- To image challenging samples such as:
  - insulating samples
  - vacuum-sensitive samples (e.g. biological samples)
  - irradiation-sensitive samples (e.g. thin organic films)
  - “wet” samples (oily, dirty, greasy)

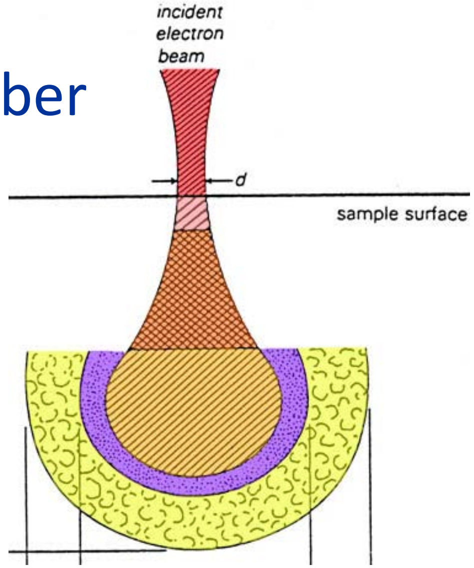
- To study and image chemical and physical processes in-situ such as:
  - mechanical stress-testing
  - oxidation of metals
  - hydration/dehydration (e.g. watching paint dry)

Some comments on resolution

- Best resolution that can be obtained: size of the electron spot on the sample surface
  - The introduction of FEG has dramatically improved the resolution of SEM
- The volume from which the signal electrons are formed defines the resolution
  - SE image has higher resolution than a BSE image
- Scanning speed:
  - a weak signal requires slow speed to improve signal-to-noise ratio
  - when doing a slow scan drift in the electron beam can affect the accuracy of the analysis

Summary

- Signals:
  - Secondary electrons (SE): mainly topography
    - Low energy electrons, high resolution
    - Surface signal dependent on curvature
  - Backscattered electrons (BSE): mainly chemistry
    - High energy electrons
    - “Bulk” signal dependent on atomic number
  - X-rays: chemistry
    - Longer recording times are needed



Summary

- The scanning electron microscope is a versatile instrument that can be used for many purposes and can be equipped with various accessories
- An electron probe is scanned across the surface of the sample, and detectors interpret the signal as a function of time
- A resolution of 1 – 2 nm can be obtained when operated in a high resolution setup
- The introduction of ESEM and the field emission gun have simplified the imaging of challenging samples

Next class

- *SEM experiment*
- Provide your samples, e.g. your hair, or anything curious – needs to be dry and solid-state; Other samples in your research – solid state, basically conductive
- 助教：童安宇
- 综合科研楼125室(一楼)
- 1:00-1:30：第1、2组
- 1:30-2:00：第3、4组
- 2:00-2:30：第5、6组
- 2:30-3:00：第7、8组