

SIGMA 500

FESEM Training

Practical Electron Microscopy

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We make it visible.

Introduction to Electron Microscopy

Course Aim

To provide the SEM user with an understanding of the operational principles, procedures and practical experiences to enable good quality micrographs to be achieved.

- Combination of theory and practical
- Theoretical background of SEM
- Practical aspects of SEM operation
- Hands on experience of SEM using real specimens



Some SEM History & Origin of Zeiss SEMs

- 1847 Carl Zeiss begins making optical microscopes
- 1881 Cambridge Scientific Instruments founded by Horace Darwin
- 1931 Germans Max Knoll & Ernst Ruska invent the TEM and later STEM
- 1938 Von Ardenne publishes the seminal theoretical and experimental work on the SEM. Von Ardenne is largely credited as being the true father of the SEM
- 1942 Zworykin independent work at RCA in the USA advances the technology but still not high resolution except in STEM mode
- 1953 Dan McMullan, the first of three research students (later also K.C.A. Smith & O. Wells) under the direction of Sir Charles Oatley at Cambridge University builds the first viable high resolution SEMs. This leads directly to the first commercial SEM.
- 1965 First commercial SEM (*Cambridge Instruments Stereoscan Mark I*)

Early Scanning Electron Microscopes

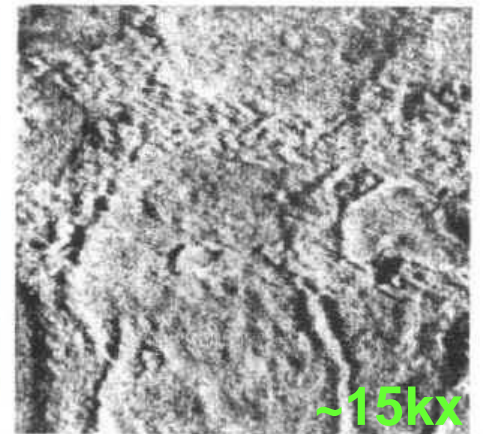
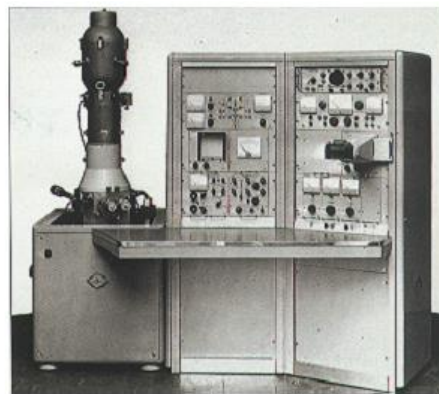
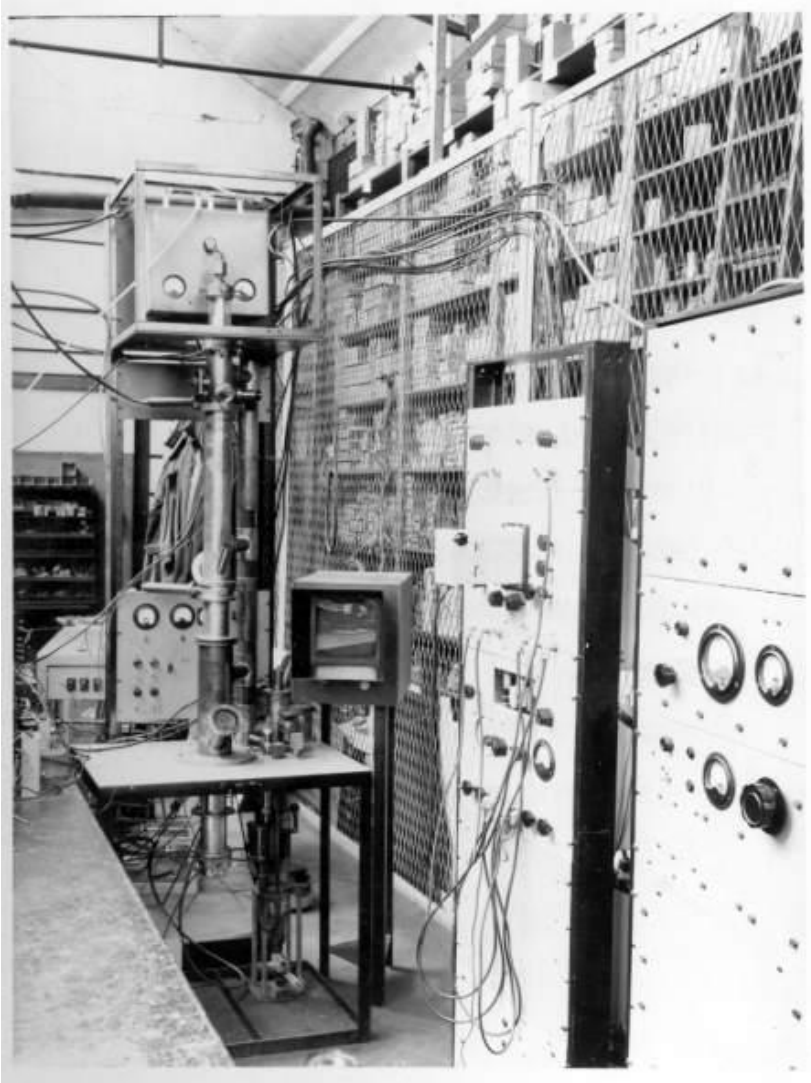


FIG. 8 Micrograph of etched brass produced by the SEM of Zworykin *et al.* (1942a); horizontal field width = 18 μm .

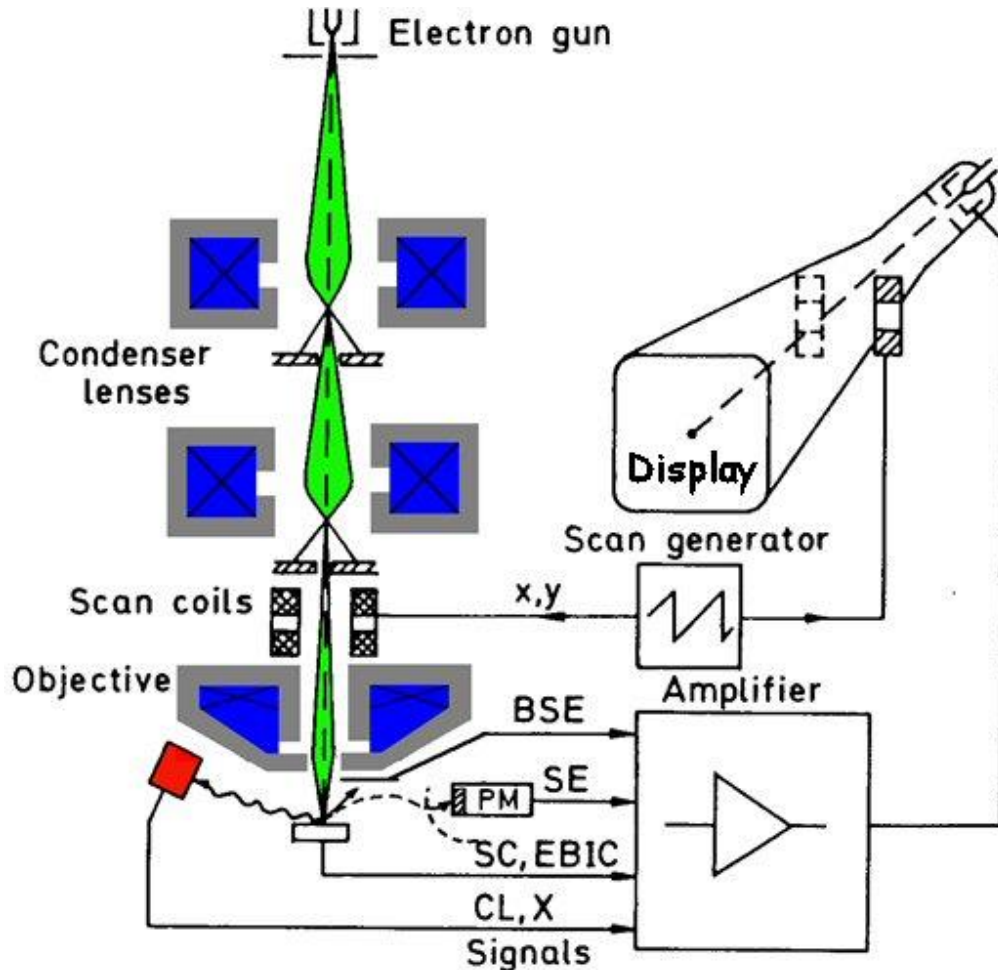


Some SEM History & Origin of Zeiss SEMs

- 1985 First all digital SEM: ZEISS DSM 950
- 1986 First software controlled SEM: Cambridge Stereoscan 360
- 1990 Leica Electron optics formed, a merger between Cambridge Instruments and Wild Leitz
- 1992 Second generation software controlled SEM (*Leica 400*)
- 1996 LEO Electron Microscopy formed as a joint venture between Leica and Carl Zeiss
- 1999 Third generation software controlled SEM (*LEO 1400*)
- 2001 Carl Zeiss acquires all of LEO and SEMs are branded ZEISS

Scanning Electron Microscope

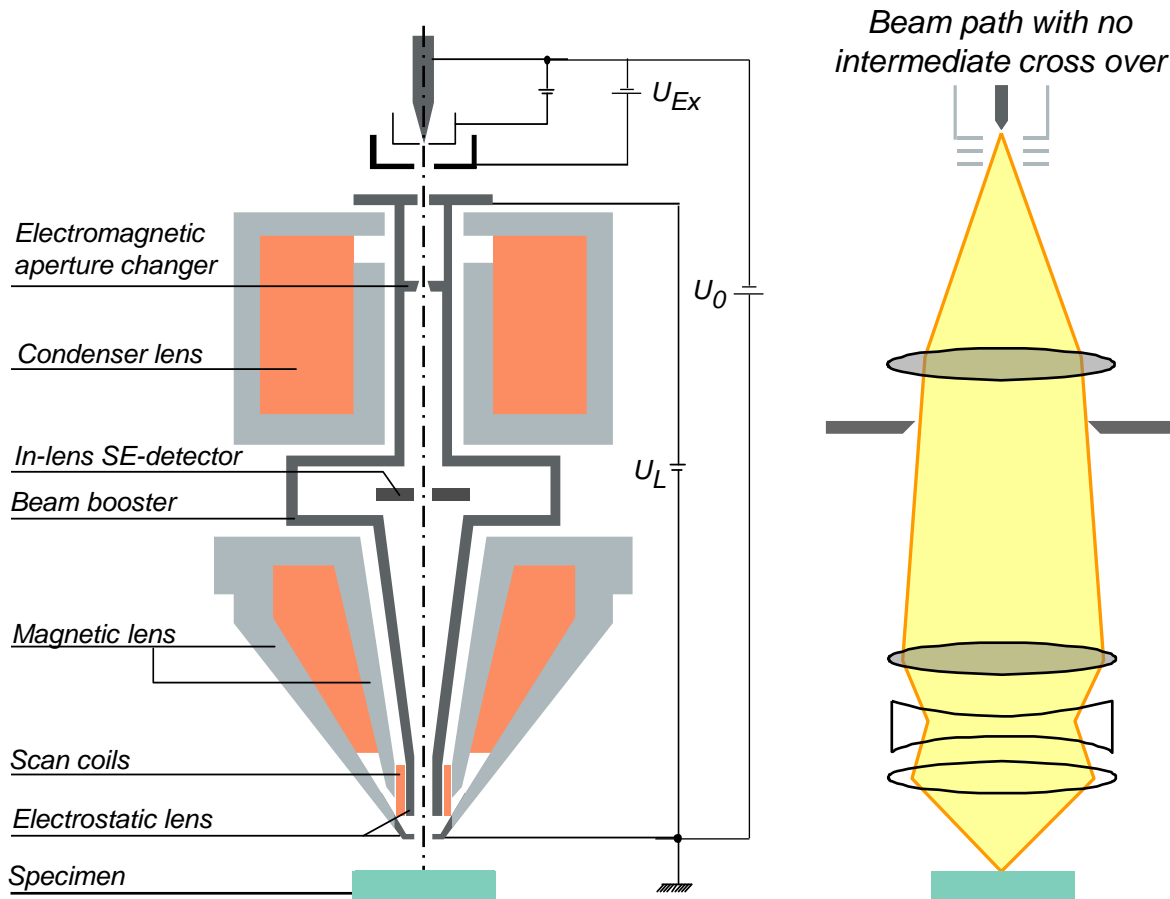
General Operating Principle of a SEM



Sigma 500
Sigma 500 VP

Electron Optics

Operating principle of the Gemini column



Features

- **highly stable thermal FEG**
< 0.5 % /h variation
- **low beam noise**
< 1 %
- **cross over free beam path**
no significant Boersch effect,
high depth of field
- **beam booster**
superb image resolution
throughout the whole
beam energy range,
particularly down to 100 eV.
High resistance to ambient
magnetic stray fields

Sigma 500

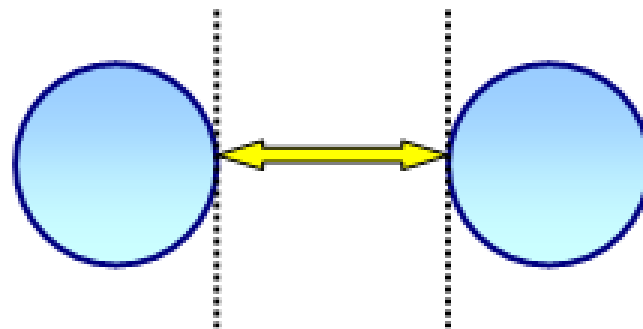
Specification & Option Overview



Sigma 500	
Res	0.8 nm @ 15 kV 1.6 nm @ 1 kV
Detection	Clear View Detection Suite Inlens SE or Inlens Duo ETSE VPSE-G4 C ² D AsB / HDBSD / SenseBSD CL YAG Detector BSD4 aSTEM
Other	32 x 24 k image store Plasma Cleaner Eucentric or Cartesian stage 80 mm or 200 mm Airlock

What is the meaning of Resolution

What is Resolution? Resolution is defined as the smallest distance between two particles that can be distinguished.



Resolution measurement

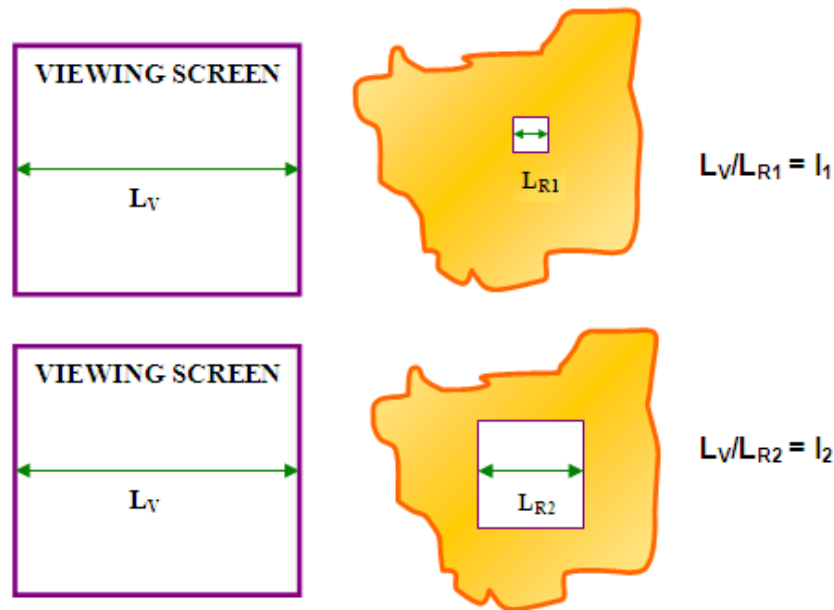
Resolution is not only dependent on the SEM, but on the sample itself and operating conditions used in its observation.

Concept of Magnification in the SEM

- ✓ In the SEM, the magnification is the ratio of the area scanned on the sample to the output device
- ✓ The smaller the area scanned, the higher the magnification and visa versa
- ✓ Historically, the output device was the Polaroid film (4"x5")

The Concept of Magnification

$$I (\text{Image}) = \frac{\text{Linear size (viewing screen)}}{\text{Linear size (raster on sample)}}$$



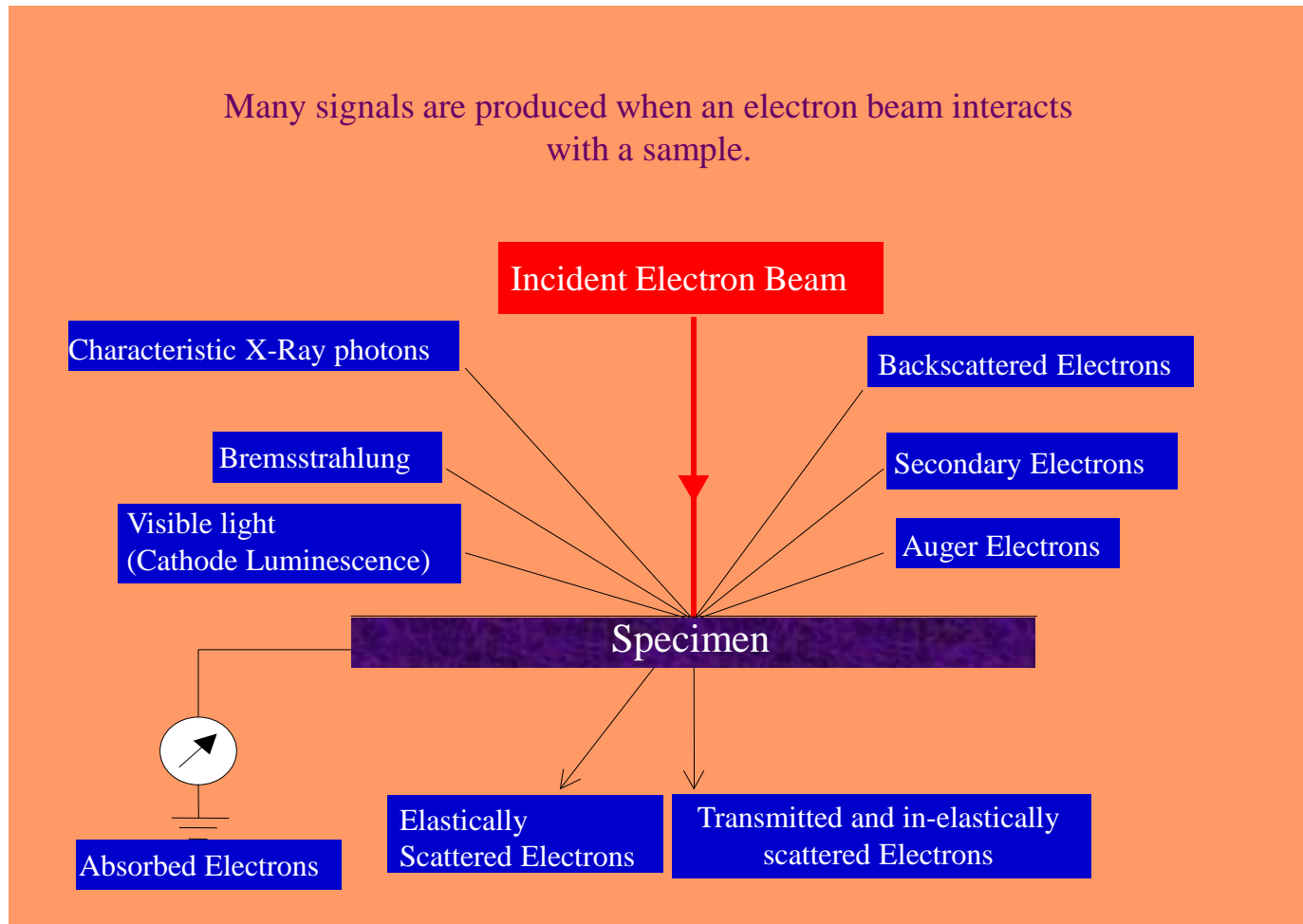
For example, if $L_V = 500$ pixels, $L_{R1} = 5$ pixels, and $L_{R2} = 50$ pixels, then

$$I_1 = 500/5 = 100 \times$$

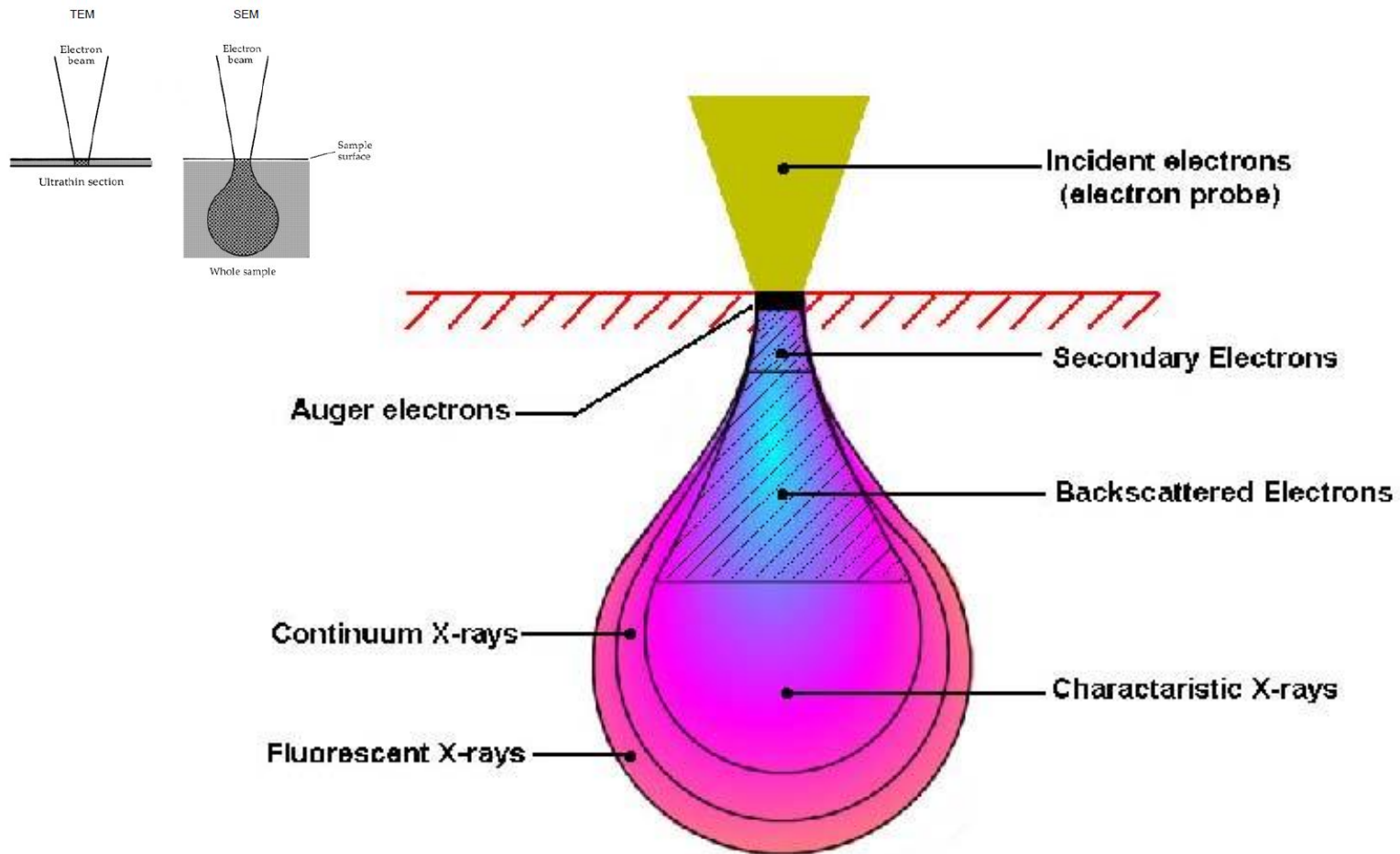
$$I_2 = 500/50 = 10 \times$$

Beam Specimen Interactions

Many signals are produced when an electron beam interacts with a sample.



Beam Interaction Diagram



Electron Beam Interaction Diagram

Electron Gun Types



	W	LaB6	Schottky FE	Cold FE
Beam diameter	1 - 2 μm	1 - 2 μm	10 - 25 nm	3 - 5 nm
Temperature	2300° C	1500° C	1500° C	Room temp
Brightness	1	10	500	1000
Energy spread, ΔE	2.0 eV	1.5 eV	0.5 eV	0.2 eV
Stability, %/h	0.1 %	0.2 %	0.1 % ★	5 %
Probecurrent	1 μA	1 μA	>100 nA ★	10 nA
Life time	1 month	6 months	18 months	5 years
Gun vacuum	10^{-5} Torr	10^{-7} Torr	10^{-9} Torr ★	10^{-11} Torr

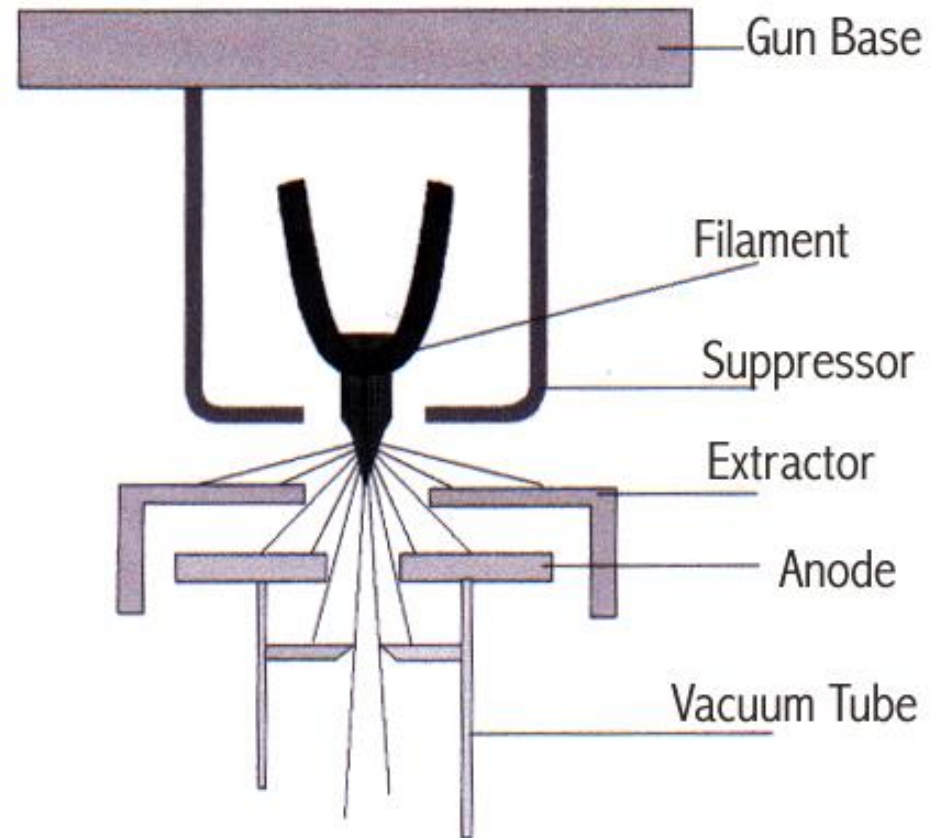


Major Benefits of Schottky FE

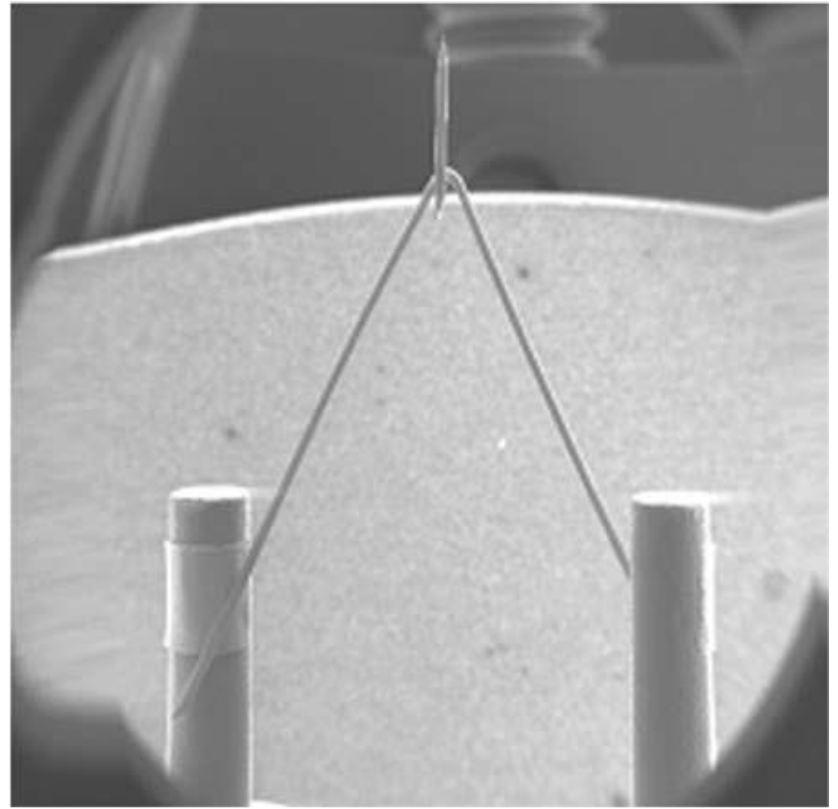
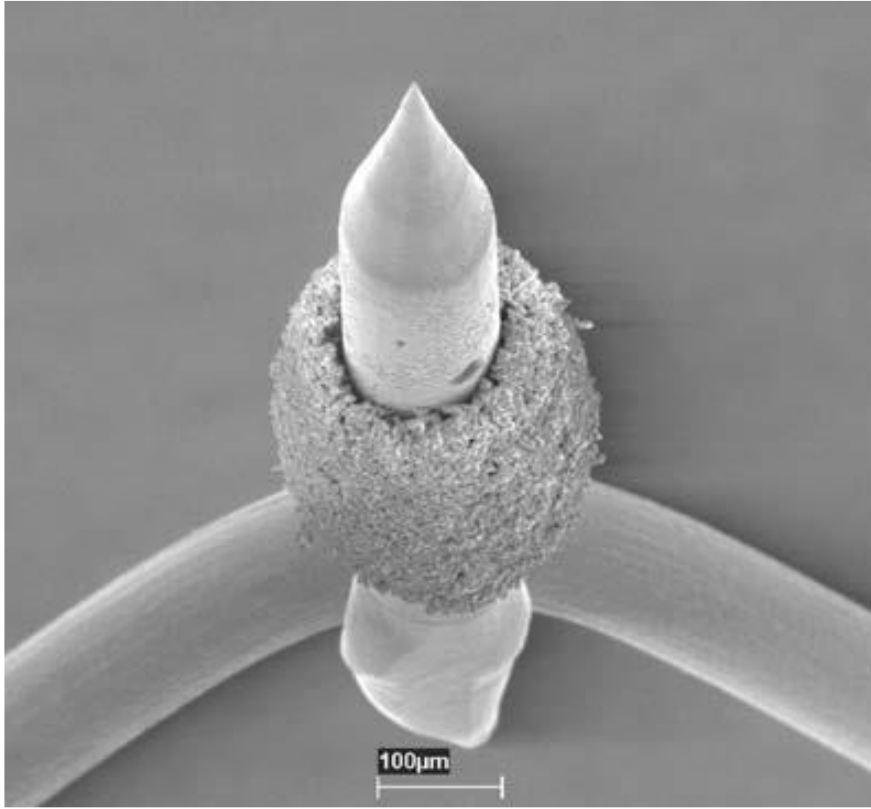
Electron Gun

The Schottky Emitter

- ✓ The tip is welded to a filament and is then centered mechanically in the Suppressor electrode which prevents stray thermionic emission from passing down the column
- ✓ The voltage on the extractor electrode controls the emission current from the gun
- ✓ The Anode is at ground potential (positive)



Schottky FE Emitter



**Filament tip with collar-like ZrO reservoir (left),
ZrO depleted tungsten filament (right).**

Detectors Types

✓ **Secondary Electron Detector (SED)**

- In-chamber Everhart Thornley
 - *Best for surface topography*
- In-column (in-lens) or InLens Duo (backscatter, energy filtered)
 - *Best for surface information (low energy)*

✓ **Backscattered Detector (BSE)**

- AsB solid state (below objective lens)
 - *Best for channeling contrast (3kV and above)*
- EsB in-column
 - *Best for Z contrast (3kV and below)*
- NTSBSD (4 or 5 Segment)
 - *Suited for Z contrast and topographic imaging*
- SenseBSD - TEM-like images at low kV (7kV and below)

✓ **Scanning Transmitted Electron Detector - aSTEM**

✓ **Variable Pressure Detector – VPSE G4**

- Ionization Detectors used at 1Pa and higher

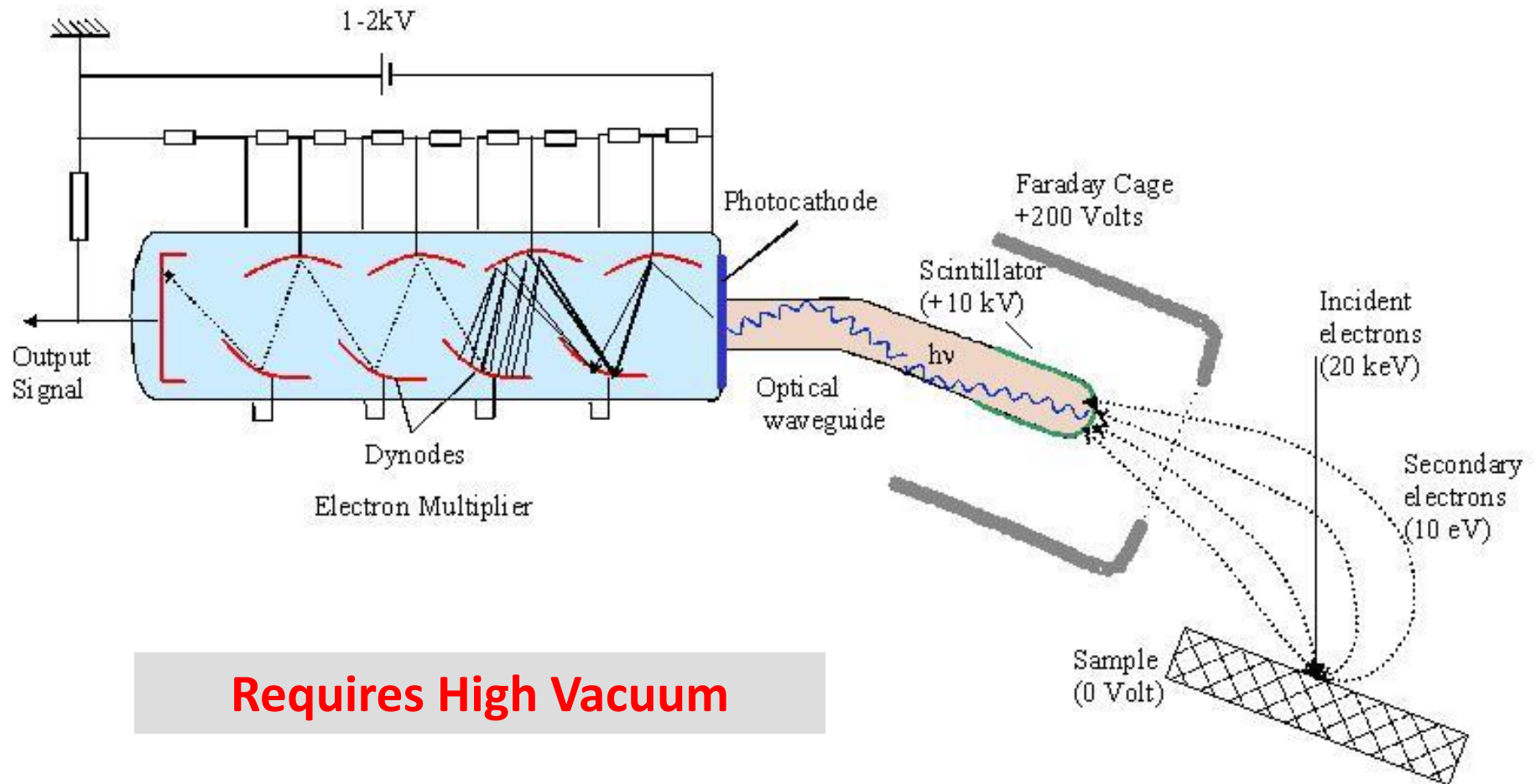
✓ **Energy Dispersive Spectrometer (EDS)**

✓ **Wavelength Dispersive Spectrometer (WDS)**

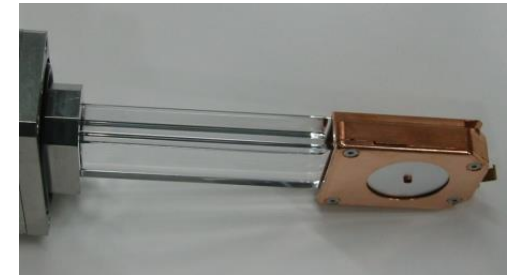
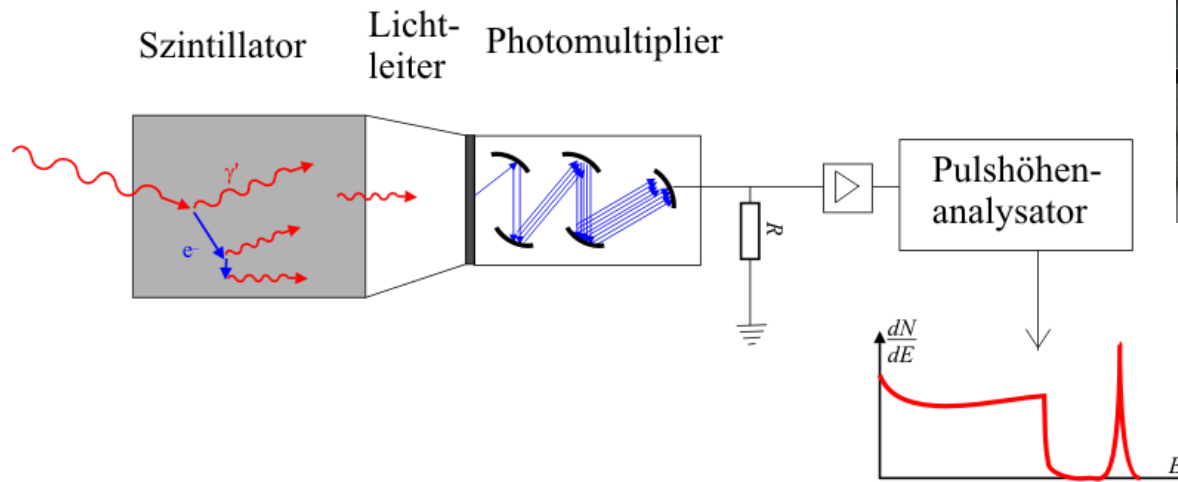
✓ **Electron Backscattered Diffraction Pattern (EBSD or EBSP)**

✓ **Cathodoluminescence Detector (CL)**

In-Chamber Everhart Thornley Secondary Electron Detector



In-Lens Everhart Thornley Secondary Electron Detector



In Lens detector

Advantage: fast, best image quality

Disadvantage: scintillator voltage is limiting use to high vacuum

e^- excite scintillator material (e.g. ZnS, NaI) (approx. 10kV acceleration voltage at crystal)

→ de-excitation by emission of light

→ emission of electrons due to photoelectric effect in photomultiplier

→ avalanche charge (signal enhancement)

→ amount of charge ~ primary signal

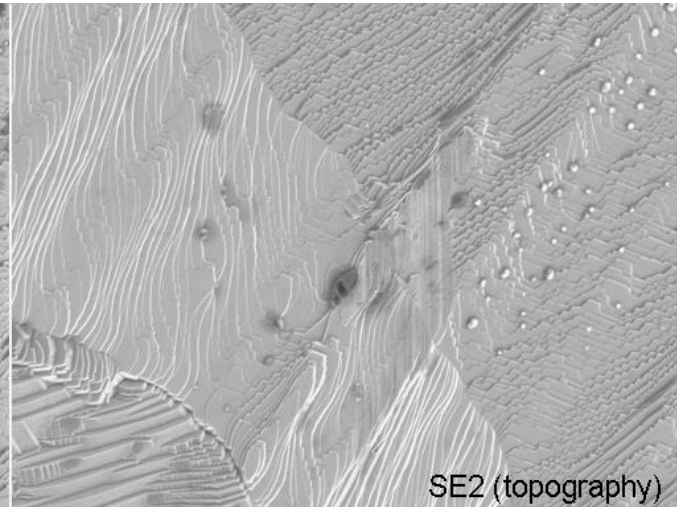
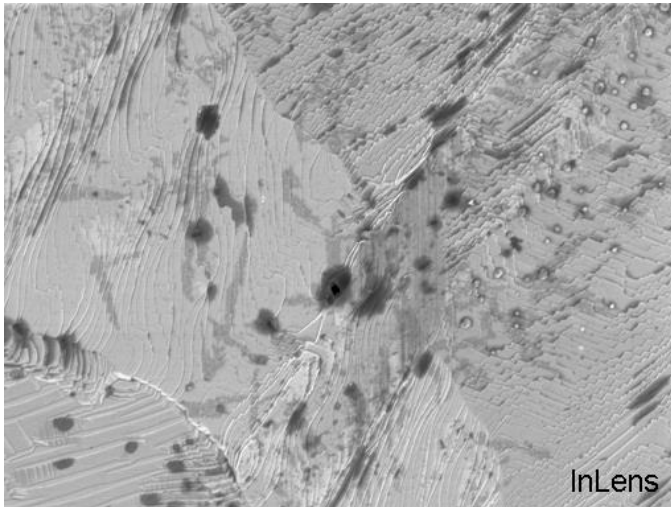
Secondary Electron Detection

InLens:

- detail surface information
- edge effects
- SE1 detection
- sensitive to charging
- highest Resolution possible
- shorter working distance (WD) – better resolution
- optimum WD-range: <5mm (for beam booster to be efficient)

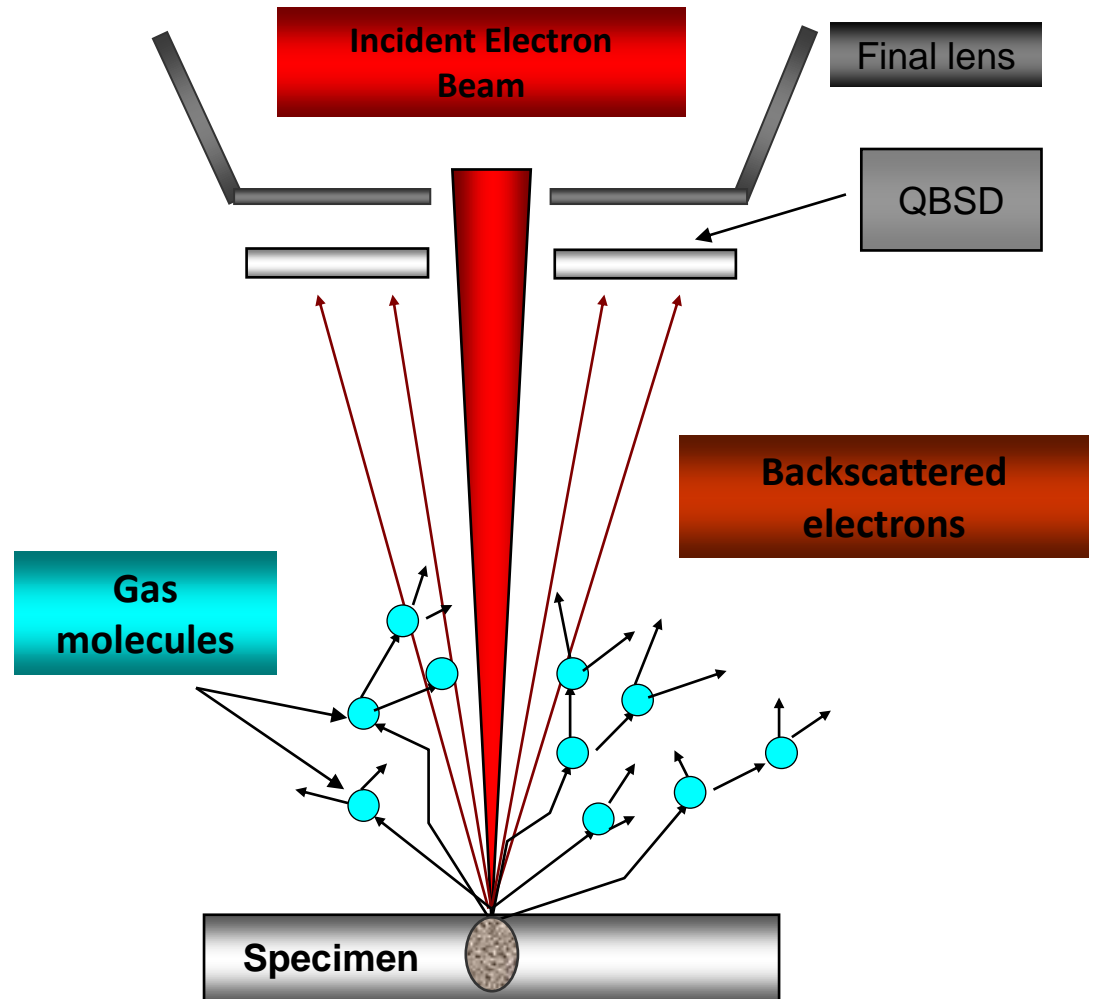
Everhart-Thornley:

- topographic surface information
- edge effects
- distinct shadows due to geometry
- SE2 detection
- insensitive to charging
- optimum WD-range: 5-12mm

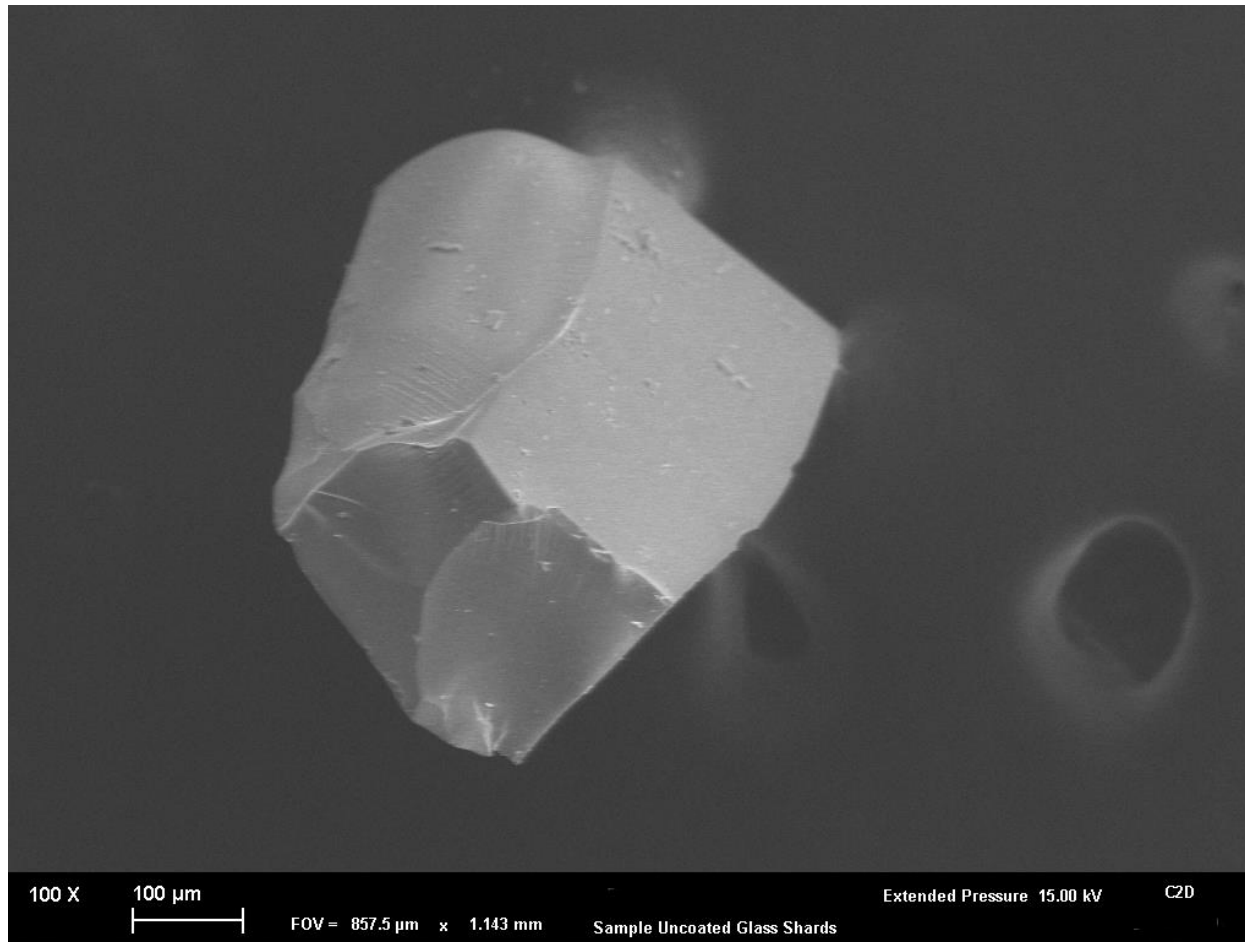


Solid State BSD Principle

- Backscattered electrons and secondary electrons collide with the introduced gas molecules and produce ions that dissipate the charge on the sample surface.
- Higher energy electrons (BSE) travel upwards.
- Backscattered electrons are detected by QBSD and converted to electrical signal.
- Amplified signal by head amplifier to signal processor; used to form an image.



Uncoated glass shard – VP mode, 15 kV



When to use which detector

Detector	Information	Opt.Working Distance	Energy
Everhart-Thornley (high vacuum) VPSE-G4 (VP/high pressure)	Topographic surface information	5-12mm	0.02-30kV
InLens/Duo	Surface details, High Resolution Imaging	<5mm	0.02-20kV
AsB/CZ NTS BSD/	Z-Contrast Channeling contrast (crystallographic information, strain, deformation)	5-10mm 2-5mm (material dependent)	>5kV
InLens Duo	Z-Contrast (sharp due to energy filter) Low Loss BSE: Compositions, Bondings	<5mm	<1.5kV
SenseBSD	Low kV high contrast TEM-like imaging	4-6mm	<=7kV (absolute)

The Three Basic Parameters

The Three Basic Parameters

- ✓ **Accelerating Voltage** (EHT)
- ✓ **Aperture Size** (diameter) which controls
Spot Size (beam or probe current)
- ✓ **Working Distance** (sample-to-lens distance)

Accelerating Voltage

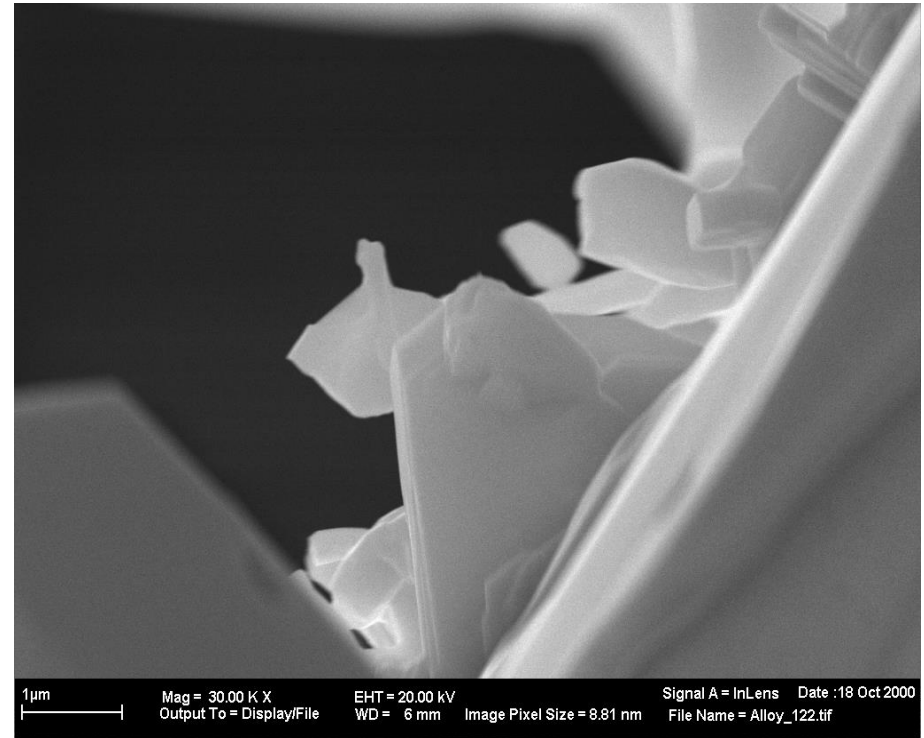
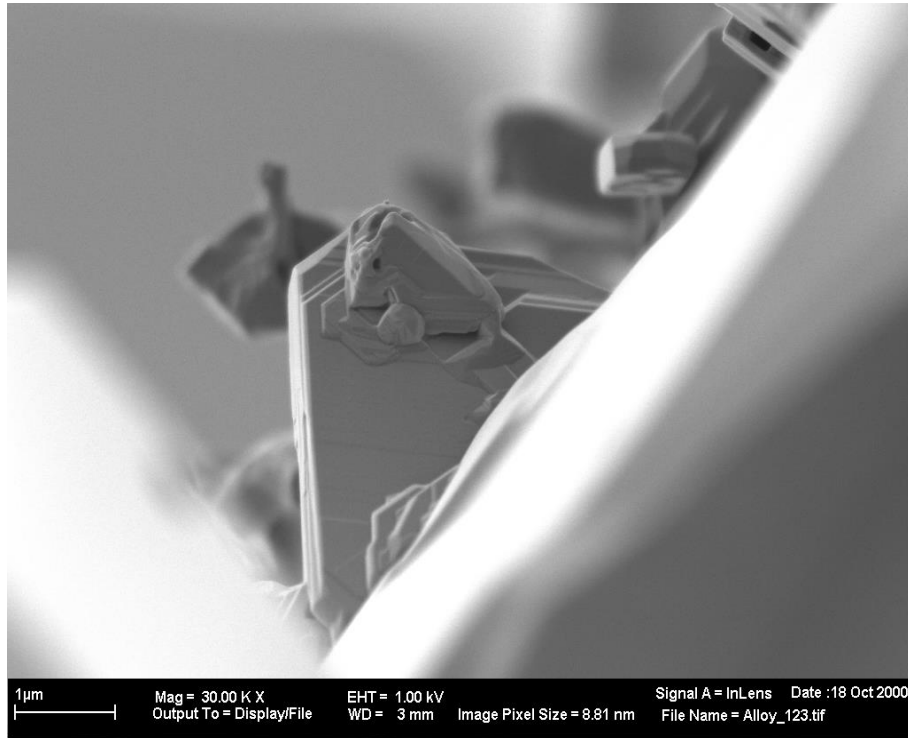
High kV

- Higher resolution
- Greater beam penetration
- Higher beam current
- Higher signal to noise
- More x-ray yield
- Good for VP

Low kV

- Lower resolution
- Less beam penetration
- Lower beam current
- Lower signal to noise
- Lower x-ray yield
- Not so good for VP

Beam Sample Interaction – Influence of Beam Energy



Platinum Rhodium Alloy Crystals at 1kV (left) and at 20kV (right)

Aperture Size (changes Spot Size)

Small Aperture

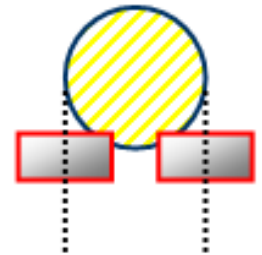
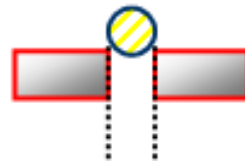
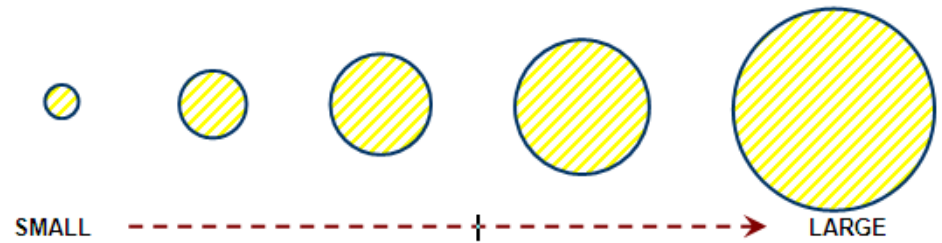
- Higher resolution
- Higher depth of focus
- Lower beam current
- Lower signal to noise
- Less x-ray yield
- May have smaller field of view (aperture cut-off)

Large Aperture

- Lower resolution
- Less depth of focus
- Higher beam current
- Higher signal to noise
- Higher x-ray yield

Spot Size

- The aperture diameter controls the final spot size on the specimen
- The smaller the spot size, the better the resolution; however, this smaller probe comes at the sake of losing beam current
- With a small spot size, the area between the two blocks can be distinguished; with the larger spot size, it cannot
- OptiProbe adjusts spot size



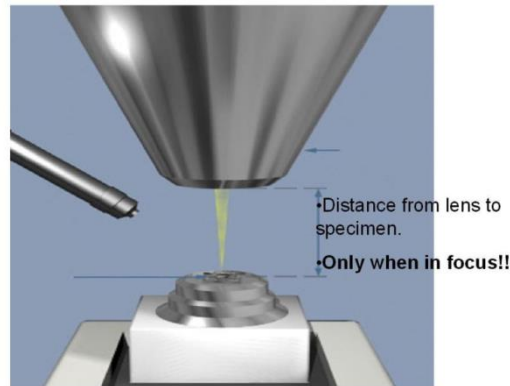
Working Distance

Short Working Distance

- Higher resolution
- Less Depth of Focus
- Better collection efficiency for In-Lens/Duo, AsB, NTSBSD, SenseBSD, and EsB detectors

Long Working Distance

- Lower resolution
- Greater Depth of Focus
- Better collection efficiency for In-Chamber SE



Variables Involved in Image Formation

- ✓ Acceleration Voltage (kV)
- ✓ Final Aperture (size)
- ✓ Probe Current (I_{probe})
- ✓ Working Distance (WD)
- ✓ Specimen
- ✓ Column/Aperture Alignment
- ✓ Detector
- ✓ Specimen geometry
- ✓ Scanning speed
- ✓ Signal processing
- ✓ Noise reduction
- ✓ Contrast / brightness
- ✓ Chamber pressure
- ✓ System cleanliness

Questions to Ask Yourself

What is information is required from the specimen

- High Magnification
- Low Magnification
- Compositional information
- Topographical information
- X-ray analysis

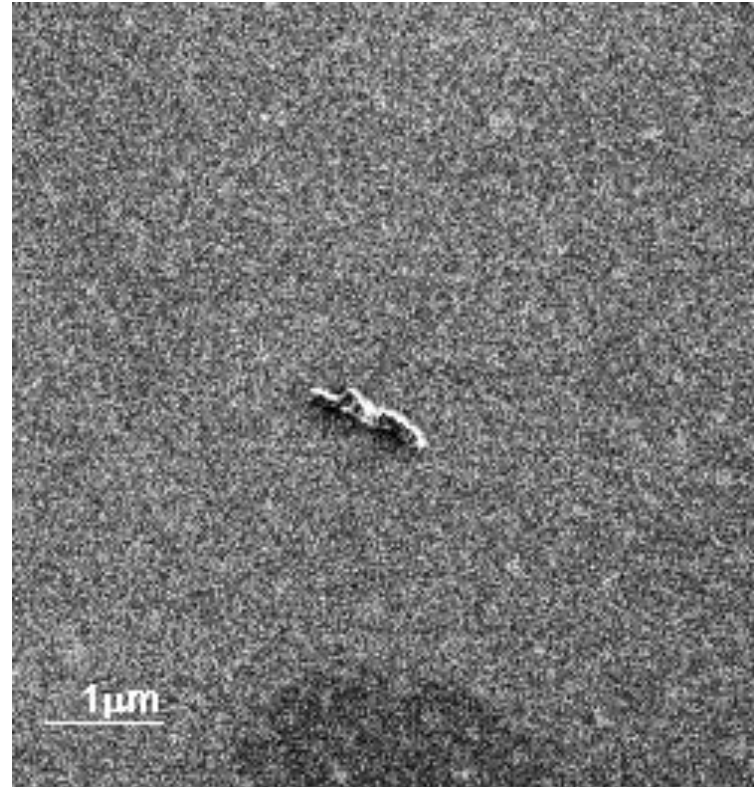
Other Factors

- Stability
- Beam sensitivity
- Electrical conductivity

Charging and Contamination

Choosing a detector

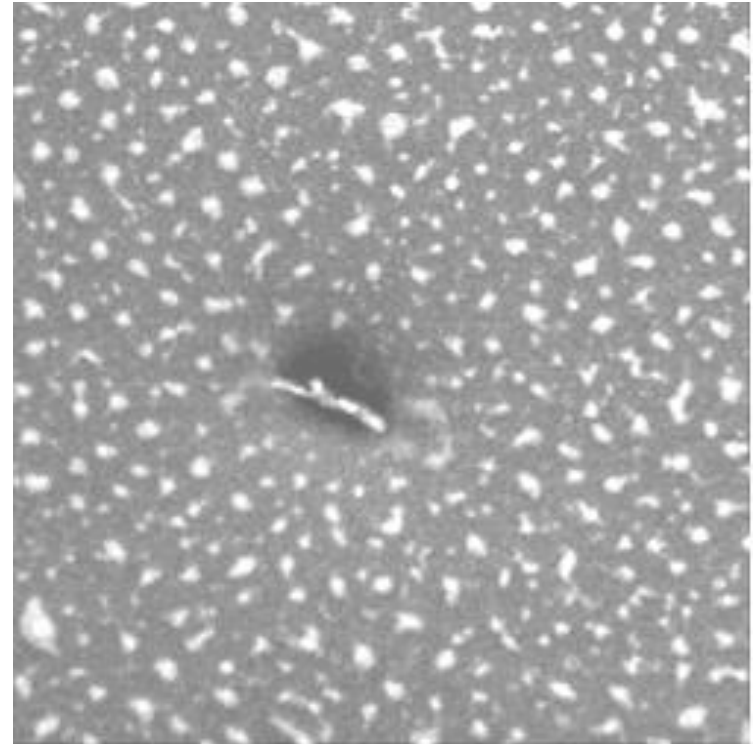
- The choice of detector can have a significant effect on the apparent severity of charging
- The conventional ET (Everhart - Thornley) detector is much less sensitive to charging than...



Individual polymer macro-molecules on Si at 1.5keV - Lower (ET) detector

Upper detector

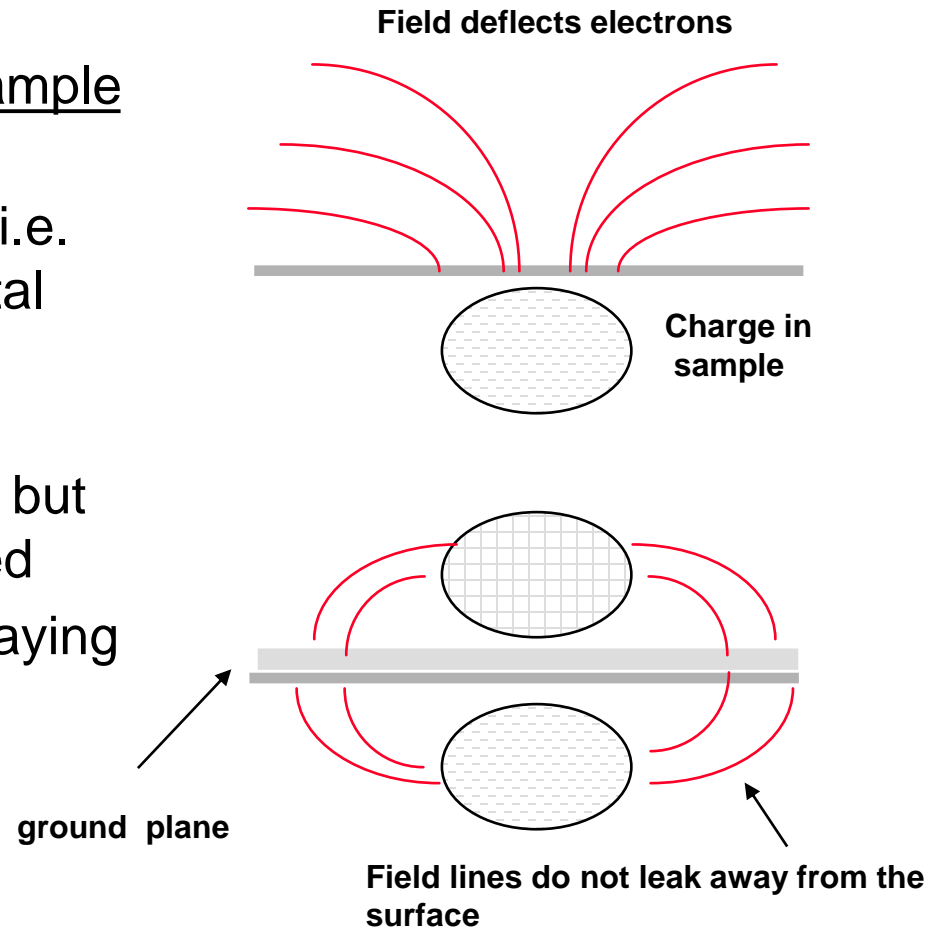
- ...the InLens detector. This is because these systems act as simple SE spectrometers and preferentially select low energy electrons
- Note however that charging can be a useful form of contrast mechanism when properly employed
- Backscattered electrons are less affected by charging and offer the same resolution at LV



**Same area as before using
InLens detector**

If all else fails.....coat the sample

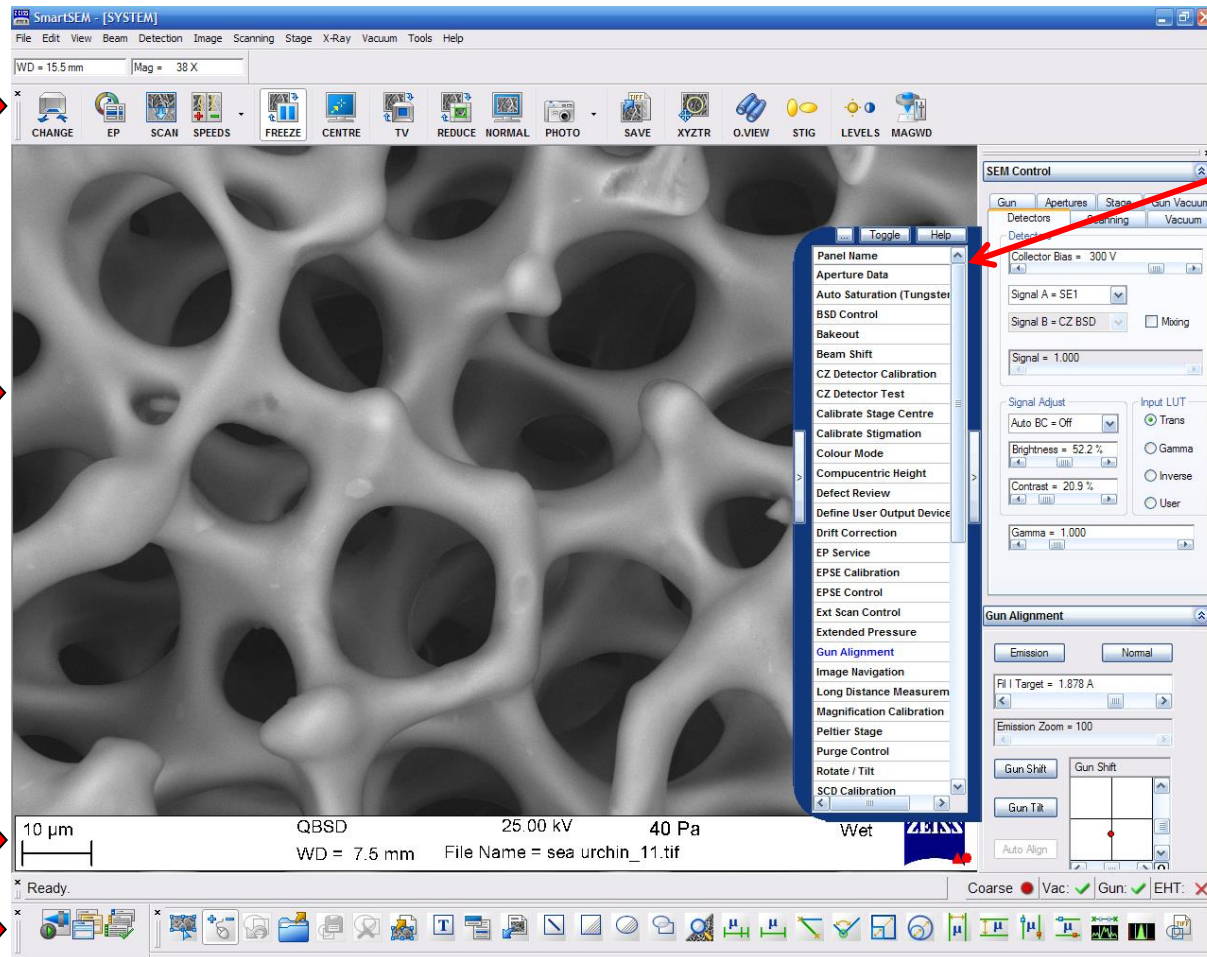
- Coatings do not make the sample a conductor
- They form a ground plane - i.e. the free electrons in the metal move so as to eliminate the external field
- The charge is not eliminated but the disruptive field is removed
- Successful coating means paying attention to the details...



Software

SmartSEM[®] software

ZEISS



Tool Bar

Imaging Zone

Data Zone

Recipes

Main Menu

Docking panel

Status Bar

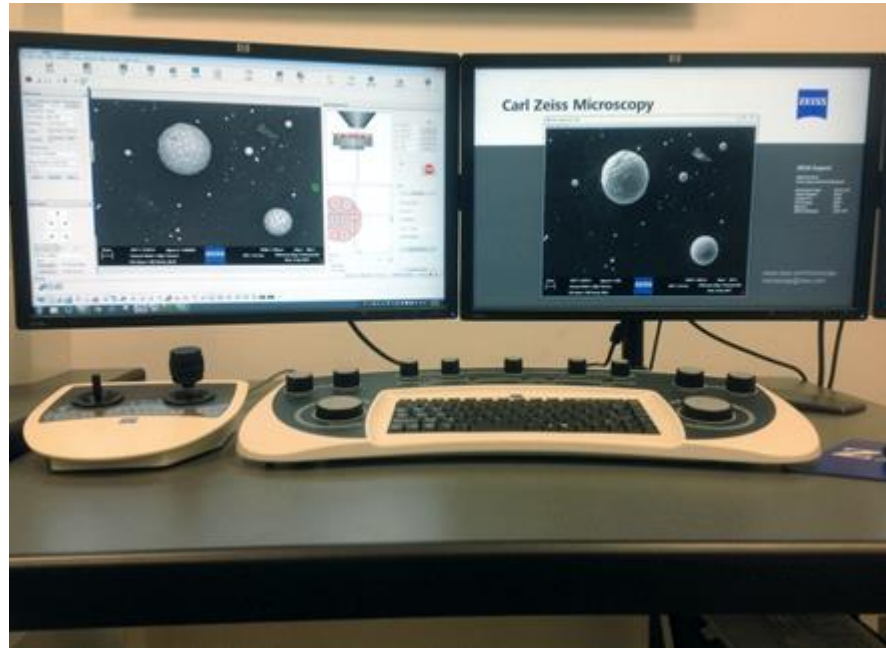
Annotation panel

SmartSEM® software

- Dual Channel:
The possibility of detecting and displaying image information simultaneously from two different detectors allows users to quickly distinguish small differences on the sample.
- Control Panel:
An optional hardware control panel is available with rotary control for improved tactile feedback.

InLens

BSD



SmartSEM[®] software

➤ Recipes

Using the recipes option, users can easily define and store protocols. The recipes enable the users to run the entire imaging process at a click of the mouse.



➤ Macros

Creating and running macros make routine procedures to be performed easily and quickly. Various macros in the software are easily accessible and linked to the icons in the toolbar. Using the assigned macros reduces the imaging time, aids reproducibility and accuracy.

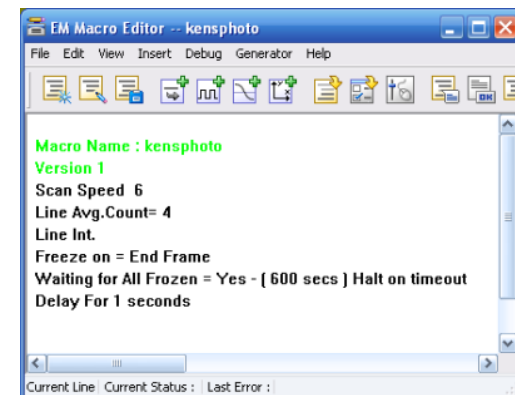
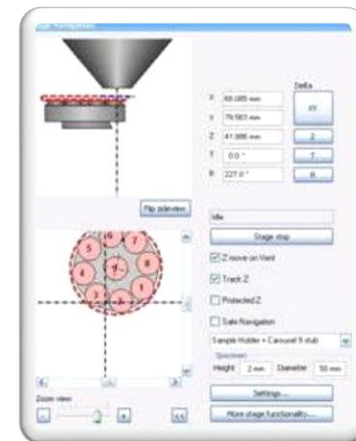


Image Navigation

- Locating the region of interest on a specimen in the SEM might sometimes be challenging, especially when identifying small features on large specimens.
- SmartSEM® graphical user interface facilitates easy and fast navigation using built-in camera as well as images from other sources as below
- Images can be imported from:
 - A live, or stored, SEM image
 - A digital camera, webcam or light microscope
 - A CAD package





ZEISS Sample Holders

Carousel 9 stubs



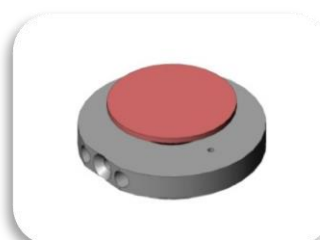
Carousel 8 stubs



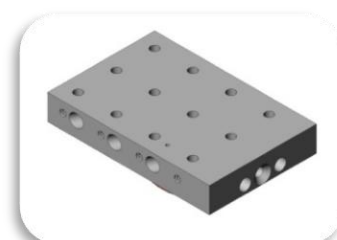
Single stubs



Single stubs



12 x single stubs



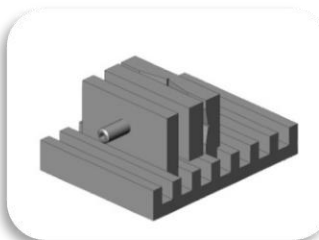
Universal 45° holder



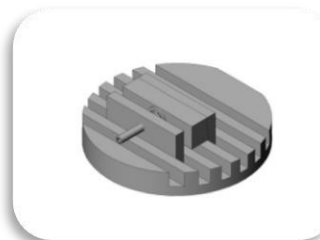
Universal 75° angled



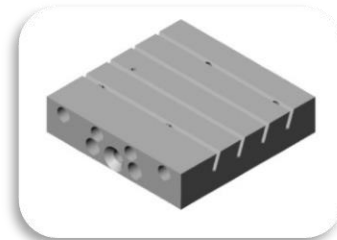
Large multi purpose



Multi purpose variable



70° angled
Multi slice wafer section



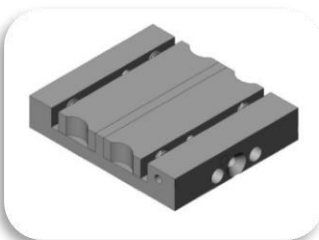
Flat cleaved:
clips for wafer sections



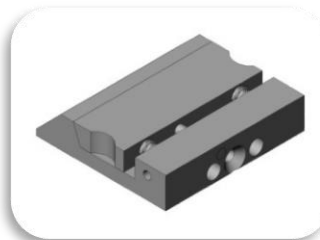
60° angled
Multi slice wafer section



wafer sections



20° angled wafer section

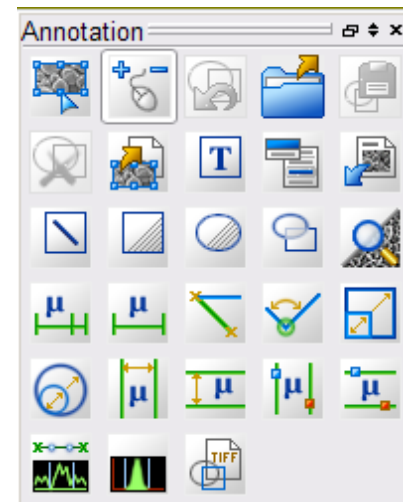


4" or 5" wafer holder



SmartSEM[®] software

- Image store:
Image resolutions up to 32k x 24k pixels images can be acquired and stored as TIF, BMP or JPG files.
ATLAS (optional) 32k x 32k pixel images
- Annotation and measurements:
A paint style control panel enables point to point, line width, line height, radial, and angle measurements on the image.
Annotation of images with text, bitmaps, and micron markers is possible using all Windows[™] fonts and colors.
Insertion/recall of TIFF data from saved images is also possible.

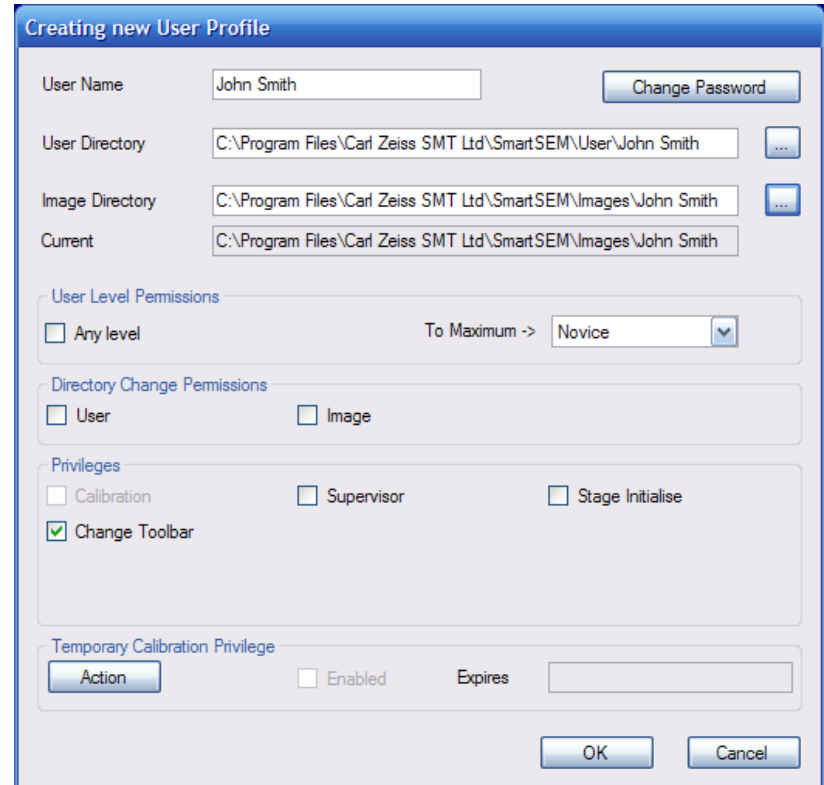


SmartSEM[®] software

➤ Administrator function:

New users accounts can be easily created with various levels of access to the functionality of the instrument.

The usage of the system can also be monitored for individual users.



The image shows a Windows-style dialog box titled "Creating new User Profile". It contains several input fields and checkboxes for configuring a new user.

- User Name:** A text box containing "John Smith". To its right is a "Change Password" button.
- User Directory:** A text box containing "C:\Program Files\Carl Zeiss SMT Ltd\SmartSEM\User\John Smith". To its right is a browse button (three dots).
- Image Directory:** A text box containing "C:\Program Files\Carl Zeiss SMT Ltd\SmartSEM\Images\John Smith". To its right is a browse button (three dots).
- Current:** A text box containing "C:\Program Files\Carl Zeiss SMT Ltd\SmartSEM\Images\John Smith".
- User Level Permissions:** A section with a checkbox for "Any level" and a dropdown menu labeled "To Maximum ->" currently showing "Novice".
- Directory Change Permissions:** A section with two checkboxes: "User" and "Image", both of which are currently unchecked.
- Privileges:** A section with three checkboxes: "Calibration", "Supervisor", and "Stage Initialise", all unchecked. Below them is a checked checkbox for "Change Toolbar".
- Temporary Calibration Privilege:** A section with an "Action" button, an "Enabled" checkbox (unchecked), and an "Expires" text box.

At the bottom right of the dialog are "OK" and "Cancel" buttons.

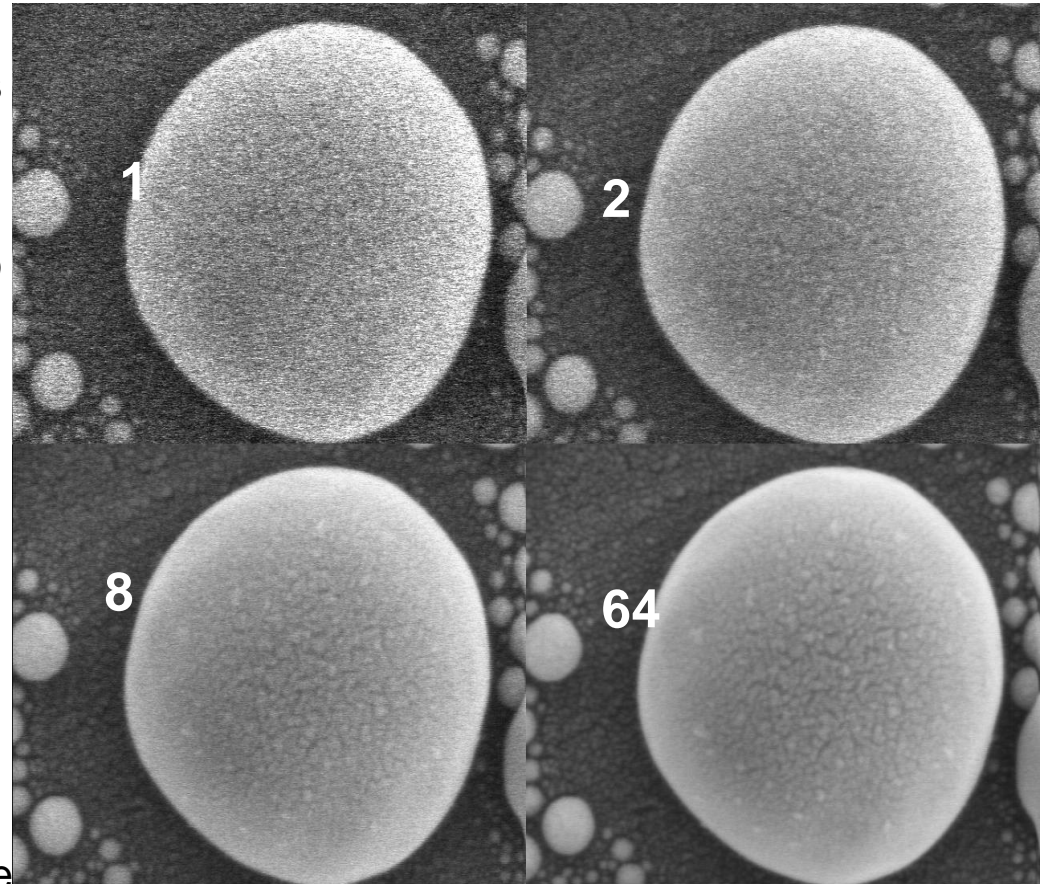
Image Optimization

Image Optimization

- ✓ What information and magnification will provide the data you are seeking
- ✓ Start off with low voltage and increase as resolution requires – never start at high voltage unless you know your sample
- ✓ Know the principles of "the three parameters"
 - Accelerating voltage
 - Aperture/Spot Size
 - Working Distance
- ✓ Make sure your sample is securely mounted to the specimen stub
- ✓ Make alignment and image adjustments at highest possible magnification
- ✓ Check aperture alignment
- ✓ Find center of focus (very important) before adjusting the stigmators – avoid edges or straight lines
- ✓ Repeat focus and stigmatation
- ✓ Lower magnification, adjust C&B and select an appropriate scan mode

Noise Reduction

- The **line** and **frame averaging** options for the framestore can greatly reduce noise
- This should not be used as an excuse to use beam currents that are too low!
- Whenever possible take a **single slow speed scan** rather than accumulating **multiple high speed scans**
- This eliminates blurring due to drift, and distortions in the video amplifier chain and usually produces a higher signal to noise ratio and better contrast
- Higher pixel resolution images require longer acquisition times compared to low pixel resolution images – dwell time should remain the same



Effect of the averaging on noise

Hands On Operation



We make it visible.