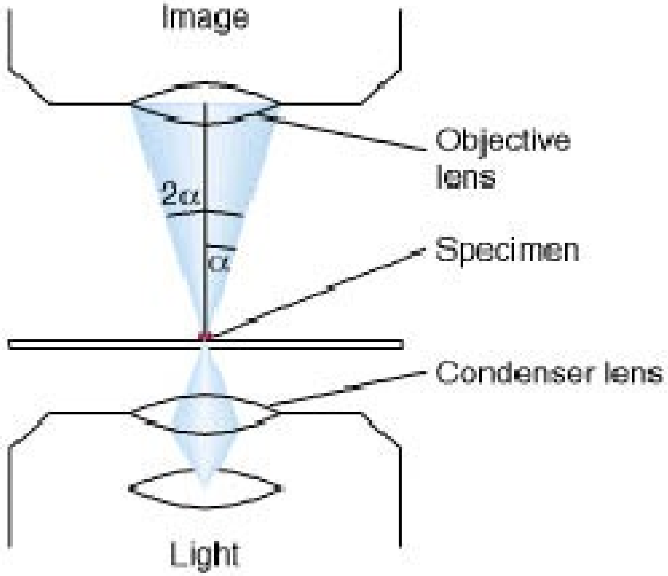
Limite di risoluzione di un microscopio

(b)



$$D = \frac{(0.61\lambda)}{N\sin\alpha}$$

$$N\sin\alpha = \mathrm{Apertura\,Numerica}$$

Risoluzione del microscopio elettronico

$$E = h\nu,\ E = h\frac{c}{\lambda},\ \lambda = h\frac{c}{E}$$

Microscopio ottico: $\lambda = 450nm$ (blu)

Microscopio elettronico: $\lambda = 0.6\,nm - 0.001nm$

Nel microscopio elettronico il fascio e‘ composto da elettroni che vengono accelerati con un potenziale che va da 40kV fino a 3000kV

Risoluzione

Perché usare gli elettroni?

Esempio – calcolare la lunghezza d'onda degli elettroni accelerati da un potenziale di 10 kV:

$$\tfrac{1}{2}mv^2 = eV$$

$$v = \sqrt{\frac{2eV}{m}}$$

$$\lambda = \frac{h}{m}\sqrt{\frac{m}{2eV}} = \frac{h}{\sqrt{2meV}}$$

$$\lambda = \frac{6.63\times10^{-34}\,\mathrm{J\,s}}{\sqrt{2(9.11\times10^{-31}\,\mathrm{kg})(1.60\times10^{-19}\,\mathrm{C})(10^4\,\mathrm{V})}}$$

$$\lambda = 1.23\times10^{-11}\,\mathrm{m} = \boxed{0.0123\,\mathrm{nm}}$$

La risoluzione è comunque limitata dall'aberrazione delle lenti.

Risoluzione del microscopio elettronico

Supponendo una apertura numerica di 1 con
λ=0.004nm la risoluzione sarà d=0.0025nm
Cioé 100,000 volte minore di un microscopio ottico.

Limite di risoluzione reale da circa 1 nm fino ad un minimo di 2 Angstroem

(difetti delle lenti, aberrazioni, limite di contrasto ecc....)

Electron Microscopy: Resolution

L'elettrone si muove a velocità prossime a quelle della luce

$\Downarrow$

Correzioni relativistiche

esempio – lunghezza d'onda per un potenziale di 100 kV

Usiamo la correzione relativistica:

$$\lambda = \frac{h}{m}\sqrt{\frac{m}{2eV}} = \frac{h}{\sqrt{2meV(1+\frac{eV}{2mc^2})}}$$

$$\lambda = \frac{6.63\times10^{-34}\,\mathrm{J\,s}}{\sqrt{2(9.11\times10^{-31}\mathrm{kg})(1.60\times10^{-19}\mathrm{C})(10^4\ \mathrm{V})(1+\frac{(1.60\times10^{-19}\mathrm{C})(10^4\ \mathrm{V})}{2(9.11\times10^{-31}\mathrm{kg})(3\times10^8\,m/s)^2})}}$$

$$\boxed{\lambda = 3.7\times10^{-3}\,\mathrm{nm}}$$

Alto potenziale→ alta velocità : dimensioni atomiche

Trasformazioni di Lorentz

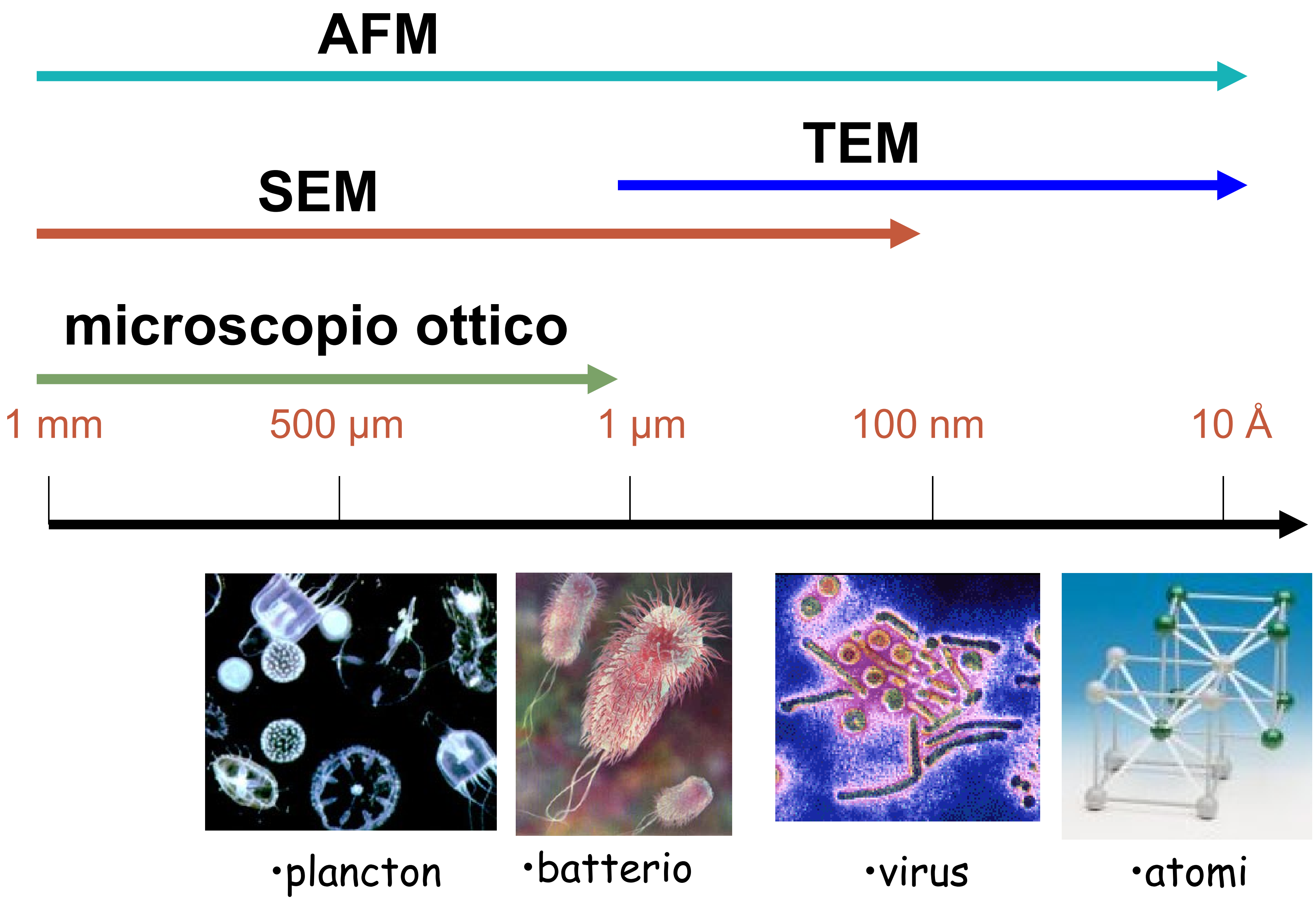
$$x^{'} = \gamma(x - vt)$$

$$t^{'} = \gamma\left(t - \frac{vx}{c^{2}}\right)$$

$$\gamma = \sqrt{1 - \frac{v^{2}}{c^{2}}}$$

$$\vec{p} = \frac{m\vec{v}}{\sqrt{1 - \frac{v^{2}}{c^{2}}}} = \gamma m\vec{v}$$

$$E = \frac{mc^{2}}{\sqrt{1 - \frac{v^{2}}{c^{2}}}} = \gamma mc^{2}$$


AFM
TEM
SEM
microscopio ottico
1 mm
500 µm
1 µm
100 nm
10 Å
•plancton
•batterio
•virus
•atomi

SEM Cathode Comparison

	•Tungsten filament	•LaB_6	•Field Emission
•**Apparent Source Size**	•100 micrometers	•5 micrometers	•<100 Angstroms
•**Brightness**	•1 A/cm^2 steradian	•20-50 A/cm^2 steradian	•100-1000 A/cm^2 steradian
•**Vacuum Required**	•10^{-5} Torr	•10^{-6} Torr	•10^{-9} Torr

Profondità di penetrazione

Fascio incidente = 20 kV

Elettroni retrodiffusi

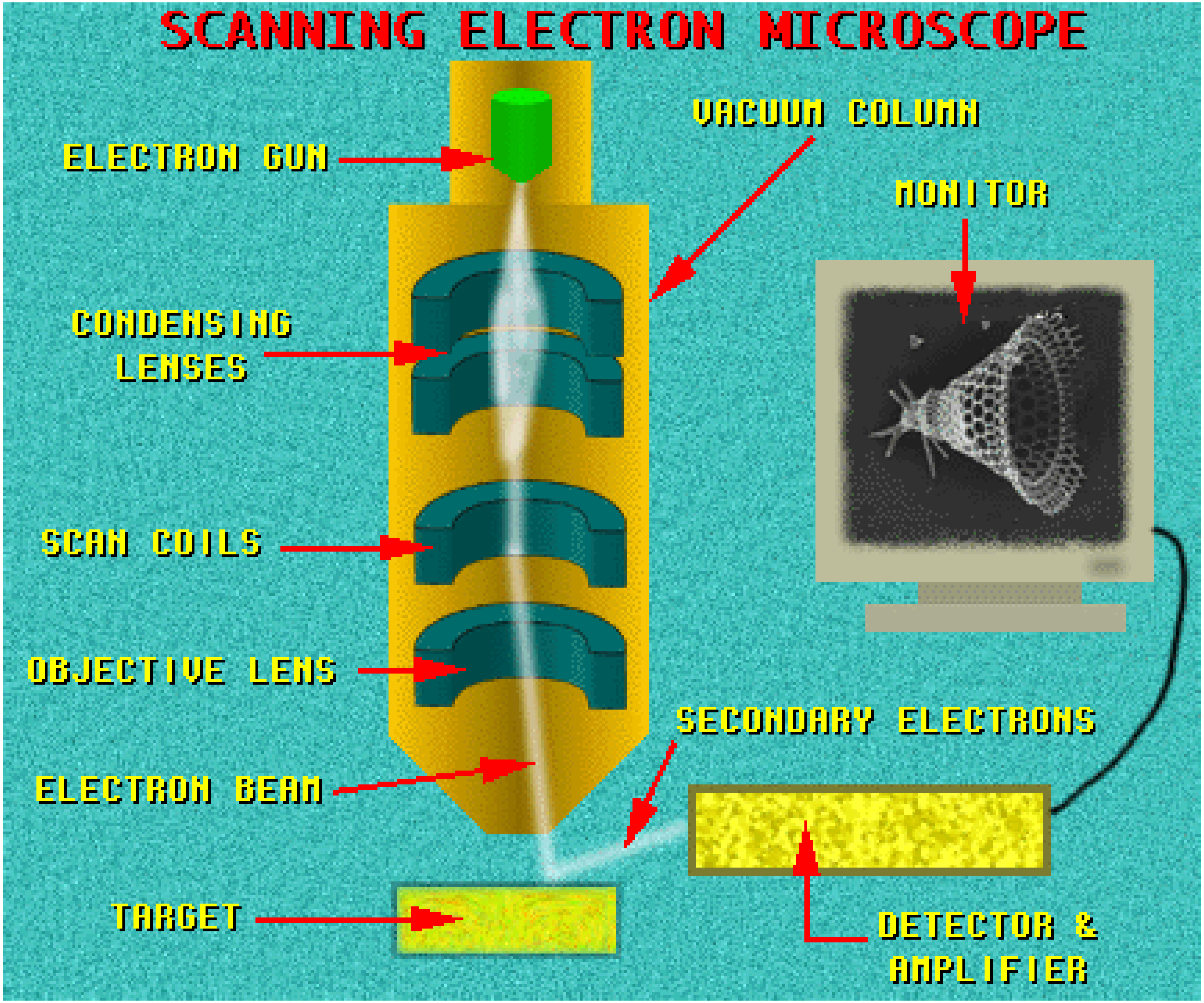
Al	0.7 μm
Au	0.07 μm

Elettroni secondari

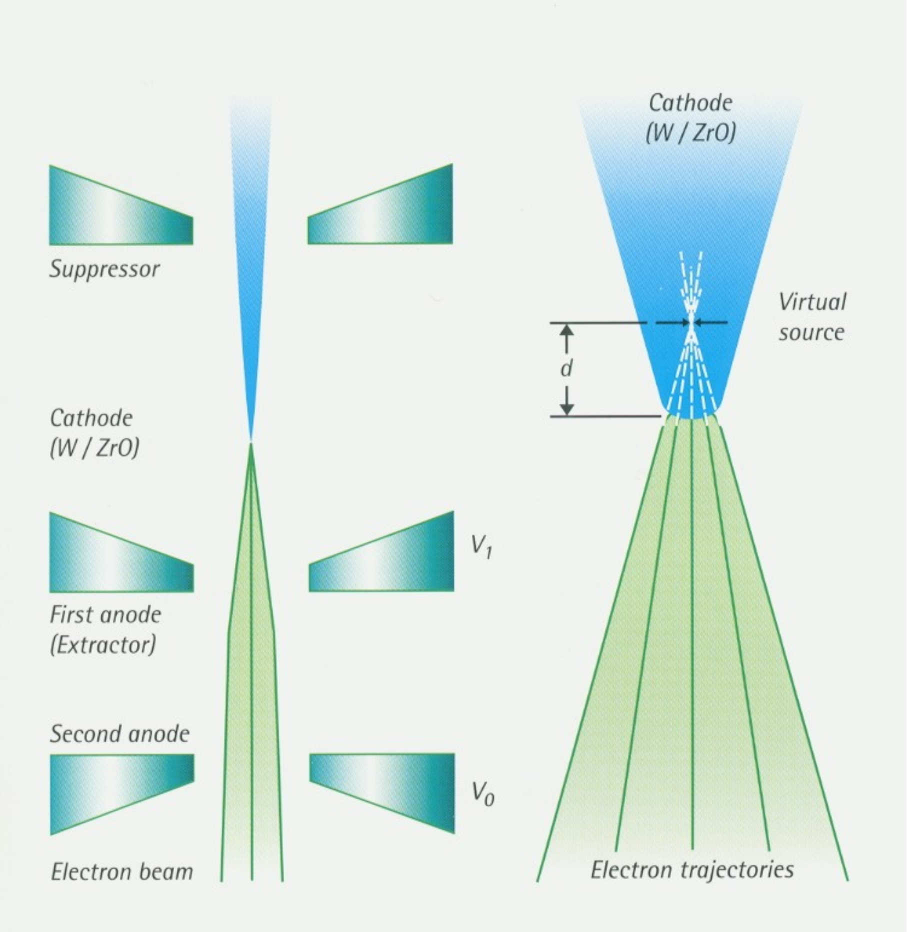
Al	50 nm
Au	5 nm

Raggi X

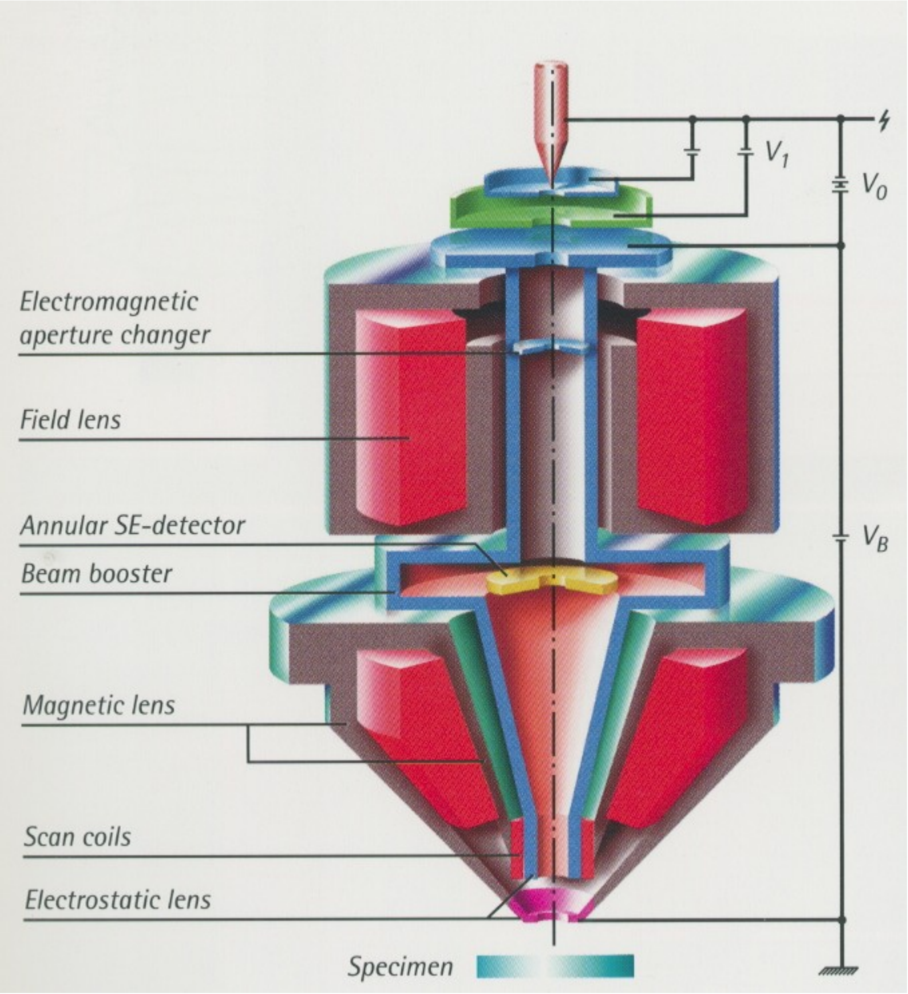
Al	2 μm
Au	0.2 μm


SCANNING ELECTRON MICROSCOPE
ELECTRON GUN
VACUUM COLUMN
MONITOR
CONDENSING LENSES
SCAN COILS
OBJECTIVE LENS
SECONDARY ELECTRONS
ELECTRON BEAM
TARGET
DETECTOR & AMPLIFIER

FEG (Field Emission Gun)



Virtual source formation in field-emission gun. Distance d depends on voltage ratio V_0/V_1.

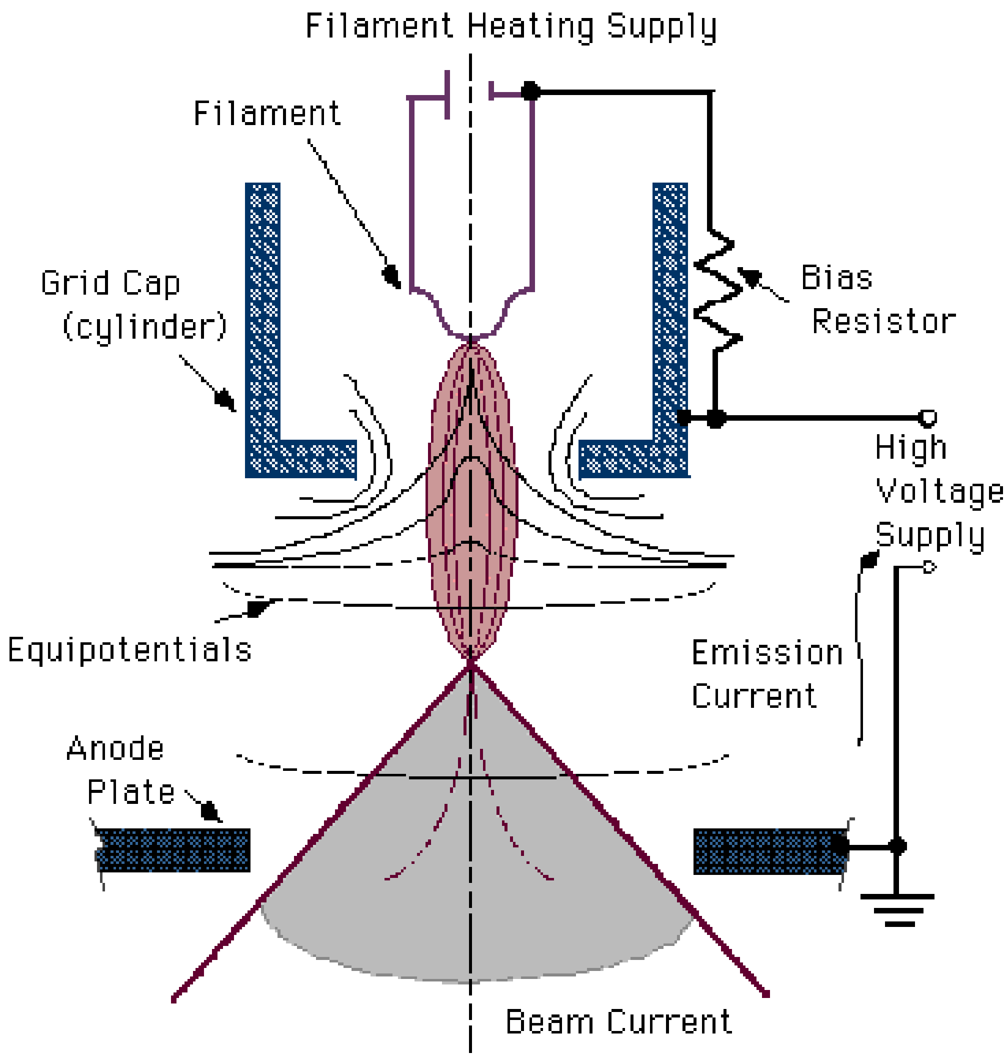


100 V - 30 kV

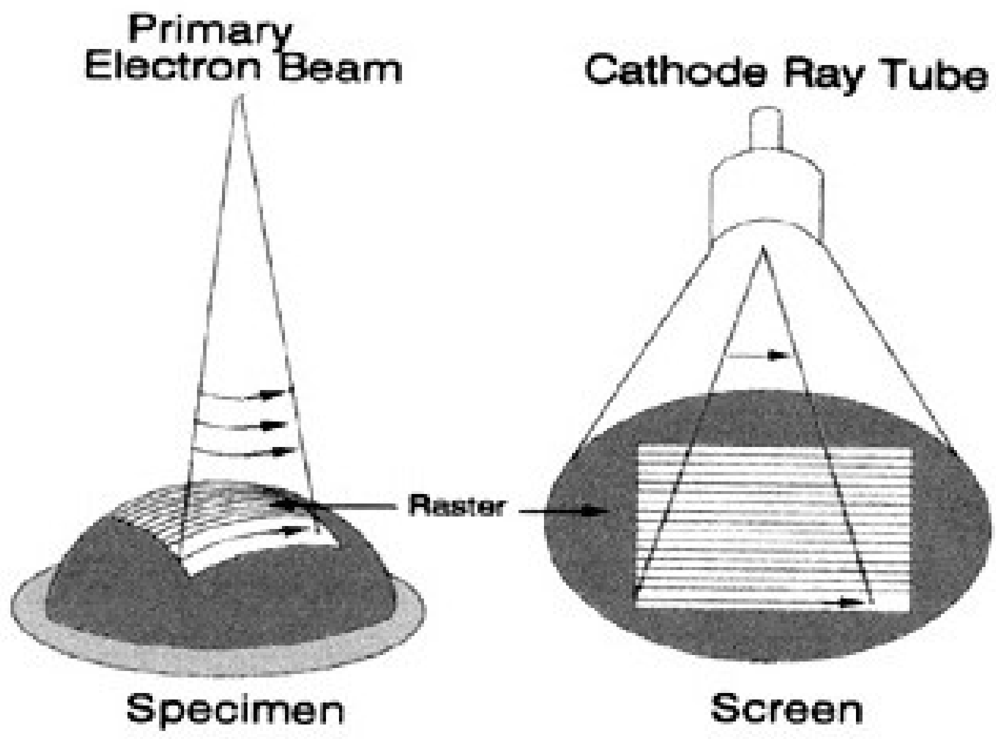
Risoluzione: 1.5 nm a 20 kV

Ingrandimenti fino a 500000

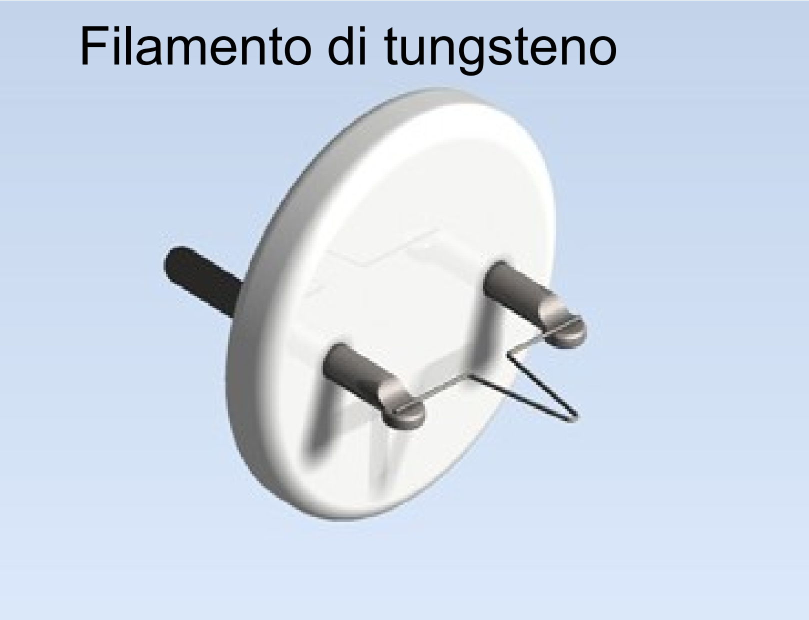
Il fascio di elettroni viene generato da un filamento riscaldato che emette elettroni (catodo), generalmente di tungsteno, per poi essere accelerati da un anodo.

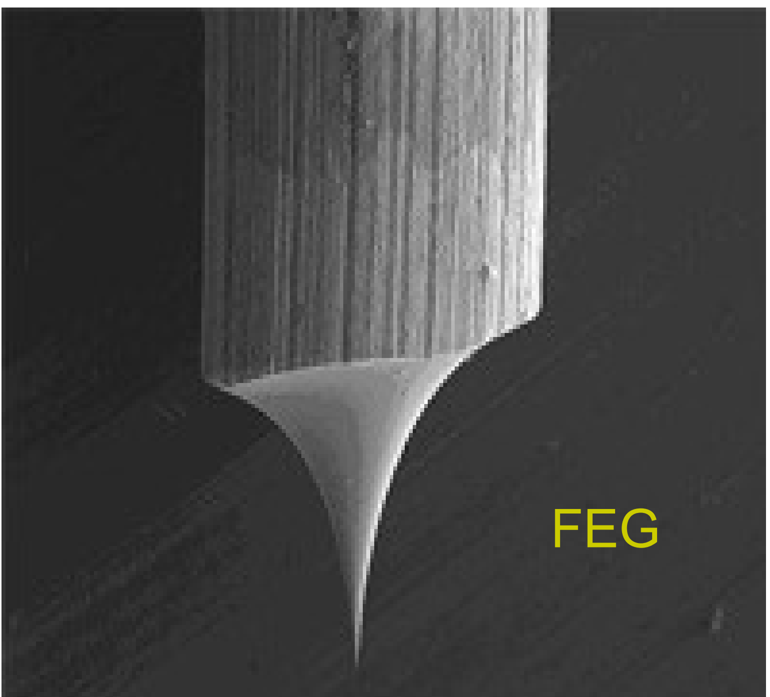


Formazione dell’immagine con il microscopio elettronico a scansione

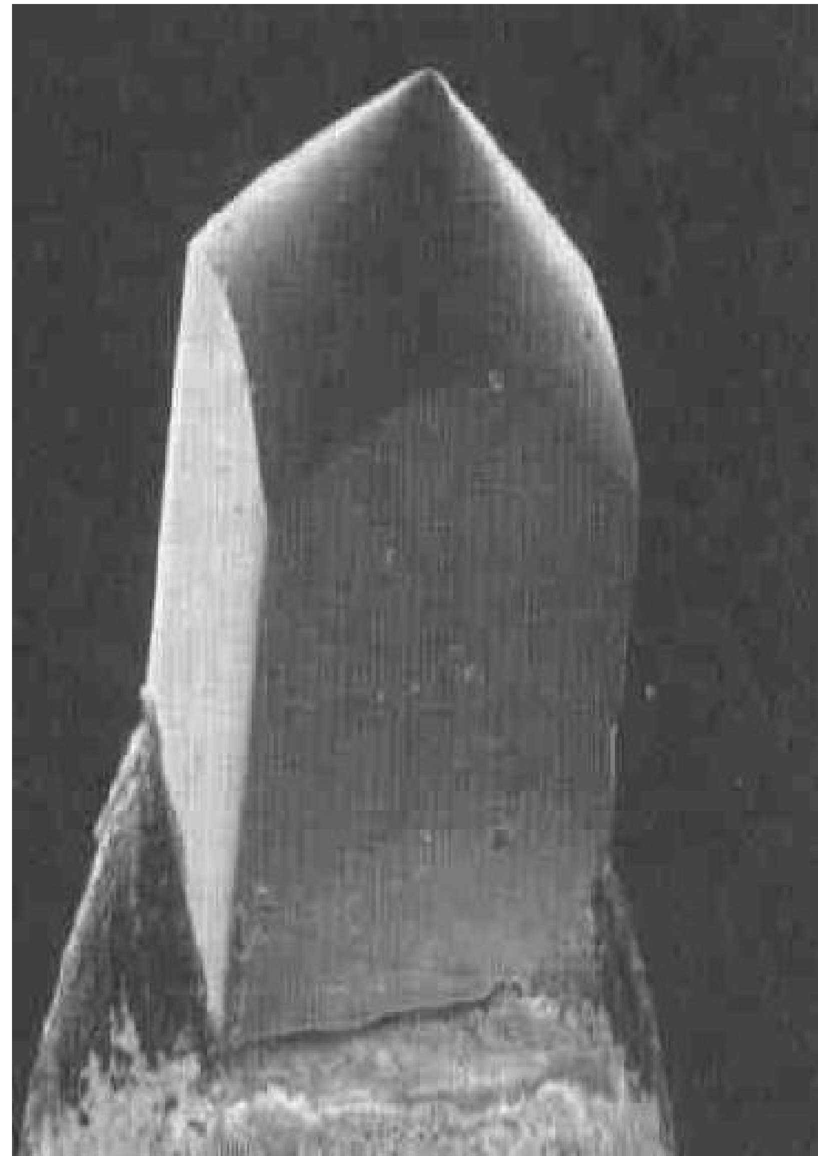


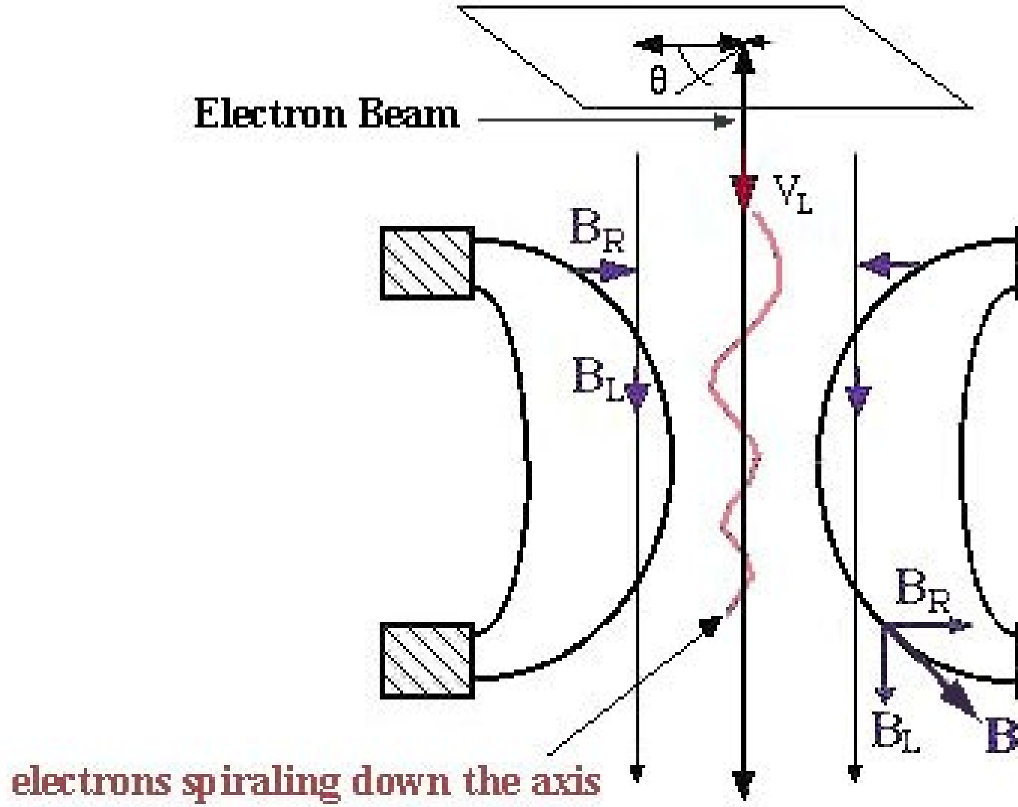
Un fascio focalizzato di elettroni viene spazzato su una superficie rettangolare detta “raster” , il fascio viene fatto passare da destra a sinistra per centinaia di volte.





Filamento di LaB_6





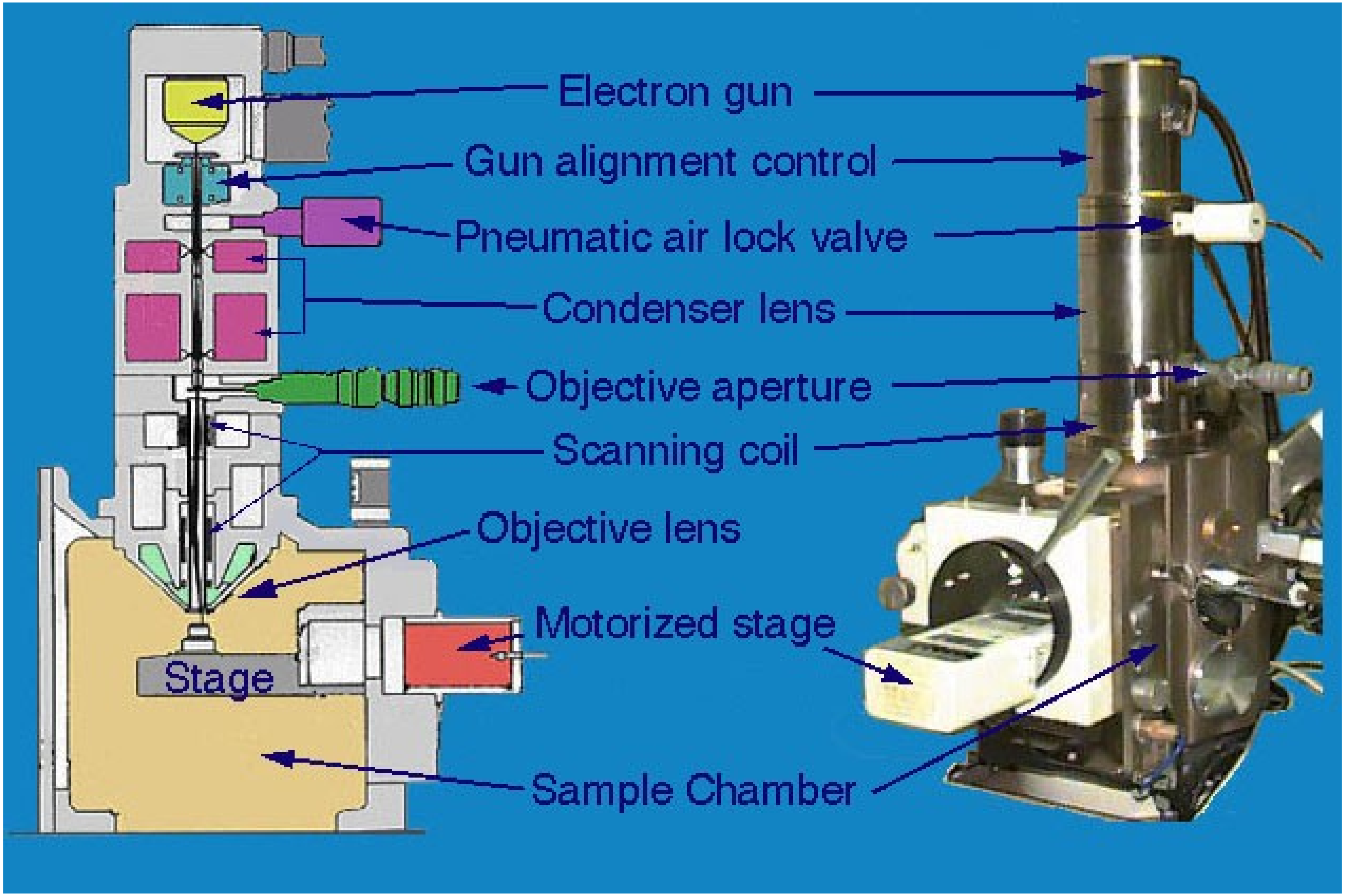
θ is a direction in the plane, V_L is ⊥ to the plane.

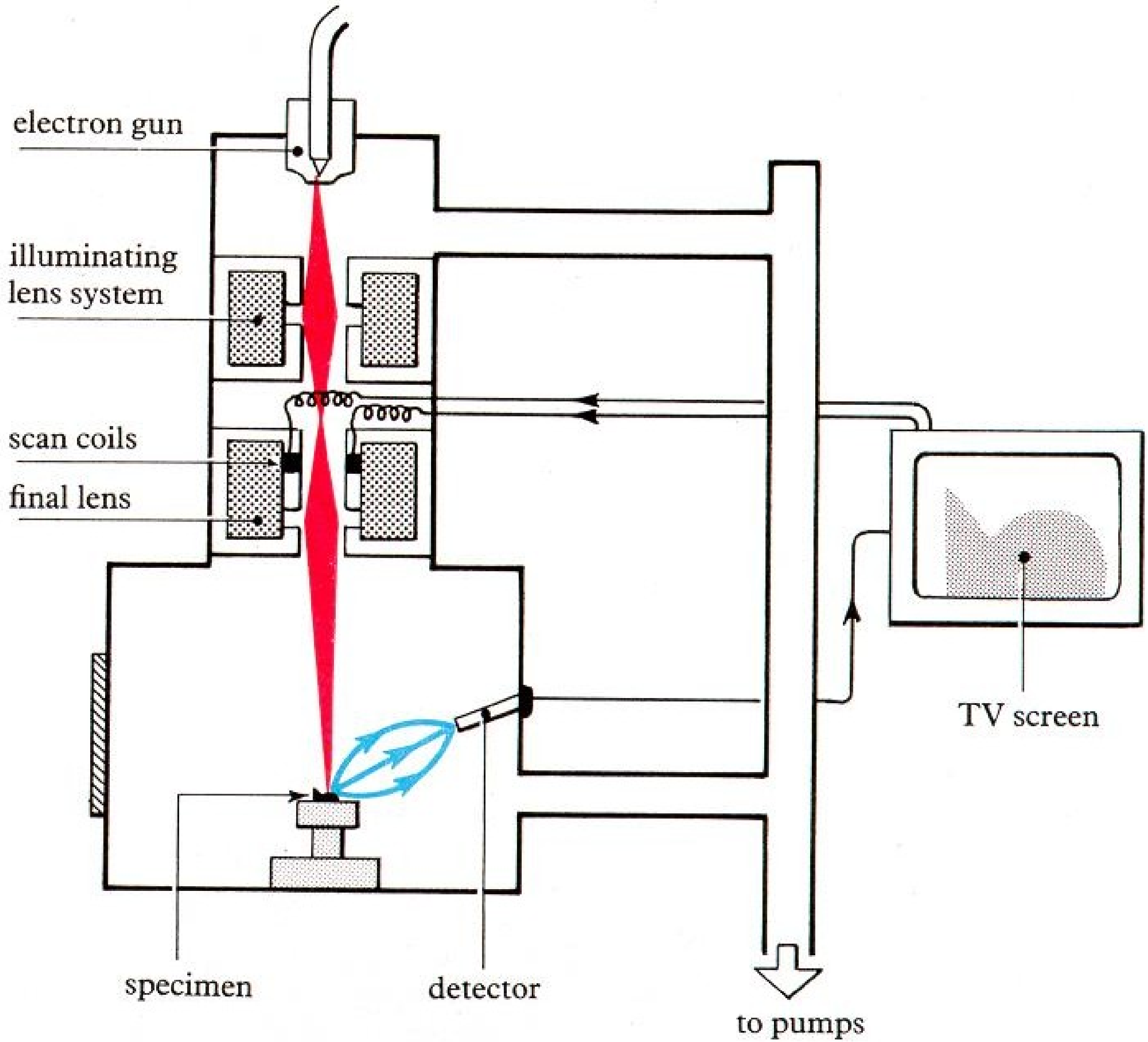
Nonaxial electrons will experience a force both down the axis and one radial to it. Only electrons traveling down the axis feel equal radial forces from all sides of the lens. The unequal force felt by the off-axis electrons causes spiralling about the optic axis.

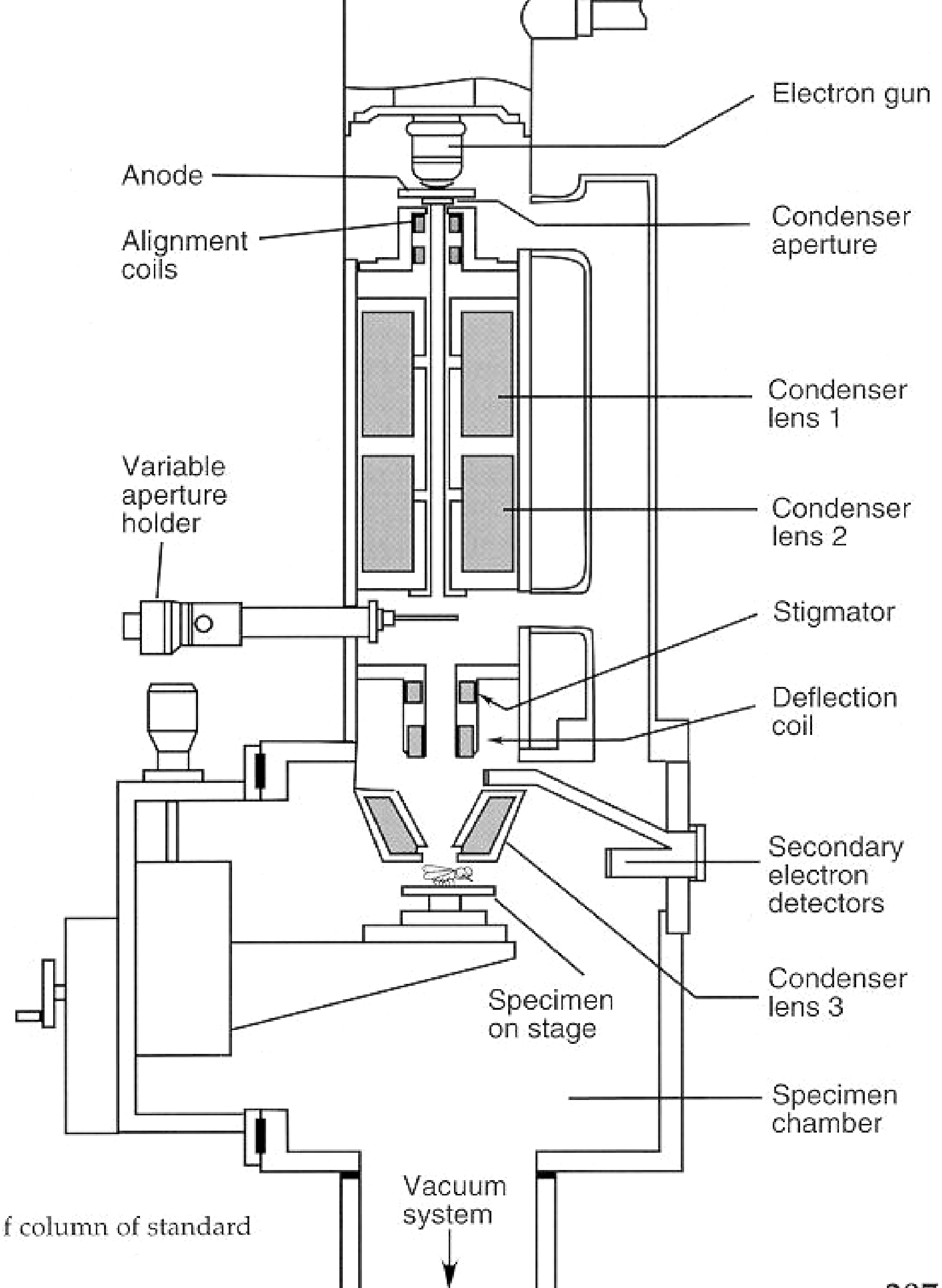
Two components to the B field:

B_L = longitudinal component (down the axis)

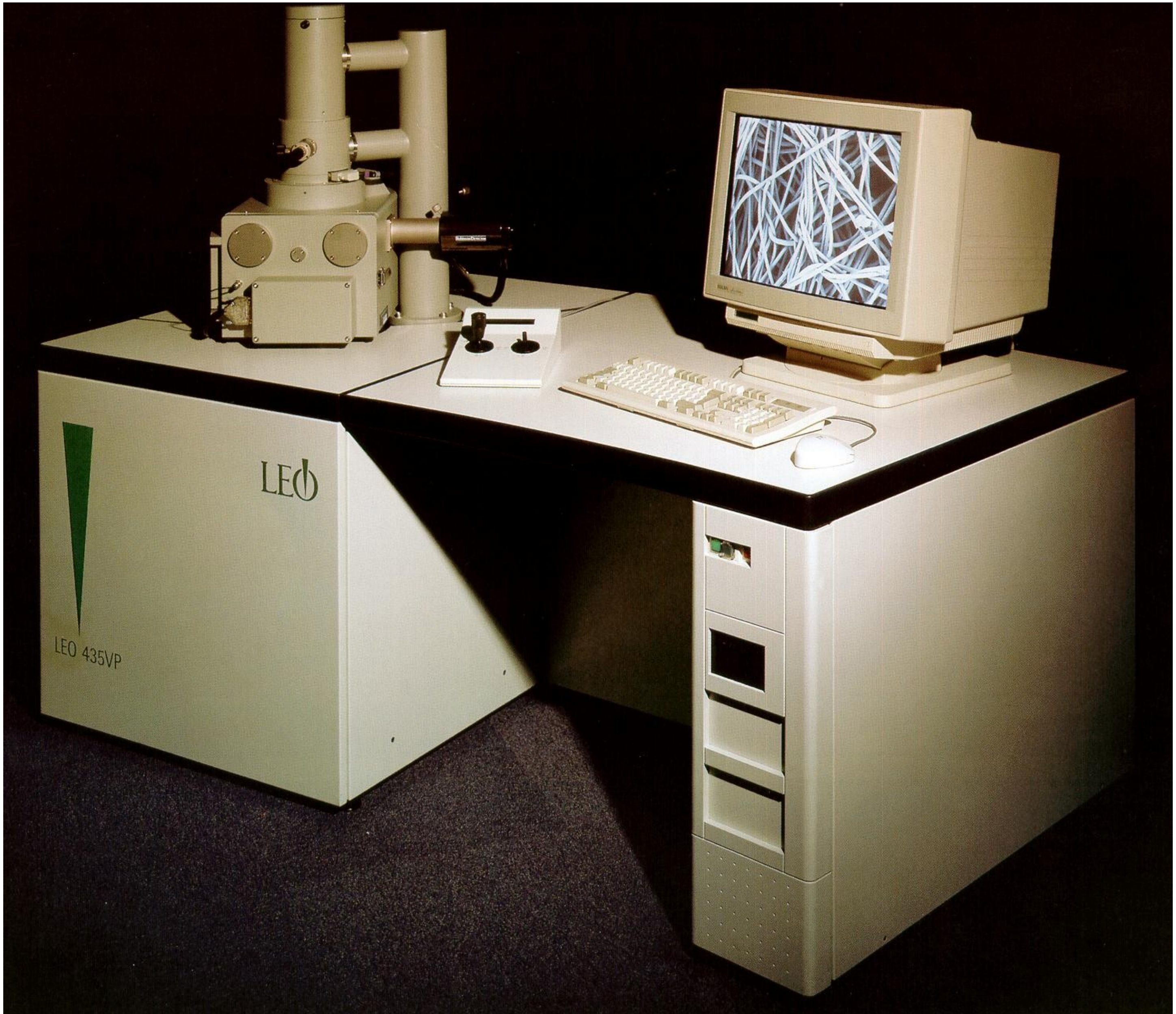
B_R = radial component (perpendicular to axis)


Electron gun
Gun alignment control
Pneumatic air lock valve
Condenser lens
Objective aperture
Scanning coil
Objective lens
Motorized stage
Stage
Sample Chamber


electron gun
illuminating lens system
scan coils
final lens
specimen
detector
to pumps
TV screen



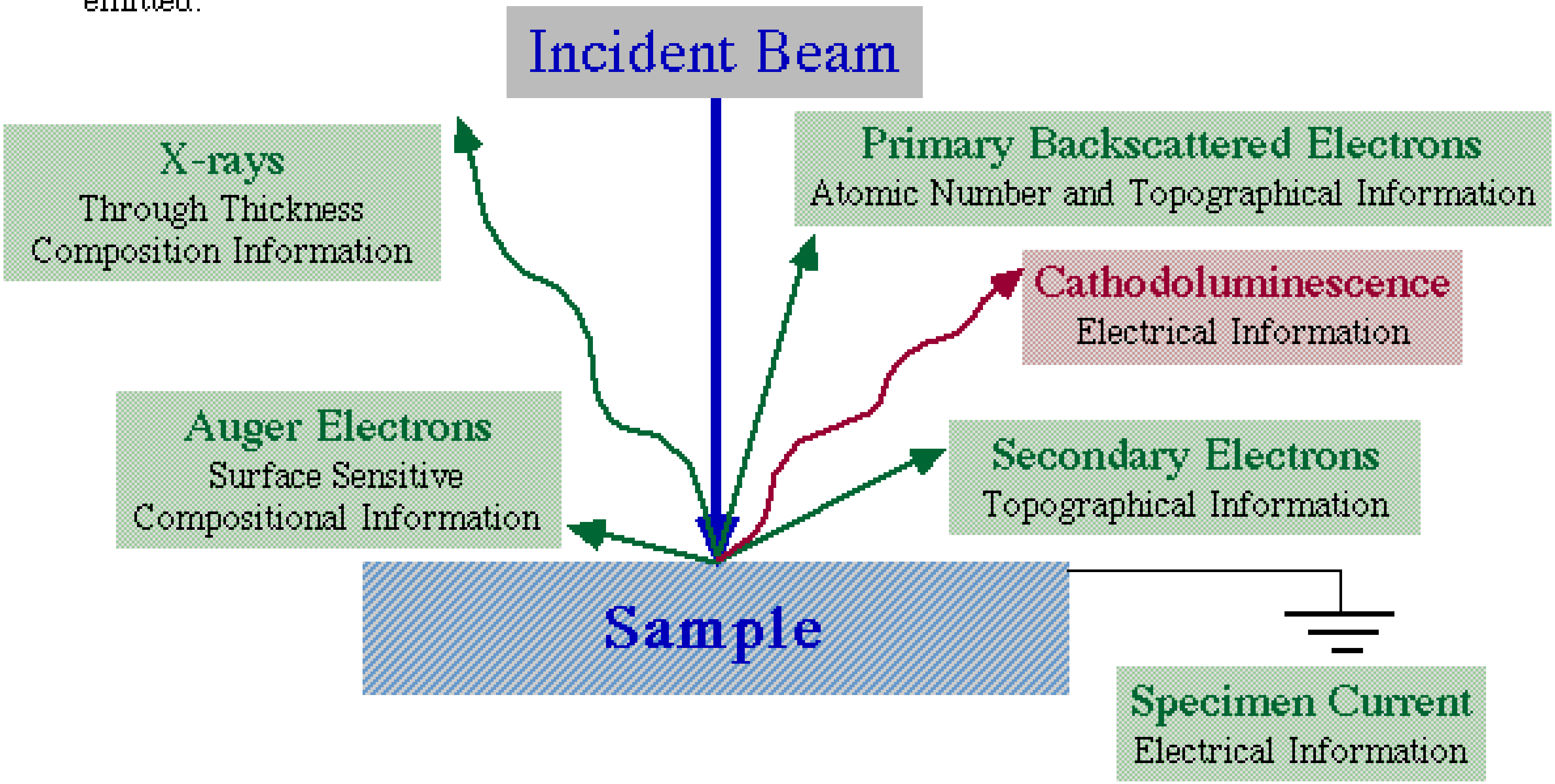
f column of standard


LEO
LEO 435VP

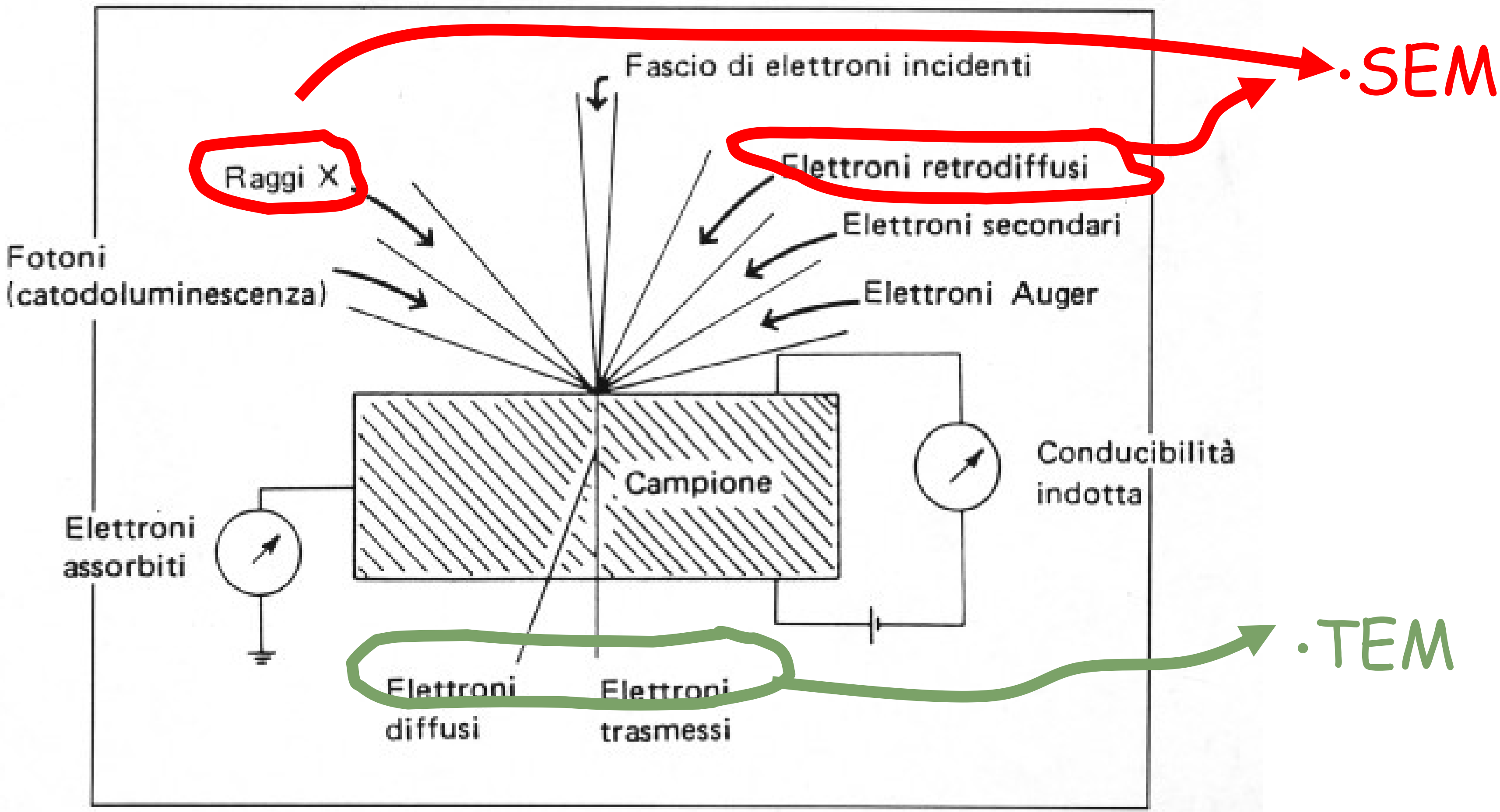
SEM Setup

Electron/Specimen Interactions

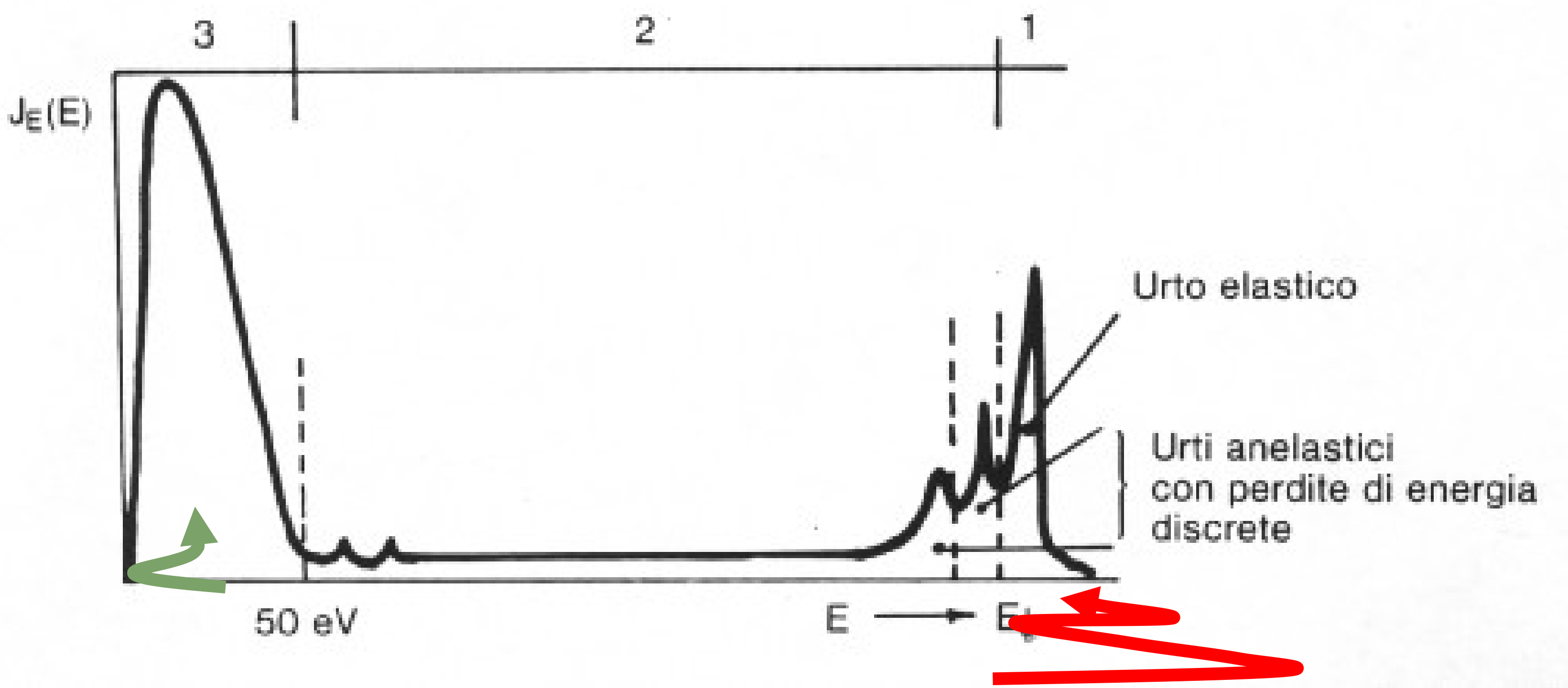
When the electron beam strikes the sample, both **photon** and **electron** signals are emitted.



fascio incidente di elettroni che interagisce con il campione



Segnali ottenuti

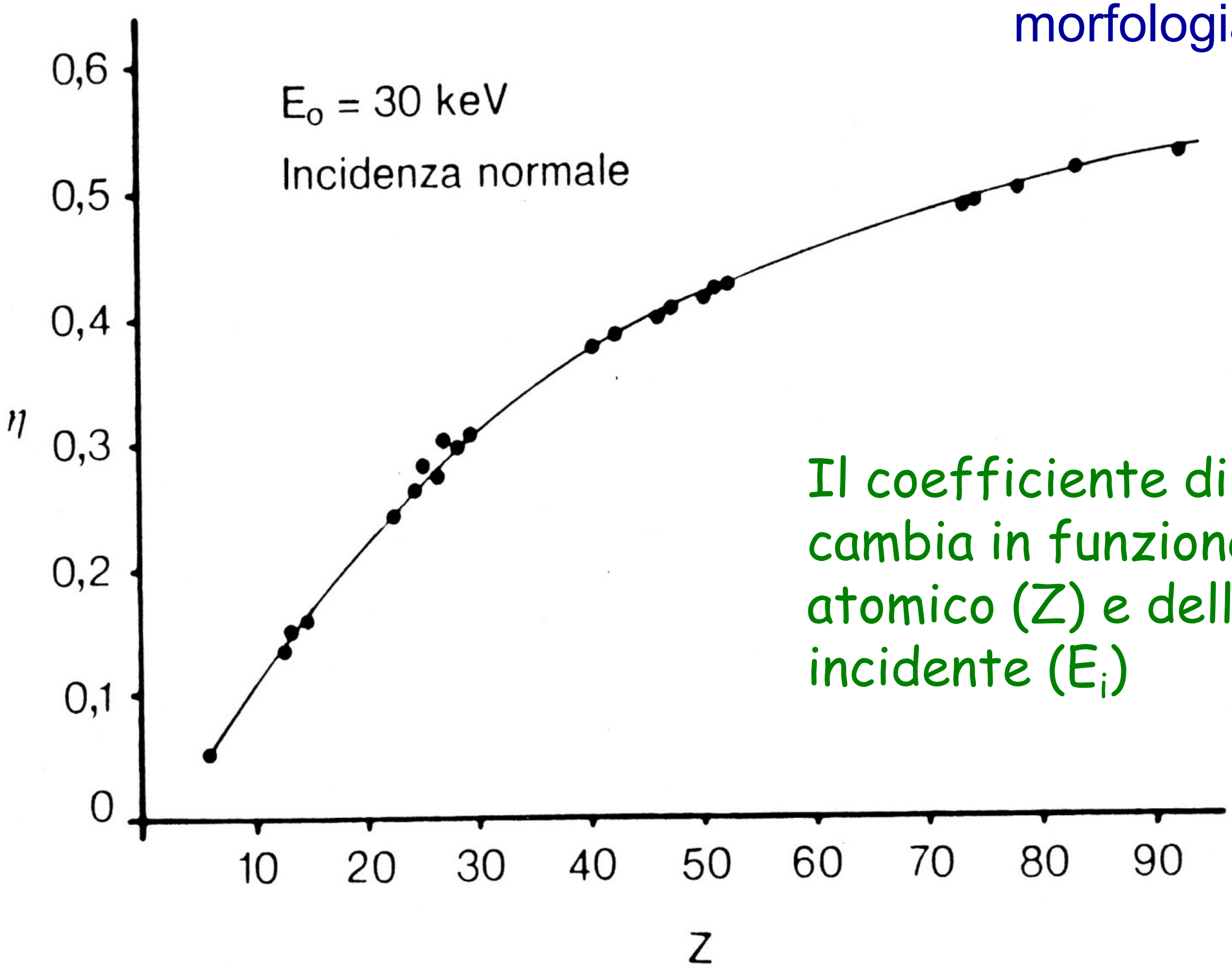


- Elettroni secondari: urti anelastici
- Elettroni retrodiffusi: urti elastici

Elettroni retrodiffusi

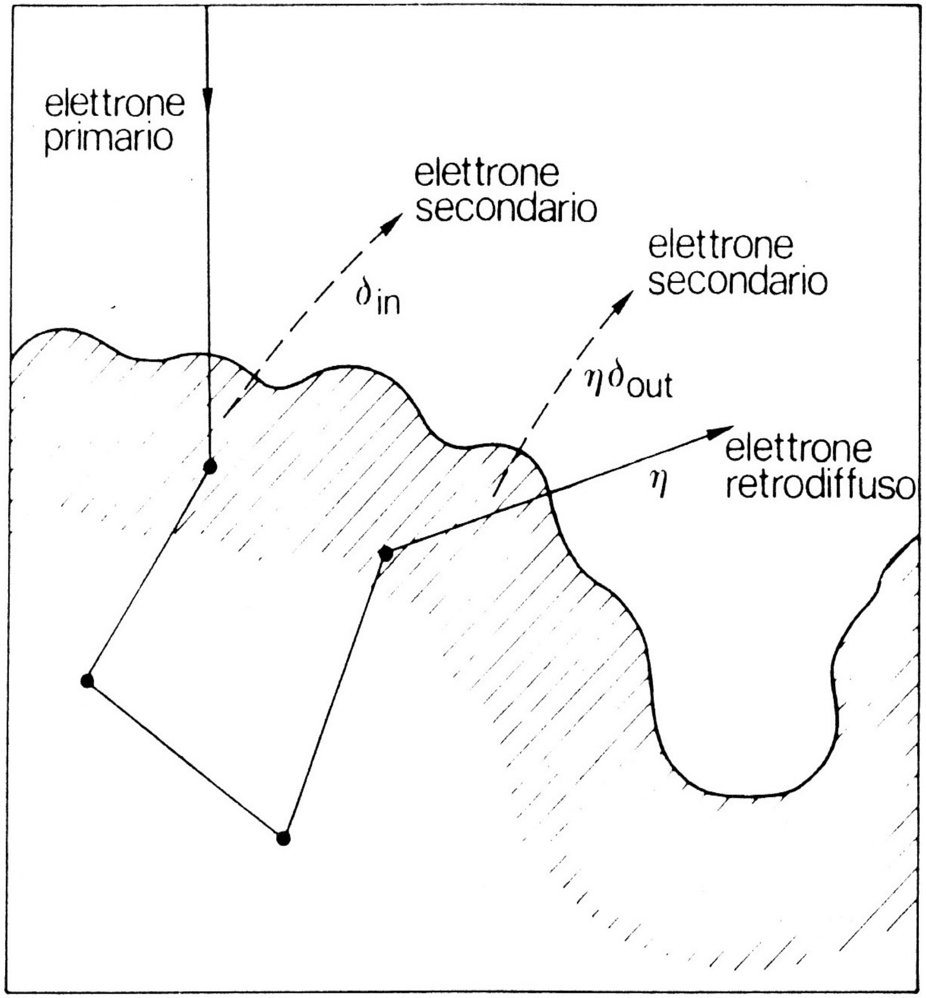
Subiscono urti
- •elastici
- •elastici ed anelastici

Gli elettroni retrodiffusi danno informazioni soprattutto sulla COMPOSIZIONE del campione e, in minor misura, sulla morfologia del campione



Il coefficiente di retrodiffusione cambia in funzione del numero atomico (Z) e dell'energia del fascio incidente (E_i)

Elettroni secondari



generati da elettroni primari e retrodiffusi

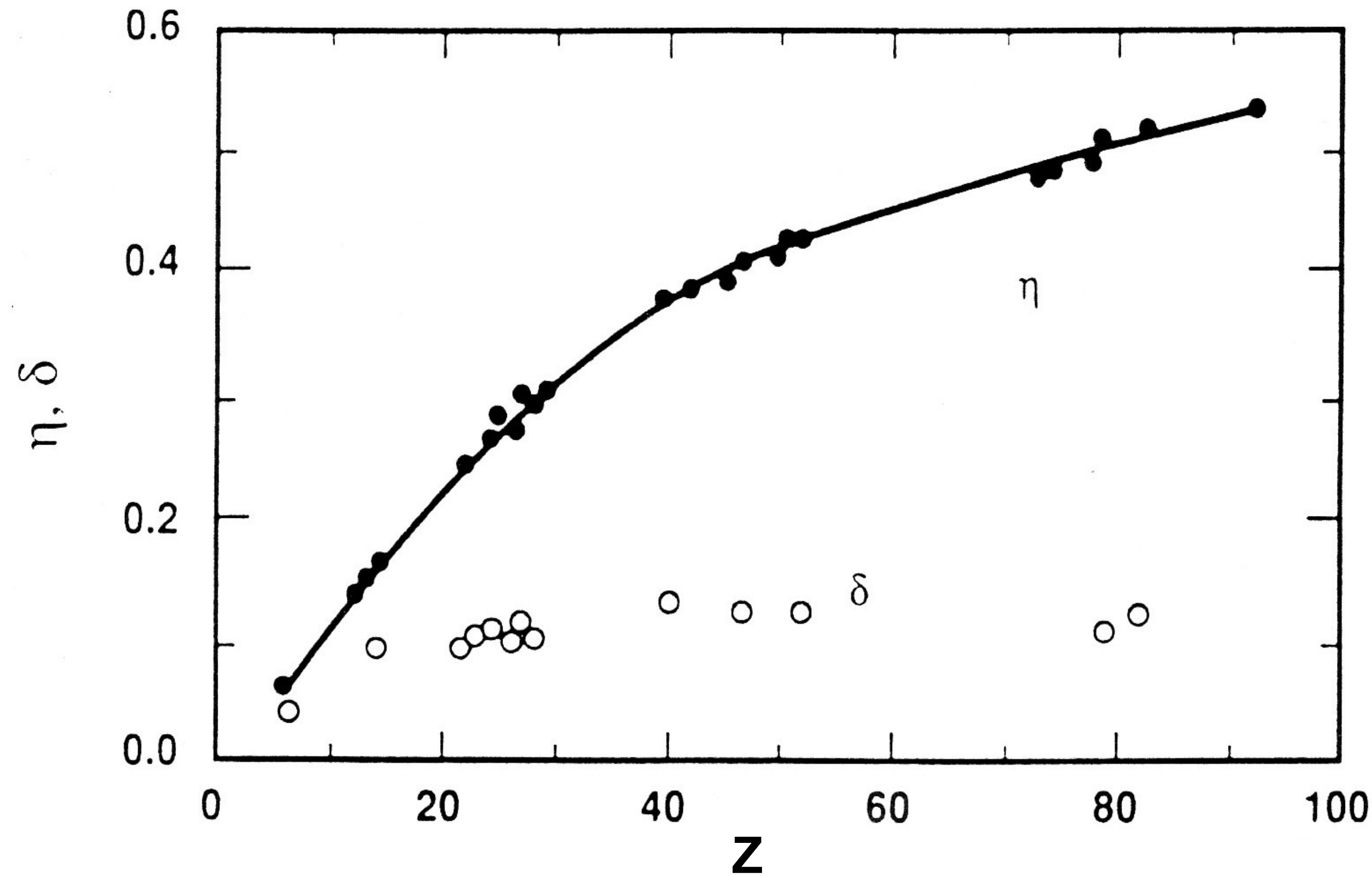
prodotti in seguito ad urti anelastici

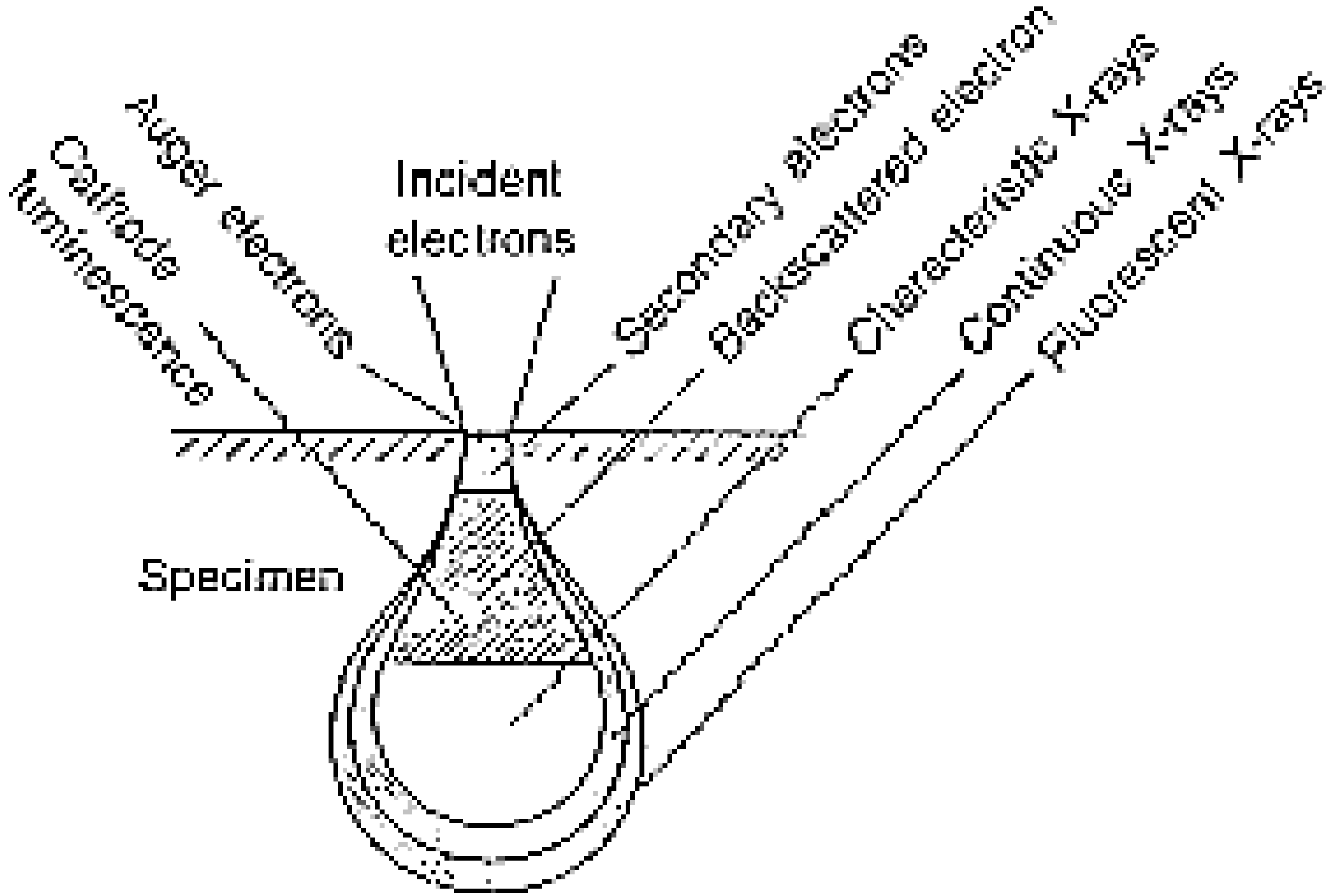
emergono da profondità inferiori a 10 nm

Elettroni secondari

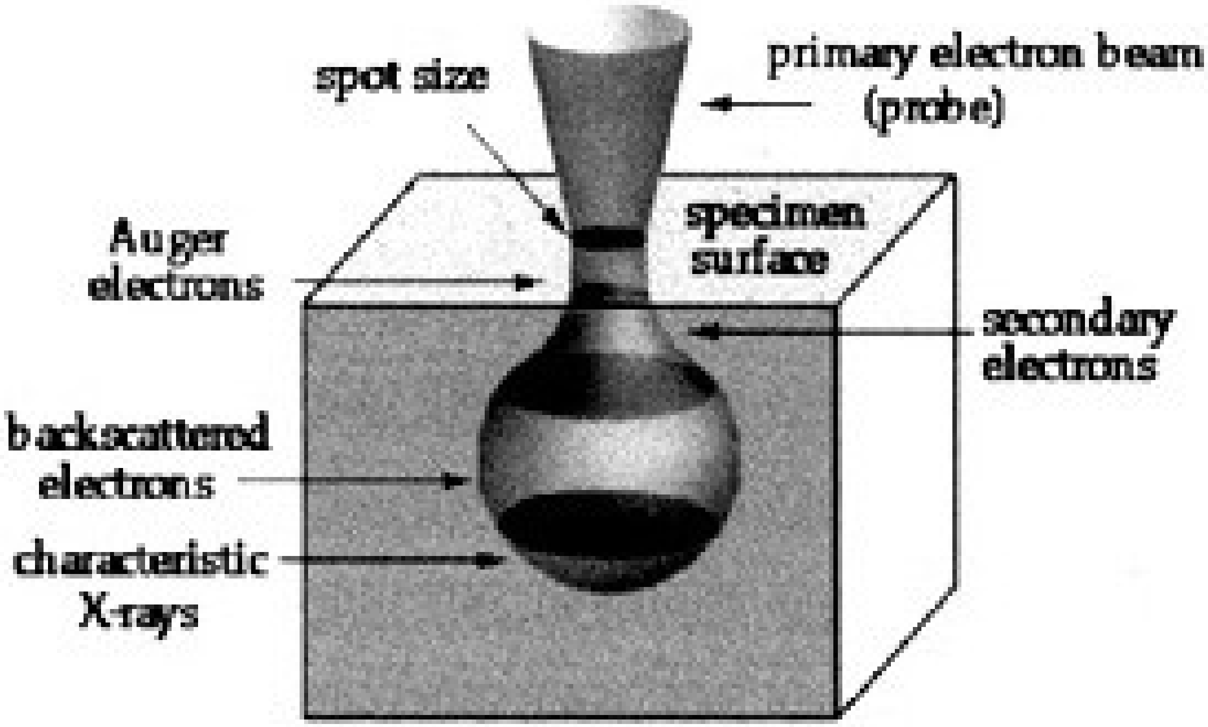
Informazioni su morfologia del campione e, in minor misura, sulla composizione del campione

Confronto fra il coefficiente di retrodiffusione (η) e il coefficiente di emissione degli elettroni secondari (δ)



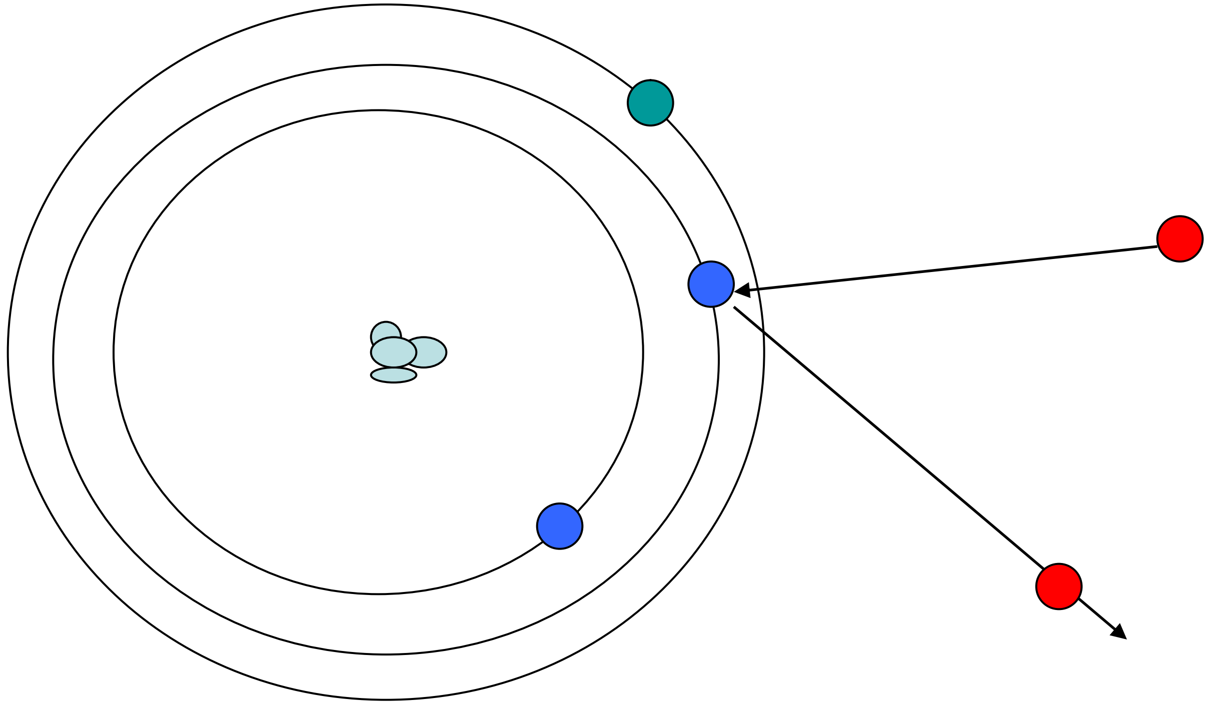


Incident electrons
Auger electrons
Cathode luminescence
Secondary electrons
Backscattered electron
Characteristic X-rays
Continuous X-rays
Fluorescent X-rays
Specimen


primary electron beam
(probe)
spot size
specimen
surface
Auger
electrons
secondary
electrons
backscattered
electrons
characteristic
X-rays

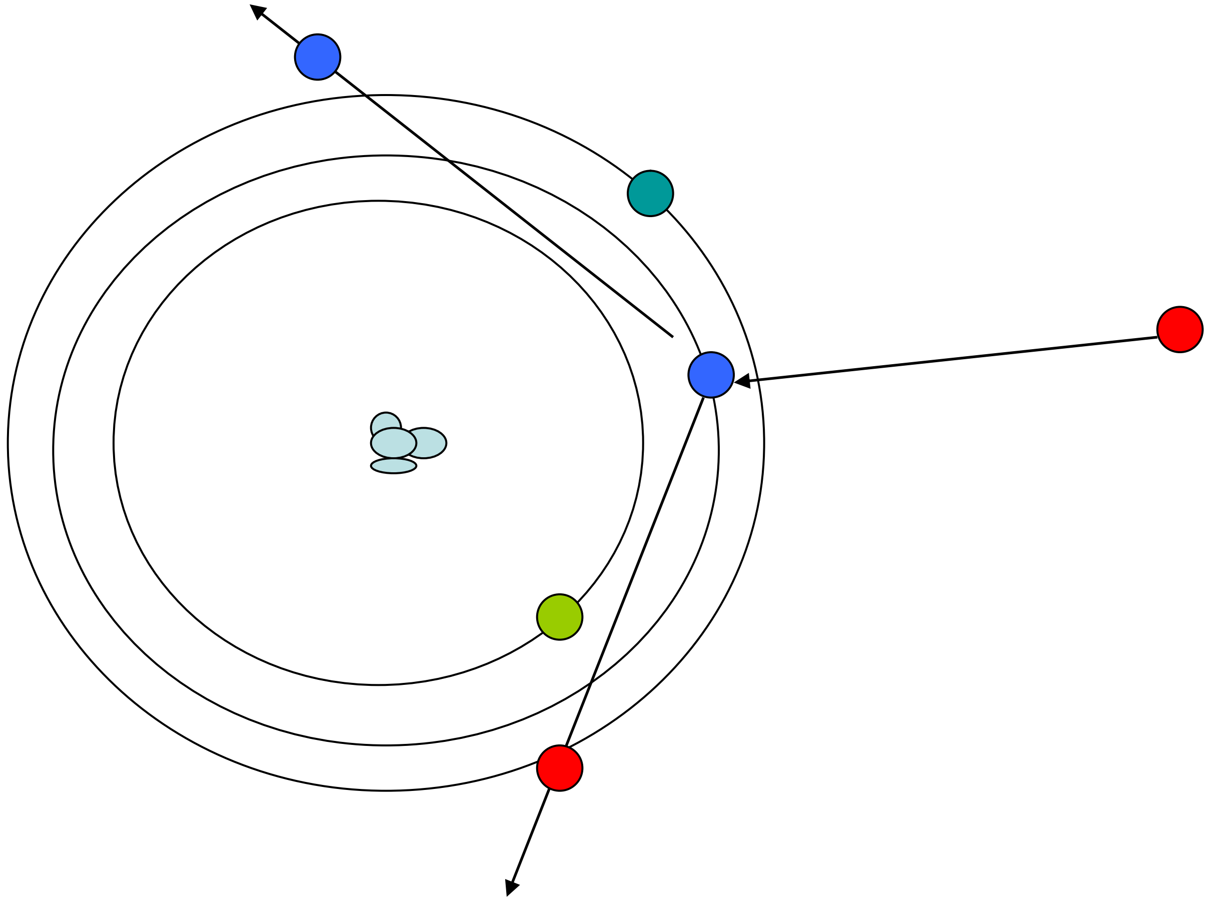
Elettroni scatterati primari

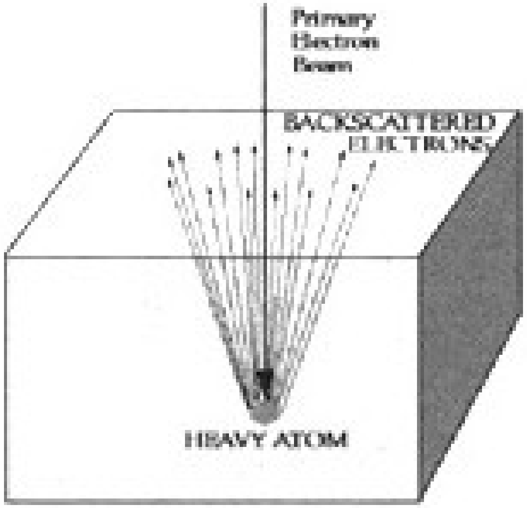
Sono gli elettroni che vengono scatterati direttamente dai livelli interni degli atomi del campione, più l'atomo è grande (numero atomico) e più atomi vengono scatterati.

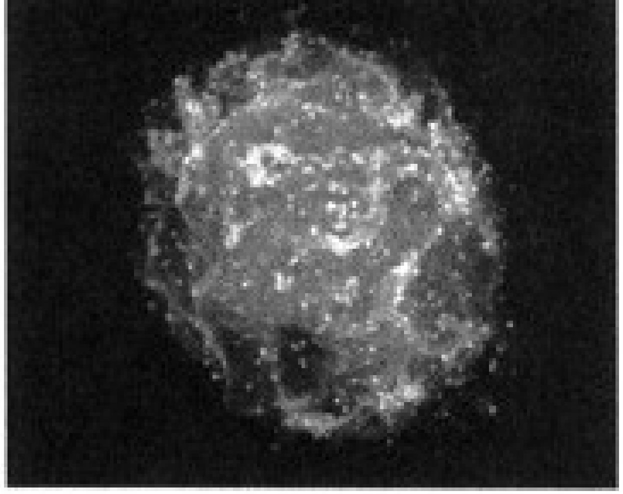


Elettroni scatterati secondari

Sono gli elettroni che vengono prodotti dalla ionizzazione degli atomi del campione data dall'interazione con gli elettroni incidenti. Avendo basse energie sono relativi solo agli atomi di superficie.



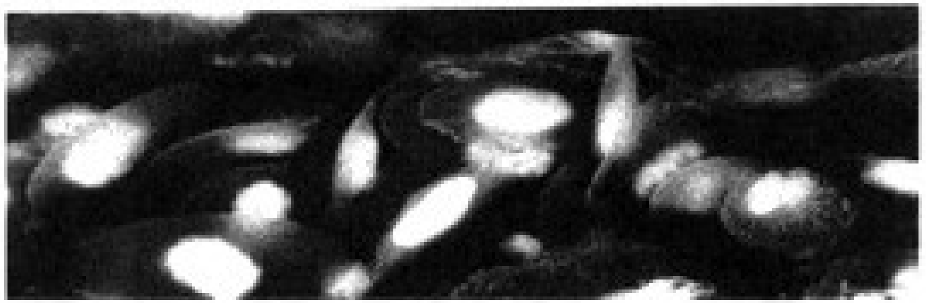




T-leukocyte labeled with colloidal-gold labeled antibodies

Red blood cells with silver-stained nuclei

Secondary Electron Mode



Backscatter Mode

Raggi X

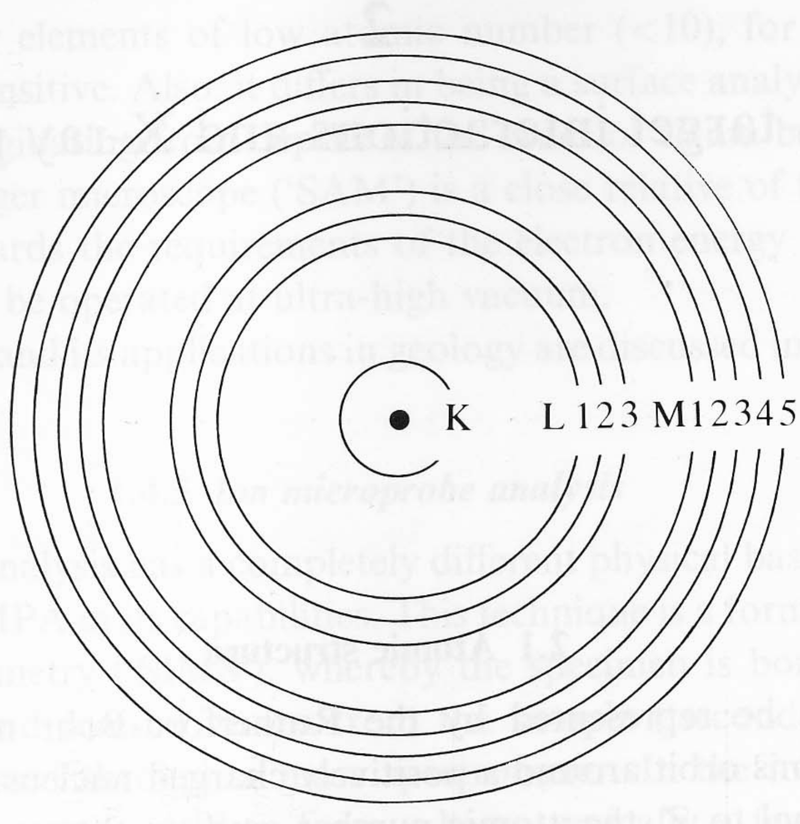


Fig. 2.1. Schematic diagram of inner atomic electron shells; characteristic X-rays are produced by transitions between these shells.

I raggi X, generati da ionizzazioni atomiche, esibiscono lunghezze d’onda (WDS) ed energie (EDS) caratteristiche dipendenti dalla composizione chimica degli elementi.

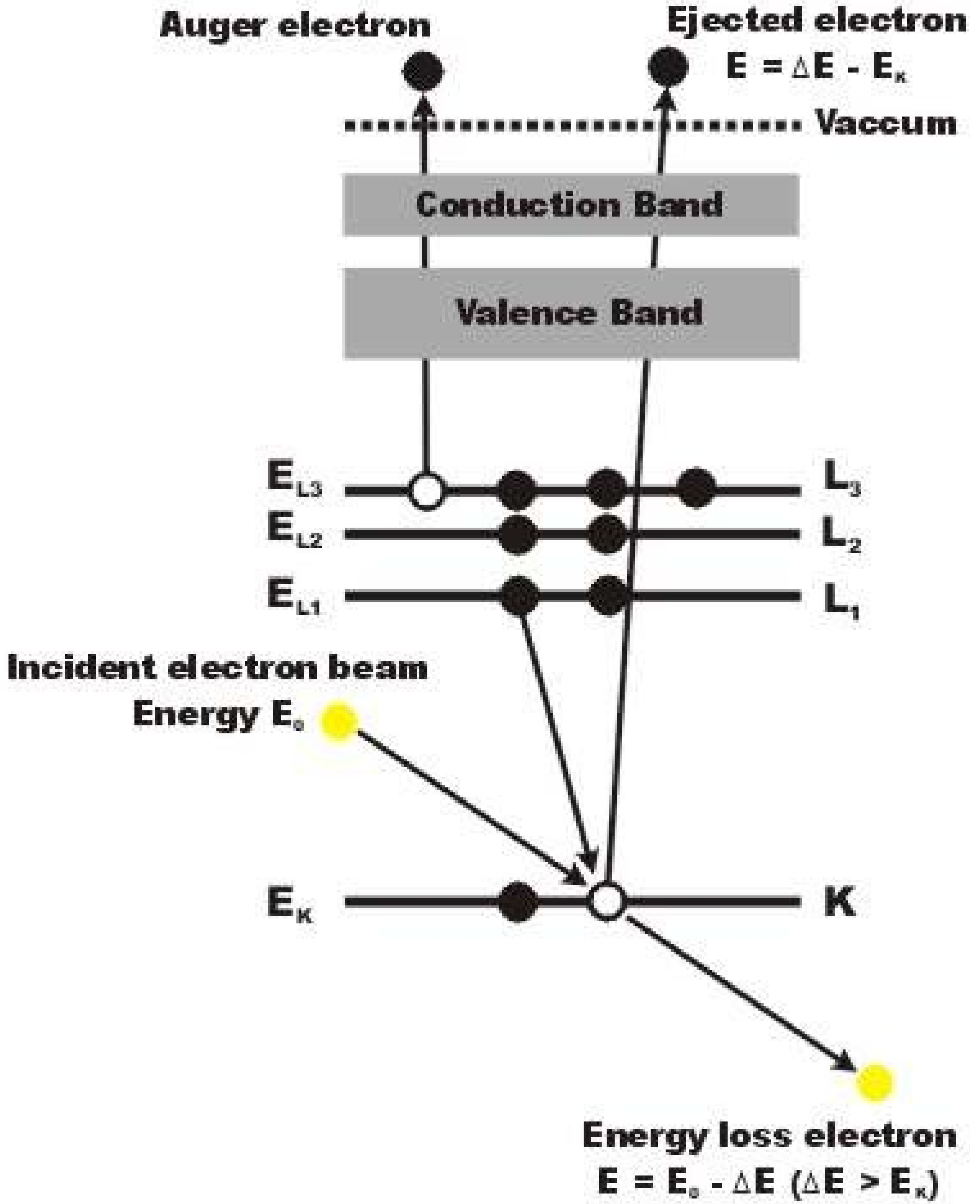
Elettroni Auger

1. **Ionizzazione:** Un elettrone incidente ad alta energia genera una lacuna in una livello energetico interno, perdendo energia che viene trasferita all'elettrone emesso.

2. **Emissione di elettroni Auger:** La lacuna sul livello K viene riempita da un elettrone su shell piu' esterne (per esempio L1). L'energia rilasciata e' trasferita ad un altro elettrone che ha quindi l'energia necessaria per uscire dall'atomo (elettrone Auger)

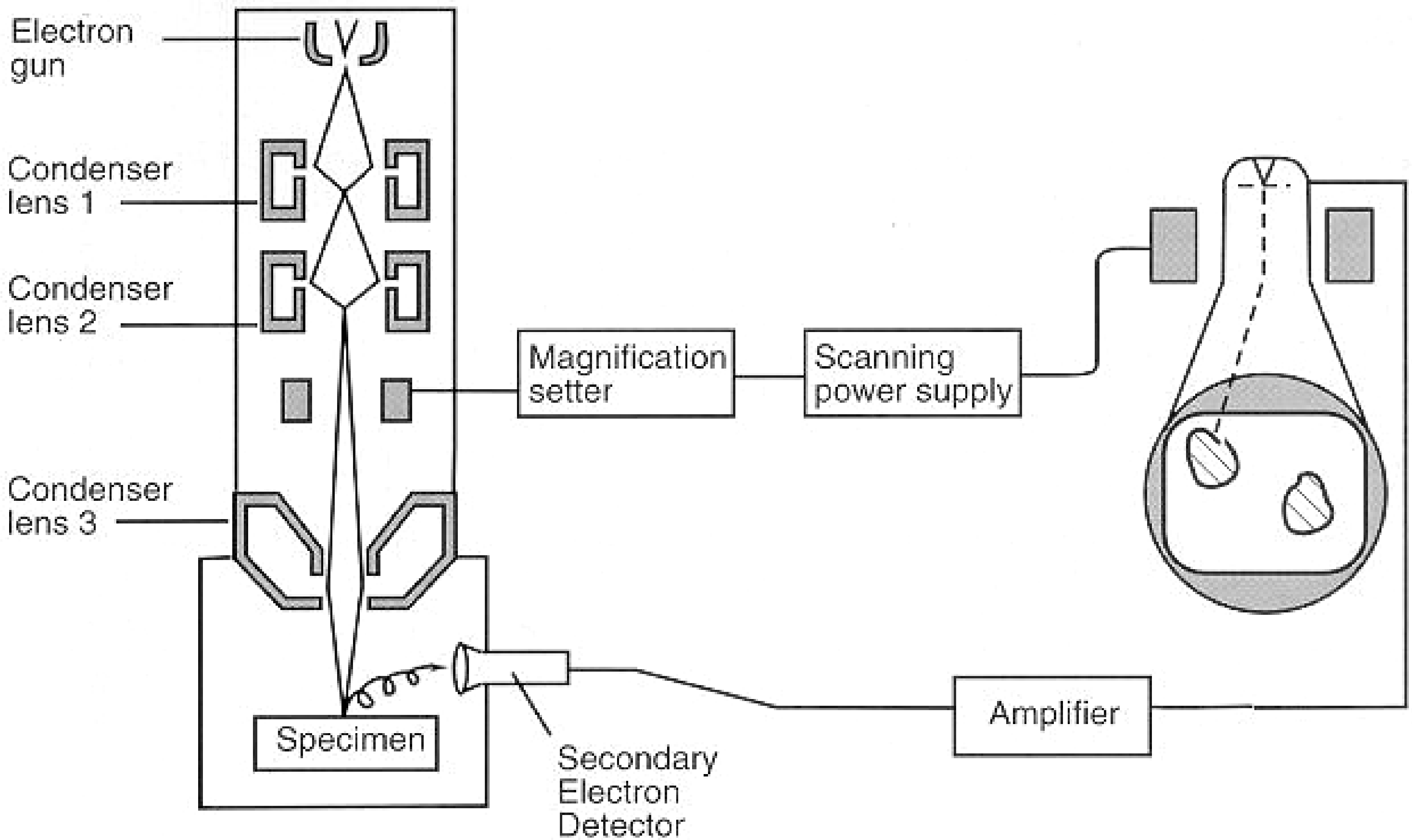
3. Poiché gli elettroni Auger hanno una energia da circa 100 eV fino a qualche KeV, vengono fortemente assorbiti dal campione. <u>Conseguentemente, solo gli elettroni Auger della superficie possono essere misurati.</u>

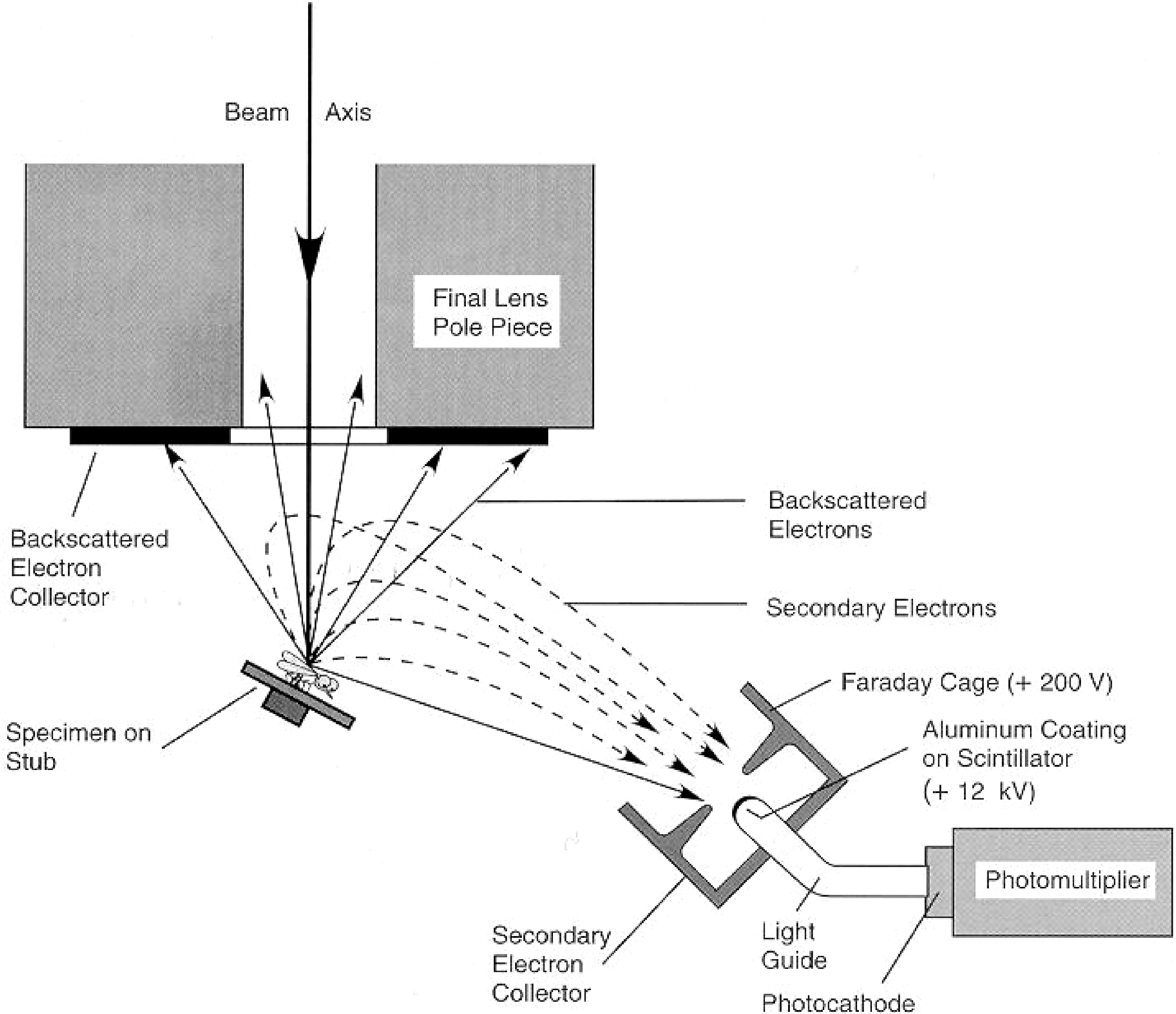
Elettroni Auger

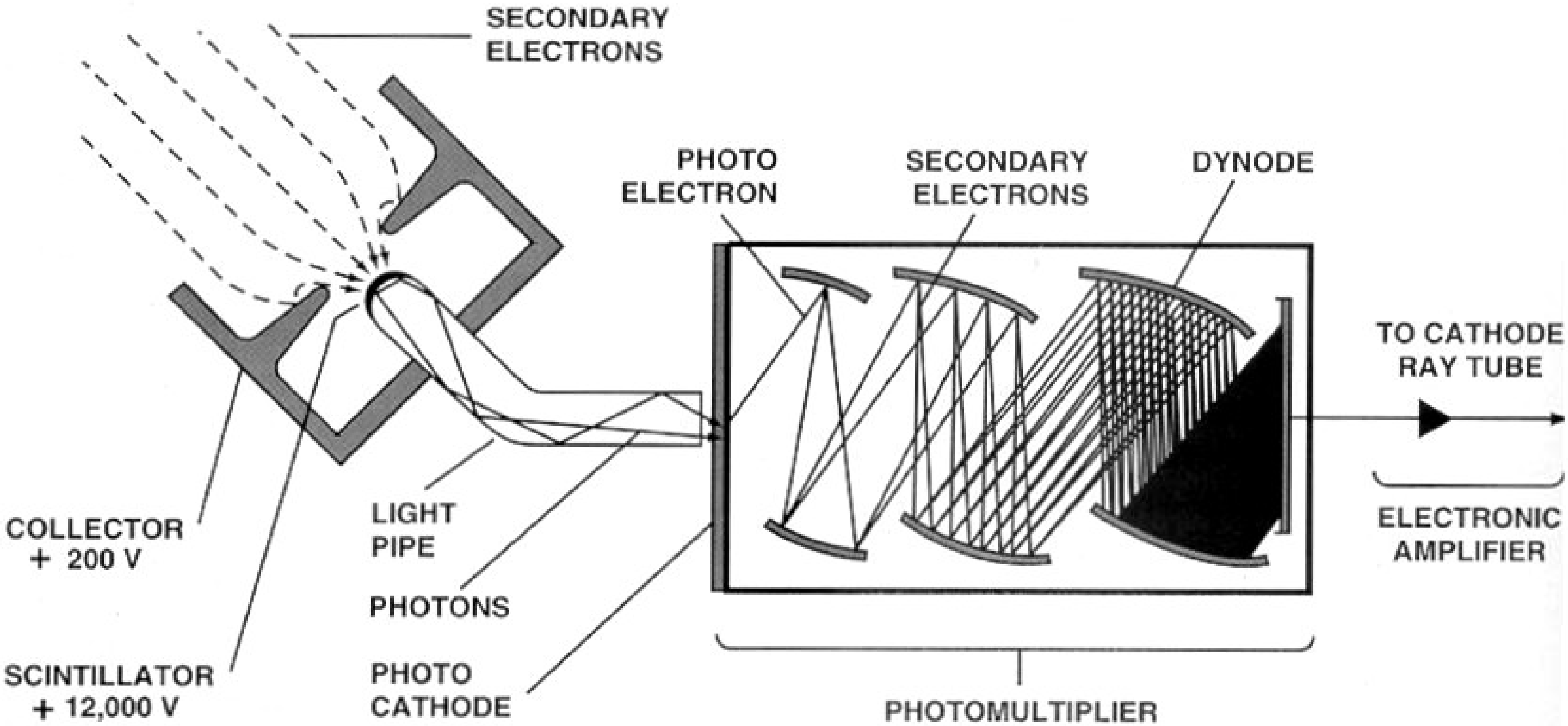


Funzionamento del SEM

- Fascio stretto di elettroni (~10 nm) passano sulla superficie del campione
- 3 lenti in fila controllano il diametro del fascio e la lunghezza del fuoco
- Le spire in fila muovono il fascio sincronizzato con il fascio sullo schermo
- Il campione opportunamente sistemato rende gli elettroni secondari conseguentemente alle collisioni dal fascio elettronico
- Gli elettroni secondari colpiscono il collettore e poi sono convertiti in fotoni dallo scintillatore
- I fotoni sono poi convertiti in corrente elettrica dal fotomoltiplicatore
- La corrente elettrica viene amplificata e utilizzata per pilotare il fascio nel tubo catodico dello schermo che riproduce l’immagine


Electron gun
Condenser lens 1
Condenser lens 2
Condenser lens 3
Magnification setter
Scanning power supply
Specimen
Secondary Electron Detector
Amplifier

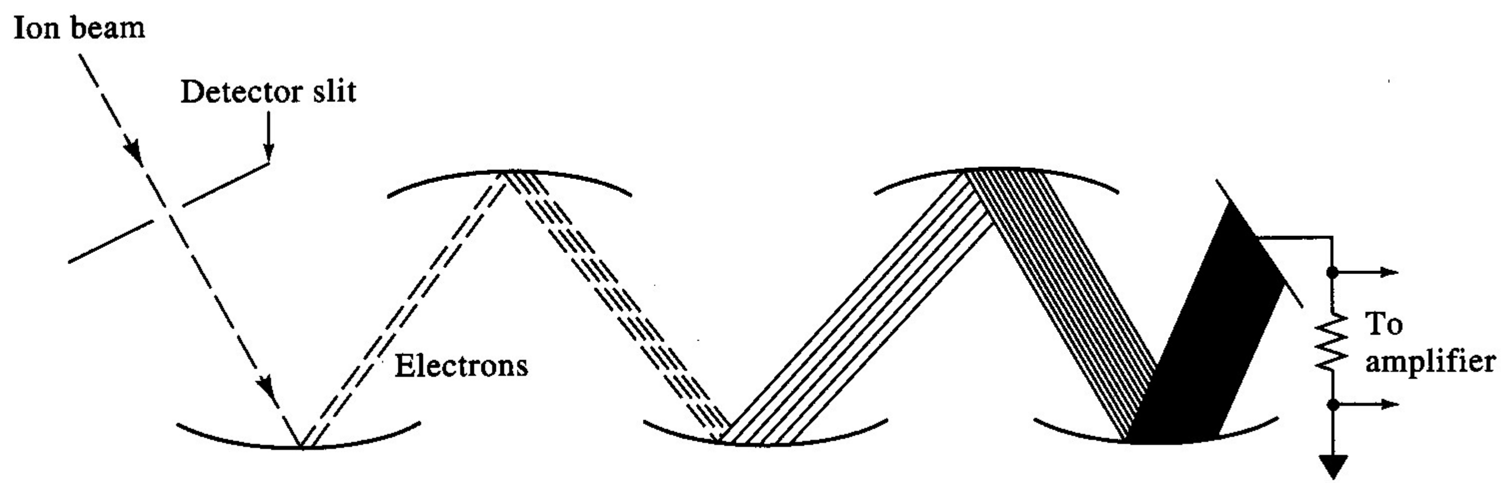

Beam
Axis
Final Lens
Pole Piece
Backscattered
Electron
Collector
Backscattered
Electrons
Secondary Electrons
Specimen on
Stub
Faraday Cage (+ 200 V)
Aluminum Coating
on Scintillator
(+ 12 kV)
Photomultiplier
Secondary
Electron
Collector
Light
Guide
Photocathode


SECONDARY
ELECTRONS
PHOTO
ELECTRON
SECONDARY
ELECTRONS
DYNODE
TO CATHODE
RAY TUBE
ELECTRONIC
AMPLIFIER
COLLECTOR
+ 200 V
LIGHT
PIPE
PHOTONS
SCINTILLATOR
+ 12,000 V
PHOTO
CATHODE
PHOTOMULTIPLIER

IV. Detectors

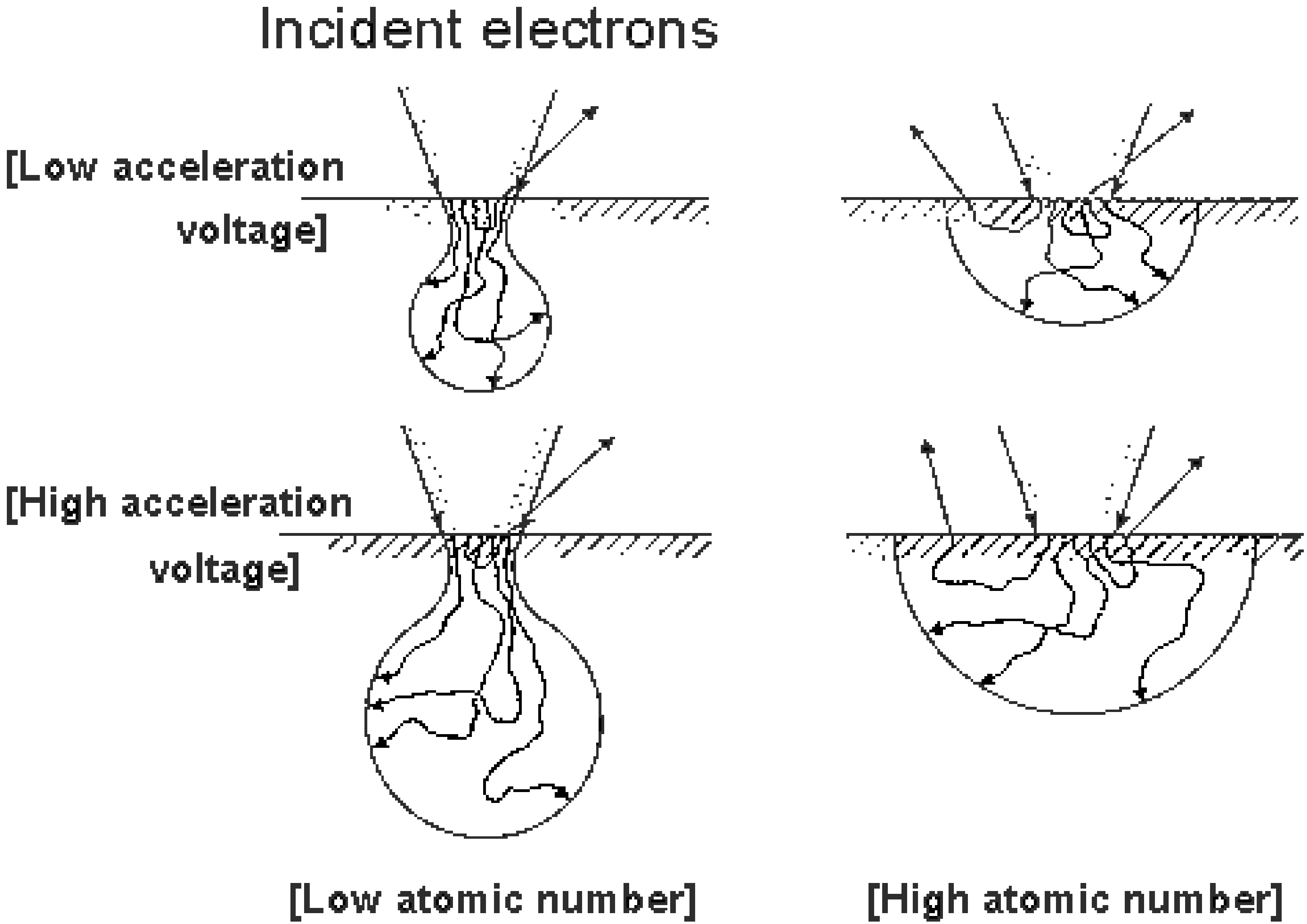
Electron Multipliers

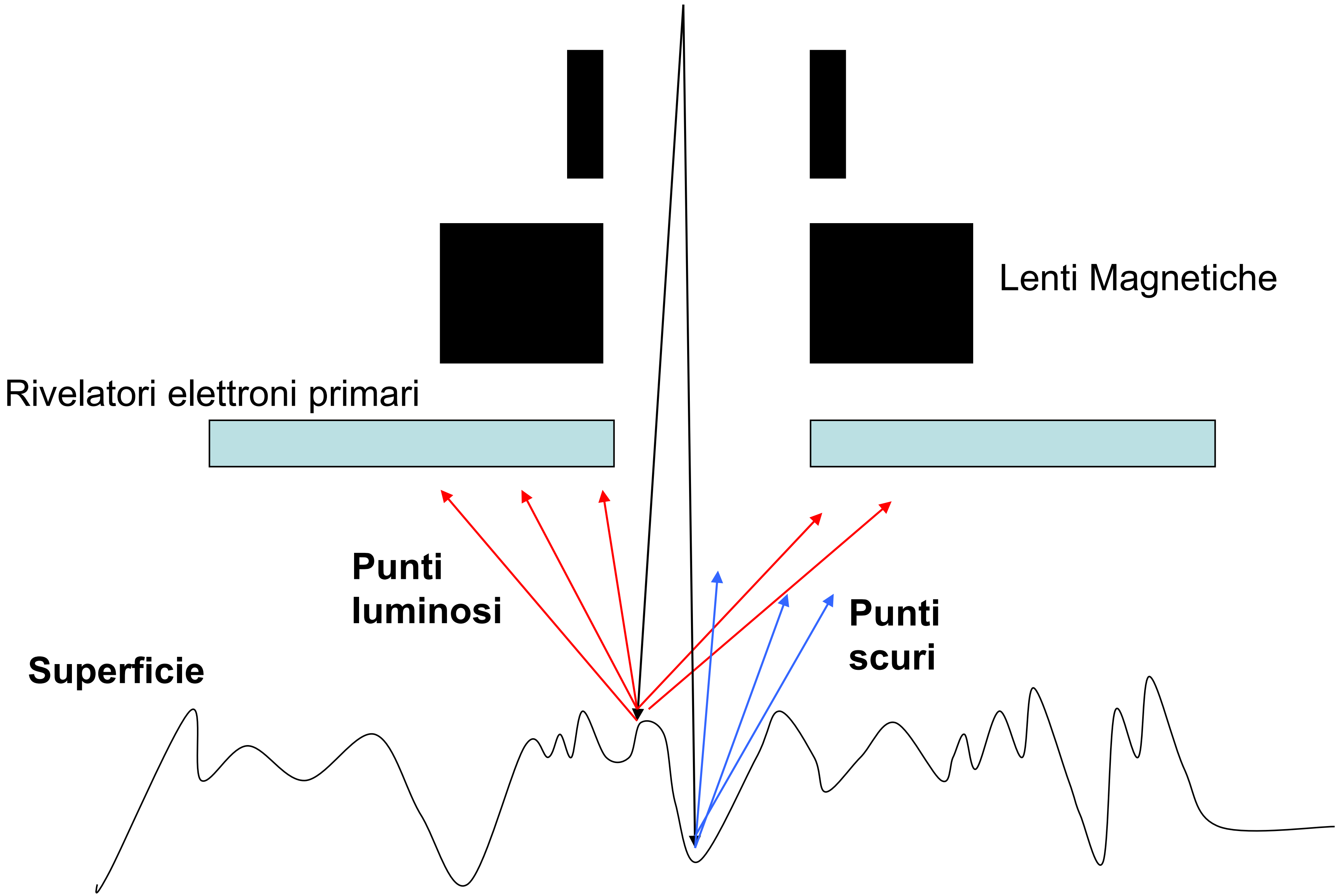
Discrete Dynode

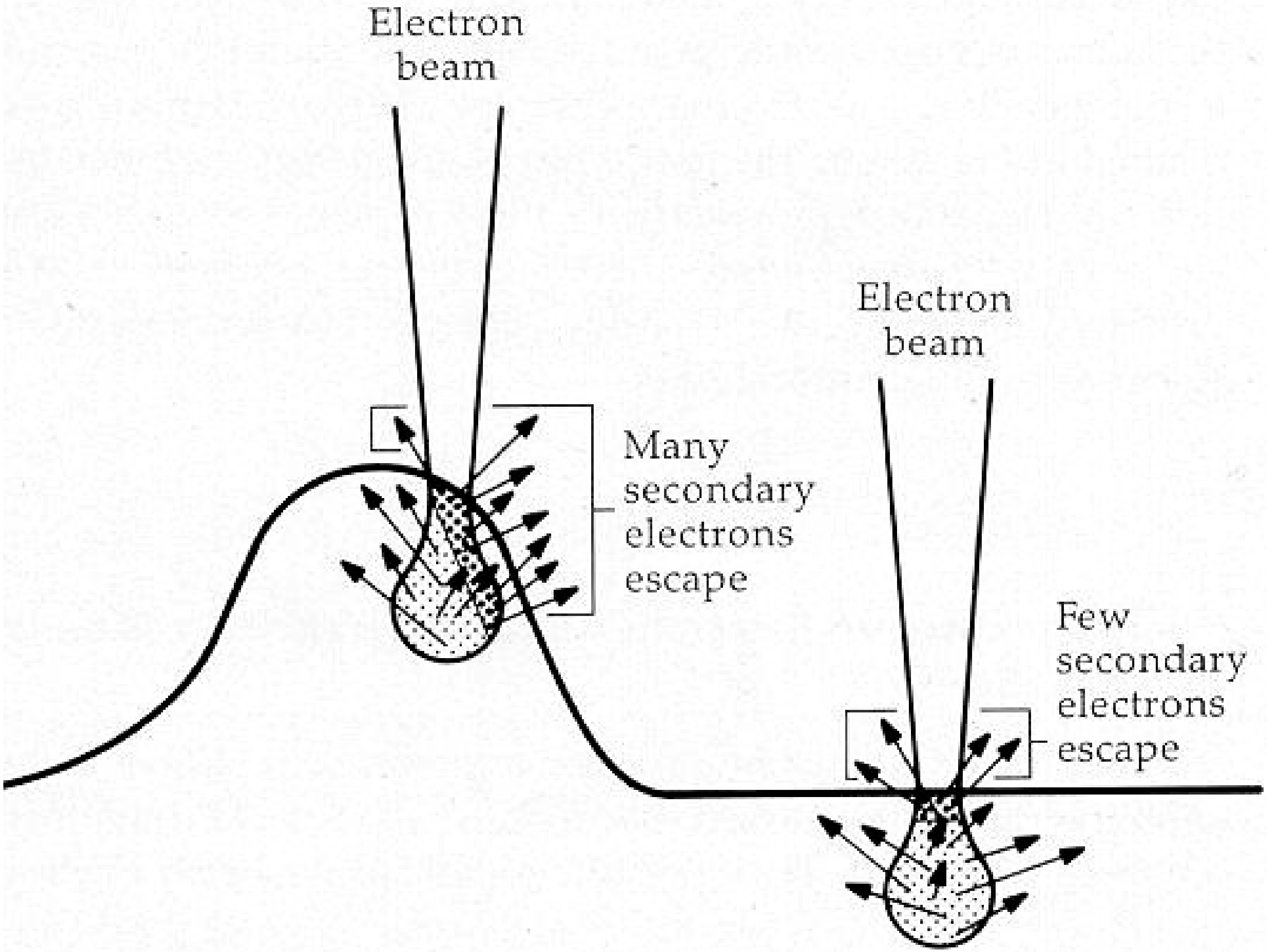


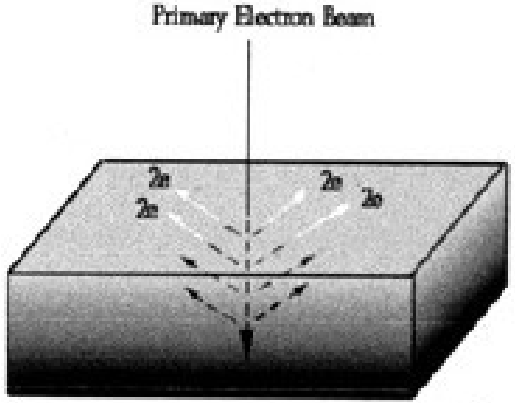
(a)

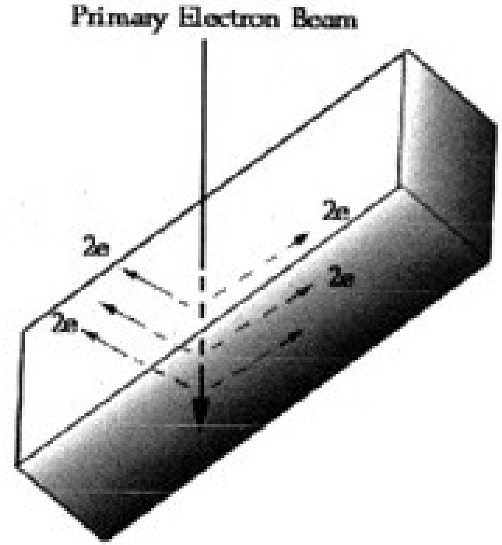
- Molecular ions cause the release of electrons from an ion sensitive dynode.
- Electrons cascade through a series of discrete dynodes to amplify the signal ~10^7 times.


Incident electrons
[Low acceleration voltage]
[High acceleration voltage]
[Low atomic number]
[High atomic number]


Lenti Magnetiche
Rivelatori elettroni primari
Punti luminosi
Punti scuri
Superficie


Electron beam
Many secondary electrons escape
Electron beam
Few secondary electrons escape





More secondary electrons will also reach the surface if it is rough rather than smooth. The raised parts will therefore appear bright in the image (relief contrast).

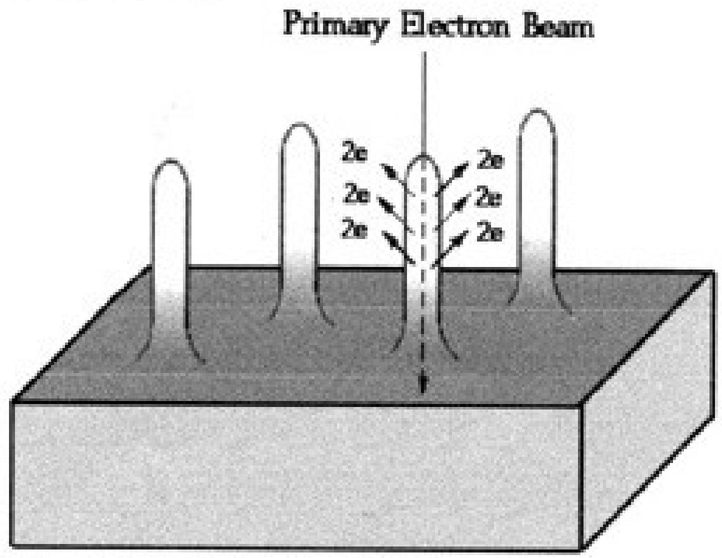


TABLE 7.2 Working Distance and Quality of Image

Working Distance (mm)	5	10	20	35
Resolution	Best	→	→	Worst
Depth of Field	Shallow	→	→	Deep
Signal Strength	Strong	→	→	Weak

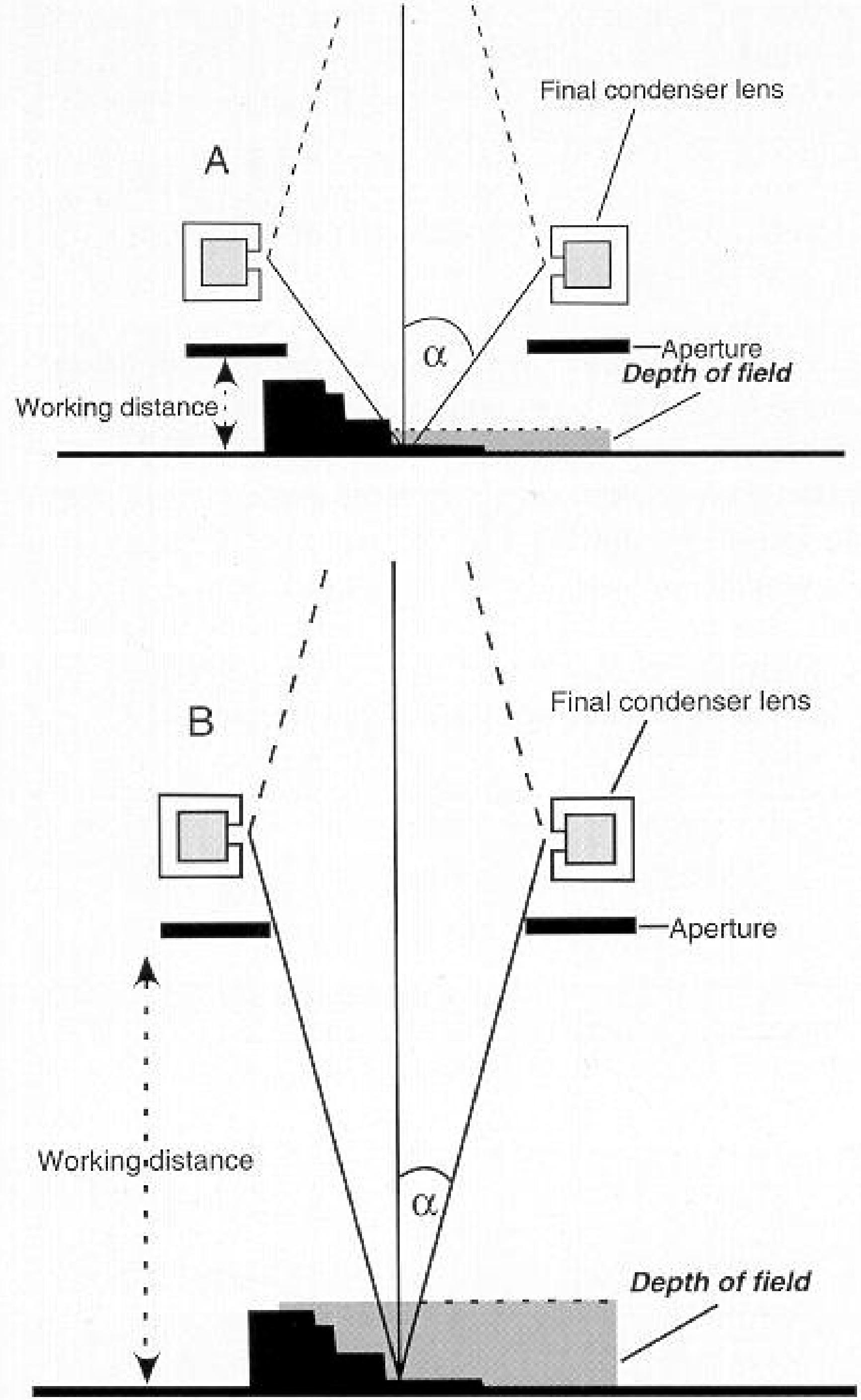


TABLE 7.4 Aperture Size and Quality of Image

Aperture Diameter (μm)	30	200	400	600
Resolution	Best	→	→	Worst
Depth of Field	Deep	→	→	Shallow
Signal Strength	Weak	→	→	Strong

TABLE 7.1 Effect of Aperture Size on Depth of Field at Various Magnifications and at a 10 mm Working Distance

	Depth of Field		
Mag	100 μm Aperture	200 μm Aperture	600 μm Aperture
10×	4 mm	2 mm	670 μm
50×	800 μm	400 μm	133 μm
100×	400 μm	200 μm	67 μm
100,000×	0.4 μm	0.2 μm	0.067 μm

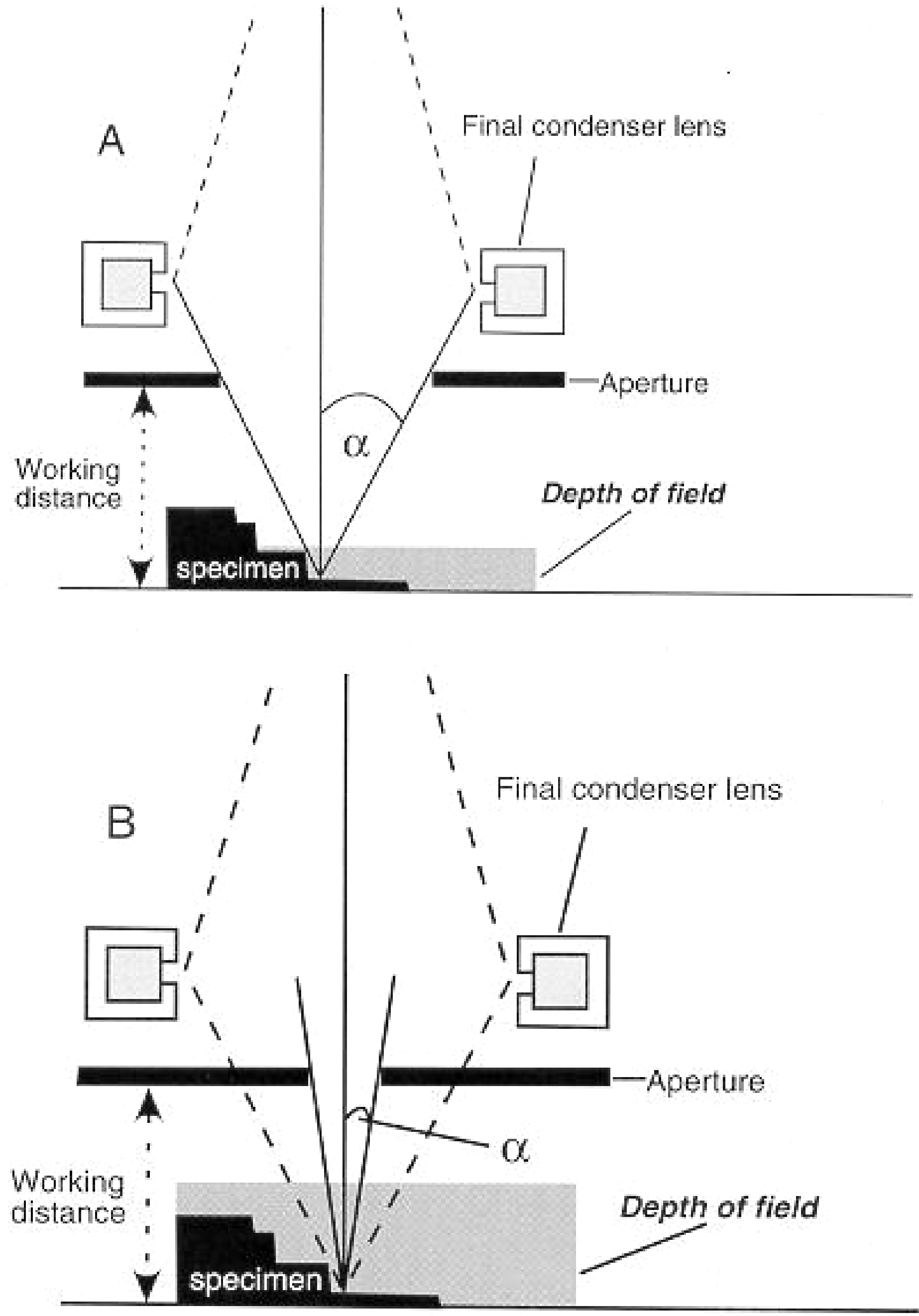
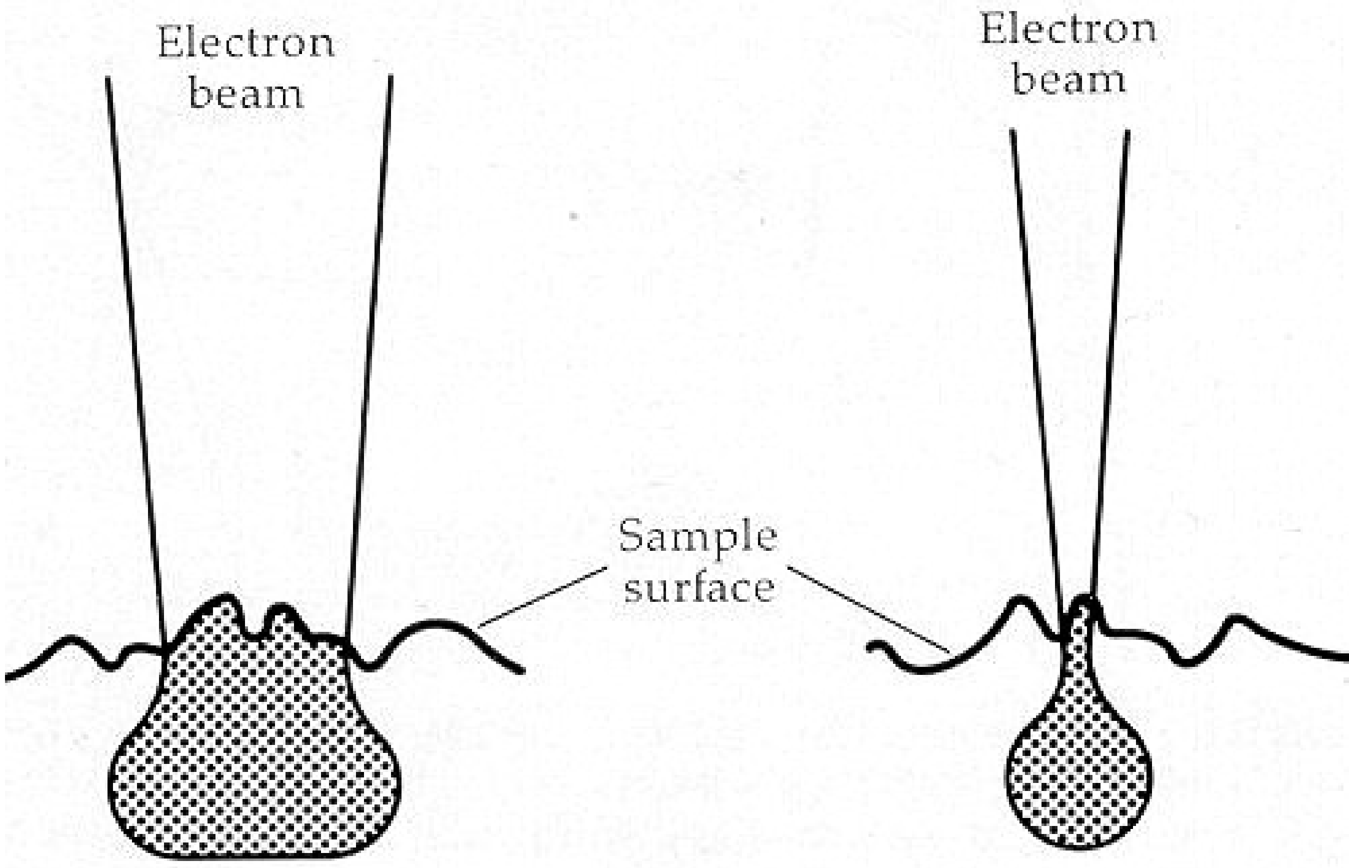
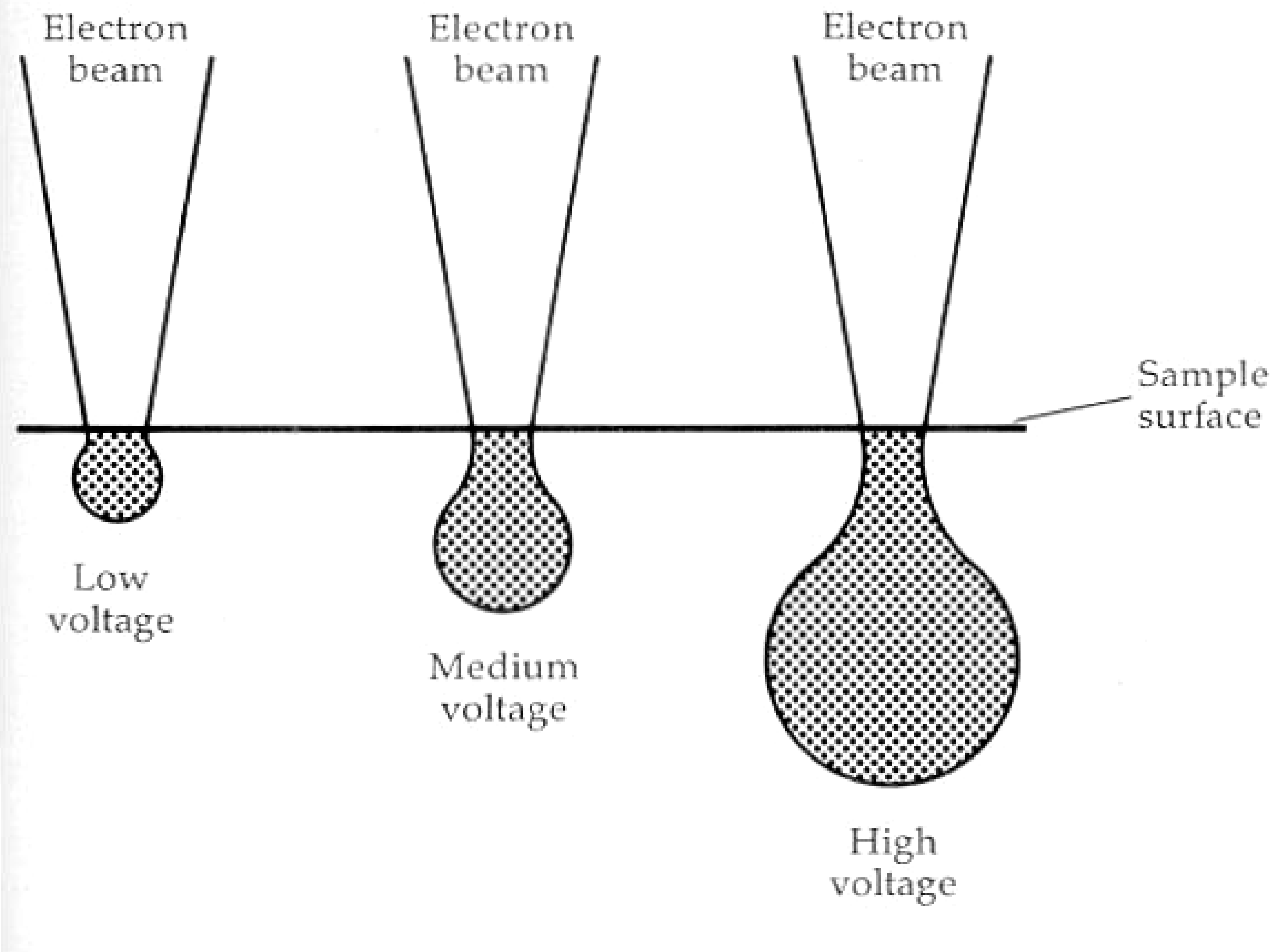


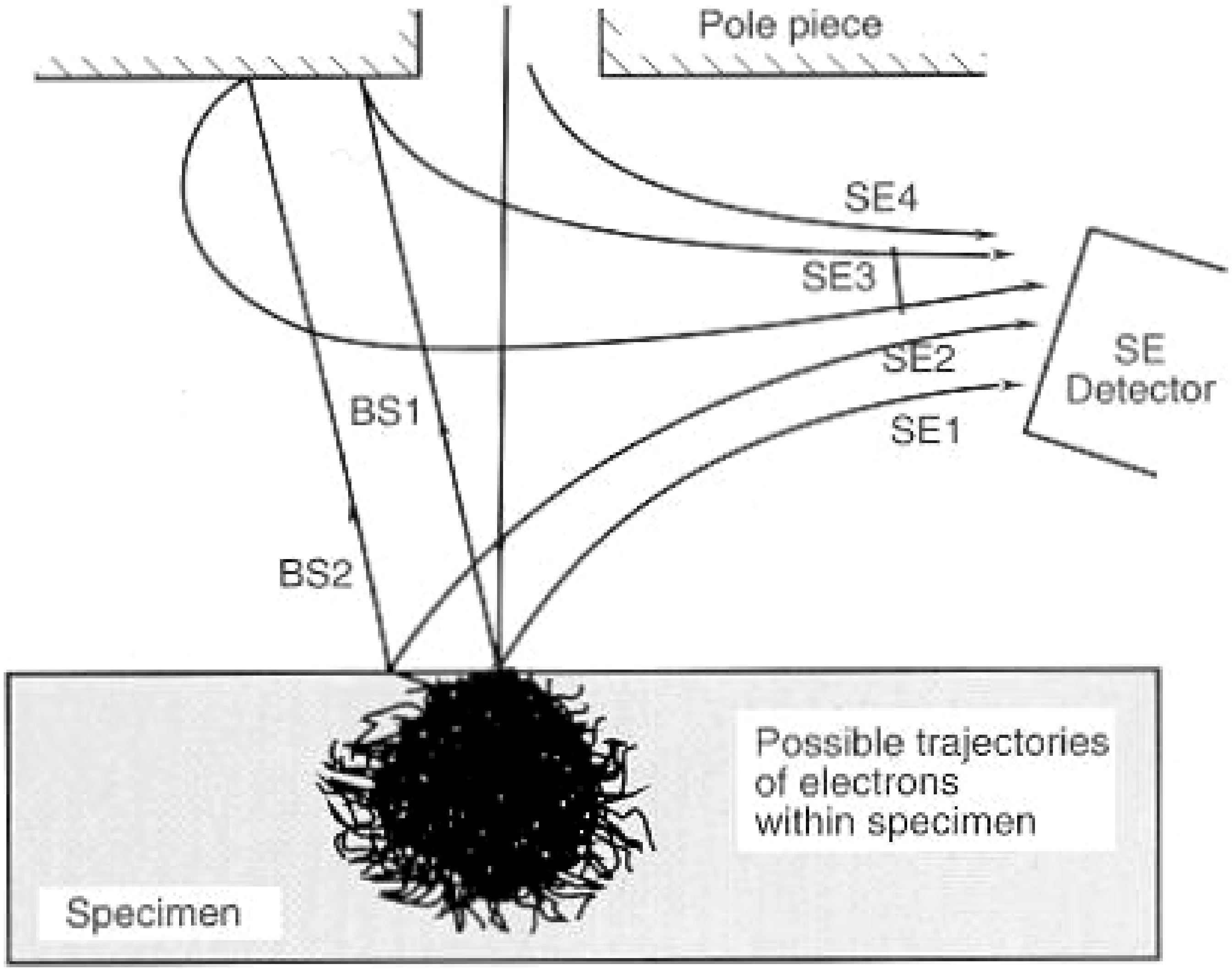
TABLE 7.3 Beam Spot Size and Quality of Image

Beam Spot Diameter (nm)	1	100	500
Resolution	Best	→	Worst
Signal Strength	Weak	→	Strong

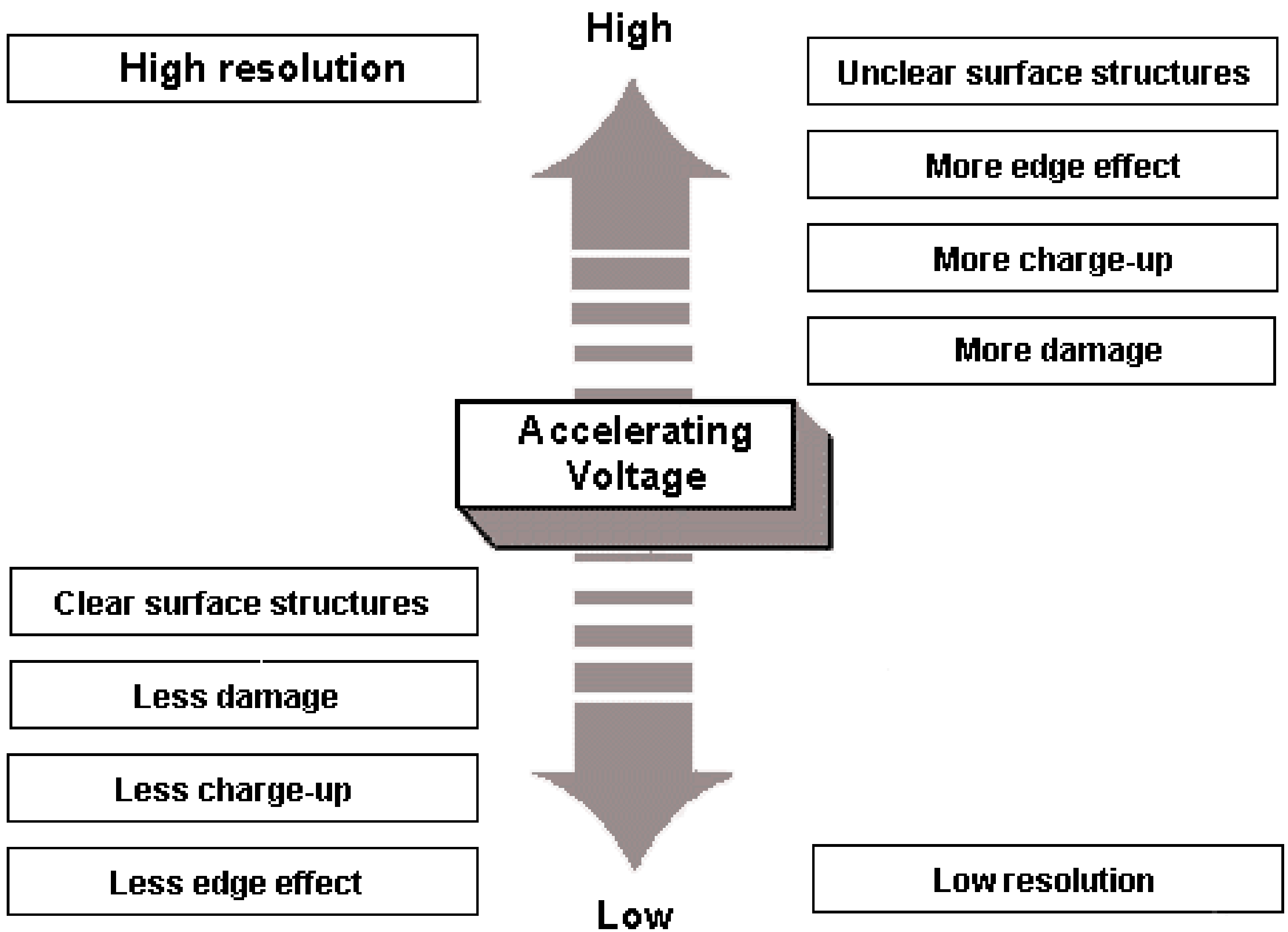


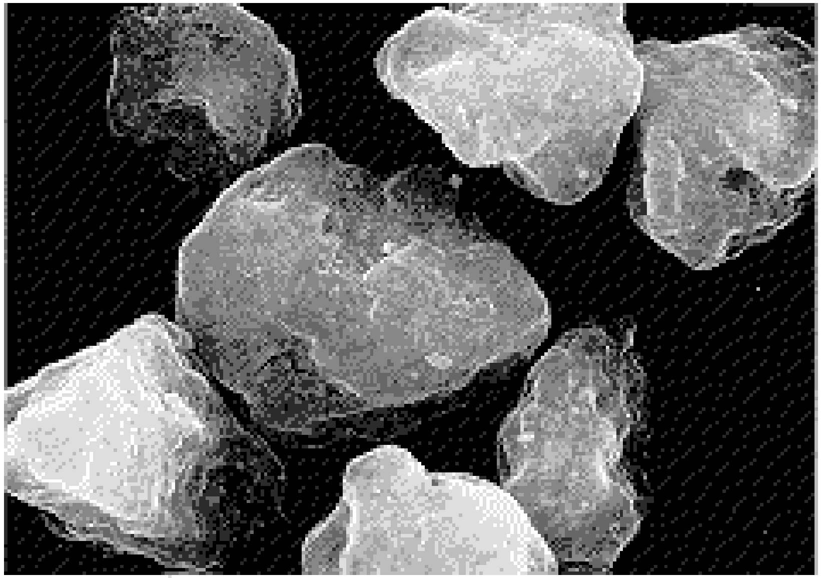


Electron beam
Electron beam
Electron beam
Sample surface
Low voltage
Medium voltage
High voltage

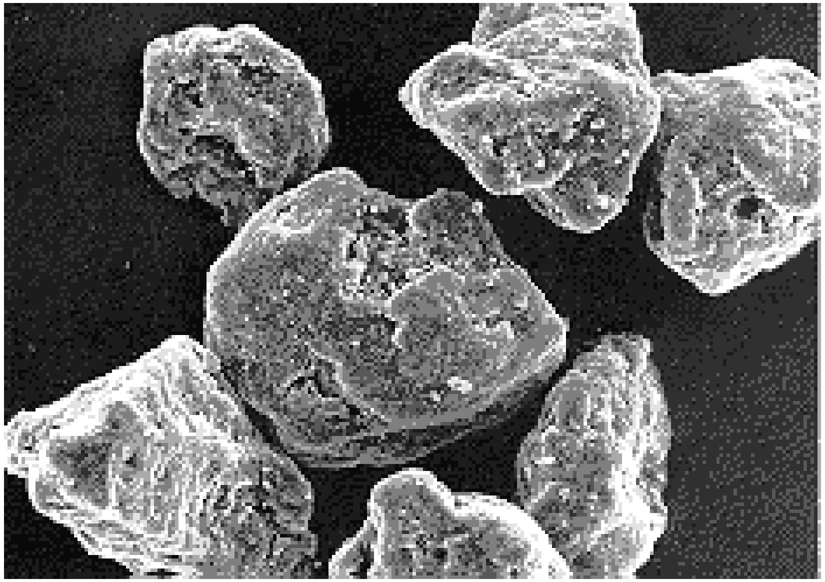


Rumore nel segnale SEM


High
High resolution
Unclear surface structures
More edge effect
More charge-up
More damage
Accelerating
Voltage
Clear surface structures
Less damage
Less charge-up
Less edge effect
Low resolution
Low



(a) 30 kV x 2,500



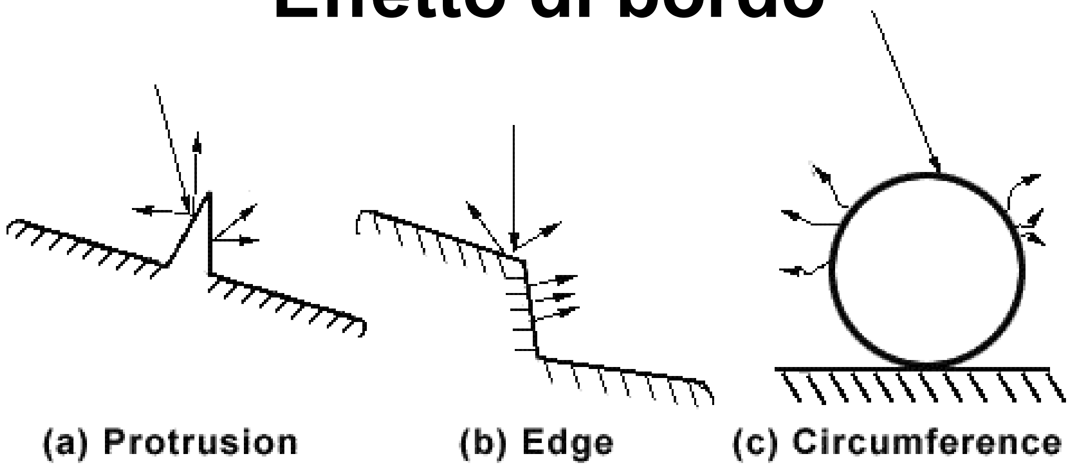
(b) 5 kV x 2,500

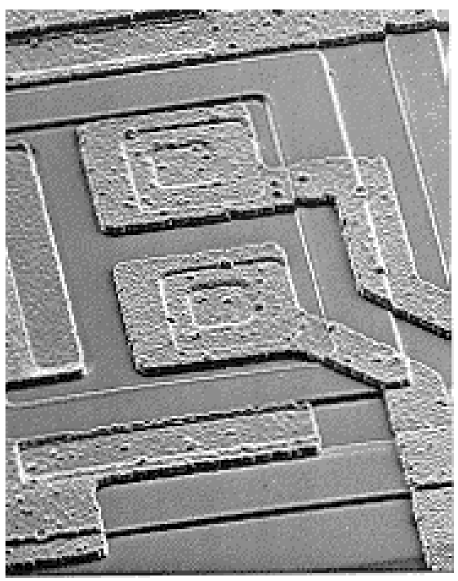
Per minimizzare le aberrazioni delle lenti, le lenti magnetiche nel microscopio elettronico hanno una piccola apertura di circa 0.1 mm in diametro.

Questo porta ad una elevata profondità di campo e profondità di fuoco che limita la visione tridimensionale degli oggetti:
Il punto più alto e quello più basso (distanza di 100 nm) sono sovrapposti con la stessa nitidezza.

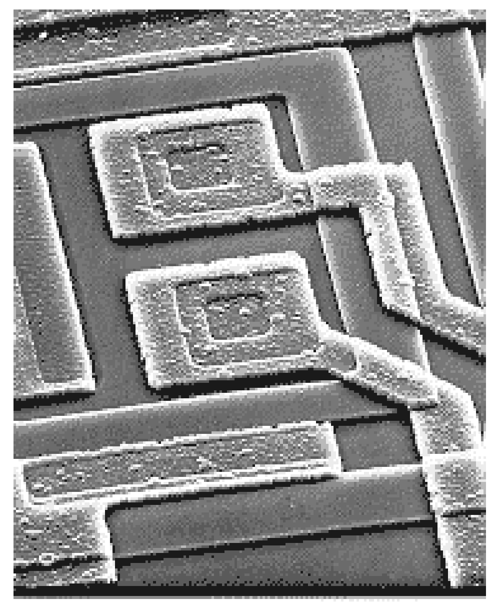
Per avere informazioni 3D il campione deve essere osservato obliquamente e le varie immagini possono essere raccolte e poi rielaborate al computer.

Effetto di bordo



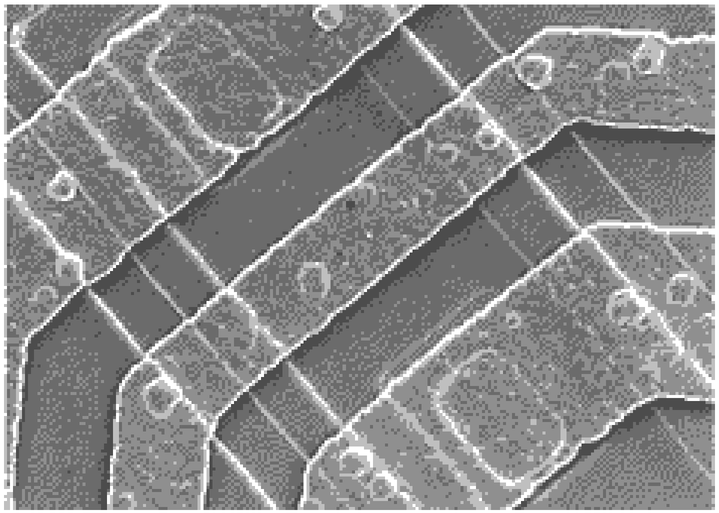


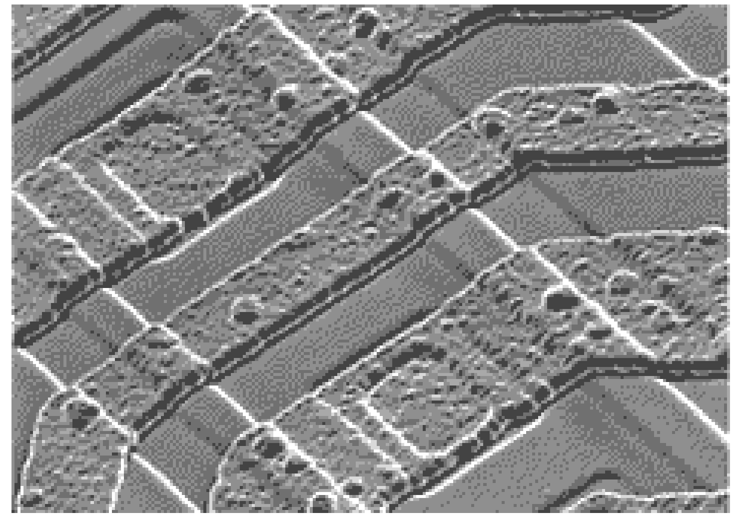
(a) 5 kV x720
Tilt angle: 50°



(b) 25 kV x720
Tilt angle: 50°

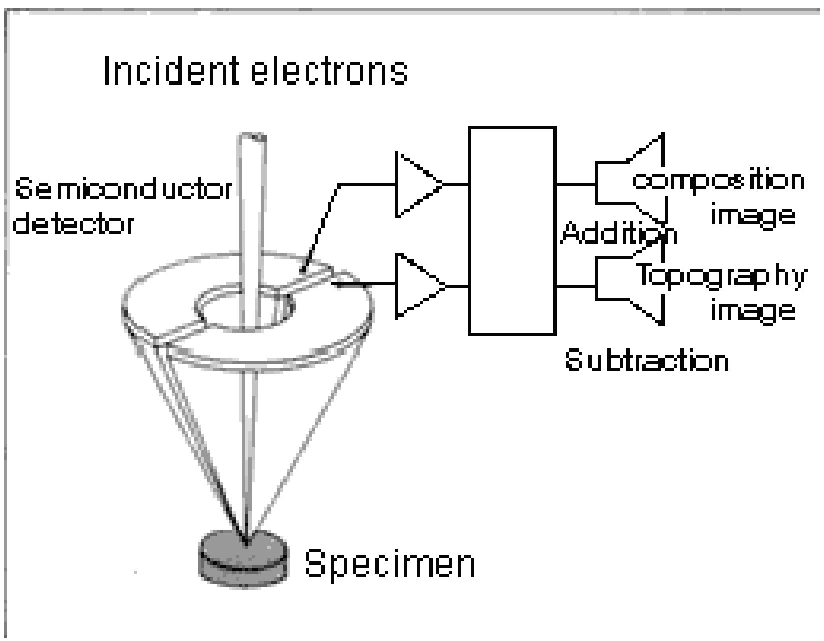
Angolazione del campione

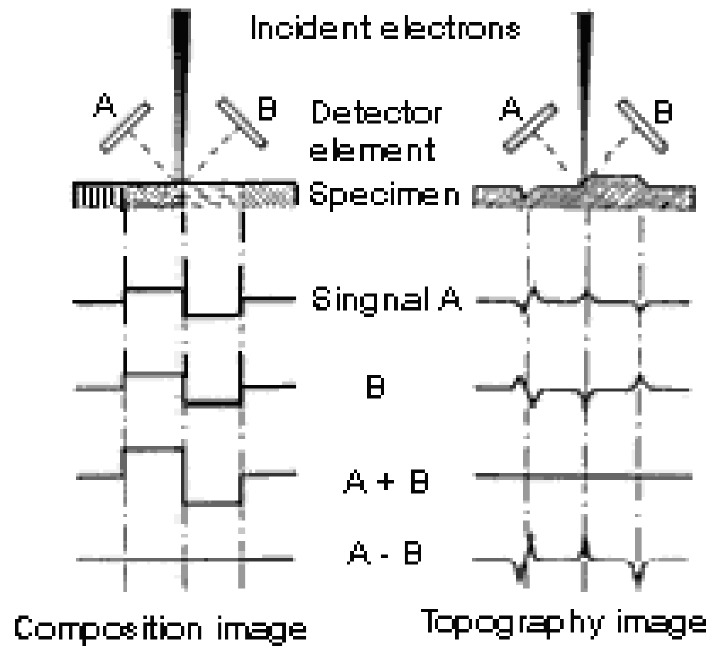

(a) Tilt angle: 0°

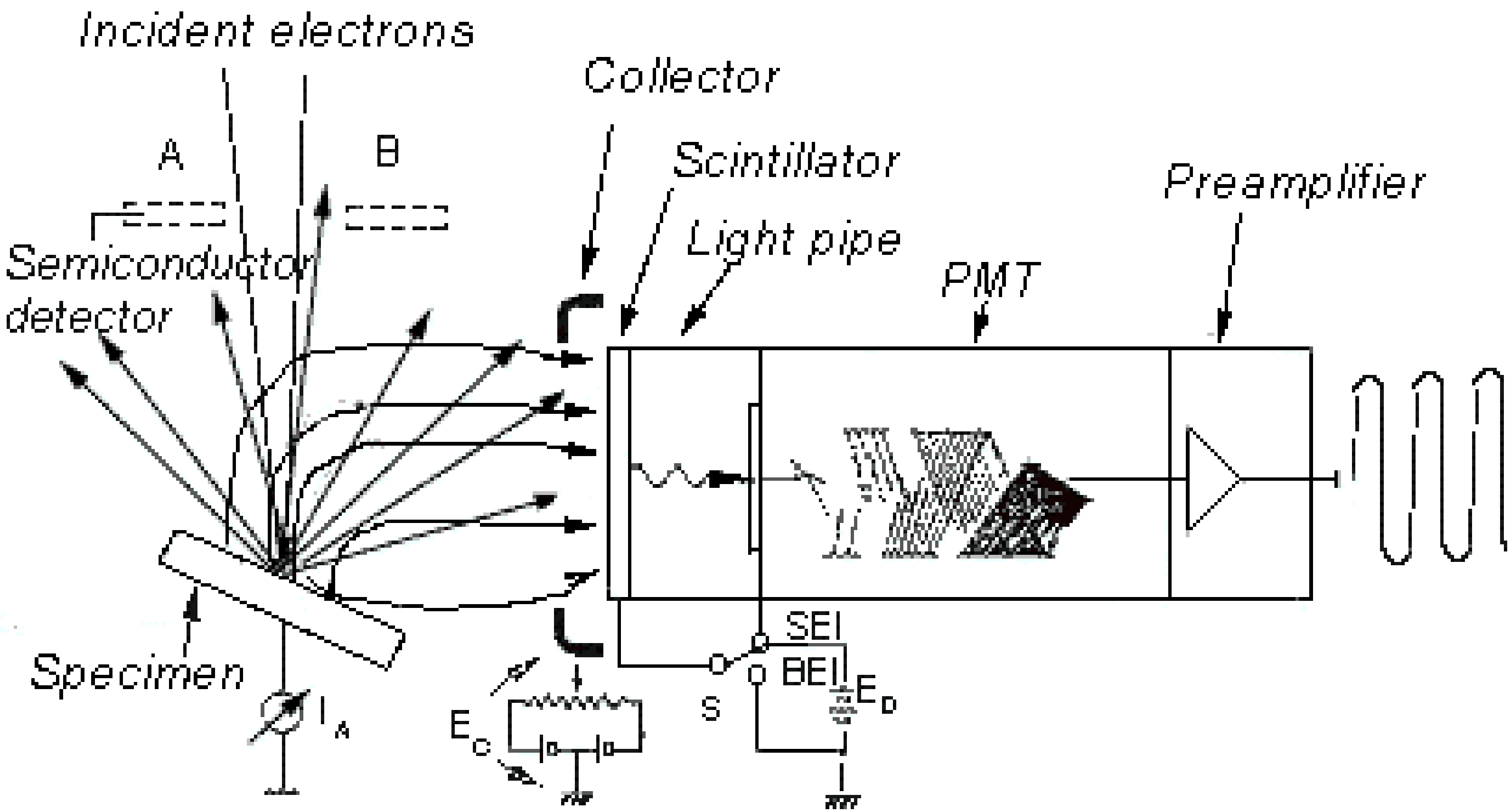

(b) Tilt angle: 45°

Specimen: IC chip.
5 W x 1,100

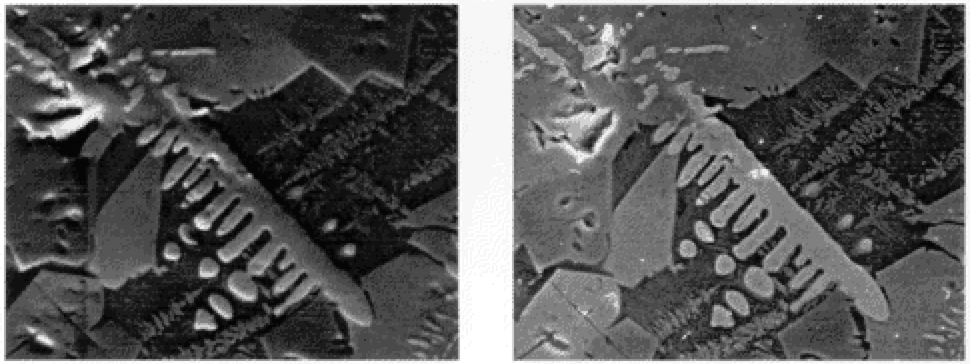
Backscattered electrons: Principles of composition detector image & topography image



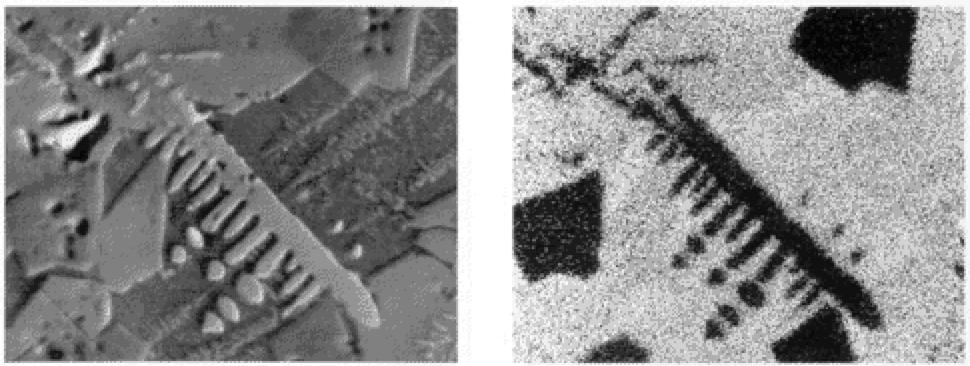




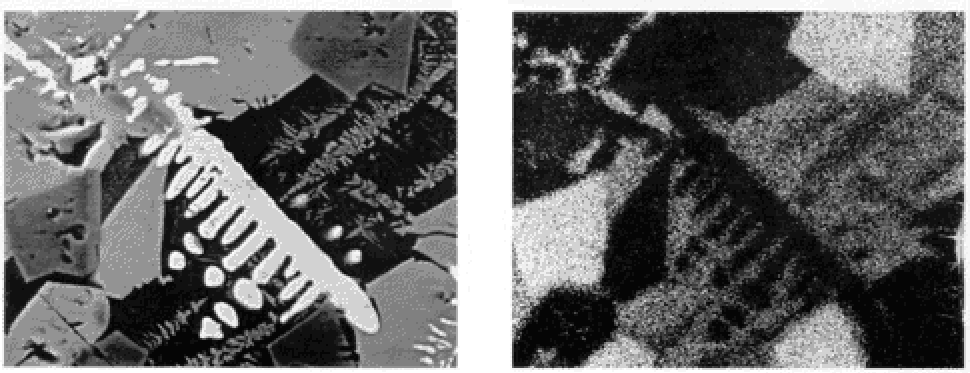
Incident electrons
Collector
A
B
Scintillator
Preamplifier
Light pipe
Semiconductor
detector
PMT
SEI
BEI
Specimen
E_D
S
I_A
E_C



(left) Backscattered electron image (BEI)
(right) Secondary electron image (SEI)

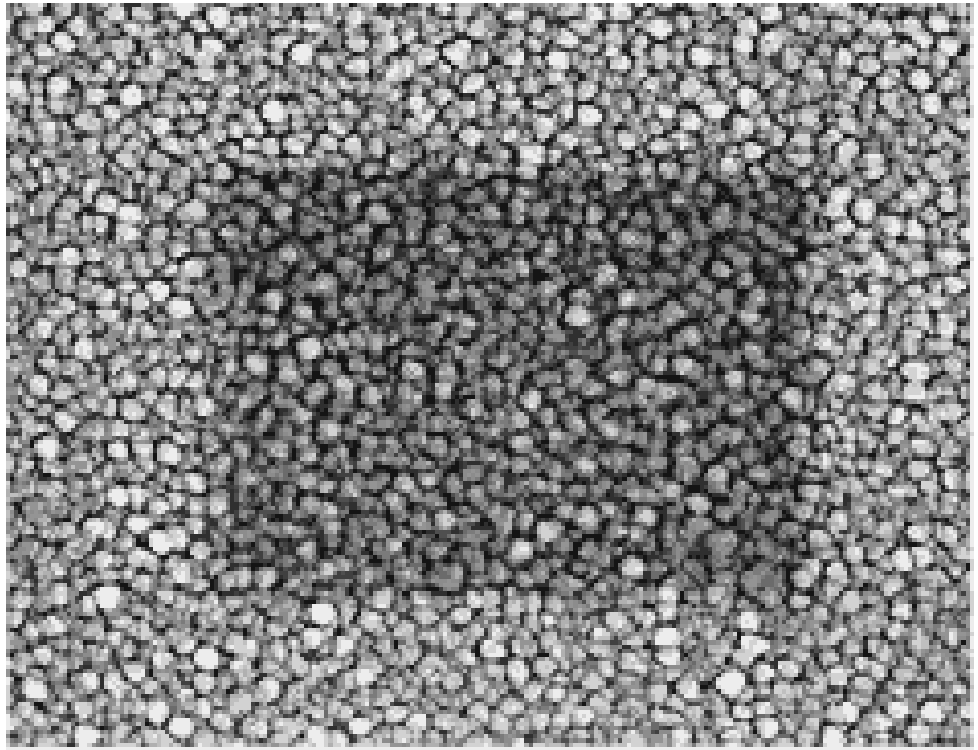


(left) Topography image (TOPO)
(right) X-ray image (Si)



(left) Composition image (COMPO)
(right) X-ray image (Al)

Contaminazione del campione



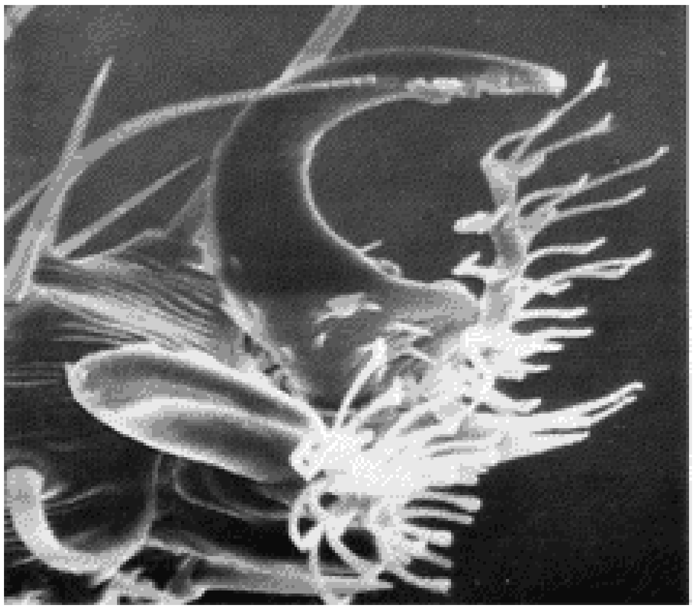
I gas nella camera del campione, che causano contaminazione sono:

1. Gas causato dallo strumento stesso

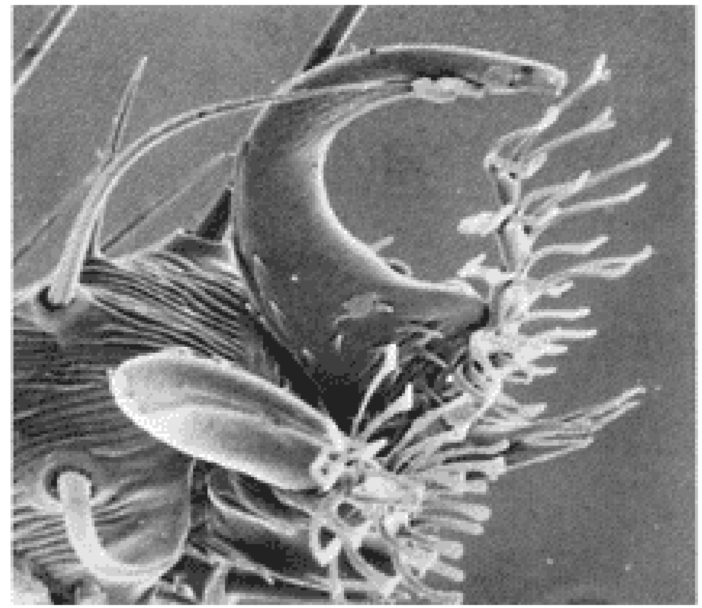
2. Gas portato dal campione nello strumento

3. Gas che il campione rilascia una volta bombardato da elettroni

Caricamento elettrico del campione



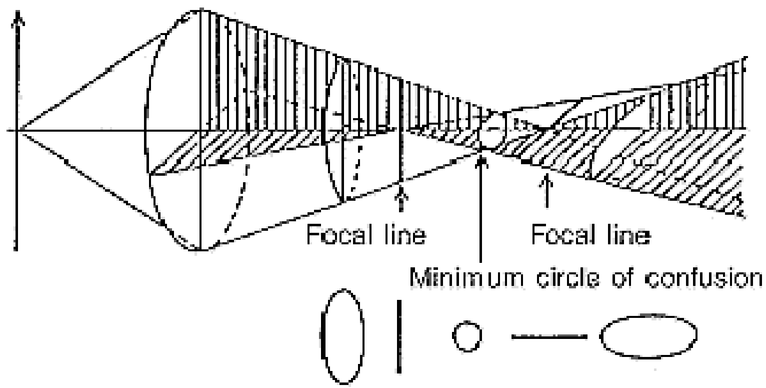
(b) 10 kV

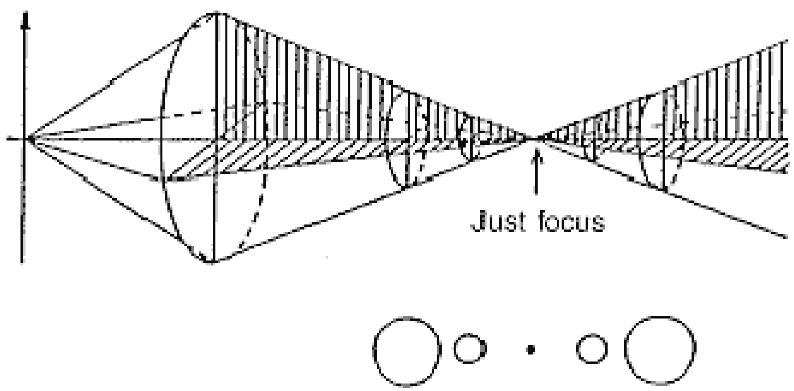


(a) 4 kV

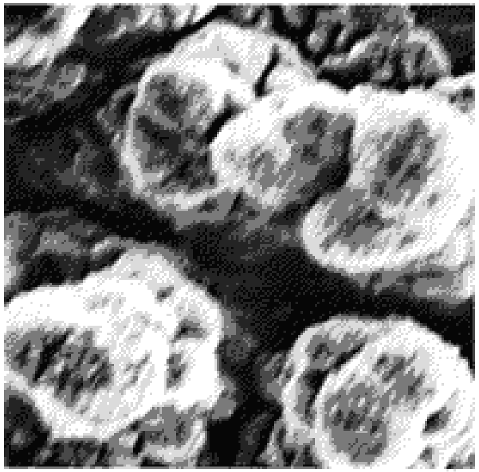
Charging

Astigmatismo

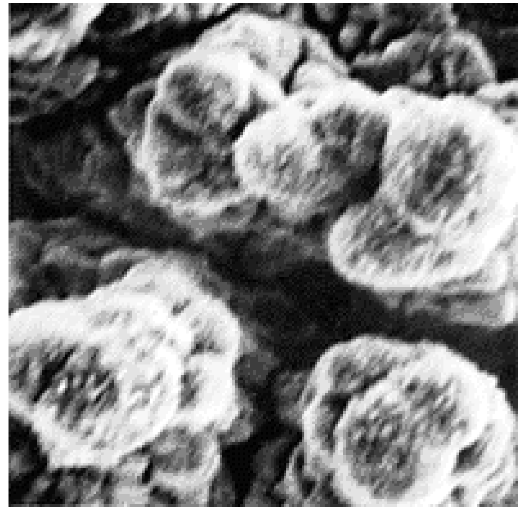




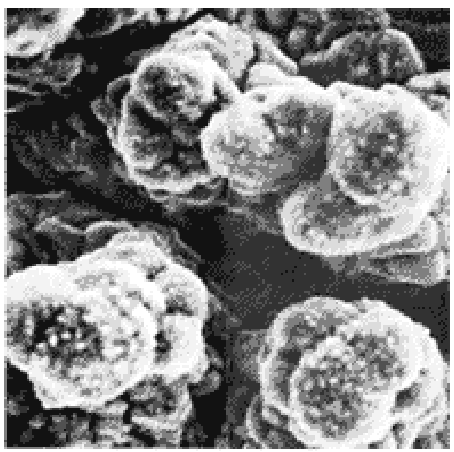
Con astigmatismo



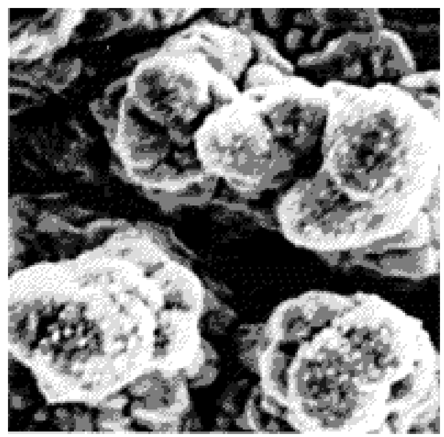
(a) under focus



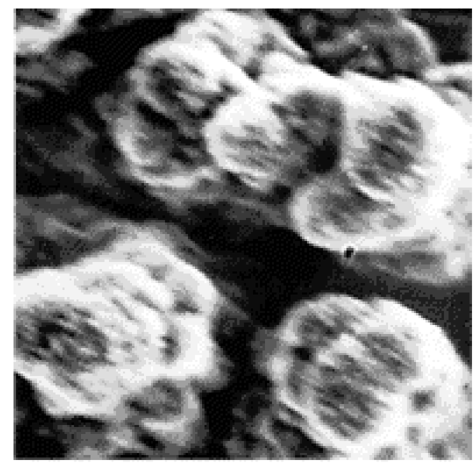
(b) under focus



(c) just focus

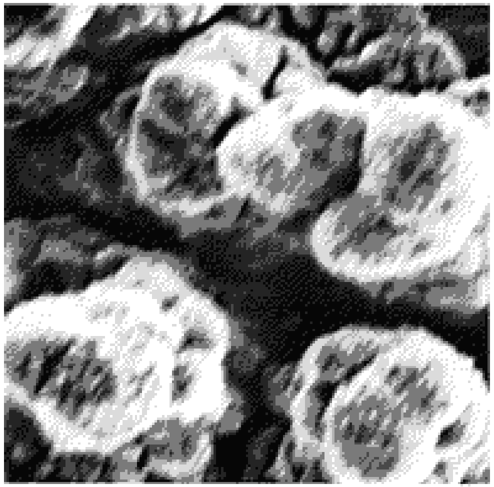


(d) over focus

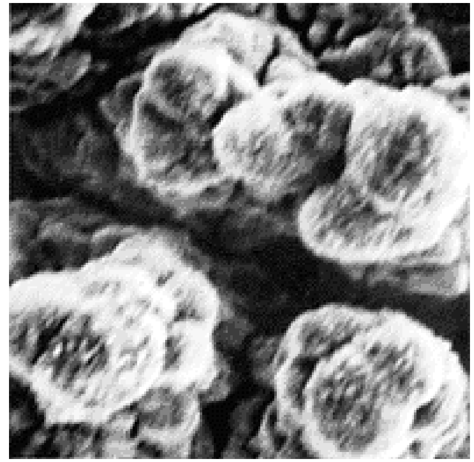


(e) over focus

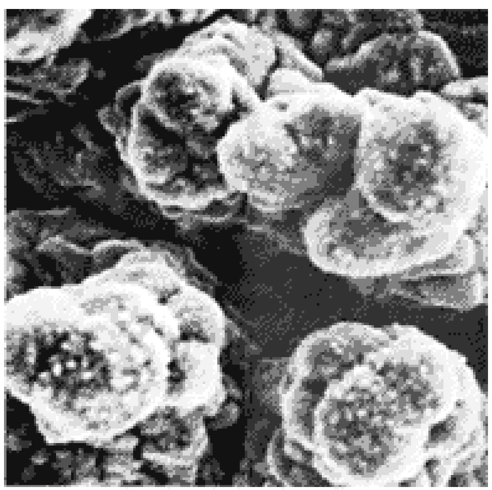
Senza astigmatismo



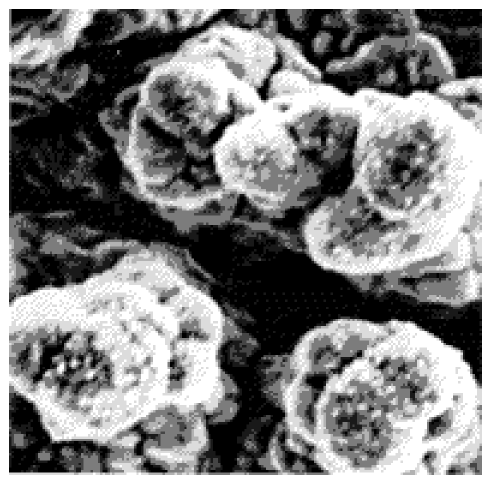
(a) under focus



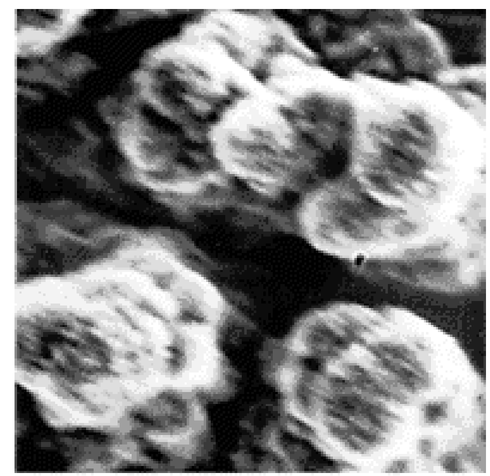
(b) under focus



(c) just focus



(d) over focus



(e) over focus