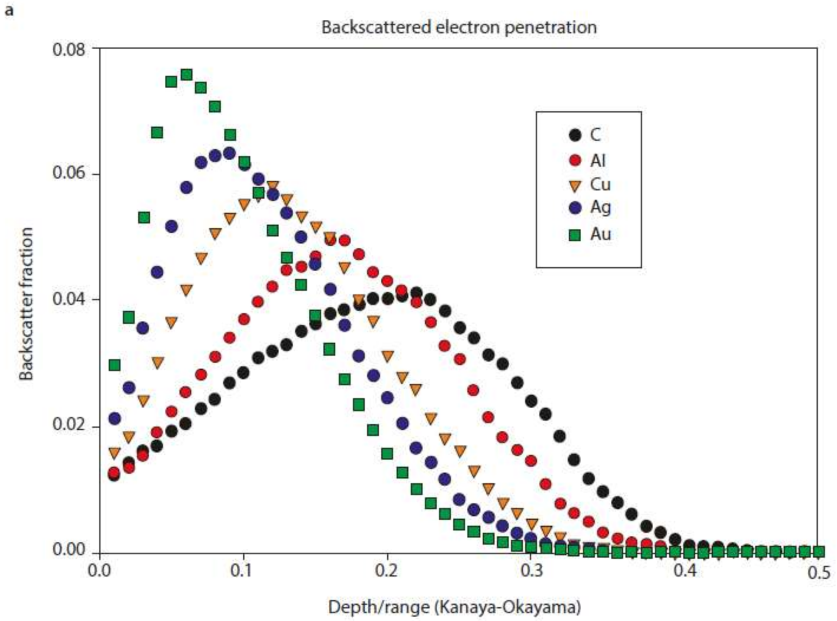
Backscattered electrons (BSE)

Depth Distribution of Backscattering dependence on Z

The maximum depth of penetration into the specimen of a primary electron before it eventually escaped as a BSE depends on the nature of the element.

A comparison between different elements can be conveniently done by plotting the contribution to the overall backscatter coefficient as a function of the depth of penetration expressed as the ratio depth/RKO as independentent variable



For heavy elements the backscattering events take place in lager part at upper part of the interaction volume. For lighter elements a sizable fraction of the backscattering events take place deeper inside the specimen.

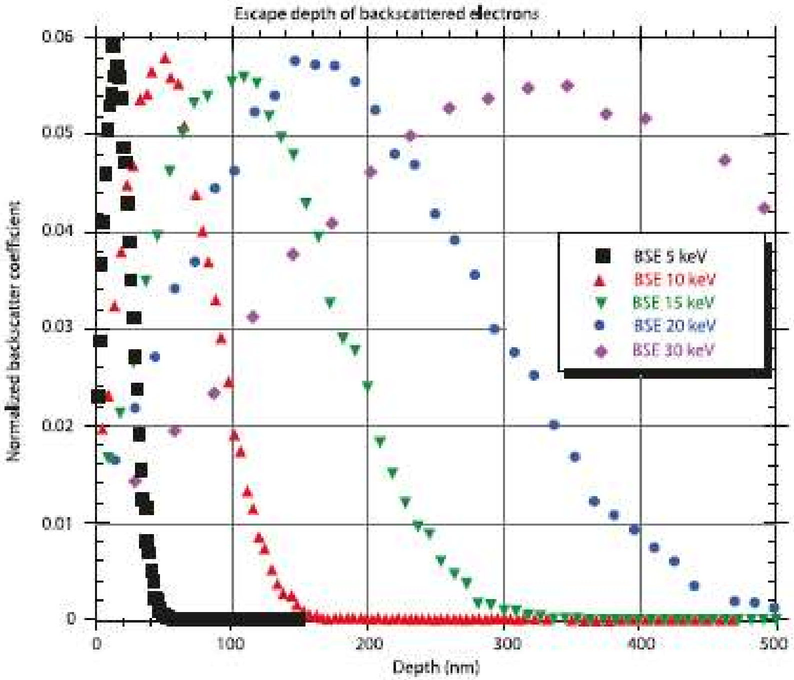
For this reason very thin specimens of materials made of light elements may not be suited to imaging using the BSE as signal.

Backscattered electrons (BSE)

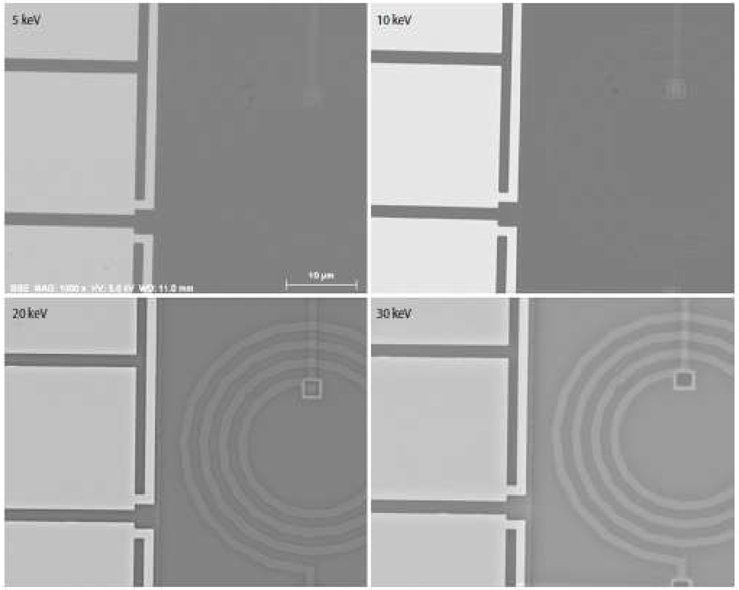
Depth Distribution of Backscattering dependence on accelerating voltage

Given a sample, by increasing the accelerating voltage the depth of generation of BSE increases (the kinetic energy of the primary electrons increases as the accelerating voltage).

This gives us the possibility of ‘seeing through’ samples to analyse hidden features.



Backscattered electron depth distributions for copper at various energies in copper at 0° tilt



BSE image at different incident beam energies of a semiconductor device consisting of silicon and various metallization layers at different depths.

Backscattered electrons (BSE)

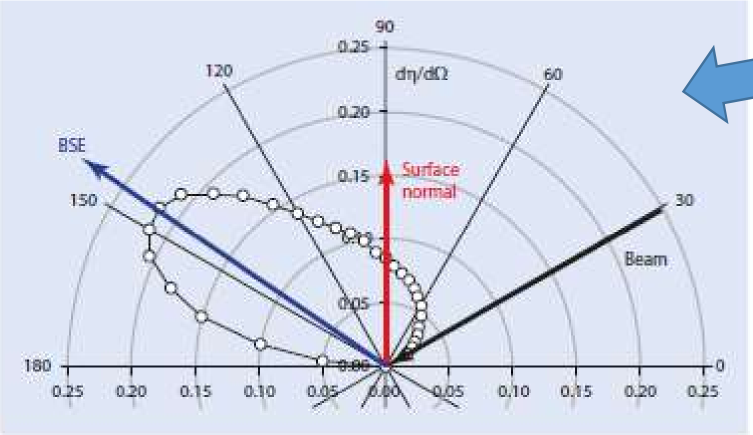
The angular distribution of BSE

The angular distribution of the trajectories of backscattered electrons is described by the cosine function: η(θ)=η′ cos θ, where η′ is the value of the backscattering coefficient of η along the surface normal and θ is the angle between the surface normal and the direction of measurements. This means that the number of backscattered electrons is maximum along the beam direction and becomes practically zero when θ increases to 90°.

However

This is true when the beam incident perpendicular to the Specimen Surface (0° Tilt)

If the sample is tilted respect to the beam a different situation occurs, it is easier for BSE to escape at higher tilt angles



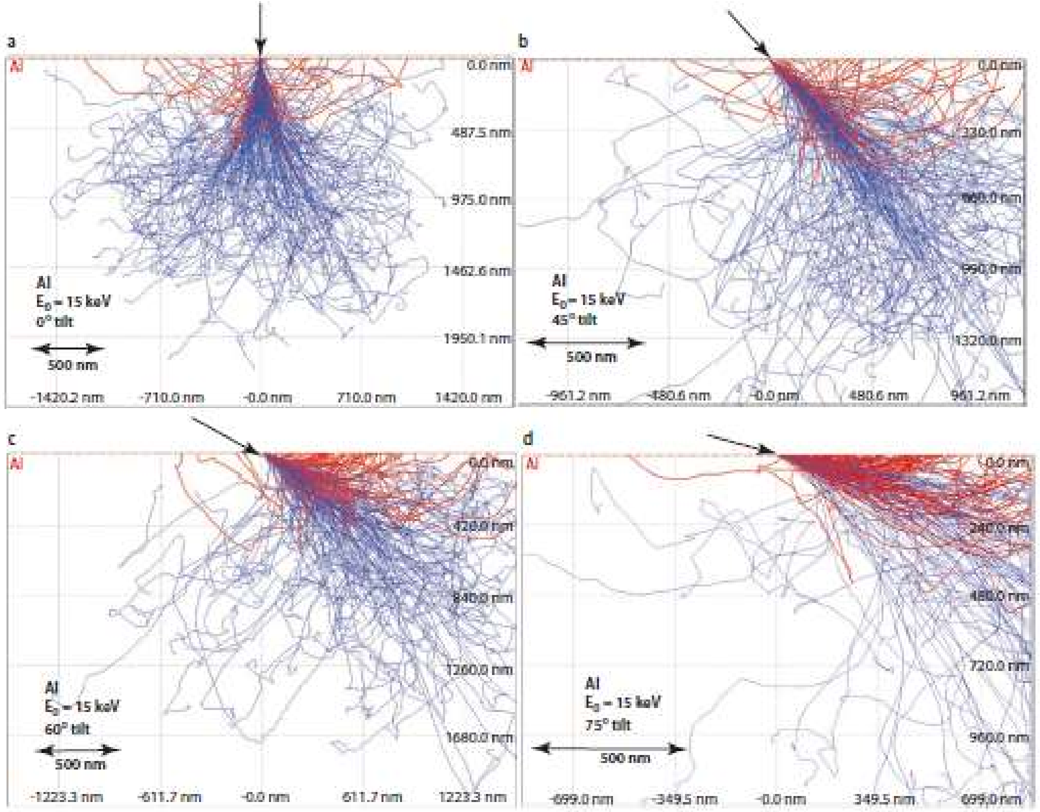
Angular distribution for a 60 degrees specimen tilt = beam incidence 30 degrees above surface

The backscattering coefficient depends on the angle between the incident beam and the sample surface

If you look carefully, it looks like BSE are simply ‘reflected’ by the specimen surface.

Backscattered electrons (BSE)

The angular distribution of BSE



Simulation for aluminum at E0 = 15 keV for a tilt angle of 0°. b for a tilt angle of 45°. c for a tilt angle of 60°. d for a tilt angle of 75°

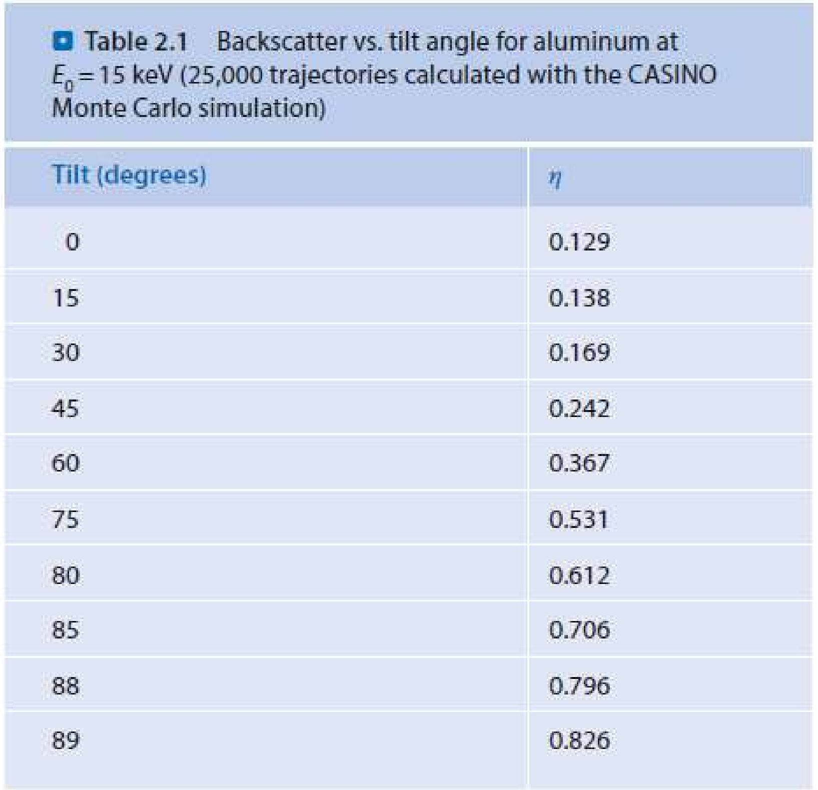


Table 2.1 Backscatter vs. tilt angle for aluminum at E_0 = 15 keV (25,000 trajectories calculated with the CASINO Monte Carlo simulation)

Tilt (degrees)	η
0	0.129
15	0.138
30	0.169
45	0.242
60	0.367
75	0.531
80	0.612
85	0.706
88	0.796
89	0.826

Backscattered electrons (BSE) Recap

- They are formed in elastic or quasi-elastic scattering events between the primary electrons and the atoms in the specimen. They actually are the primary electrons scattered back from the sample.

- Are high energy electrons, their kinetic energy is in the order of magnitude of the primary electrons kinetic energy.

- Their yield, the backscattering coefficient η, increases as the atomic number increase (heavy elements scatter better than lighter ones) and depends on the accelerating voltage, the dependence is however weak.

- BSE do not have all the same energy, there is an energy distribution (or spectrum) that is peaked close to the PE energy for heavier elements. For lighter elements the energy distribution is broader because multiple scattering events are necessary prior the PE to becomes a BSE.

- The BSE originate from a sizable fraction of the interaction volume, at a depth of about 10-20% of the Kanaya-Okayama range, this means ≈ 100 nm or more from the specimen surface. This depth depends on the accelerating voltage and can be tuned by acting on this parameter. The higher the accelerating voltage the deeper is the position at which BSE are generated.

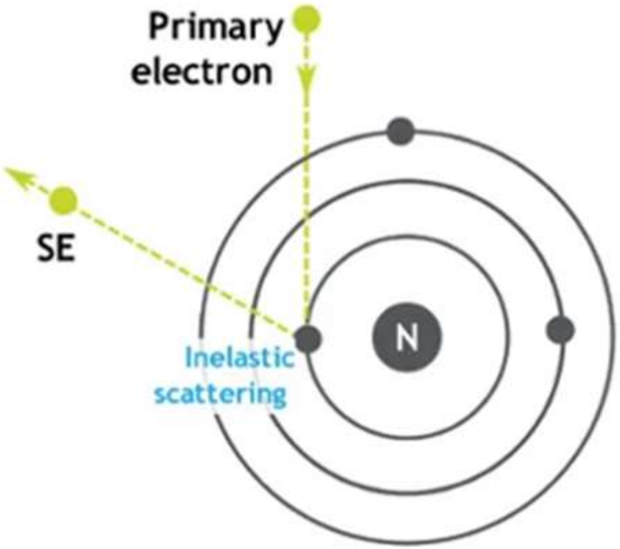
- An analysis of the angular distribution of BSE pinpoints that they can be considered as PE ‘reflected from the specimen surface. However, the yield of BSE increases as the tilt angle increase.

Secondary Electrons (SE)

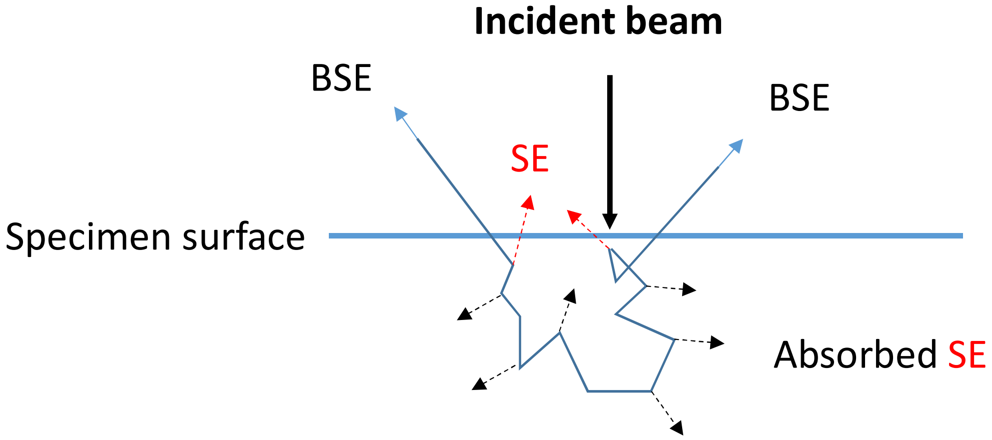
Interaction of the electron beams with materials

Secondary electrons (SE)

Secondary electrons are generated when a primary electron displaces a specimen electron from the sample surface. Secondary electrons can also be generated by other secondary electrons. SE are weakly bound valence electrons for ionic or covalent analytes or conduction band electrons for metals usually with energies of 1–15 eV, usually below 50 eV.



Secondary electrons have a low energy level of only a few electron eV, thus, they can only be detected when they are formed near the surface of the interaction volume. Therefore, secondary electrons cannot escape from deep within the interaction volume. Secondary electrons that are generated but do not escape from the sample are absorbed by the sample.



Incident beam

BSE

BSE

SE

Specimen surface

Absorbed SE

Secondary electrons (SE)

The most important characteristic of SE is, as mentioned, their extremely low kinetic energy. Because of the large mismatch in relative velocities between the primary beam electron (incident energy 1–30 keV) and the weakly bound atomic electrons (1–15 eV ionization energy),

Secondary electrons are quantified by the parameter δ, which is the ratio of secondary electrons emitted from the specimen, N_{SE}, to the number of incident beam (primary) electrons: **$\delta = N_{SE} / N_B$**

The mean free path length of secondary electrons in many materials is 10 Å (exceptions exist). Thus, although electrons are generated throughout the region excited by the incident beam, only those electrons that originate less than 10 Å deep in the sample escape to be detected as secondary.

Attention here!

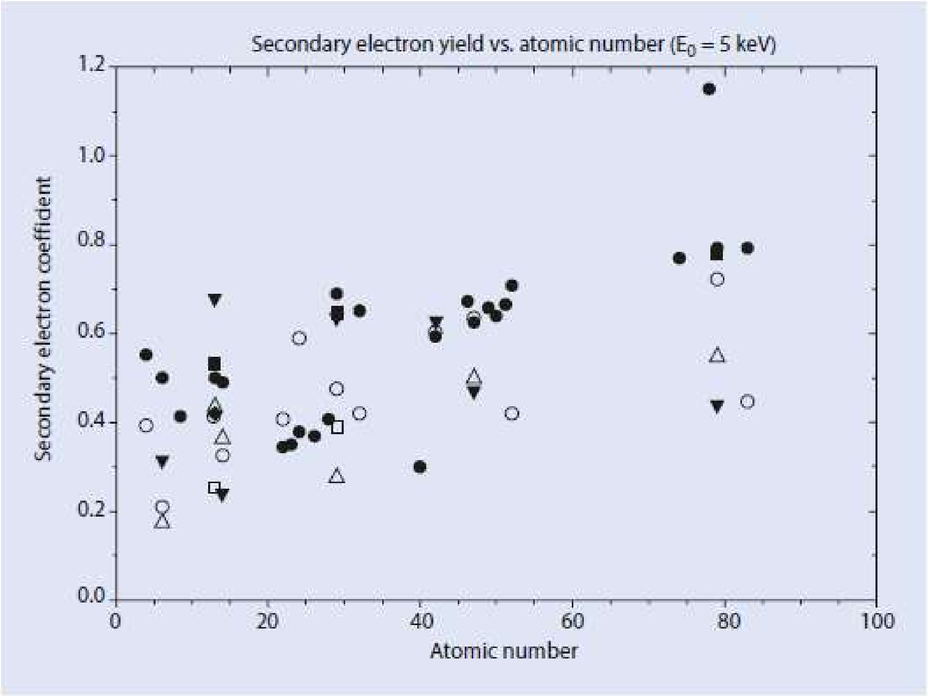
The parameter **δ** can be greater than 1 under certain circumstances

This process includes the SE cascade whereby scattered primary electrons collide with other electrons within the material, which produce further SEs and so on. This process generates many more SEs than otherwise would have been the case.

Secondary electrons (SE)

the parameter δ, the secondary electron yield

Secondary emission coefficient as a function of Z



In this graph, the measurements of δ seem chaotic and inconsistent.

This behavior is really an indication of how difficult it is to make a representative measurement of a property that results from very low energy electrons generated within and escaping from a very shallow layer below the surface. Thus, a surface modified by accumulations of oxide and contamination (e.g., adsorbed water, chemisorbed water, hydrocarbons, etc.) is likely to produce a value of δ that is different from the “ideal” pure element or pure compound value.

Secondary electrons (SE)

the parameter δ, the secondary electron yield

Secondary emission coefficient as a function of Z

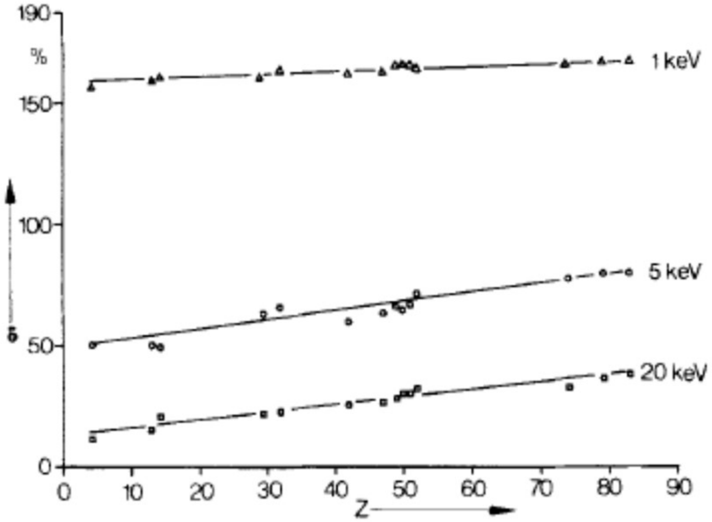


Fig. 6 Secondary electron yield δ as a function of electron energy E for different elements

SCANNING Vol. 3, 35-39 (1980)

However, under controlled conditions it appears that the δ values increase with increasing Z.

Pleas also observe that δ values seem to decrease when the primary electron energy increases.

To explain this we need to introduce the concept of escape depth.

Secondary electrons (SE)

The escape depth

The escape depth means the depth from the upper surface a specimen, from which secondary electrons can escape.

Thus, although SE are generated along the entire trajectory of a beam electron scattering in the target, only those SE generated close to a surface have a significant chance to escape.

The probability of escape depends on the initial kinetic energy, the depth of generation, and the nature of the host material. Since there is a spectrum of initial kinetic energies, each energy represents a different escape probability and depth sensitivity.

Kanaya and Ono in 1984 modeled the mean secondary electron escape depth, desc, in terms of various material parameters:

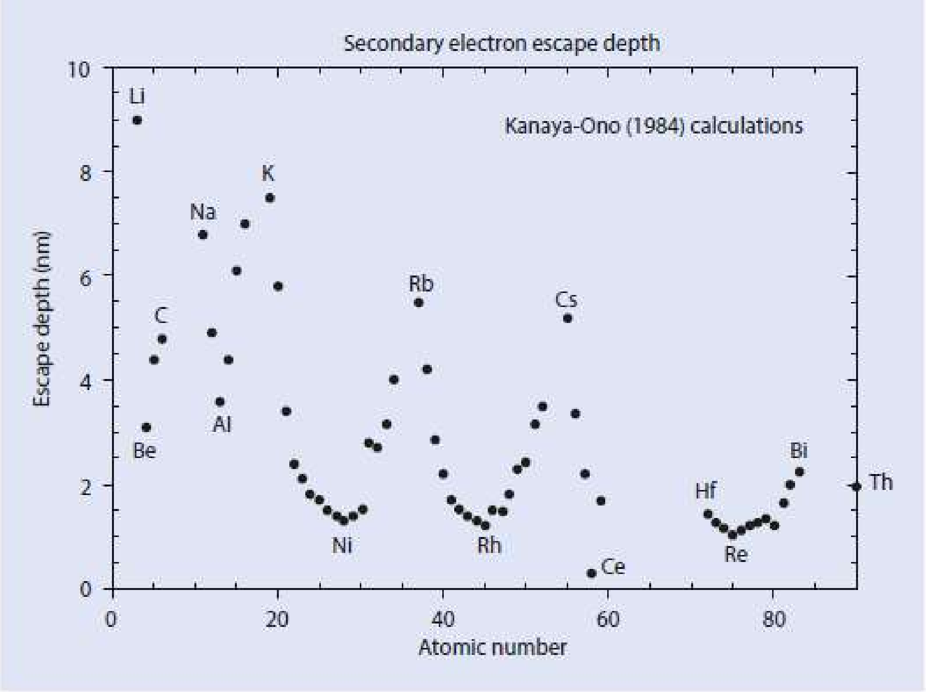
$$d_{esc}\left(\mathrm{nm}\right) = 0.267\, A\, I / \left(\rho Z^{0.66}\right)$$

where A is the atomic weight (g/mol), ρ is the density (g/ cm3), Z is the atomic number, and I is the first ionization
potential (eV).

Secondary electrons (SE)

The escape depth

Besides the actual structure of the Kanaya Ono equation, it important to recognize that the escape depth of the secondary electron for most metals is approximately 2 to 5 nm and in general below 10 nm.



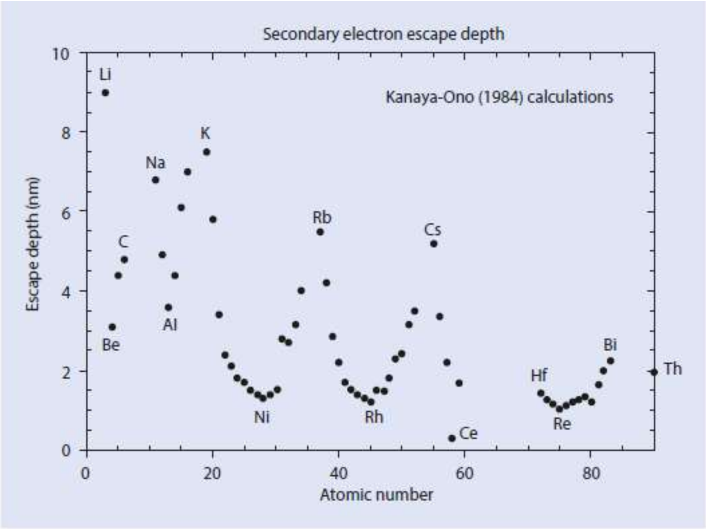
This means that secondary electrons generated deeper than 10 nm from the sample surface do not have chance to emerge from the surface and being detected.

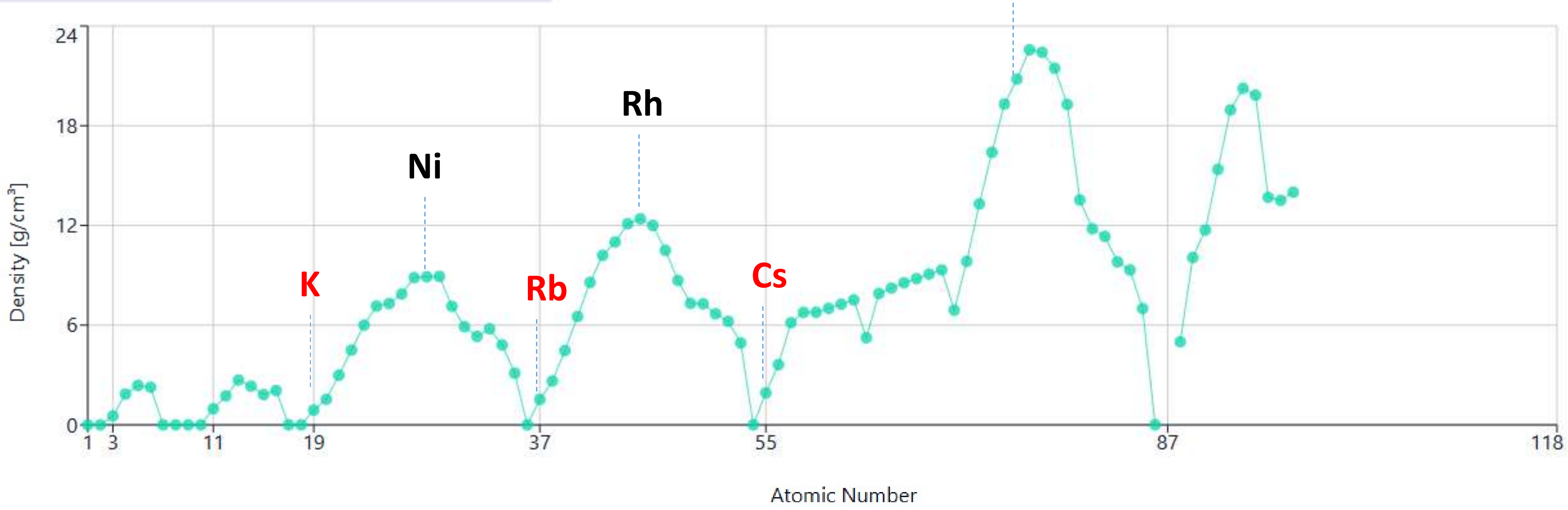
Remember: the number of secondary electrons ejected from the Target are due to inelastic scattering and therefore, the number of SE increase in proportion to the energy loss. This is more efficient for denser elements, remember the Bethe equation.

$$\mathrm{d}E/\mathrm{d}s\left(\mathrm{eV}/\mathrm{nm}\right)=-7.85\left(Z\rho/AE\right)\ln\left(1.166\,E/J\right)$$

Secondary electrons (SE)

The escape depth vs density

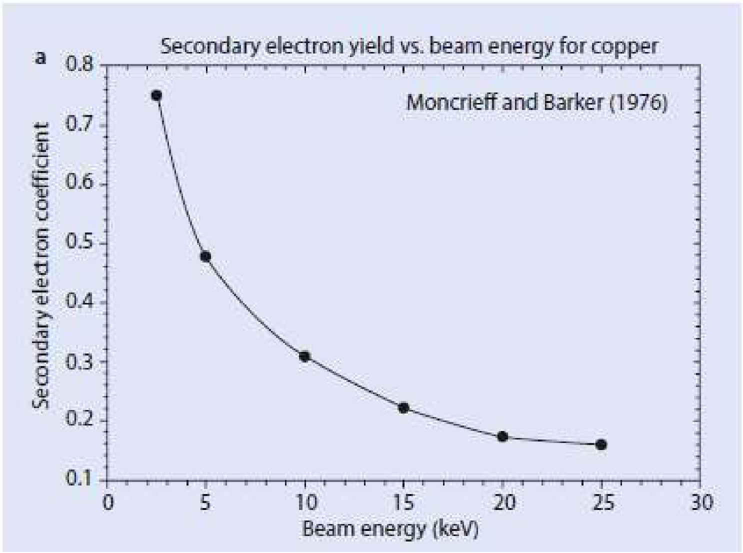




Secondary electrons (SE)

the parameter δ, the secondary electron yield

Dependence on beam energy



Remember: the number of secondary electrons ejected from the target are due to inelastic scattering and therefore, the number of SE increase in proportion to the energy loss.

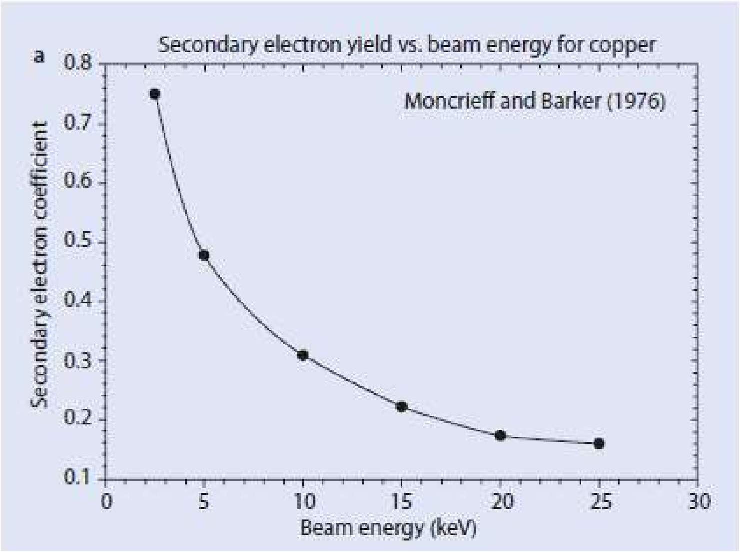
But for a given element the energy loss decreases when the primary electron energy is increased!

Furthermore, for a given element the Bethe range increases increasing the beam energy and therefore SE generated deeper into the sample cannot escape!

Overall, increasing the beam energy generates less SE at the surface but more deep inside the sample but these cannot escape. Hence the shape of the curve.

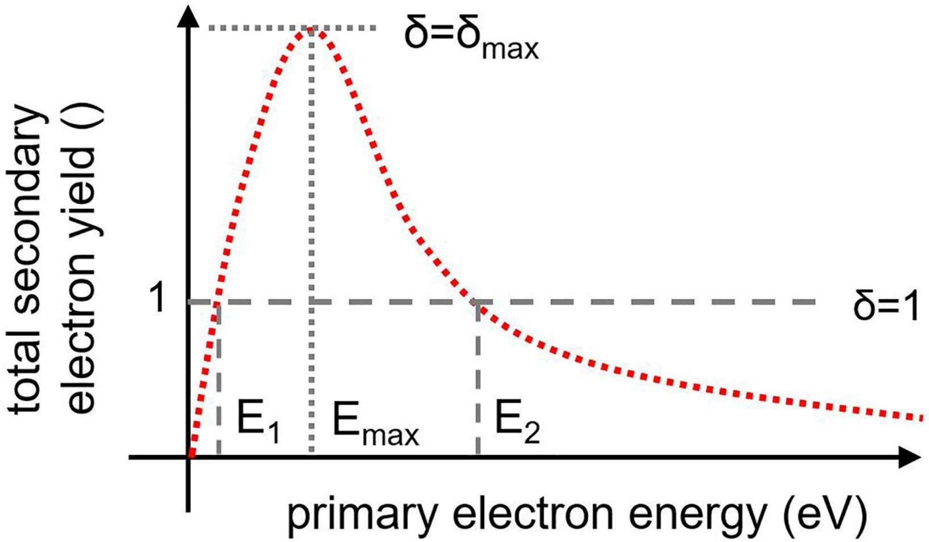
Secondary electrons (SE)

Dependence on beam energy



Please observe that this is a monotonic decrease, but since at zero beam energy there are no electrons ejected this means that somewhere in the low energy regime there has to be a maximum.

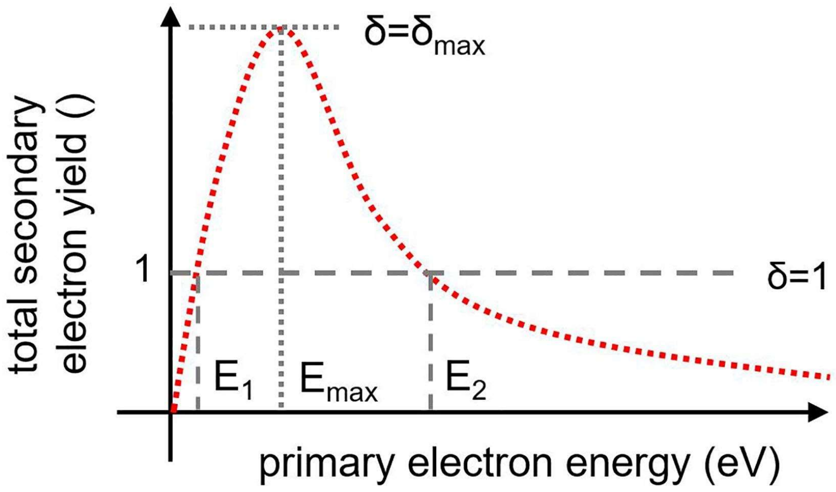
The complete curve describing δ as a function of the beam energy



Secondary electrons (SE)

the parameter δ, the secondary electron yield

Dependence on beam energy



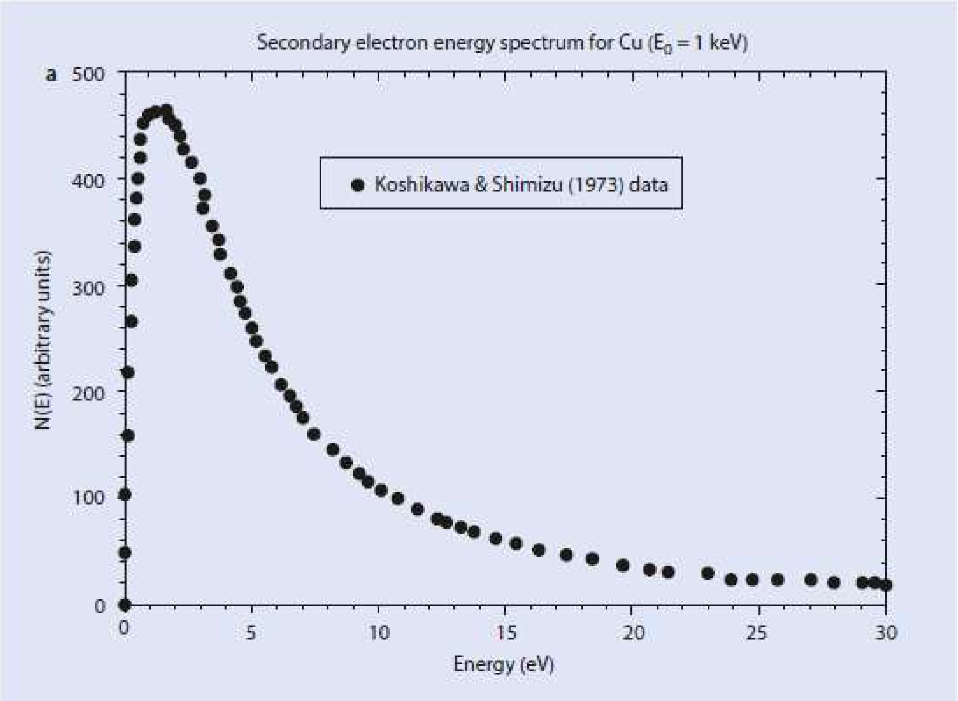
δ increases with the accelerating voltage until about several 100 eV. That is, δ takes maximum at voltages between 300 to 800 eV (different for constituent materials) and then decreases gradually with increasing the accelerating voltage.

Two important energies are the first and second crossover energies EI and EII. When E0 < EI, the penetration depth is smaller than the escape depth and the majority of the secondary electrons can escape. However, only a limited number of secondary electrons are generated, E0 is small. For E0 > EII, the amount of secondary electrons is larger, but they are generated deeper inside the material. Electron multiplication occurs for EI < E0 < EII with a maximum yield at Em.

Remember: Each primary electron is able to generate more secondary electrons as its energy increases

Secondary electrons (SE)

Secondary electrons energy spectrum



The most important characteristic of SE is their extremely low kinetic energy. Because of the large mismatch in relative velocities between the primary beam electron (incident energy 1–30 keV) and the weakly bound atomic electrons (1–15 eV ionization energy), the transfer of kinetic energy from the primary electron to the SE is relatively small, and as a result, the SE are ejected with low kinetic energy. After ejection, the SE must propagate through the specimen while undergoing inelastic scattering, which further decreases their kinetic energy.

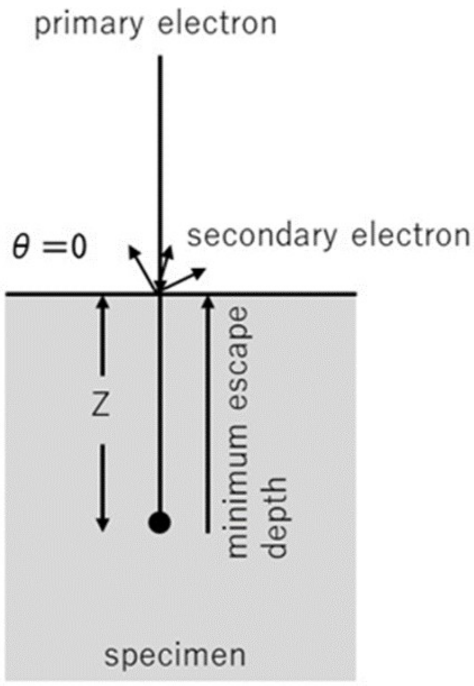
The energy spectrum of the secondary electrons that escape is peaked at only a few eV, as shown in the figure for a copper specimen and an incident beam energy of E0 = 1 keV.

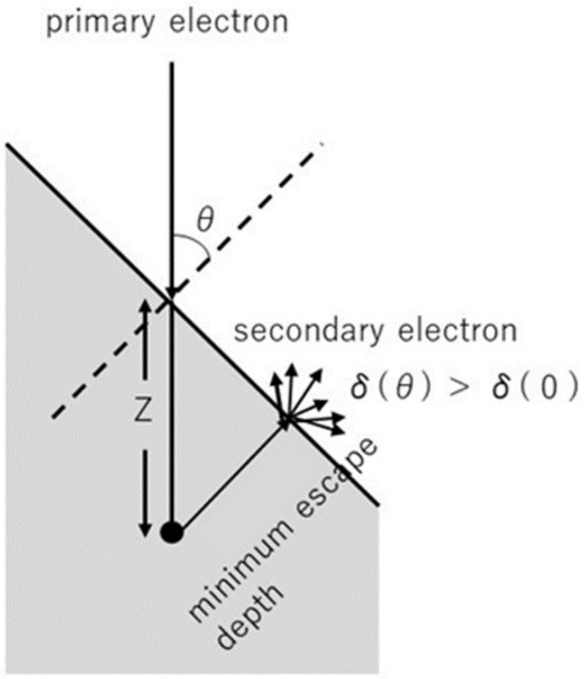
Secondary electrons (SE)

the parameter δ, the secondary electron yield

Dependence on tilt angle

When the secondary electron coefficient is measured as a function of the specimen tilt angle, θ (i.e., the specimen inclination to the beam, where a tilt of 0° means that the beam is perpendicular to the surface), a monotonic increase with tilt is observed.





This increase in δ with θ can be understood from the geometric argument.

The theoretical minimum escape path (from the point of formation to the specimen surface) decreases as the tilt angle increases.

$$\cos(\theta) = s/Z$$

Since SE remains less in the sample they are less likely to be absorbed and δ increases.

$$\delta(\theta) \approx \frac{1}{\cos(\theta)}$$

Secondary electrons (SE)

Types of Secondary Electrons

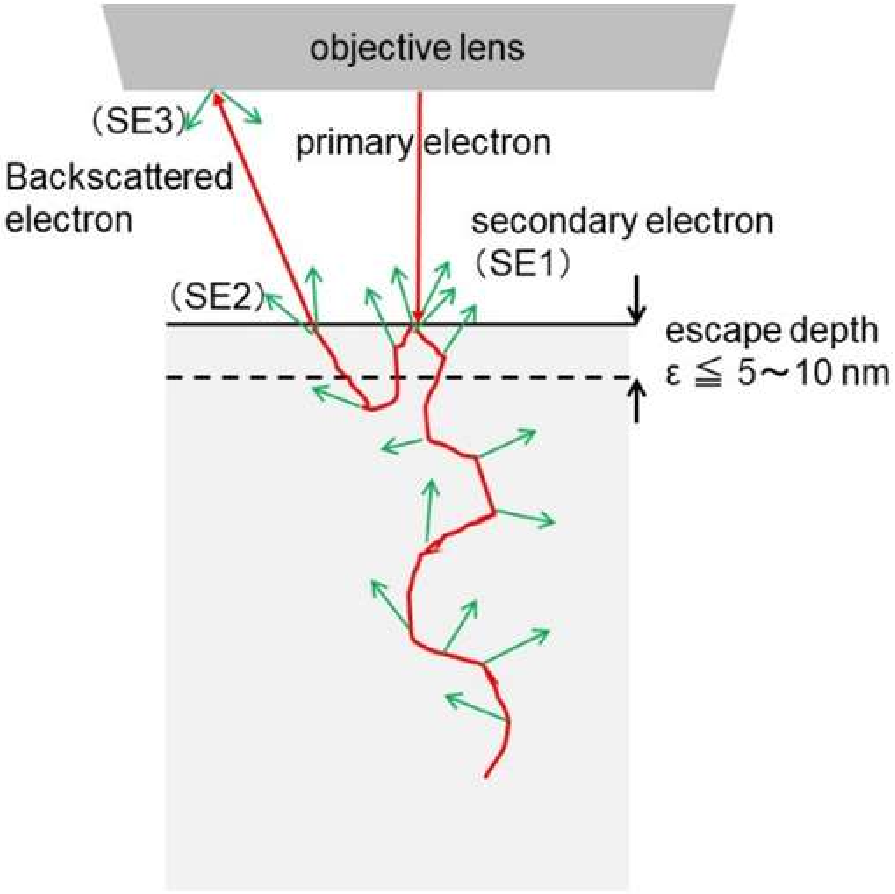
Please note: not all the secondary electrons are worth the same!

There are different 'classes' of secondary electron according to how they are produced.

SE1 are produced as the beam enters the sample, in the very narrow range close to the incoming electron beam. These are the 'high resolution' SE.

SE2 are produced by the BSE when they leave the specimen. Since BSE may have undergone multiple scattering events, E2 may originate far from the spot at which the primary electrons hit the specimen.

SE3 are produced by the BSE hitting the apparatus: lenses, chamber walls etc. They carry little if no information.



Secondary electrons (SE) Recap

- They are formed in inelastic scattering events between the primary electrons and the atoms in the specimen. They actually are the electrons from the specimen atoms, typically outer shell electrons, that are ‘hit’ by the primary electrons and acquire a certain kinetic energy.

- Are low energy electrons, their kinetic energy is in the order of few eV, in general below 50 eV and typically in the range of 1-15 eV.

- Their yield, is the secondary electron coefficient δ, δ can be higher than 1. δ depends on the atomic number but the dependence is weak. δ also depends on the accelerating voltage. Below a few hundreds eV in increases with the increasing of the accelerating voltage while above this threshold it starts decreasing.

- The yield of SE increases when the specimen surface is tilted respect to the impinging beam.

- The SE originate from a small fraction of the interaction volume, at a depth of about 1 nm. SE generated deeper inside the specimen do not have enough kinetic energy to leave the sample. This explains the behavior of δ as a function of the accelerating voltage.

X-Rays emission (Characteristic and continuous)

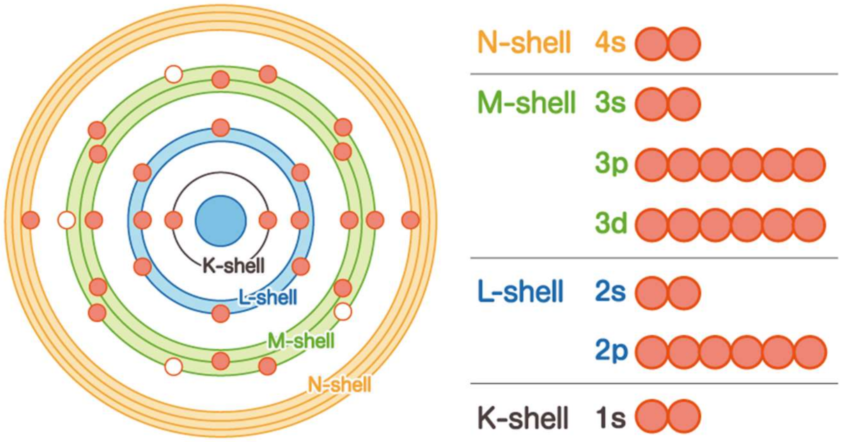
Interaction of the electron beams with materials

X-ray emission

Beforehand we need to consider that a different nomenclature of electron energy levels in atoms is required.

Electrons are divided in ‘shells’ : K, L, M, N. Luckily it is easy to map what we know to this different nomenclature

Shell K: all electrons with principal quantum number **1** (1s) 2 electrons in total;
Shell L: all electrons with principal quantum number **2** (2s, 2p) 8 electrons in total;
Shell M: all electrons with principal quantum number **3** (3s, 3p, 3d) 18 electrons in total;
Shell N: all electrons with principal quantum number **4** (4s, 4p, 4d, 4f) 32 electrons in total.



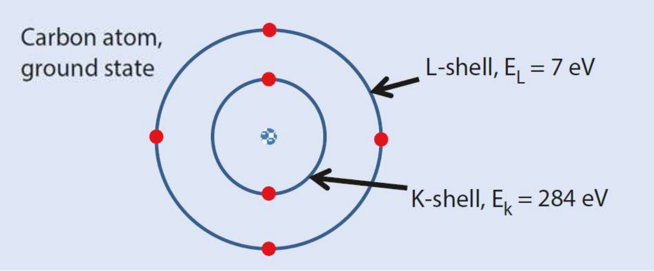
Schematic Diagram of the Electron Orbit of an Iron Atom

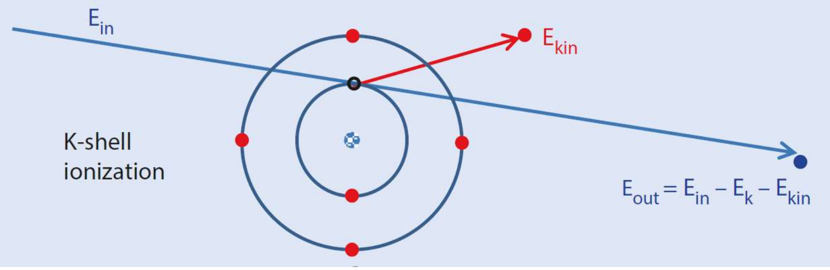
Interaction of the electron beams with materials

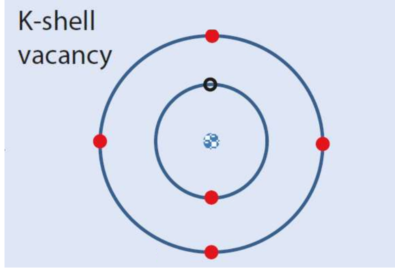
X-ray emission

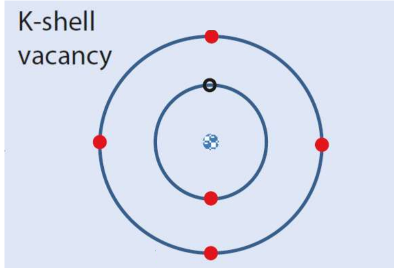
Where does characteristic X-ray emission come from?

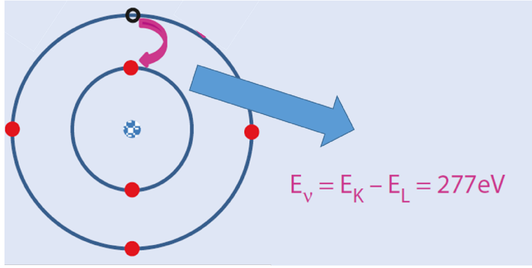
When electrons are displaced from a specific shell of an atom in the sample, X-rays of a well-defined wavelength are emitted. The wavelength of the emitted x-rays is specific for the element involved.











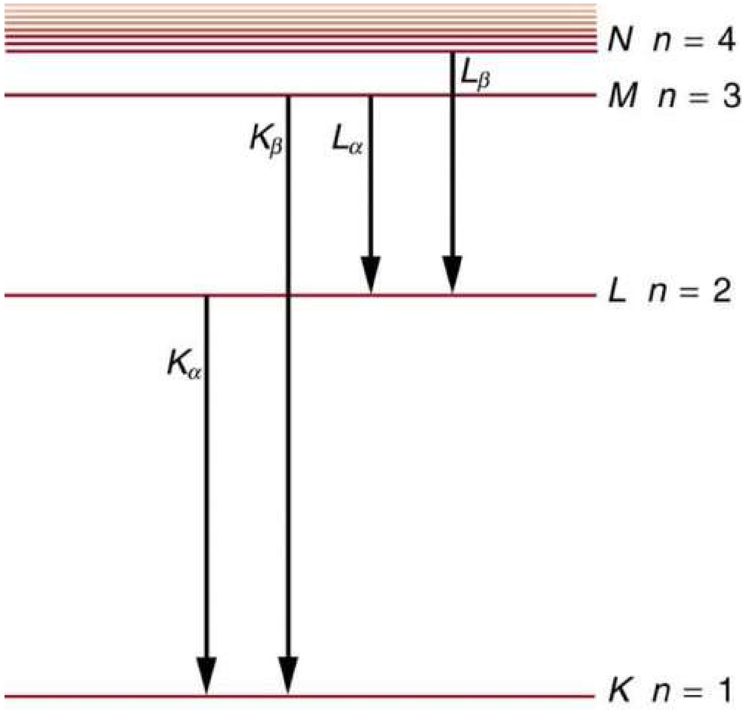
X-ray emission

X-rays come from the whole interaction volume which may have sizes in the order of microns.

Interaction of the electron beams with materials

X-ray emission

Types of emitted lines



Some nomenclature

K-alpha emission lines (Kα) result when an electron transitions to a vacancy in the innermost "K" shell (principal quantum number n = 1) from an electron in the "L" shell (n = 2), leaving a vacancy there.

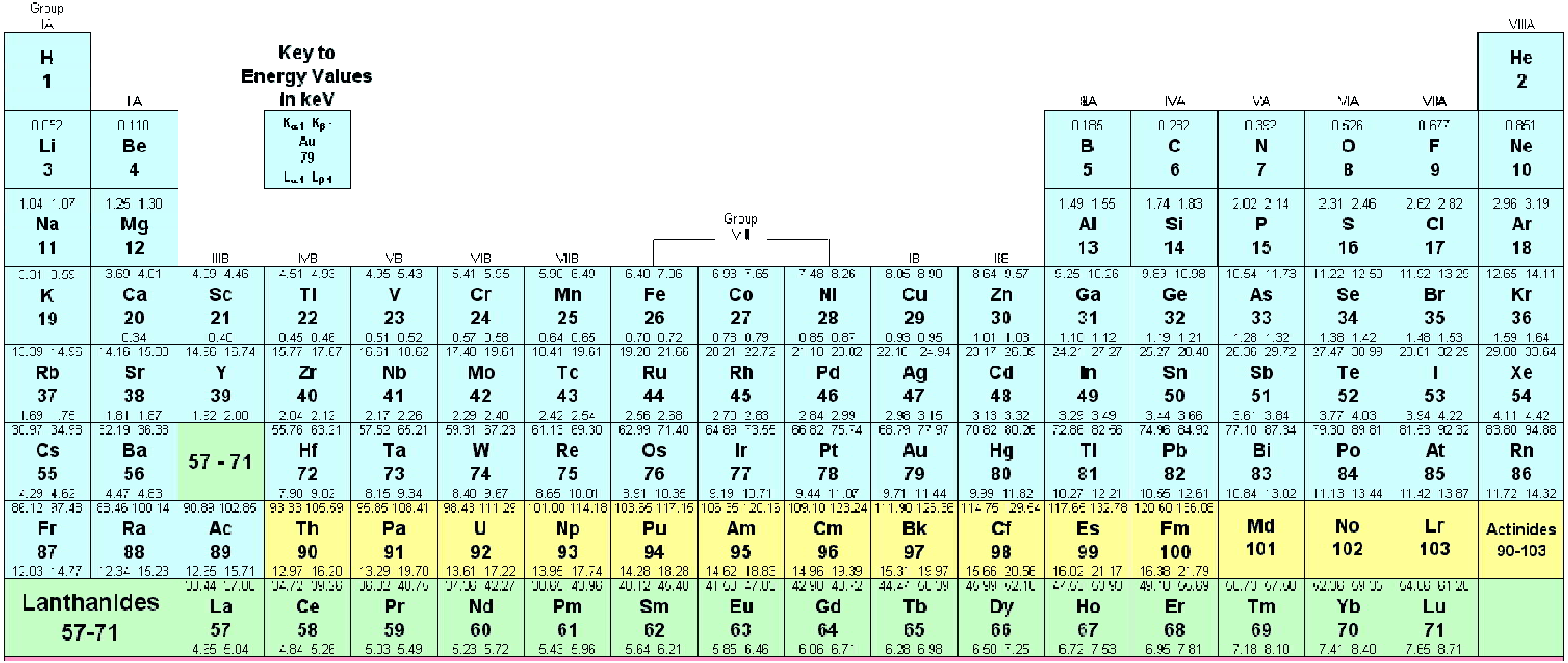
K-beta emissions (Kβ), similar to K-alpha emissions, result when an electron transitions to the innermost "K" shell (principal quantum number 1) from an electron in the "M" shell (with principal quantum number 3).

L-alpha (Lα) line result when an M shell electron fills a vacancy in the L shell and the L-beta (Lβ) line results when an N shell electron fills an L shell vacancy.

Since the energy difference between shells is specific for each element, the x-rays emitted (their energies) are informative of the composition of the sample.

Characteristic X-rays

Characteristic X-ray lines may have energies from as low as 1 keV for lighter elements up to about 100 keV for very heavy elements. If we want to excite a given transition, the energy of the primary electrons needs to higher than the energy of this transition.



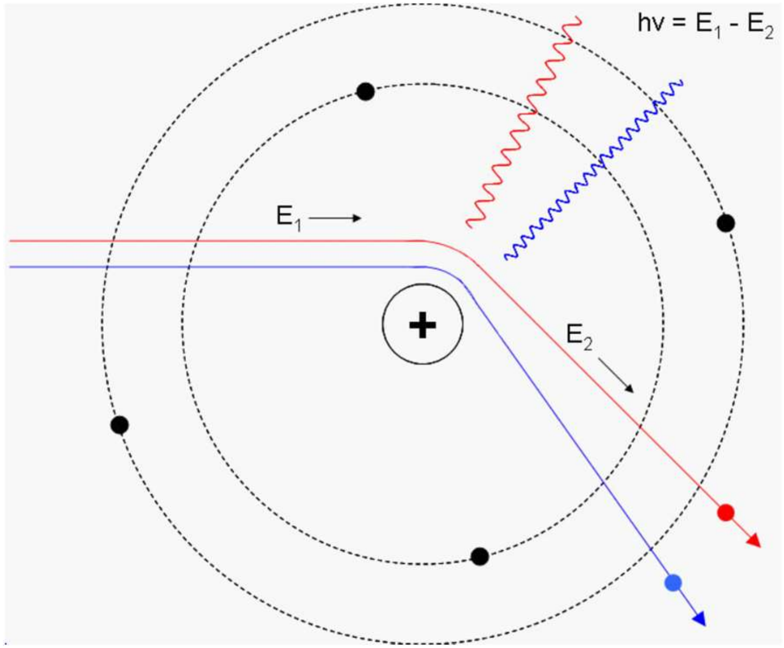
Group IA	IA	IIIB	IVB	VB	VIB	VIIB	Group VIII			IB	IIB	IIIA	IVA	VA	VIA	VIIA	VIIIA
H **1**			**Key to Energy Values in keV**														**He** **2**
0.052 **Li** **3**	0.110 **Be** **4**		$K_{\alpha1}$ $K_{\beta1}$ Au 79 $L_{\alpha1}$ $L_{\beta1}$									0.185 **B** **5**	0.232 **C** **6**	0.392 **N** **7**	0.526 **O** **8**	0.677 **F** **9**	0.851 **Ne** **10**
1.04 1.07 **Na** **11**	1.25 1.30 **Mg** **12**											1.49 1.55 **Al** **13**	1.74 1.83 **Si** **14**	2.02 2.14 **P** **15**	2.31 2.46 **S** **16**	2.62 2.82 **Cl** **17**	2.96 3.19 **Ar** **18**
3.31 3.59 **K** **19**	3.69 4.01 **Ca** **20** 0.34	4.09 4.46 **Sc** **21** 0.40	4.51 4.93 **Ti** **22** 0.45 0.46	4.95 5.43 **V** **23** 0.51 0.52	5.41 5.95 **Cr** **24** 0.57 0.58	5.90 6.49 **Mn** **25** 0.64 0.65	6.40 7.06 **Fe** **26** 0.70 0.72	6.93 7.65 **Co** **27** 0.78 0.79	7.48 8.26 **Ni** **28** 0.85 0.87	8.05 8.90 **Cu** **29** 0.93 0.95	8.64 9.57 **Zn** **30** 1.01 1.03	9.25 10.26 **Ga** **31** 1.10 1.12	9.89 10.98 **Ge** **32** 1.19 1.21	10.54 11.73 **As** **33** 1.28 1.32	11.22 12.50 **Se** **34** 1.38 1.42	11.92 13.29 **Br** **35** 1.48 1.53	12.65 14.11 **Kr** **36** 1.59 1.64
13.39 14.96 **Rb** **37** 1.69 1.75	14.16 15.83 **Sr** **38** 1.81 1.87	14.96 16.74 **Y** **39** 1.92 2.00	15.77 17.67 **Zr** **40** 2.04 2.12	16.61 18.62 **Nb** **41** 2.17 2.26	17.48 19.61 **Mo** **42** 2.29 2.40	18.41 19.61 **Tc** **43** 2.42 2.54	19.28 21.66 **Ru** **44** 2.56 2.68	20.21 22.72 **Rh** **45** 2.70 2.83	21.18 23.82 **Pd** **46** 2.84 2.99	22.16 24.94 **Ag** **47** 2.98 3.15	23.17 26.09 **Cd** **48** 3.13 3.32	24.21 27.27 **In** **49** 3.29 3.49	25.27 28.48 **Sn** **50** 3.44 3.66	26.36 29.72 **Sb** **51** 3.61 3.84	27.47 30.99 **Te** **52** 3.77 4.03	28.61 32.29 **I** **53** 3.94 4.22	29.80 33.64 **Xe** **54** 4.11 4.42
30.97 34.98 **Cs** **55** 4.29 4.62	32.19 36.38 **Ba** **56** 4.47 4.83	**57 - 71**	55.76 63.21 **Hf** **72** 7.90 9.02	57.52 65.21 **Ta** **73** 8.15 9.34	59.31 67.23 **W** **74** 8.40 9.67	61.13 69.30 **Re** **75** 8.65 10.01	62.99 71.40 **Os** **76** 8.91 10.35	64.89 73.55 **Ir** **77** 9.19 10.71	66.82 75.74 **Pt** **78** 9.44 11.07	68.79 77.97 **Au** **79** 9.71 11.44	70.82 80.26 **Hg** **80** 9.99 11.82	72.86 82.56 **Tl** **81** 10.27 12.21	74.96 84.92 **Pb** **82** 10.55 12.61	77.10 87.34 **Bi** **83** 10.84 13.02	79.30 89.81 **Po** **84** 11.13 13.44	81.53 92.32 **At** **85** 11.42 13.87	83.80 94.88 **Rn** **86** 11.72 14.32
86.12 97.48 **Fr** **87** 12.03 14.77	88.46 100.14 **Ra** **88** 12.34 15.23	90.89 102.85 **Ac** **89** 12.65 15.71	93.33 105.59 **Th** **90** 12.97 16.20	95.85 108.41 **Pa** **91** 13.29 19.70	98.43 111.29 **U** **92** 13.61 17.22	101.00 114.18 **Np** **93** 13.95 17.74	103.65 117.15 **Pu** **94** 14.28 18.28	106.35 120.16 **Am** **95** 14.62 18.83	109.10 123.24 **Cm** **96** 14.96 19.39	111.90 126.36 **Bk** **97** 15.31 19.97	114.75 129.54 **Cf** **98** 15.66 20.56	117.65 132.78 **Es** **99** 16.02 21.17	120.60 136.08 **Fm** **100** 16.38 21.79	**Md** **101**	**No** **102**	**Lr** **103**	**Actinides** **90-103**
Lanthanides **57-71**		33.44 37.80 **La** **57** 4.65 5.04	34.72 39.26 **Ce** **58** 4.84 5.26	36.03 40.75 **Pr** **59** 5.03 5.49	37.36 42.27 **Nd** **60** 5.23 5.72	38.65 43.96 **Pm** **61** 5.43 5.96	40.12 45.40 **Sm** **62** 5.64 6.21	41.53 47.03 **Eu** **63** 5.85 6.46	42.98 48.72 **Gd** **64** 6.06 6.71	44.47 50.39 **Tb** **65** 6.28 6.98	45.99 52.18 **Dy** **66** 6.50 7.25	47.53 53.93 **Ho** **67** 6.72 7.53	49.10 55.69 **Er** **68** 6.95 7.81	50.73 57.58 **Tm** **69** 7.18 8.10	52.36 59.35 **Yb** **70** 7.41 8.40	54.06 61.28 **Lu** **71** 7.65 8.71	

Interaction of the electron beams with materials

X-ray emission   The non characteristic Braking radiation or ‘Bremsstrahlung’

Simultaneously with the inner shell ionization events that lead to characteristic X-ray emission, a second physical process operates to generate X-rays, the “braking radiation,” or bremsstrahlung.

Because of the repulsion that the beam electron experiences in the negative charge cloud of the atomic electrons, it undergoes deceleration and loses kinetic energy, which is released as a photon of electromagnetic radiation.

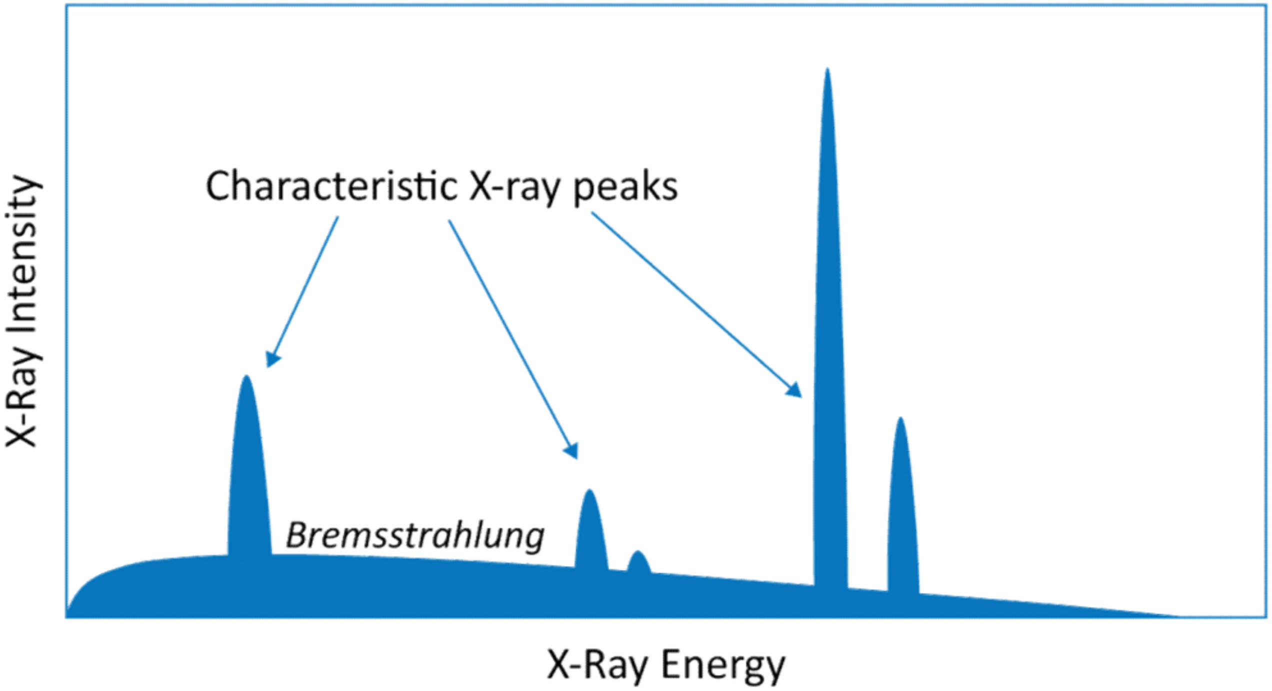


The braking radiation process thus forms a continuous energy spectrum, also referred to as the “X-ray continuum,” from 100 eV to E0, which is the so-called Duane–Hunt limit.

The X-ray continuum forms a background beneath any characteristic X-rays produced by the atoms.

Interaction of the electron beams with materials

X-ray emission, the overall spectrum



The X-ray continuum forms a background beneath any characteristic X-rays produced by the atoms.

X-Rays emission Recap

- X-rays can be characteristic or continuous. The characteristic X-rays are due to electron transitions after a PE ejects a inner core electron of the specimen atoms creating a vacancy. Continuous X-rays are due to the deceleration of PE by the electric fields generated by the atom electrons of the specimen.

- The continuous radiation ‘Bremsstrahlung’ cover a range of 100 eV up to the energy of the primary electrons .

- The characteristic X-rays are discrete, their energy depends on what element is present and therefore and they carry information on composition.

- The lines are named Xω according to the shell in which the vacancy was created and the shell from which the electron filling the vacancy comes. The shells are named K, L, M, N, (principal quantum numbers 1, 2, 3, 4 respectively), these are X. The ω can be α if the electron comes from the next, β if they come from the second next shell, etc.

- The energies of the characteristic x-ray lines span an interval from 1 keV up to 100 keV, the energy of the primary electrons has to be higher than the energy of the x-ray we want to excite.

- X-rays come form the whole interaction volume (which may be several microns wide).

Contrast in SEM Images

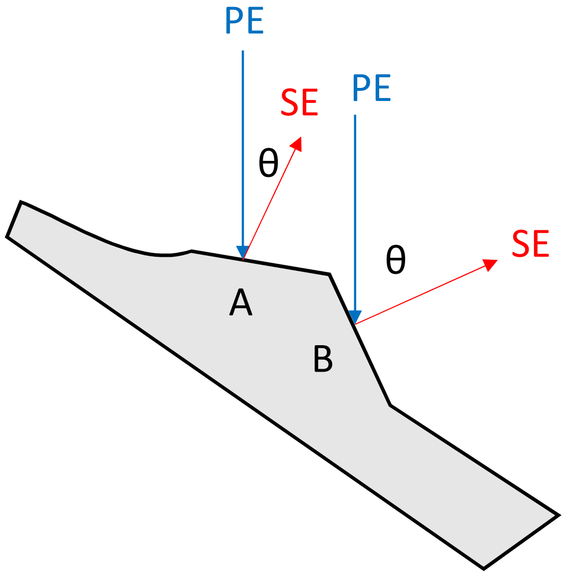
Contrast in SEM images

Topography contrast, Secondary electrons

In the SEM, the direction of the incident electrons is fixed, However, because of the surface features of the specimen, the angle of incidence of the primary electrons may be different from place to place. This is the source of what is known as topographic contrast. This is also a very important application of SEM.

It is the monotonic dependence of the secondary electron coefficient on the local surface inclination is an important factor in producing topographic contrast that reveals the shape of an object.

$$\delta(\theta) \approx \frac{1}{\cos(\theta)}$$



A SE detector positioned sideways receives more SE from spot B respect to spot A, B will be brighter than A

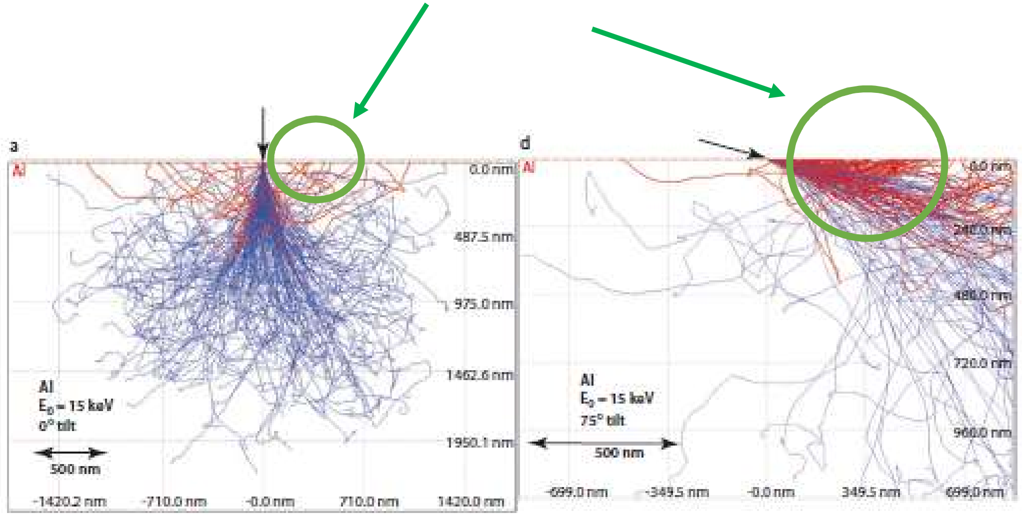
To obtain topographic contrast it may also be useful to tilt the sample

Contrast in SEM images

Topography contrast, Backscattered electrons

Remember that is easier for BSE to escape at higher tilt angles.

More BSE escape at high tilt angles.



However they may also escape from a larger surface area and therefore this has a negative impact on the image resolution.

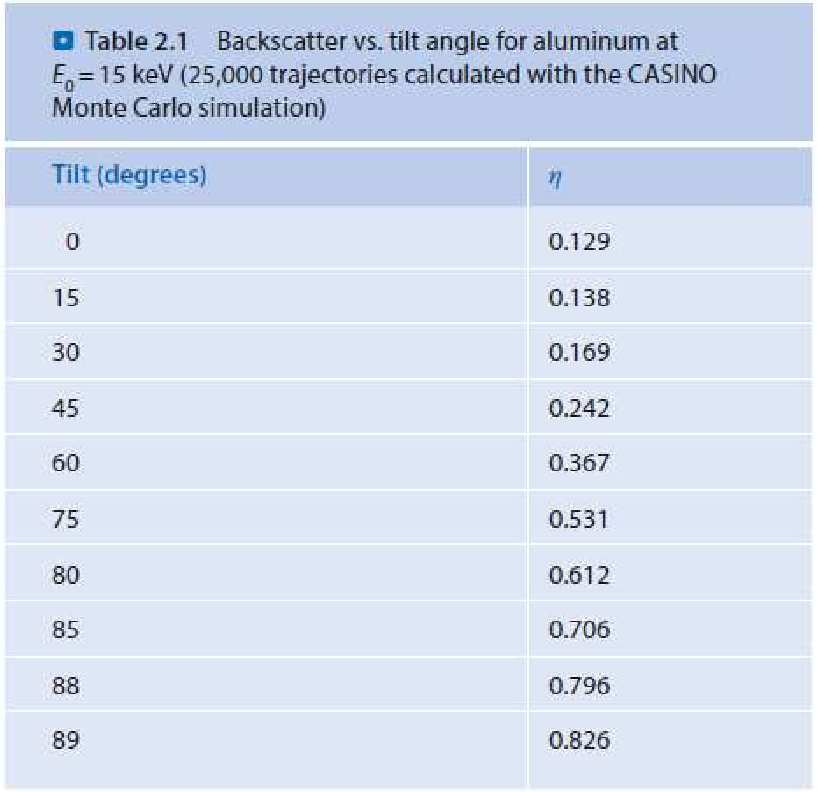


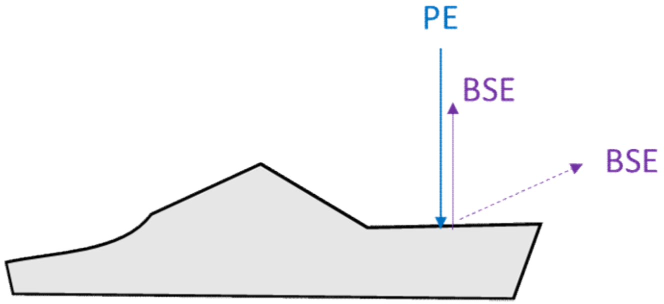
Table 2.1 Backscatter vs. tilt angle for aluminum at $E_0 = 15$ keV (25,000 trajectories calculated with the CASINO Monte Carlo simulation)

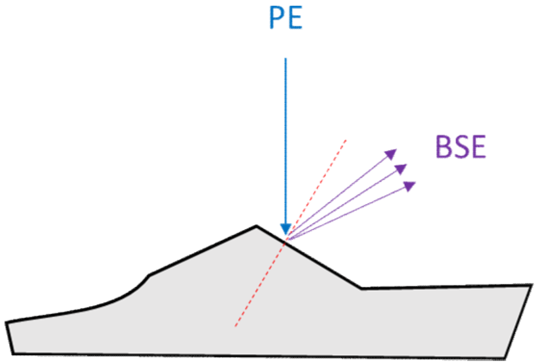
Tilt (degrees)	η
0	0.129
15	0.138
30	0.169
45	0.242
60	0.367
75	0.531
80	0.612
85	0.706
88	0.796
89	0.826

Contrast in SEM images

Topography contrast, Backscattered electrons

Since more electrons escape when inclined incidence occurs, surfaces inclined respect to the primary electron beam appear brighter than those orthogonal.



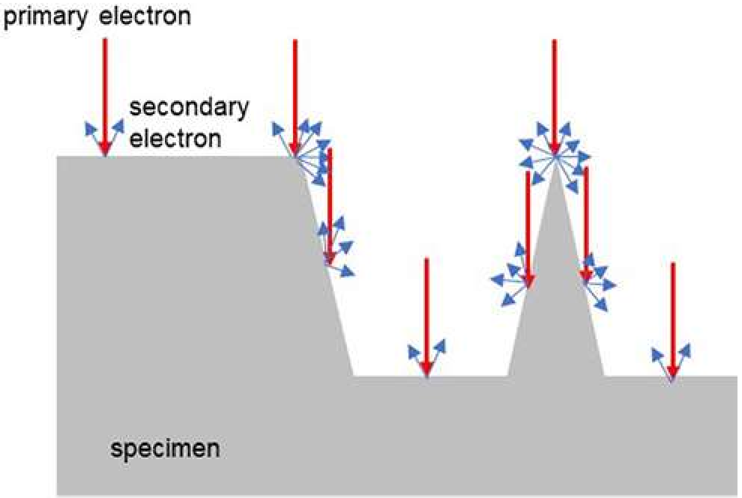


but due to the deep exit depth of the backscattered electrons and the relatively large emission area, the spatial resolution is much lower than that of the secondary electrons, and its three-dimensional perception is not as good as that of the secondary electrons

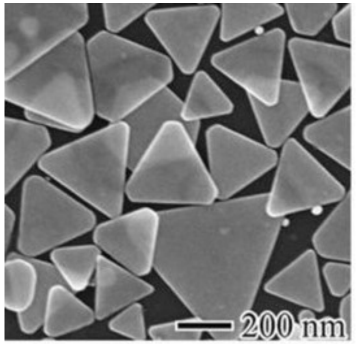
Contrast in SEM images

Edge and tip effects

At tips, sharp steps or steep inclined planes, there are more efficient paths for secondary electrons to escape from the surface than at normal incidence, so the yield of secondary electrons tends to be much higher than in normal planes. This phenomenon gives rise to very bright areas.

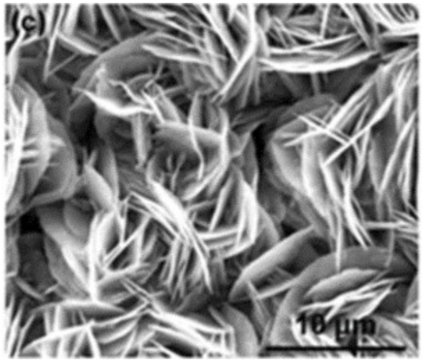


Given the dependence on the SE coefficient on the tilt angle or surface inclination respect to the electron beam, there are obvious edge effects and tip effects in the topography contrast of secondary electrons but also backscattered electrons



Ag nanoparticles

small 2014, 10, 1430–1437



Ag nanosheets

ACS Appl. Mater. Interfaces 2012, 4, 2752–2756

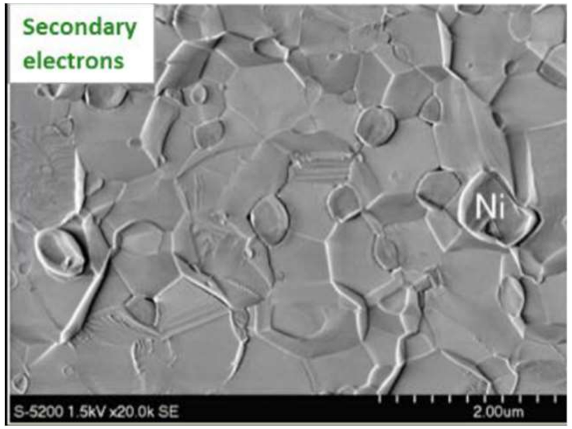
Contrast in SEM images

Atomic number Z

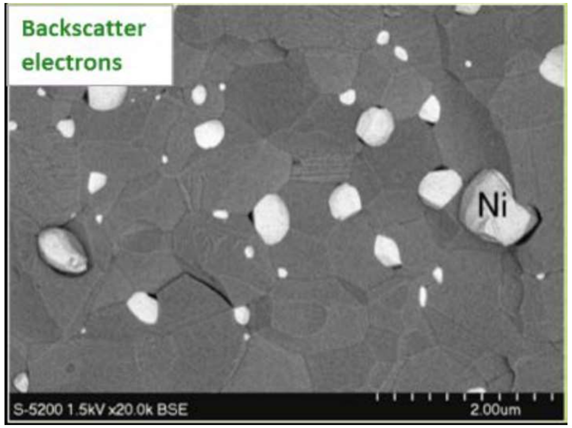
Remember that the SE yield of different substances is different. However he dependence is weak and, in practice, when the atomic number Z is greater than 20, the secondary electron yield basically does not change with the change of atomic number.

See images, $_{28}$Ni against $_{13}$Al. The SE image at top does nor allow differentiating the two elements in the composite. On the contrary the BSE image at the bottom clearly spots Ni on the aluminium oxide substrate.

Therefore SE are not very good to obtain compositional contrast. In this case better resorting to BSE imaging.



Ni on aluminium oxide



Ni on aluminium oxide