Signal detection in electron microscopy

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Contents

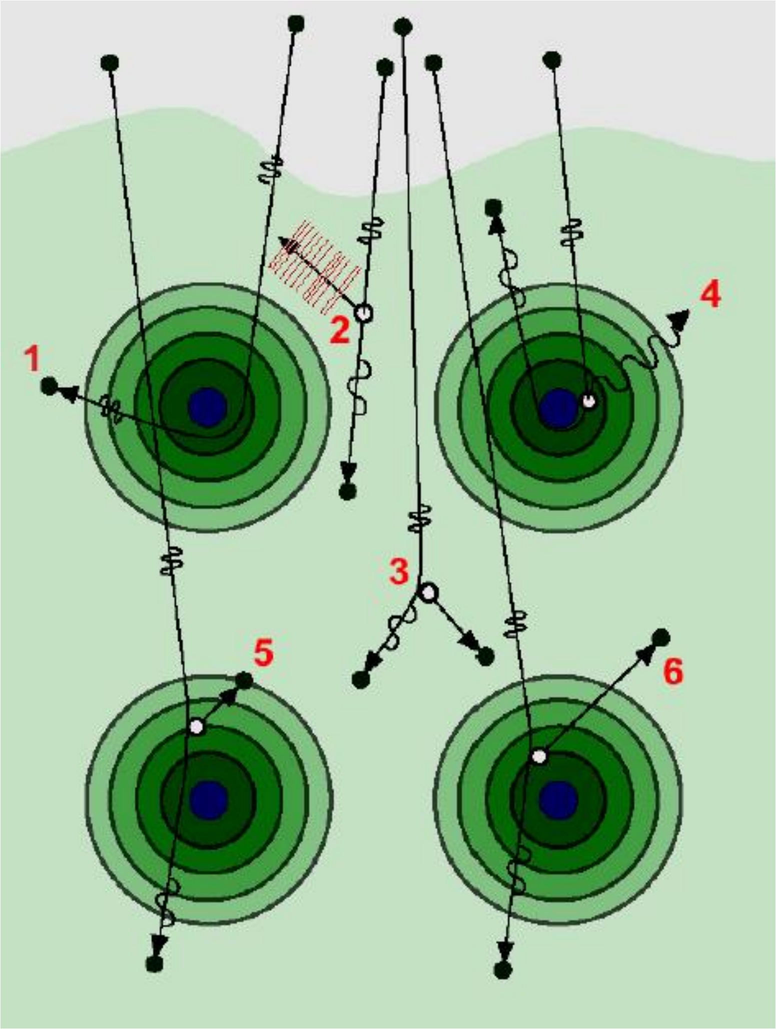
Detector basics – what is detector?

- Detector is part of imaging system of microscope
- It collects signal radiation in EM, amplifies and converts into form which can be digitized and visualized
- Detectors can be divided into different categories based on:
    - Type of signal they collect
    - Technology they use

Electron beam interaction with sample

• **Scattering processes:**

- **Elastic**
  - Rutherford or Mott scattering (1)
    -> Low Loss BSE
  - Compton scattering (3)
- **Inelastic**
  - Inner/interband transitions (5)
  - Inner shell ionization (6)
    -> **SE, AE**
  - X-ray emission (4)
  - phonon and plasmon emission (2)
    -> **BSE**

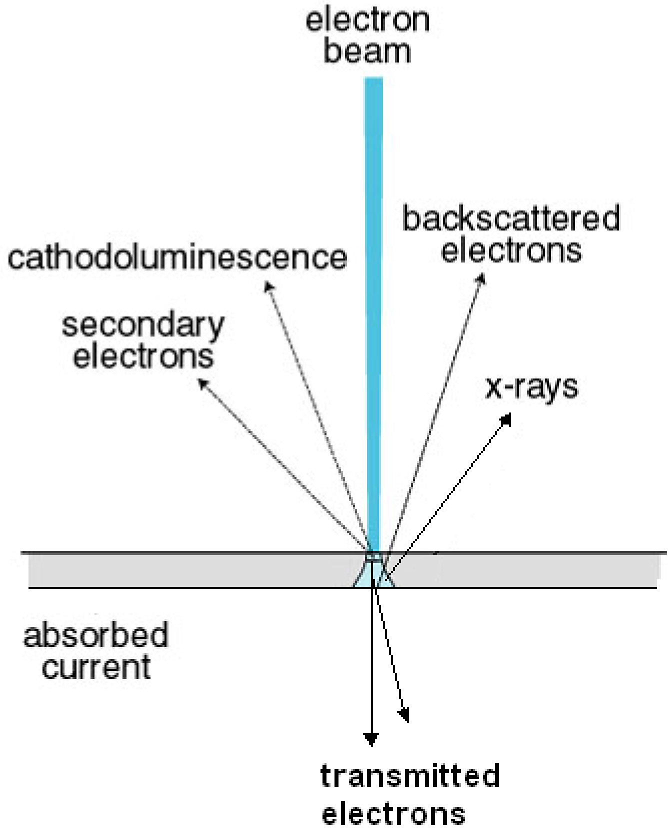


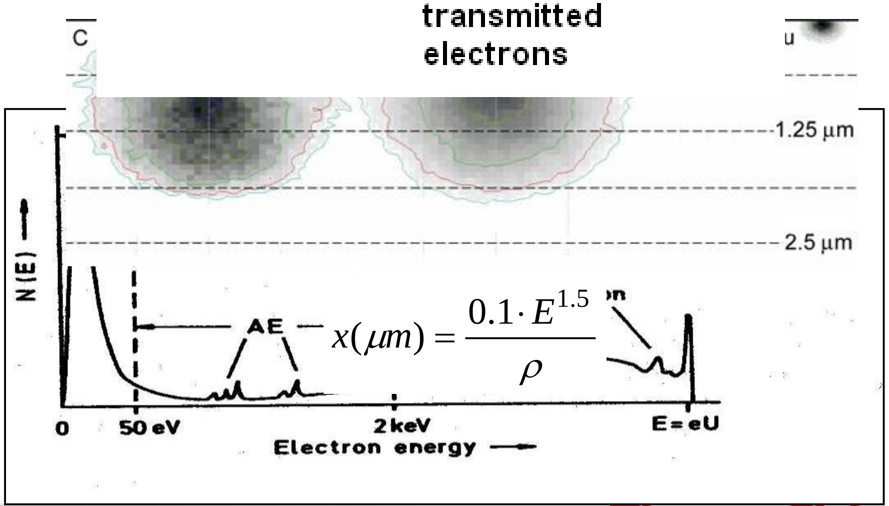
Types of signals detected in electron microscopes

- Electron or ion beam interaction with sample produce various types of particles/radiation = signal
  - Electrons
    - Secondary
    - Backscattered
    - Auger
    - Transmitted
    - Absorbed (specimen current)
  - Photons
    - Cathodoluminescence
    - X-ray
  - Ions
    - Secondary

Electrons

- Electron signals
  - Secondary electrons – (SE), E<50eV, small escape depth (~10nm) → best resolution
  - Backscattered electrons – (BSE), 50eV<E≤$E_{primary\ beam}$, large interaction volume
  - Auger electrons, E>50eV, characteristic peaks, surface material composition information
  - Transmitted electrons (sample must be thin enough)
  - Absorbed electrons/current

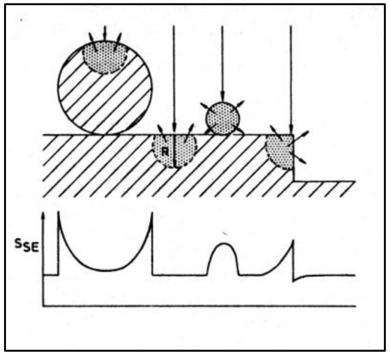


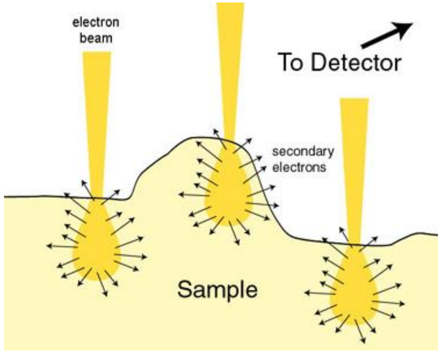


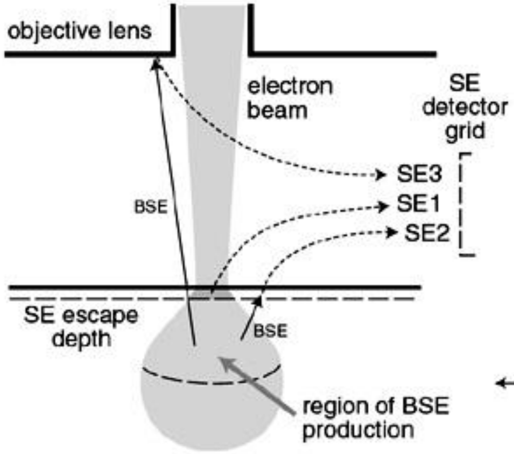
http://www4.nau.edu/microanalysis/Microprobe-SEM/Signals.html

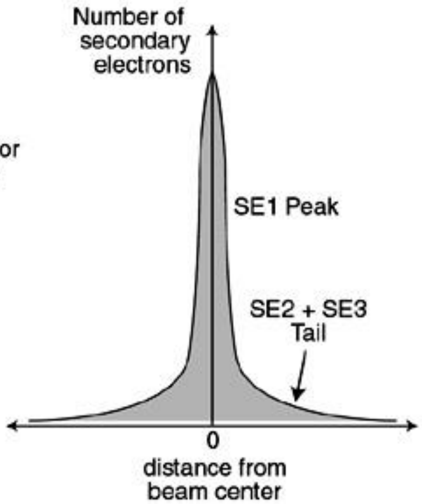
Secondary electrons

- Electrons emitted by the sample under electron beam (inner shell ionization effects)
- Small escape depth → high resolution
- Yield depends on local sample tilt → Topography contrast
- Yield depends on local magnetic or electrostatic fields
- Signal is polluted by SE created by BSE in sample – SE2, or on some other surface in specimen chamber (usually final lens) – SE3 → noise (information from different part of with different contrast)









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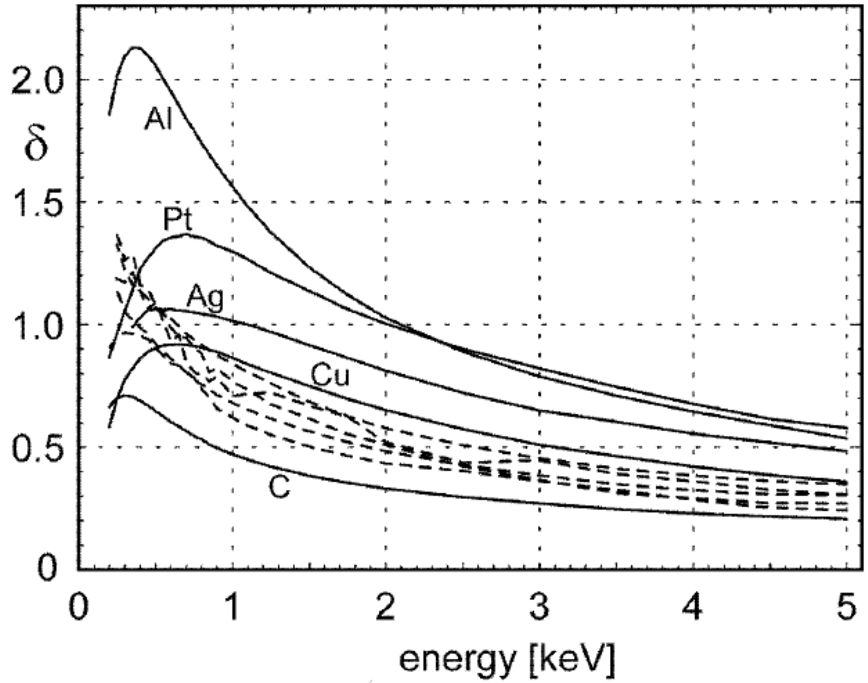
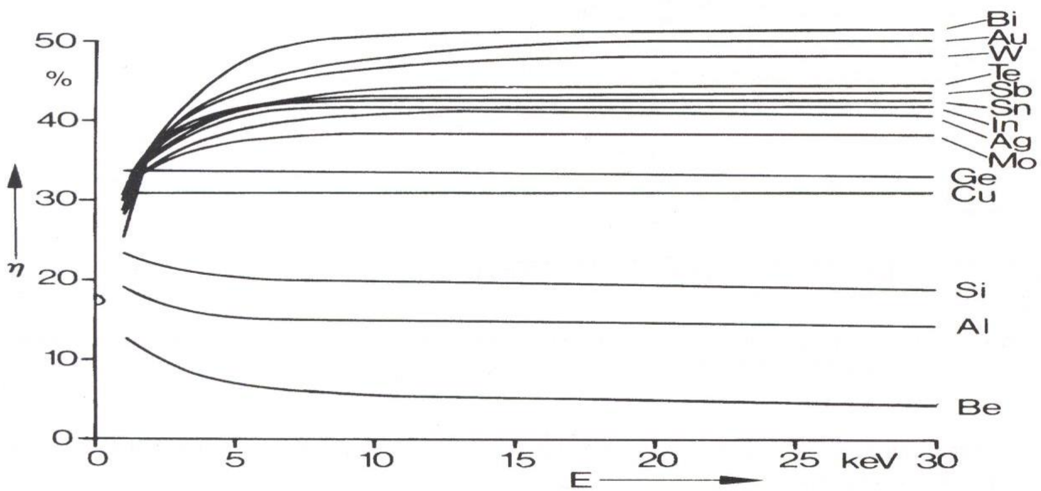
Secondary electrons

- Different yield for different materials → material contrast
- Yield changes with primary beam energy → for most materials there is equilibrium point where secondary emission balances primary beam current, i.e. no charging occurs even in case that sample is insulator.



Backscattered electrons

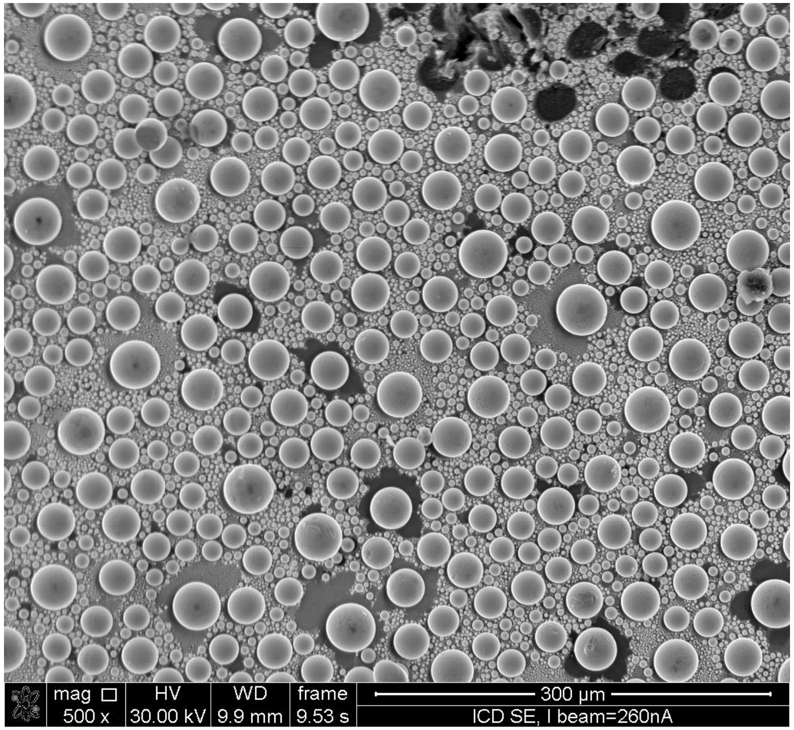
- Primary beam electrons reflected by the sample (elastically or inelastically)
- Yield depends on atomic number of sample material → low loss BSEs reflected close to beam axis – high take off angle
- Yield depends on local tilt of sample surface → BSEs reflected far from beam axis – low take off angles
- Yield depends on crystal orientation → channeling contrast & EBSD(P) = Electron Back Scattered Diffraction (Pattern)

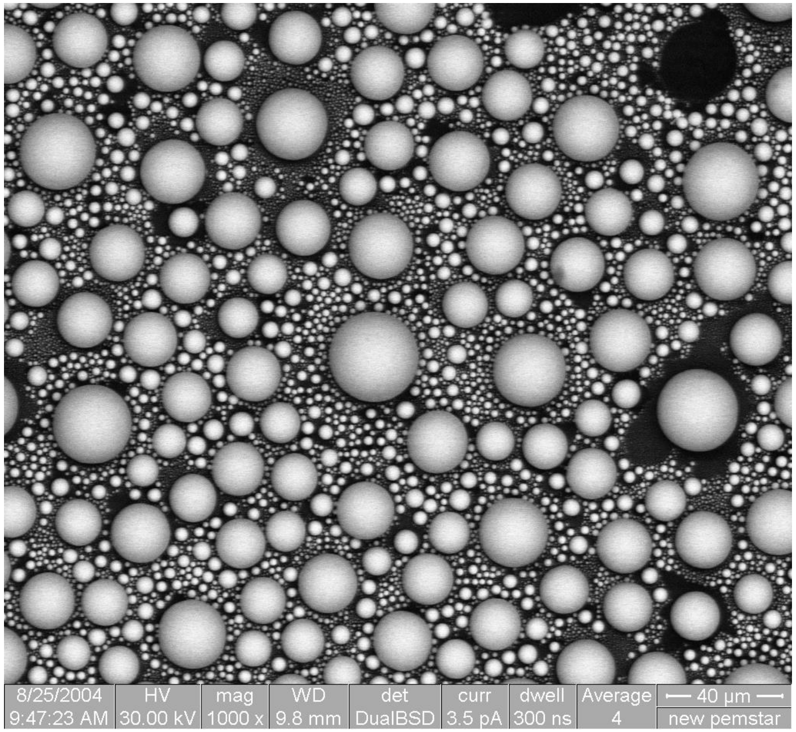


Vybrané partie z elektronové mikroskopie

Examples of SE and BSE images

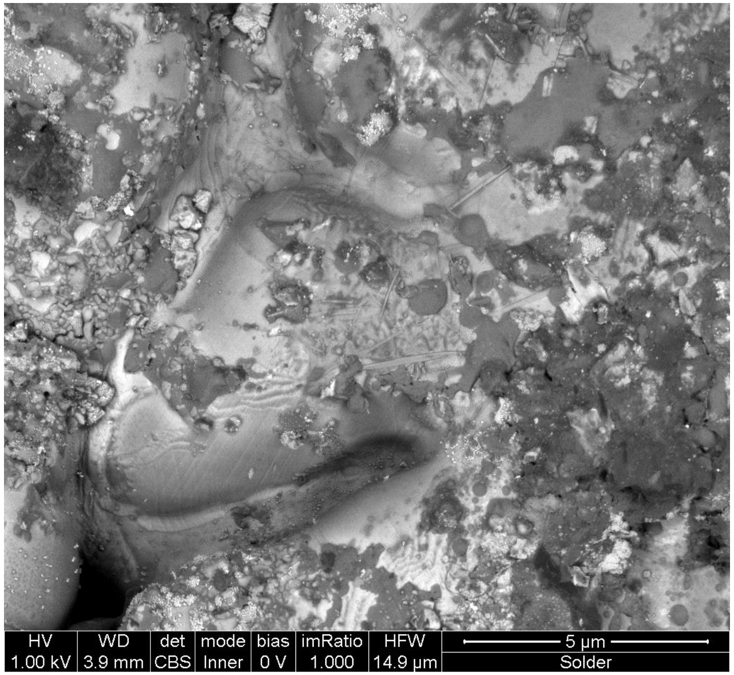
- SE image BSE image



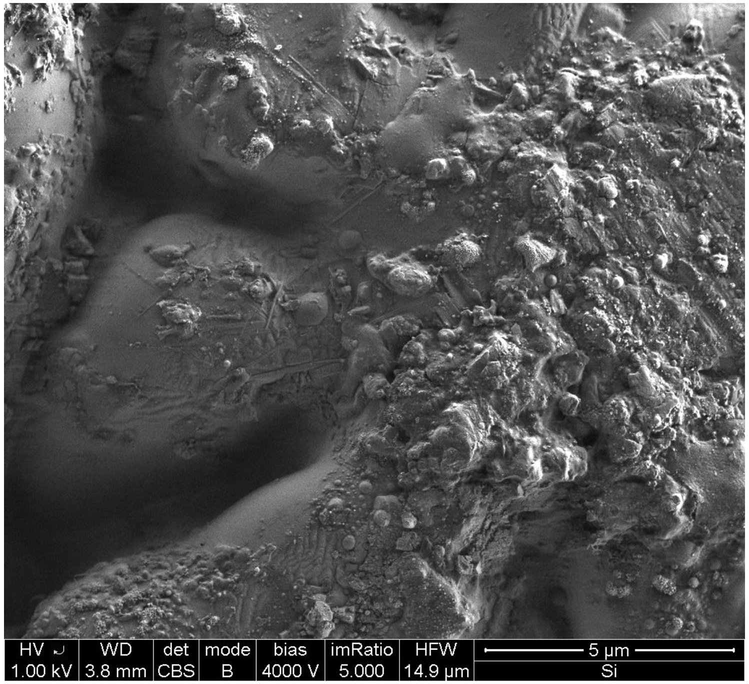


Material & topography contrast in BSE signal

Z- contrast



Topography



Auger electrons

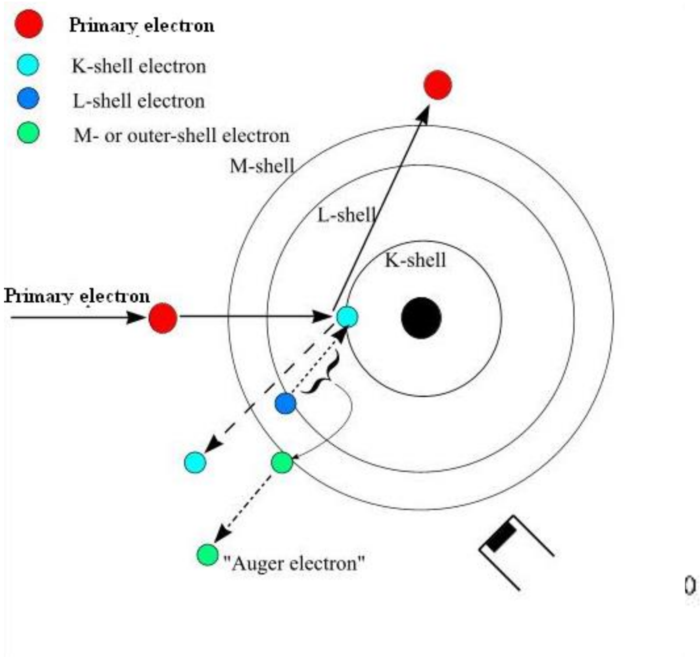
• Transition of electron in atom filling inner shell vacancy results in release of energy

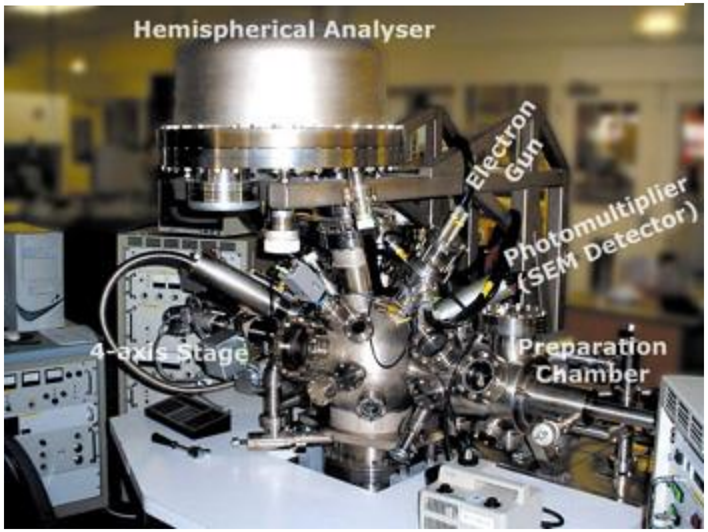
• Energy may be transferred to another electron which is ejected from the atom

• Characteristic peaks for elements – analytical method AES- Auger Electron Spectroscopy

• Low energies (50eV-3keV)-> small escape depth = surface sensitive method

• Extreme surface sensitivity and weakness of signal require usually UHV setup





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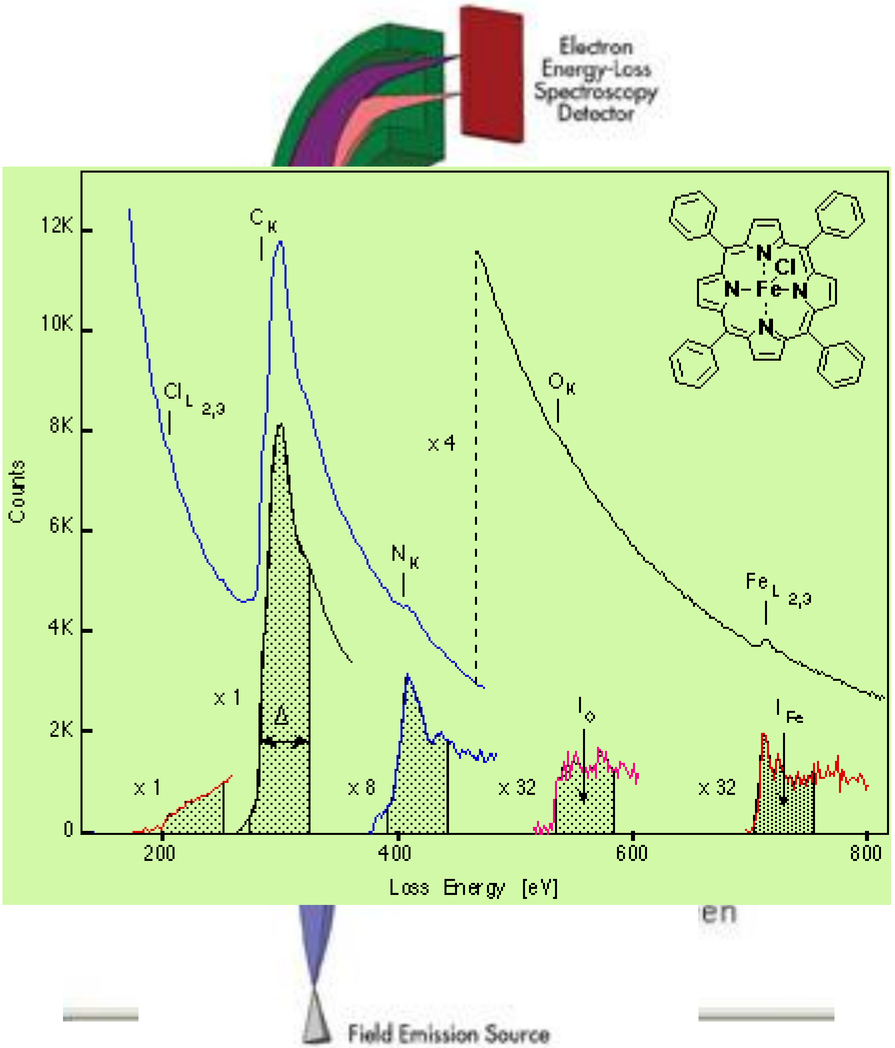
Transmitted electron - TEM

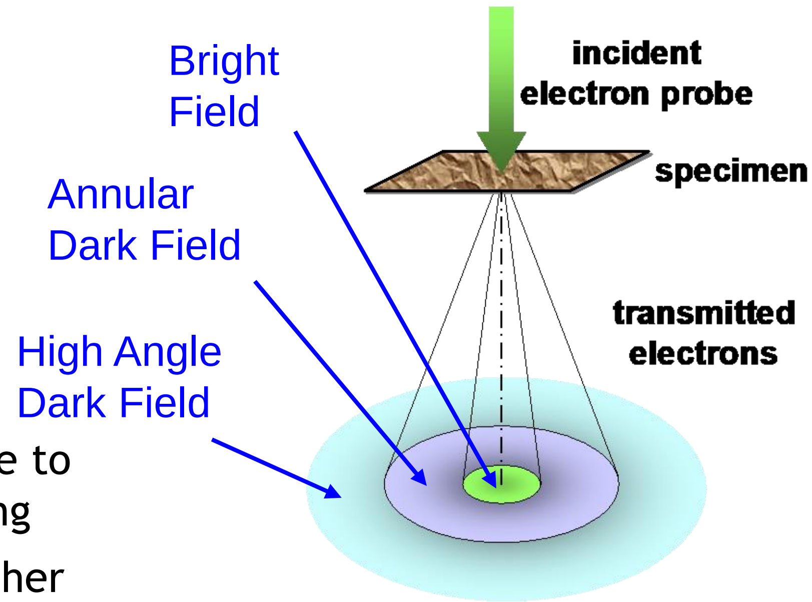
- Electrons transmitted through sample without scattering or scattered to space below sample
- Only possible for samples with thickness smaller than interaction volume
- Signal depends on:
  - Sample thickness
  - Sample material
  - Crystal orientation
- Standard imaging - TEM
- Scanning transmission electron microscopy – STEM
- Electron energy loss spectroscopy - EELS



Transmitted electrons – STEM

(Scanning Transmission Electron Microscopy)

- Electrons transmitted through sample without scattering or scattered to space below sample
- Only possible for samples with thickness smaller than interaction volume
- Signal depends on:
  - Sample thickness
  - Sample material
  - Crystal orientation
- Signals:
  - BF – Bright Field – electrons scattered close to beam axis or transmitted without scattering
  - DF – Dark Field – electrons scattered in higher angle from beam axis
  - HADF – High Angle Dark Field – Higher angle than DF



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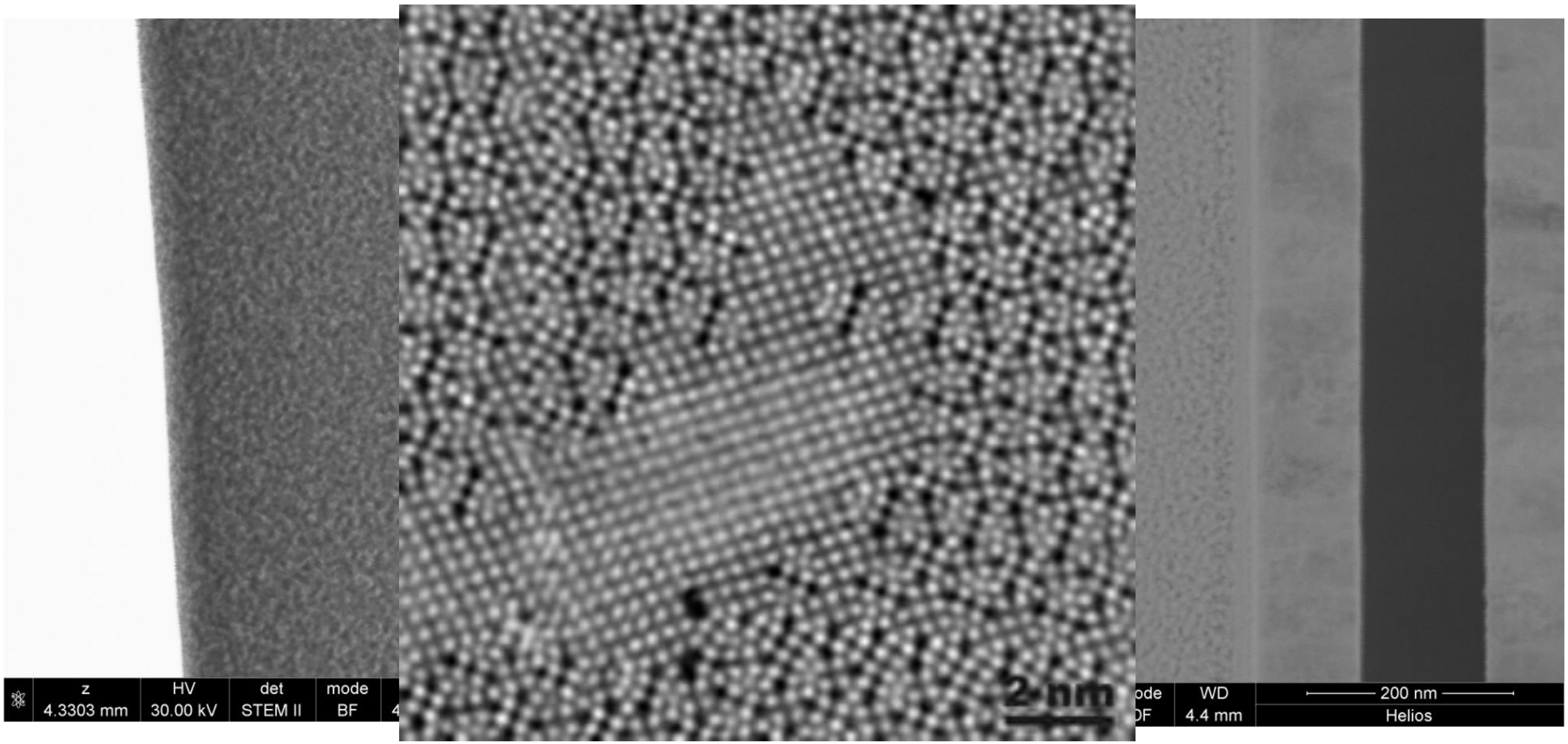
STEM - examples

- Cross section of multilayer (Pt, Mo, Si)

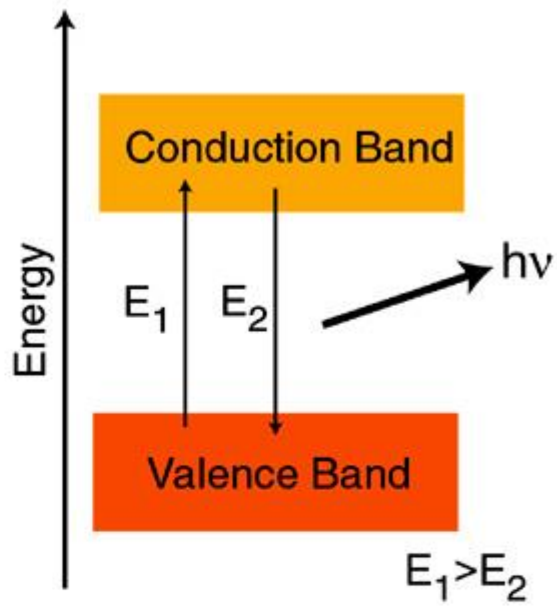
Bright Field

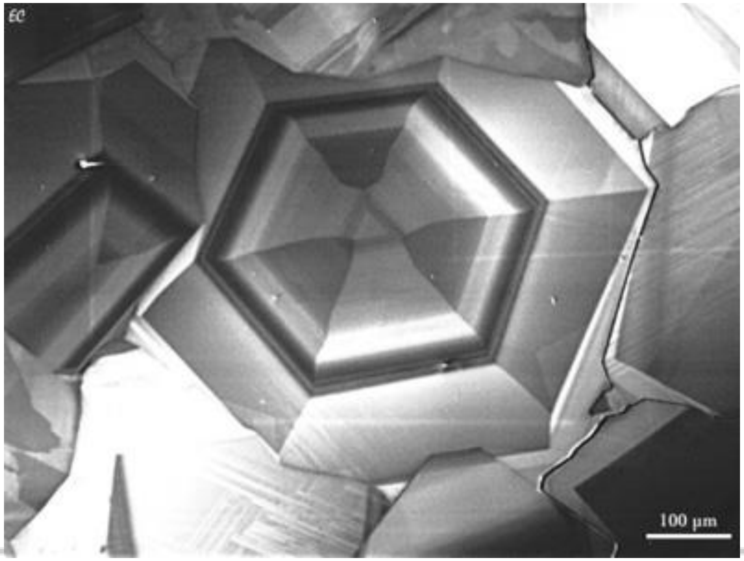
Dark Field

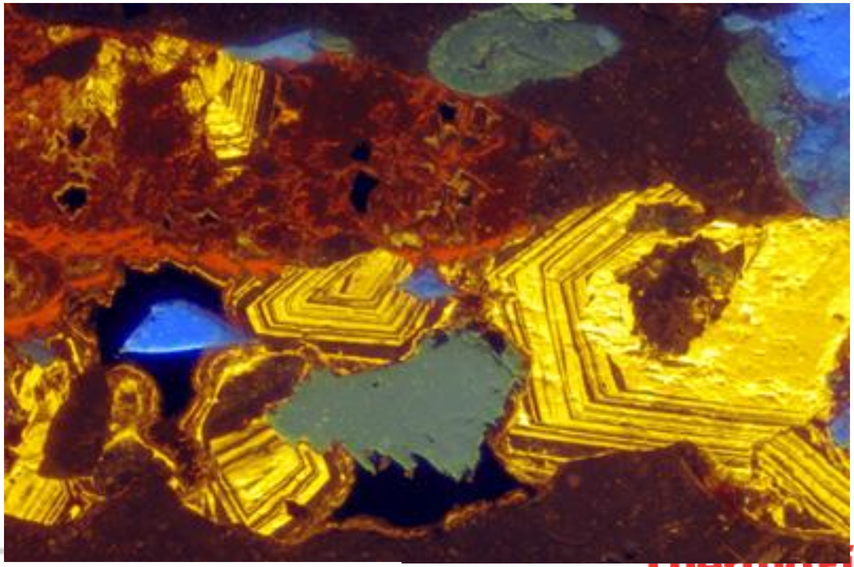


Cathodoluminescence

- UV to IR light (160nm-2000nm) emitted by the sample under electron irradiation
- Effect occurs only in certain materials (semiconductors, minerals, organic molecules)
- Direct detection of light emitted by sample, or more complex instruments with monochromator to obtain spectra of emitted light

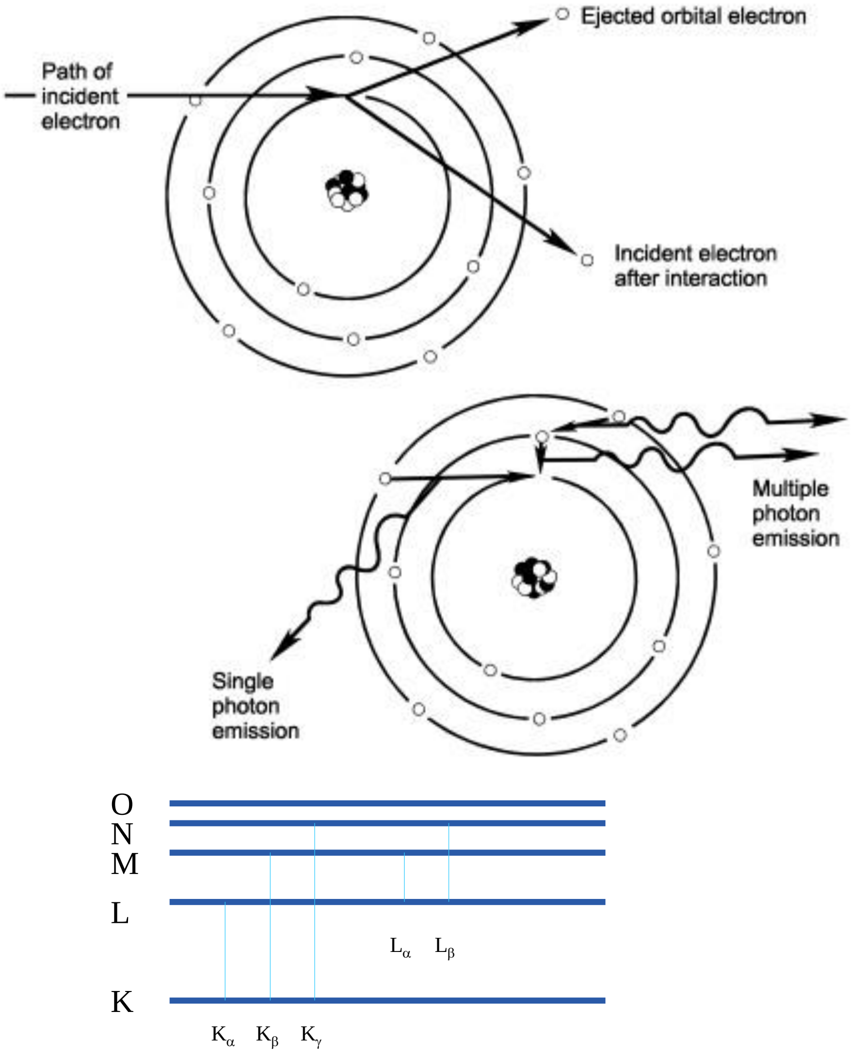






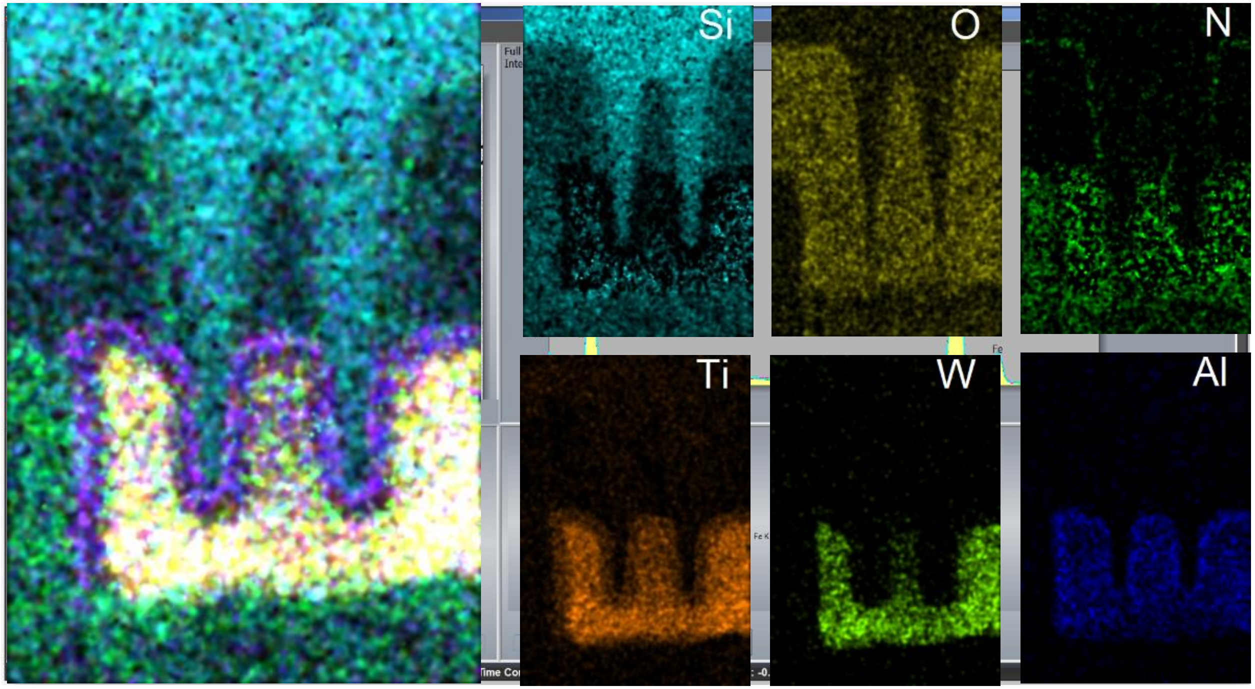
Characteristic X-Ray

- Electron beam induced emission of X-ray has two components
  - Continuous (“brehmstrahlung”)
  - Characteristic X-ray – dependent on atomic structure of sample
- Peaks of characteristic X-ray corresponds to energy emitted by electron when changing energy levels in atom, thus they enable to determine atomic compound of sample (not chemical structure)



Characteristic X-ray

- EDS or WDS (also EDX, WDX)
  - Energy (Wave) Dispersive Spectroscopy (X-ray)
  - EDS – faster x WDS - more accurate (better energy resolution)
  - X-ray spectra
  - X-ray mapping



Other

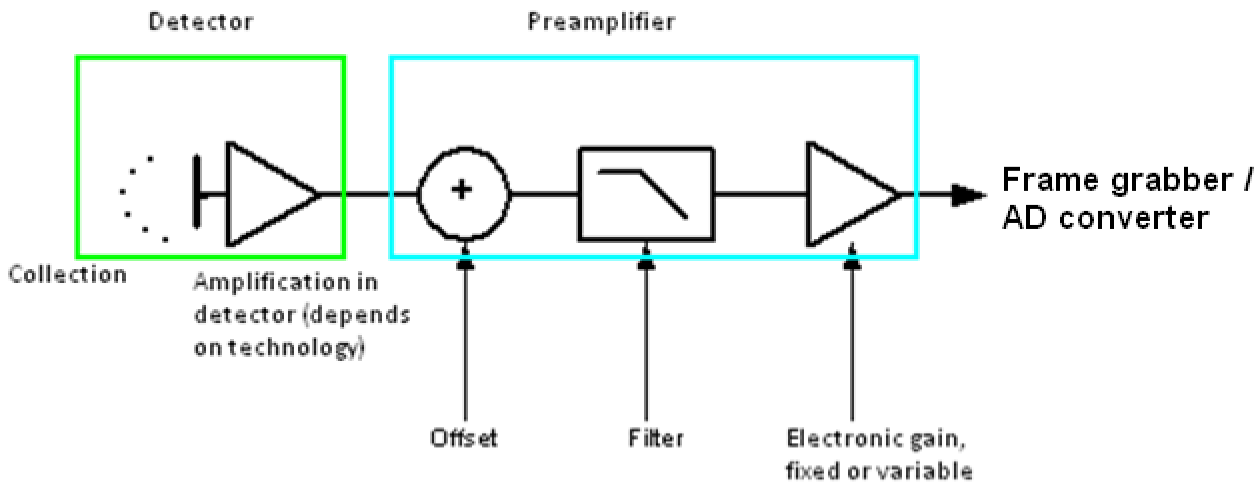
- Secondary Ions
  - Secondary ions are sputtered from sample by ion beam – only in FIB or SDB
  - Yield depends on material and crystal orientation
  - Can be used for:
    - Imaging (channeling contrast)
    - SIMS – Secondary Ion Mass Spectroscopy – enable to measure atomic compound of sputtered sample
- Specimen current
  - Signal of absorbed current is complementary to SE+BSE signal
  - Usually large currents and long dwell times are needed

Signals in EM - summary

•Topography, shape –	Secondary electrons
•Material (qualitatively) –	Backscattered electrons, Cathodoluminescence STEM
•Material (quantitatively) -	Characteristic X-ray, EELS

Basic Detector Scheme

- Each detector consist of:
  - collection part with primary amplification mechanism depending on technology
  - Preamplifier with additional electronic gain, offset and filtering capabilities which prepares signal for frame grabber

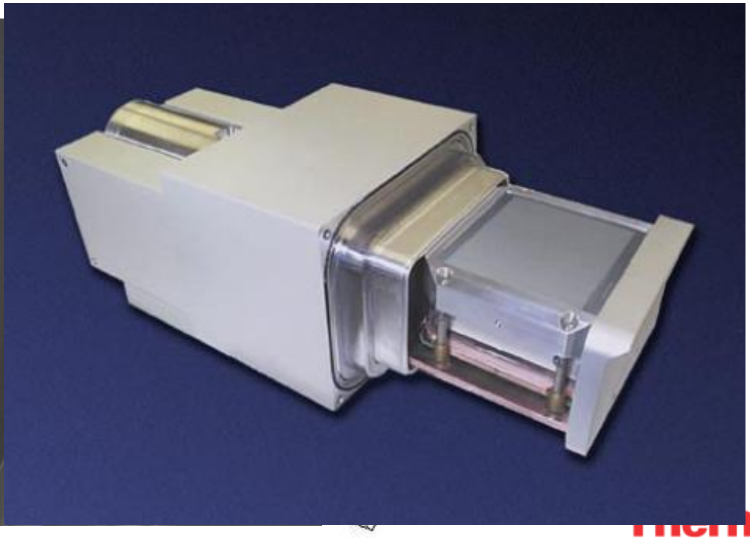


Technologies used for signal detection & amplification

- All detectors used in electron microscopy use several detection principles
  - Scintillation detectors
    - Use materials which emit light when irradiated by electrons
  - Semiconductor detectors
    - Use photodiodes or avalanche diodes with thin entrance window
  - Gaseous ionization detectors
    - Use amplification in gas environment
  - Continuous multipliers
    - Amplification in continuous dynode structure in chamber environment
  - Light detectors
    - For CL detection, uses mirrors and lightguides to get emitted light to PMT
  - Conversion detectors
    - Not special category - detectors which use some structure to convert real signal into different type of signal which can be then detected by some of the above mentioned detector schemes

Scintillation detectors

- Signal electrons are converted into light in scintillator
- Light is transported through lightguide to photomultiplier which amplifies signal and converts it to current
- SE detection - scintillator has to be biased so SE’s has sufficient energy to scintillate
- BSE detection – No bias needed
- SI (secondary ion) detection – conversion electrode needed to convert ions to SE’s, these can be amplified
- TEM phosphor screens/screens of TEM cameras



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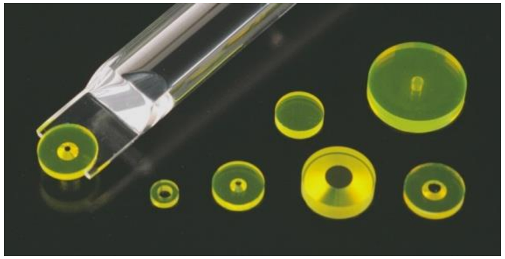
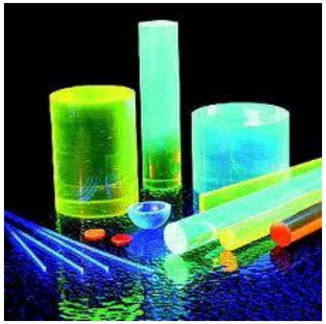
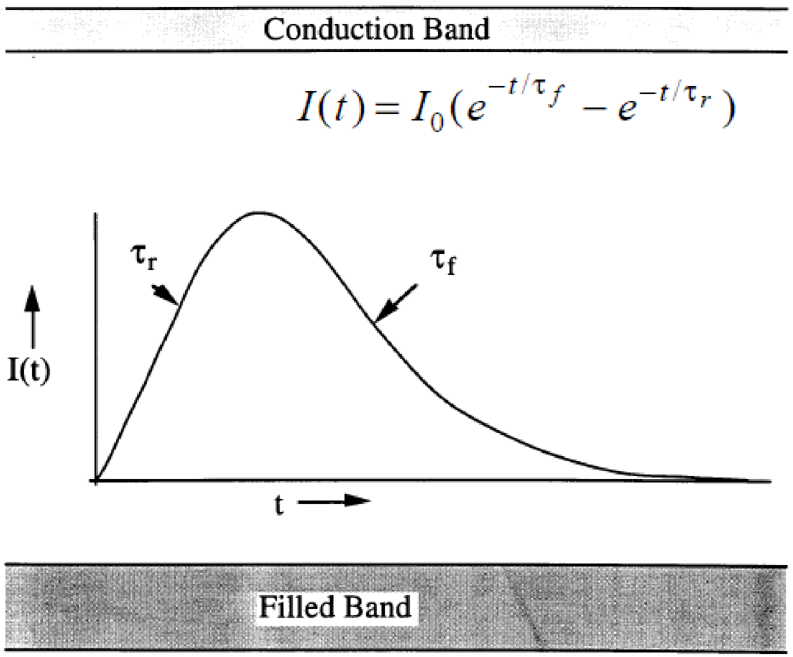
Components of scintillation detectors

- Scintillator
  - Single crystal (YAG, YAP..), Powder (P47 = Y_2SiO_5. or organic (PBO..)
  - Fluorescence caused by
    - Transition between vibrational states of organic molecules
    - De-excitation of electrons excited in states in forbidden band (inorganic scintillators)
  - Basic properties
    - Decay time
    - Light output
    - Emission spectrum
  - Electron energy needs to be in range of keV to produce light in scintillator
  - Lightguide
    - Has to be matched to scintillator (similar refraction index) and in good optical contact with scintillator and PMT

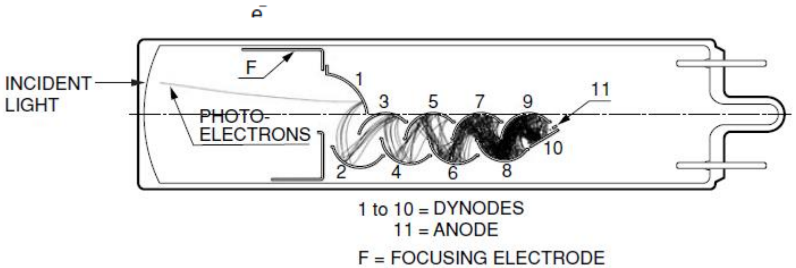


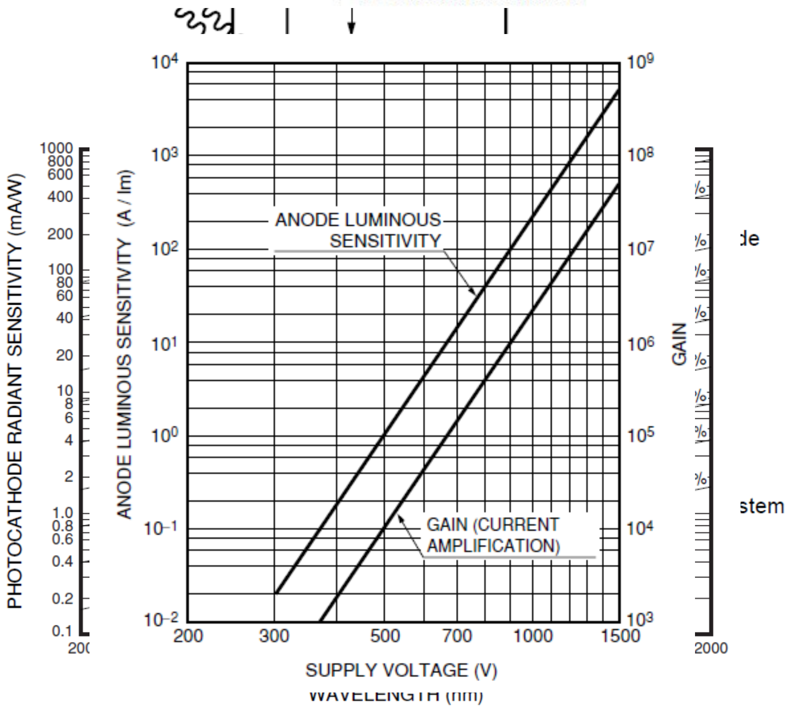




Components of scintillation detectors

- Photomultiplier (PMT)
  - Electron tube with photosensitive cathode -external photoelectric effect
  - Photocathode –> external photoelectric effect (Alkali – Sb, Cs, Te, Rb, or III-V semiconductors GaAs…)
  - Dynodes (BeO,MgO,GaP…)
  - Basic parameters
    - Spectral sensitivity of photocathode
    - Gain $\mu = A \cdot V^{kn}$
    - Linearity
    - Dark current





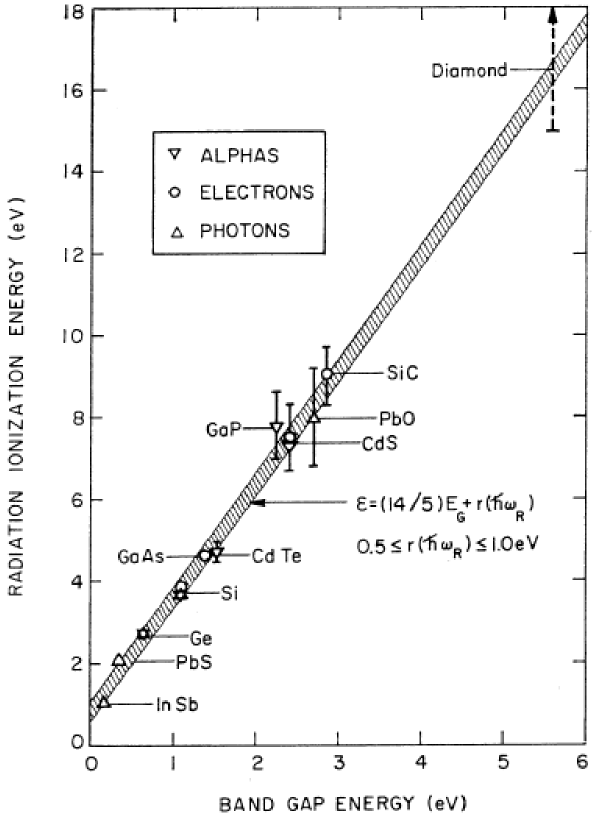
Examples of scintillation detectors

- ETD – Everhardt Thornley detector – standard detector of SEM systems
  - Detector uses biased grid to attract low energy SEs
  - Scintillator is biased to 10kV to provide enough light output
  - In-chamber or in-column versions
- Robinson type BSE detector – Scintillation BSE detector
  - Annular scintillator with central hole for electron beam

- HAADF STEM detector for TEM

Semiconductor detectors

- Charged particle passing through semiconductor causes production of electron hole pairs
- Ratio $\frac{\varepsilon_i}{E_g} \approx const.$ for all materials and types of radiation
- Ionization energy ~3eV for both Ge and Si, Fano factor ~0.1 ->good energy resolution
- Several detector types
  - P/N, PIN diodes
  - Avalanche diodes – APDs
  - CCD cameras
  - Direct detectors – CMOS
  - Hybrid pixel detectors
  - X-ray detectors for spectorscopy

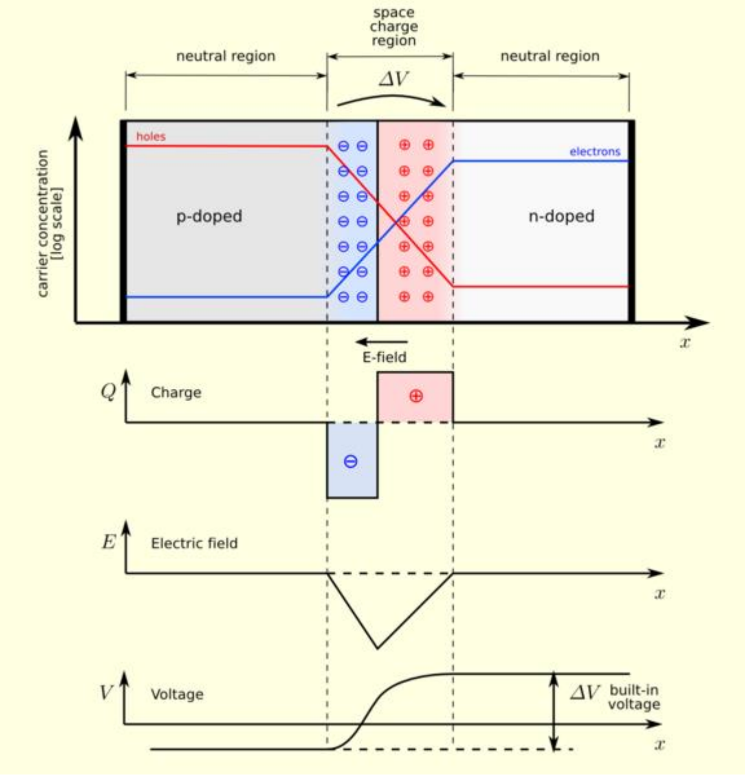


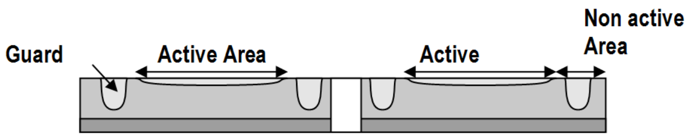
Semiconductor detector - diode

- Equilibrium of drift and diffusion current at p-n junction forms depletion region

- Reverse bias often used to enlarge depletion region (fully depleted detector)
  - Increase active volume
  - Decrease capacitance of the detector

- Shallow implant on the surface needed to not block low energy electrons





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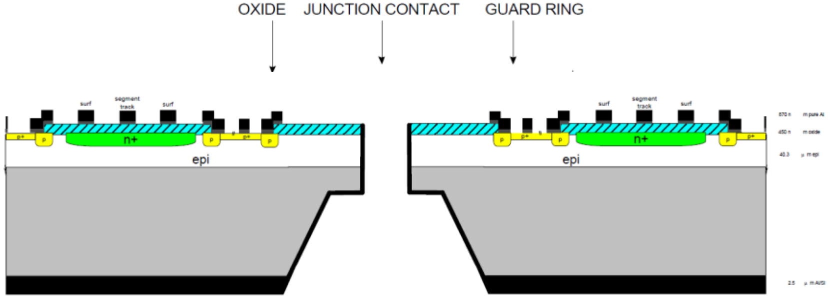
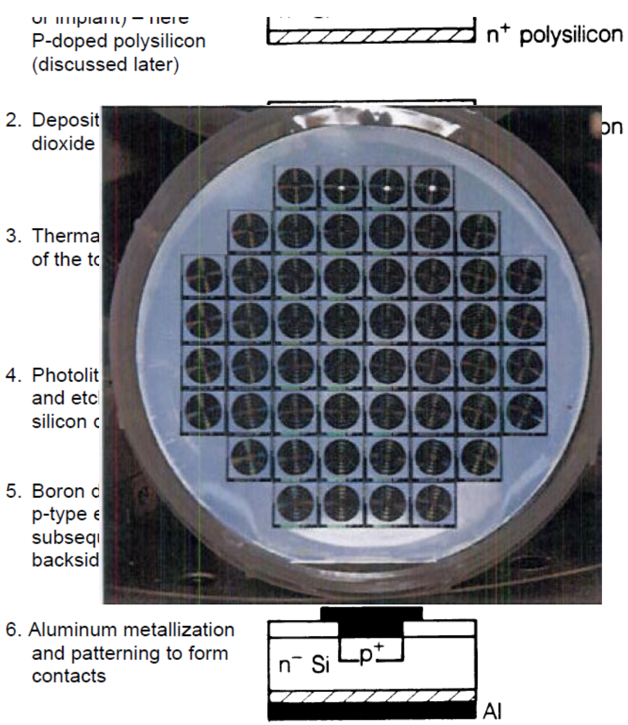
Semiconductor detector – diode Fabrication

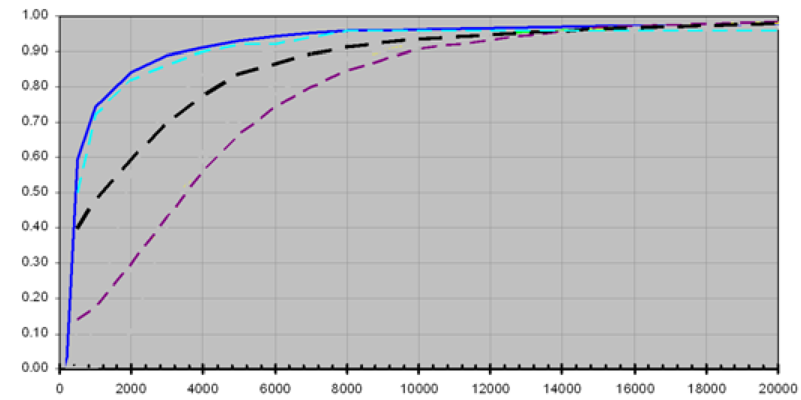
- Bulk material
    - Semiconductor wafer - Si, Ge, GaAs..
- Dopants to create p and n regions
    - Introduced from the surfaces using diffusion or ion implantation
- Metallization to make contacts
    - Evaporation or sputterring
- Passivation to protect semiconductor surfaces
- Guard ring – additional junction to isolate main junction from detector edge
- Each production step only on selected parts of surface – masks, photoresists



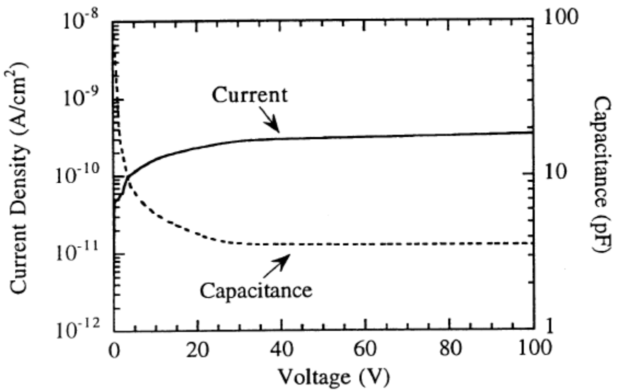


Semiconductor detector – diode Parameters

$$I_{\det} = I_{beam} \cdot \frac{E_{beam}}{3.62eV} \cdot (1-\eta) \cdot R$$

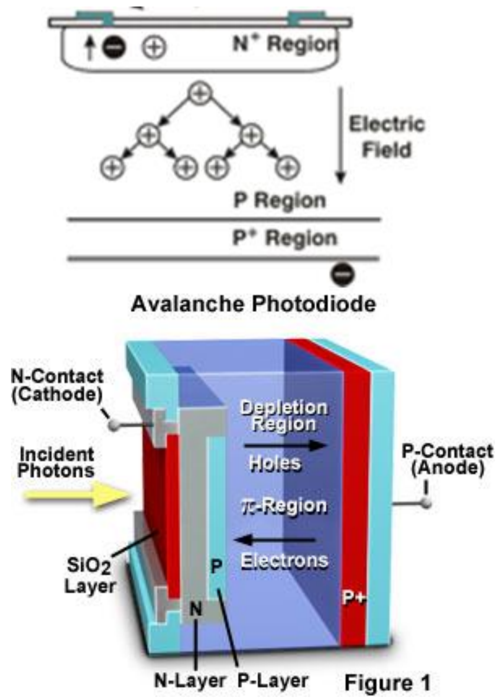


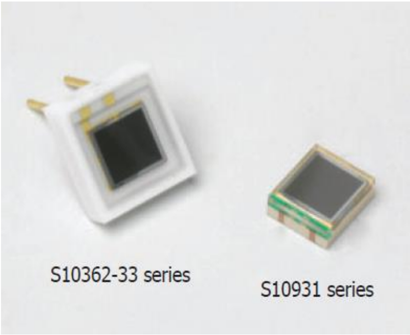
- Energy needed to create one electron – hole pair in silicon is 3.6eV → 30keV electron creates ideally > 8000 electron hole pairs, 1keV electron only ~270
- Thickness of front implant – "dead layer" – low energy electron efficiency
- Capacitance and serial resistance τ = RC (C = 10pF, R=100Ω $\rightarrow \tau = 1$ ns)
- Leakage current – Crystal imperfections, temperature, (mechanical damage, surface contamination)



Semiconductor (solid state) detectors

- Avalanche photodiodes (APDs)
  - Contrary to PIN diodes charge carriers are accelerated by high bias (up to 2500V)
  - Avalanche electron multiplication in the depletion region similar to process in PMT
  - Higher gain x Higher dark current = noise
  - Recently arrays of APDs are produced which may replace PMT in future (MPPC-multipixel photon counter, SiPM – Silicon photomultiplier)





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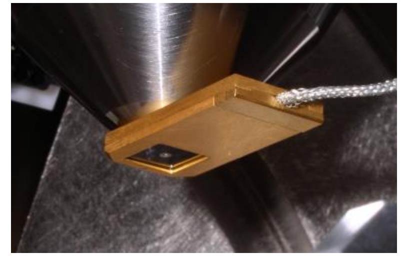
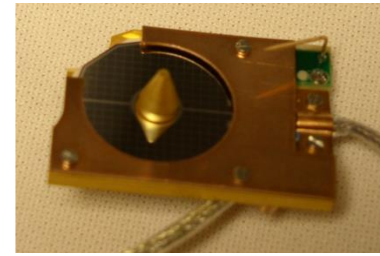
Semiconductor (solid state) detectors

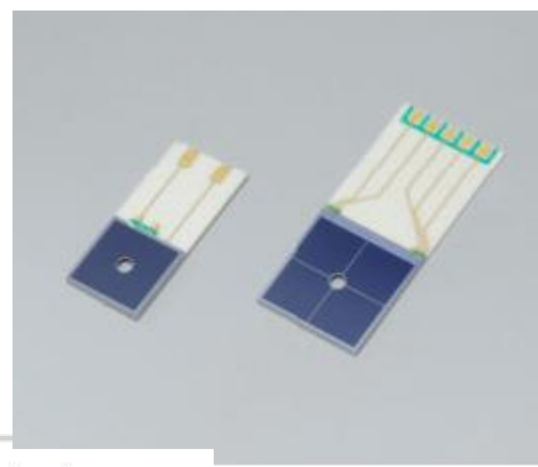
- Advantages
  - Segmentation of semiconductor detector is very easy, fabrication of multichannel detectors is easier than with scintillation detectors
  - Makes use of progress in silicon industry, not special production like scintillators or PMTs
  - Signal is led via cabling in chamber – it does not block excessive spatial angle around the sample
- Disadvantages
  - Low keV sensitivity (technology improves)
  - Sensitivity to mechanical damage of surface layer
  - Relatively higher input cost (masks, production preparation etc.)
  - Signal routing in chamber is vulnerable to EMC interference

Semiconductor detectors – examples

- Several types of BSE detectors placed below pole piece, different segmentation
- STEM detectors placed below the sample
- In column detectors mounted inside or above objective lens

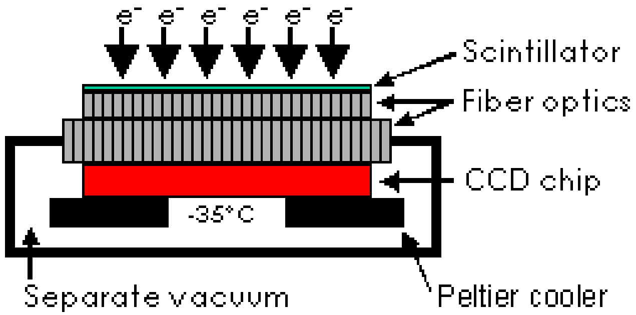




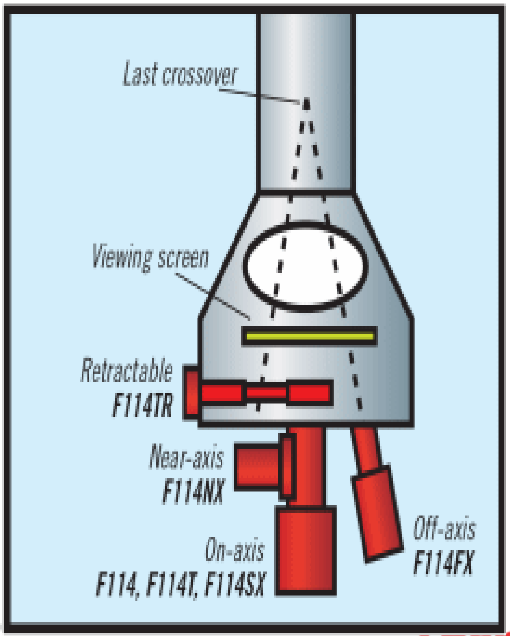


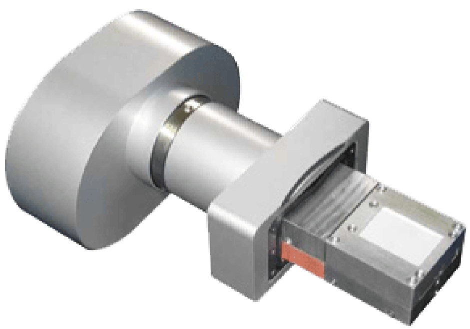
Pixelated detectors
CCD camera - TEM

- High resolution CCD cameras
- Fiber optic or lens coupling to scintillator
- Cooling to suppress thermal noise (peltier cooler)
- Variety of different position possibilities



Fiber-optically coupled CCD Camera





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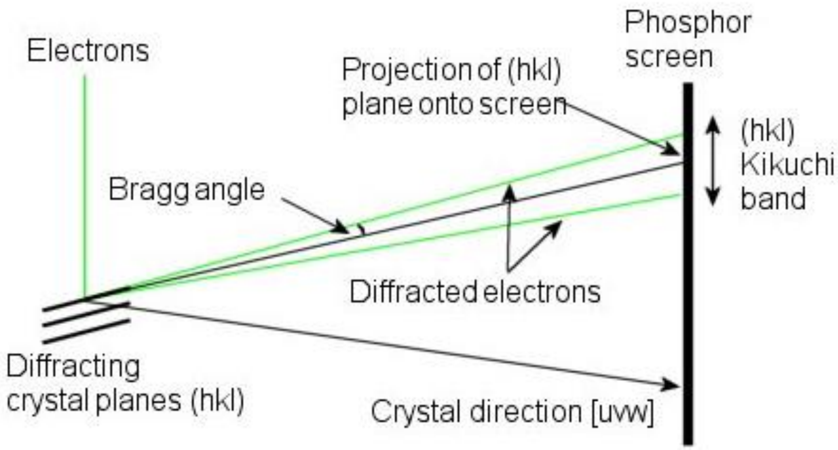
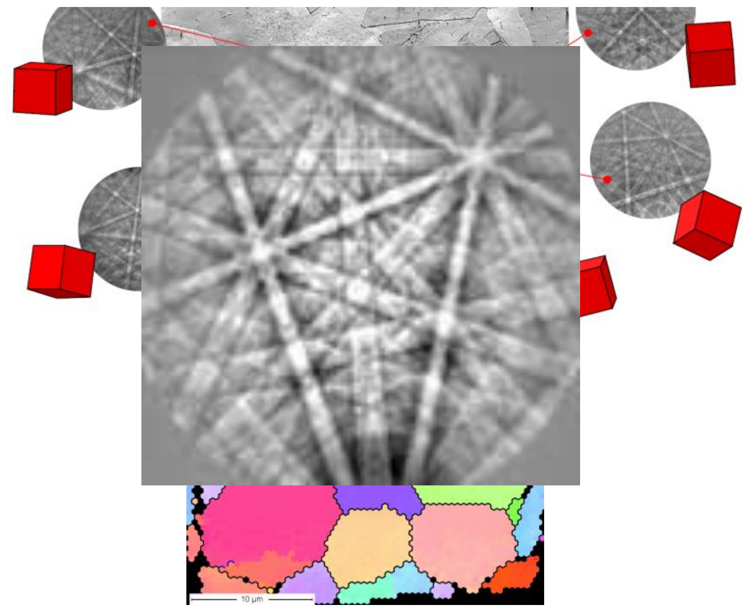
Pixelated detectors
CCD camera - EBSD

- SEM/SDB uses CCD mostly for EBSD
  - Electron Back Scattered Diffraction Pattern
  - Information about crystal lattice orientation
  - Specimen tilted to 70 degrees
  - EBSD pattern (Kikuchi bands) images taken while scanning
  - Crystal orientation determined for each part of sample (Hough transform)
  - Scintillator coupled to CCD camera





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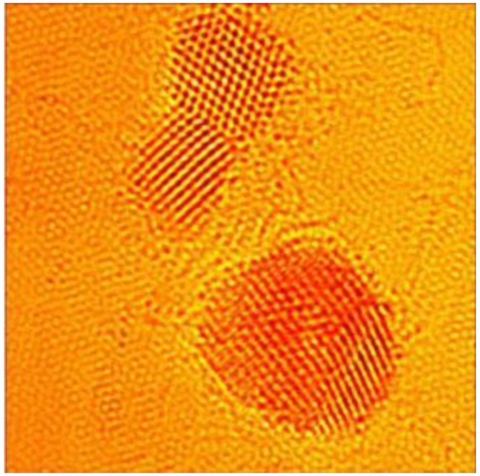
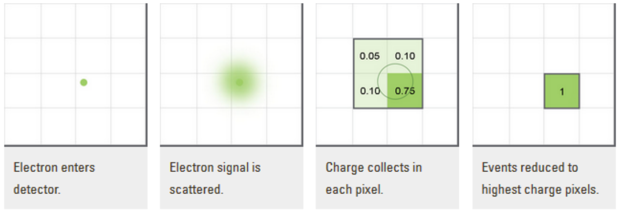
Pixelated detectors
CMOS camera – direct detection - TEM

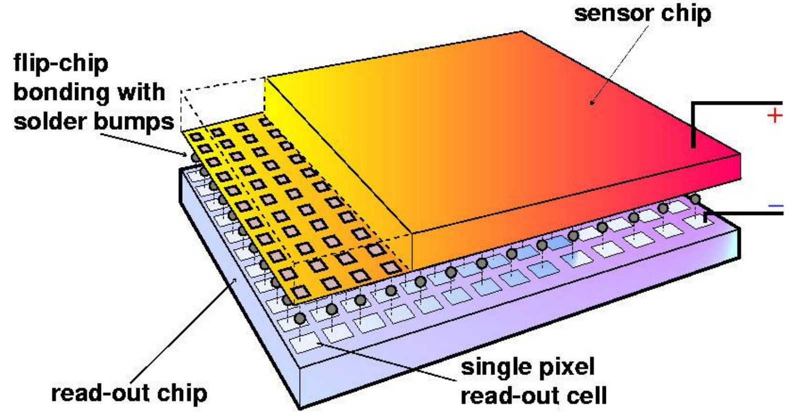
- High resolution / low dose
- Radiation hardness is important
- Pixel sizes <5um – comparable to interaction volume
- Interaction volume reduction by backthinning
- Resolution >20Mpix
- Speeds >1000fps

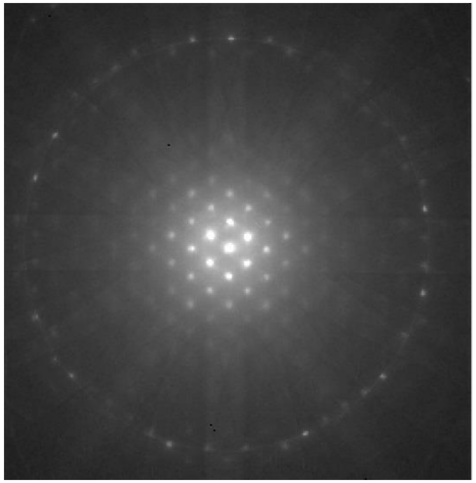




Pixelated detectors
Hybrid detectors

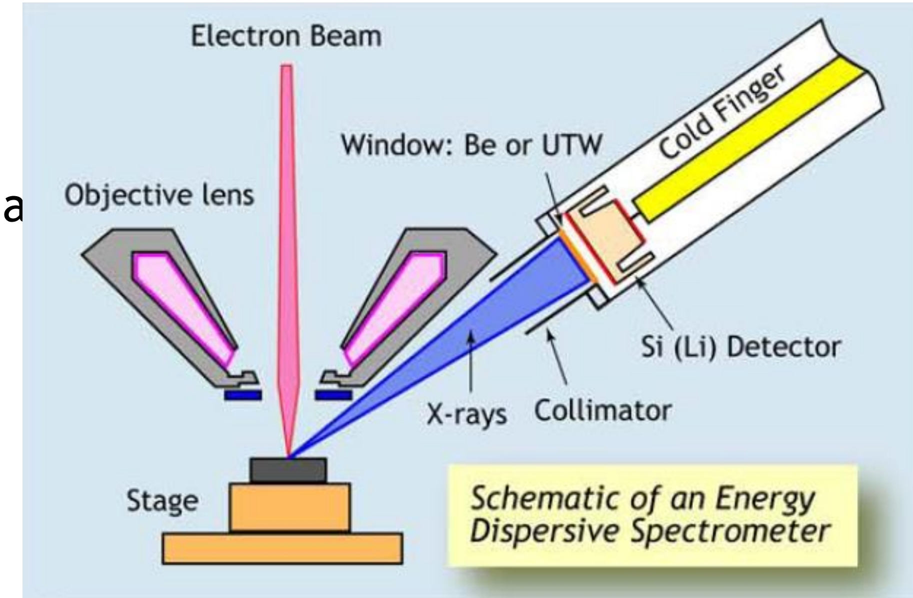
- Combination of readout chip (common) and sensor
- Sensors tailor made for application – Si, GaAs, CdTe…
- Higher radiation hardness
- Frame based or data driven mode
- Pixel sizes ~10s of um

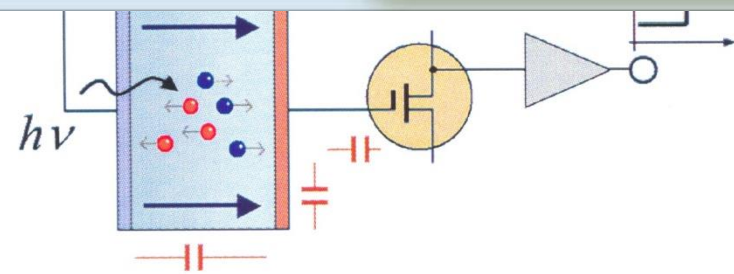




X-ray detectors

- X-ray detectors measure energy of incoming photon by the amount of ionizations it produces in detector materia
- Si(Li) and SDD detectors available
- Si(Li) – lithium drifted silicon
  - PIN diode (3-5mm thickness) with ~1000V bias
  - For noise reduction it requires liquid nitrogen cooling
  - External FET transistor to convert current to voltage
  - Resolution ~ 130eV FWHM @ Mn Ka line



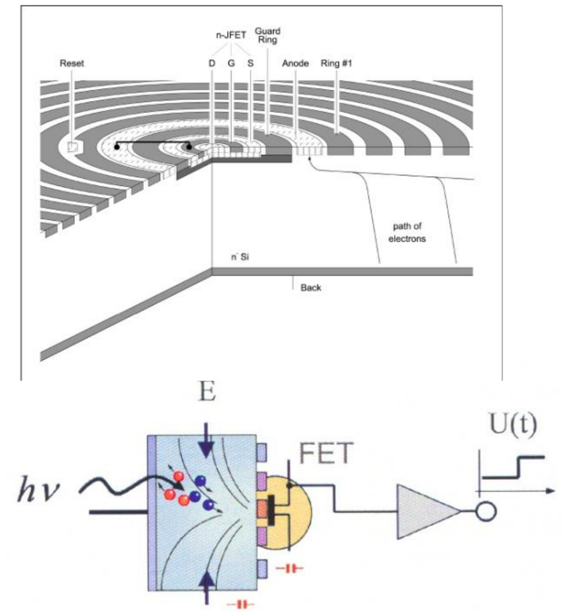


$$n_e = h\frac{\nu}{\varepsilon}$$

Fig. 8.5-9 Schematic drawing of Si(/Li) detector

X-ray detectors

- SDD detector
  - Transversal field generated by serie of ring electrodes causes charge carrier to drift to small collection electrode
  - This concept allows significantly higher countrate than Si(Li)
  - FET transistor integrated in the chip improves energy resolution and throughput
  - High purity allows to use Peltier cooling instead of liquid nitrogen
  - Resolution ~ 130eV FWHM @ Mn Ka line
  - Countrate up to 1,000,000 cps, better energy resolution at given countrate



$$n_e = h\frac{\nu}{\varepsilon}$$

Fig. 8.5-10 Schematic drawing of SDD detector

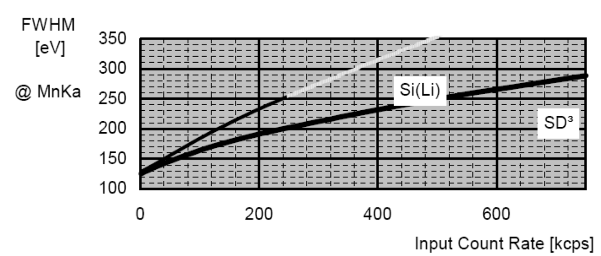
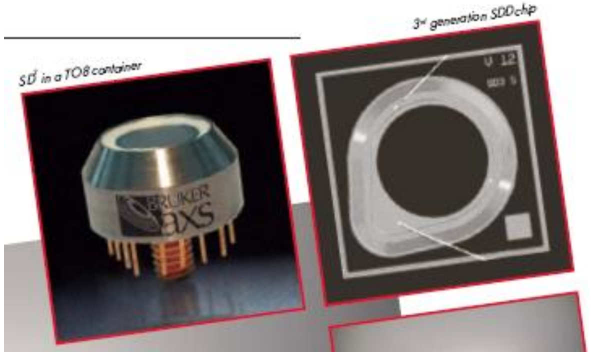


Fig. 1. Energy Resolution (FWHM) of SD³ Detectors and state-of-the-art Si(Li)

X-ray detectors - examples

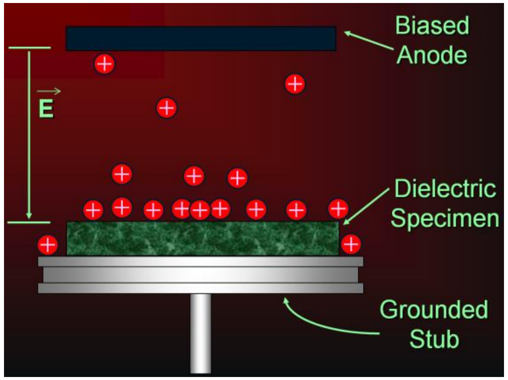
- SDD chip



- Si(Li) detector

- SDD detector

Gaseous ionization detectors



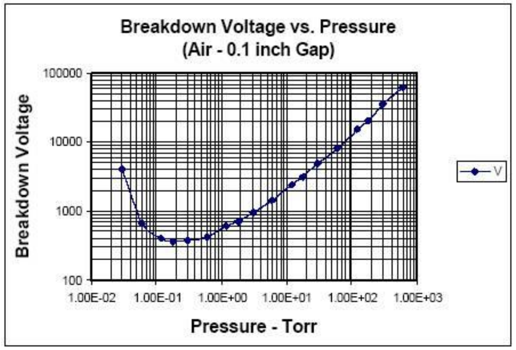
- Low Vacuum or ESEM (Environmental Scanning Electron Microscopy) mode is used for imaging of non conductive samples
- Chamber pressure vary between 10-4000 Pa -> biased scintillation detectors cannot be used
- Amplification in chamber gas environment – biased electrode (~0-800V)
- Maximum bias given by Paschen law $V = \frac{a \cdot p \cdot d}{\ln(p \cdot d) + b}$
- Gain is determined by gap distance, gas type, pressure and anode bias

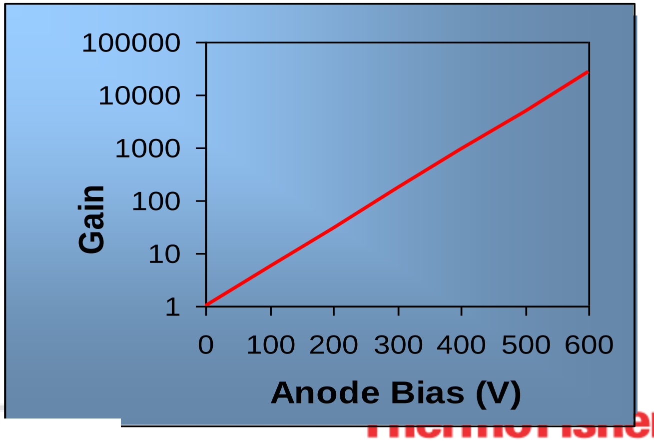
$$g = e^{\alpha d}$$

$$\alpha = APe^{-BPd/V_0}$$

- Optimum exists in gain dependence on Pd/V_0

Inelastic mean free path ~ length needed to achieve ionization energy





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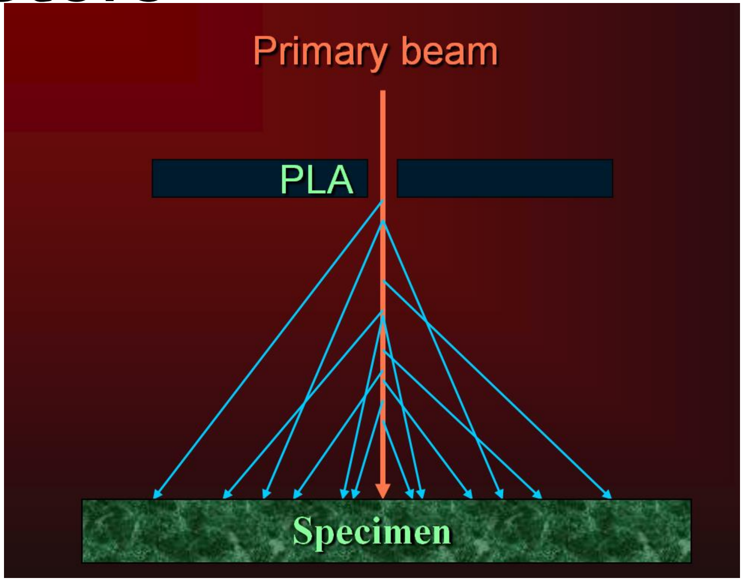
Gaseous ionization detectors

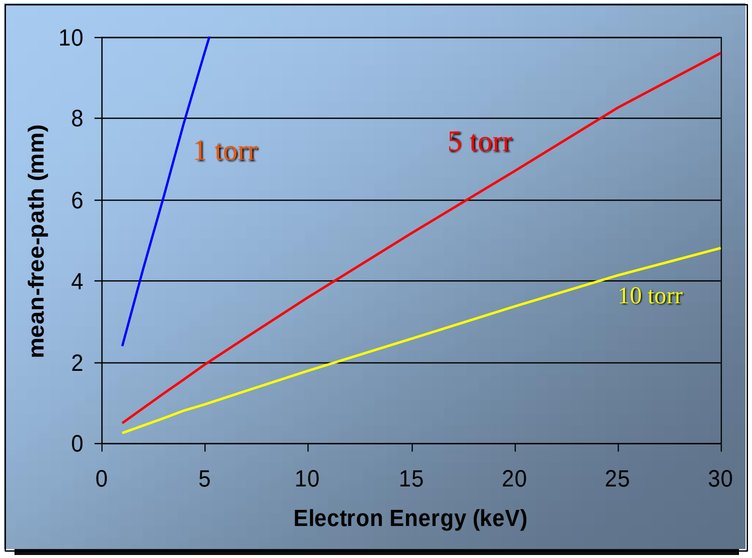


- Additional effects influencing/limiting signal of gaseous detectors
  - BSE signal amplification
  - Primary beam amplification

$$I = I_0 e^{\alpha d}\left(\delta + \frac{S_{PE}}{\alpha d} + \eta \frac{S_{BSE}}{\alpha d}\right)$$

  - Skirt effect – elastic scattering of primary beam

$$r_s = \frac{364 \cdot Z}{E}\left(\frac{p}{T}\right)^{1/2} L^{3/2}$$



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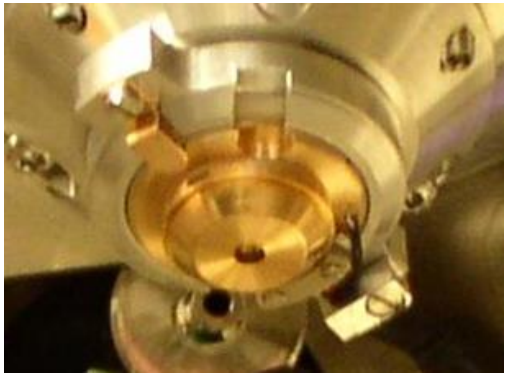
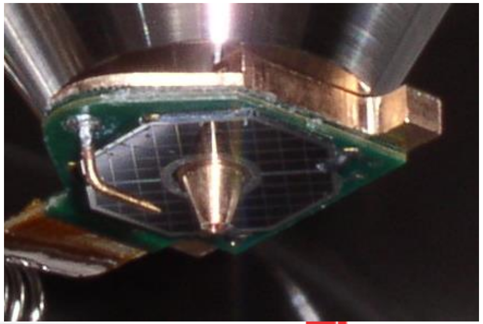
Gaseous ionization detectors

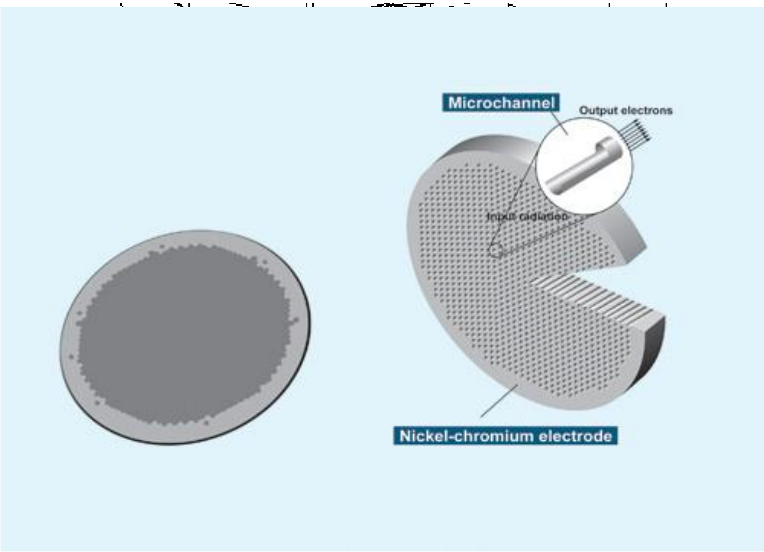
- Advantages
  - Cheap production, no special know how needed (PCB, metal electrodes)
  - Can be tailor made for given application
  - Robust and user exchangeable
- Disadvantages
  - ESEM versions of detectors (with PLA) limit field of view significantly
  - Slower response time due to current induced by slow ions produced in amplification cascade
  - Needs biased amplifier which complicates multichannel detector schemes





Continuous multipliers

- Structures created from material with high secondary emission yield (or covered with layer with this feature)
- Electric field is distributed along the channel of multiplier placed directly in SEM/SDB chamber
- Signal particle creates SE in input part of detector and this is further amplified similarly to PMT
- More channels can be combined in microchannel plate → position sensitive detectors



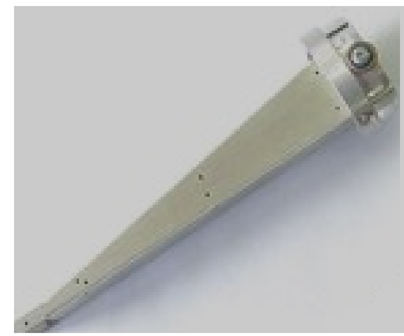
Continuous multipliers

- Advantages
  - Small size w.r.t. PMT detectors x similar gain
  - Can be used to detect both electrons and ions
- Disadvantages
  - Sensitive layer providing amplification degrades with time and use, detectors need to be exchanged after some time, when used heavily it could be a few months
  - Biased preamplifier needed for this type of detectors

Cathodoluminescence detectors

- Uses mirrors to transport light generated by electron beam on the sample to light sensor

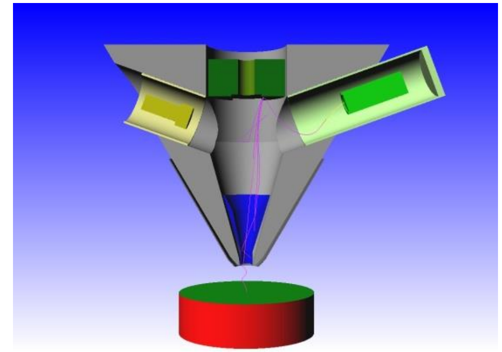
- Examples
  - Centaurus CL – version of BSE detector with exchangeable tip – instead of scintillator only mirror placed above sample the rest of detector remains the same – simple CL detecor

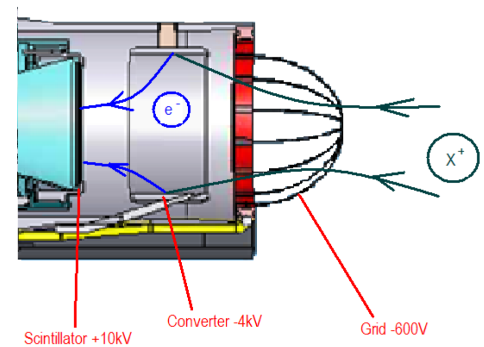


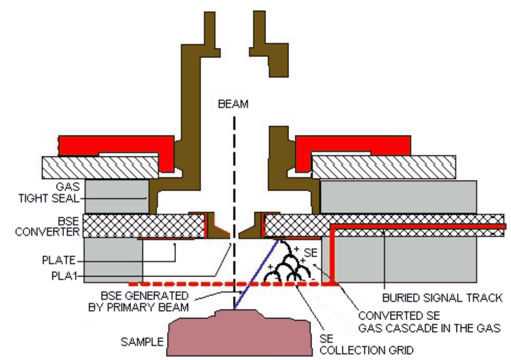
  - Gatan Chroma CL – Detector with monochromator enabling to determine also spectrum of emitted light

Conversion Detectors

- BSE mode of TLD
  - BSEs converted on mirror electrode to SEs, then detected
- SI mode of ICE
  - Secondary ions converted on conversion ring to SE's, then detected
- BSE mode of GBSD
  - BSE's converted on inner electrode of detector to SE's , then detected







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Technologies used for signal detection Summary

- Scintillation
  - Coupling to PMT or other light sensitive devices
  - SE, BSE detection
  - Fast, low noise
- Solid state – semiconductor
  - Variety of use cases, BSE, EBSD, EDS…
  - Direct detection, multichannel detection
  - Benefits from progress in semiconductor industry
- Gas ionization
  - Low Vac, ESEM, charge neutralization
- Other technologies
  - Continuous multipliers
  - Cathodoluminescence

Detector design considerations

- Collection efficiency

$$SNR \sim \sqrt{N}$$

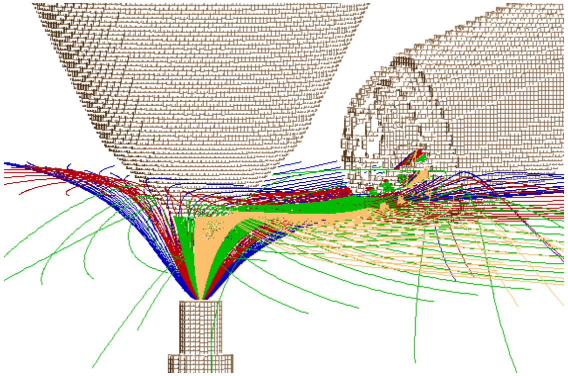
  - Simulation tools enable modeling of collection efficiency of different detector configurations including beam sample interaction

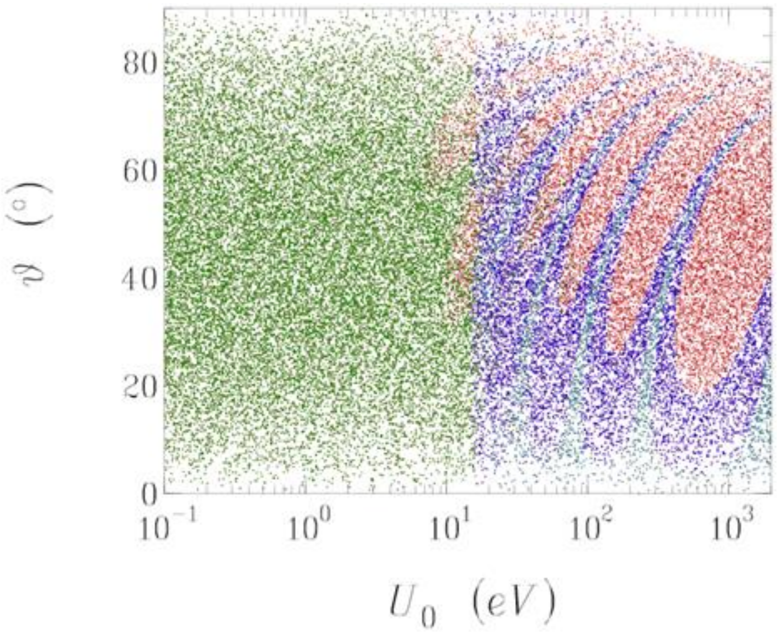
- Narrow point spread function
  - Scintillator decay time, semiconductor sensor capacitance

- Low additional noise
  - Low losses in signal transport (scintillator photocathode matching, lightguide transport efficiency, signal wiring capacitance)

$$DQE = \frac{SNR_{out}^2}{SNR_{in}^2}$$

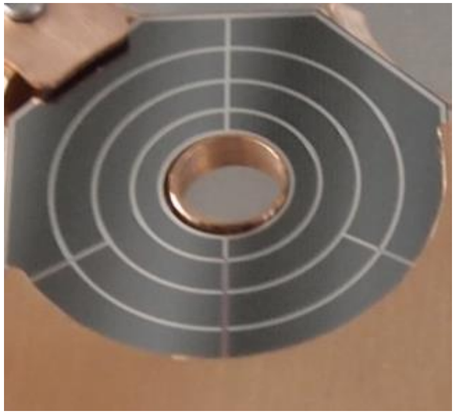
  - Noise free amplification, reduced electronic noise

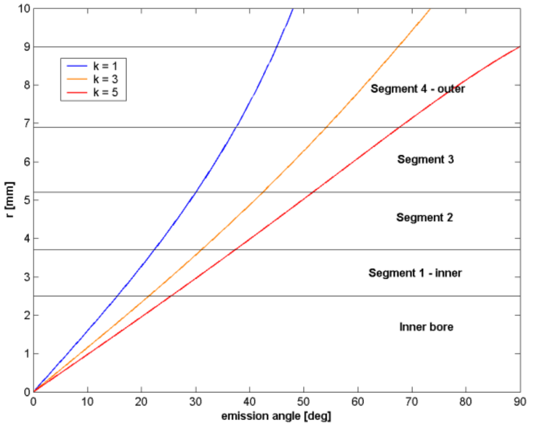


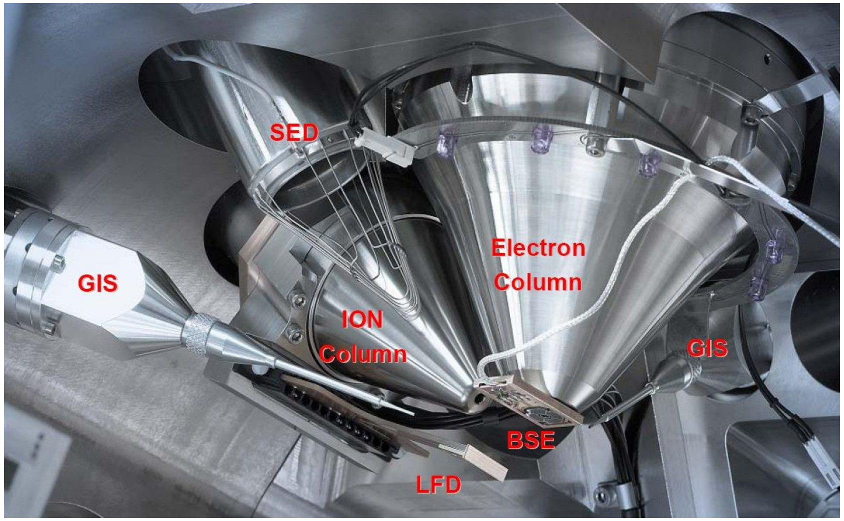


Detector design considerations

- Possibility of signal filtering
  - Spatial (angular distribution)
  - Energy
- Simultaneous acquisition
  - Variety of signals at the same time
  - Multichannel detection, acquisition and processing
- Spatial requirements
  - Space around the sample is limited
  - Number of SEM/SDB/TEM accessories is increasing

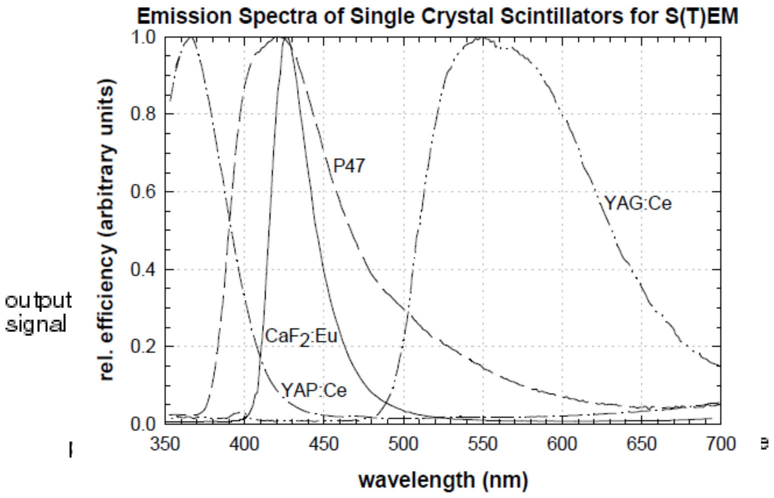


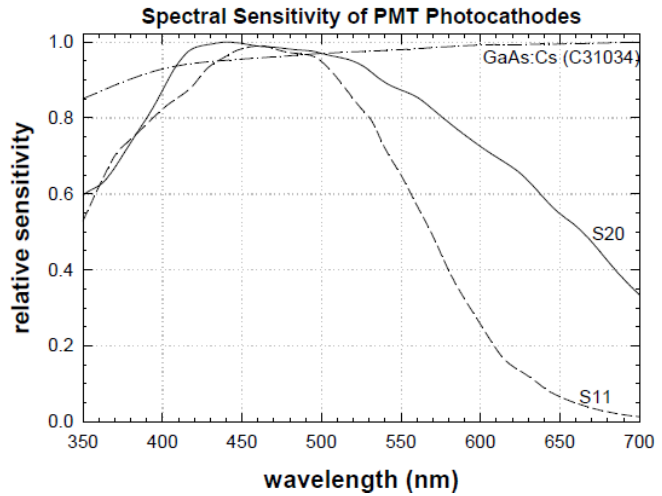




Detector design example

- ETD
  - Collection efficency ~30%
  - P47 scintillator 1signal electron ~100photons – decay time ~80ns
  - Losses in scintillator, lightguide and interfaces ~20-30 photons reaching photocathode
  - According to cathode DQE (30-50%) ~10 photoelectrons produced
  - Amplification in PMT => 10^6 electrons at PMT anode





Summary

- Detectors are key part of the electron microscope – eyes of the system
- Detect signals produced by interaction of electron or ion beam with sample
  - Electrons (secondary, backscattered, Auger…)
  - Photons (X-Ray, Cathodoluminescence)
  - Ions
- Use different technologies to convert and amplify particle signal into current/voltage which can be digitized and visualized
  - Scintillators
  - Semiconductors
  - Gas amplification

Literature

- Knoll, G. F.: Radiation detection and measurement, ISBN 0-471-07338-5
- Spieler, H.: Introduction to radiation detectors, http://www-physics.lbl.gov/~spieler/physics_198_notes_1999/
- Reimer L.: Scanning Electron Microscopy – Physics of Image Formation and Microanalysis, ISBN: 3-540-63976-4
- Hamamatsu photonics K.K.: Photomultiplier Tubes – Basics and Applications http://sales.hamamatsu.com/assets/applications/ETD/pmt_handbook_complete.pdf