Skaneeriv elektronmikroskoopia (SEM)

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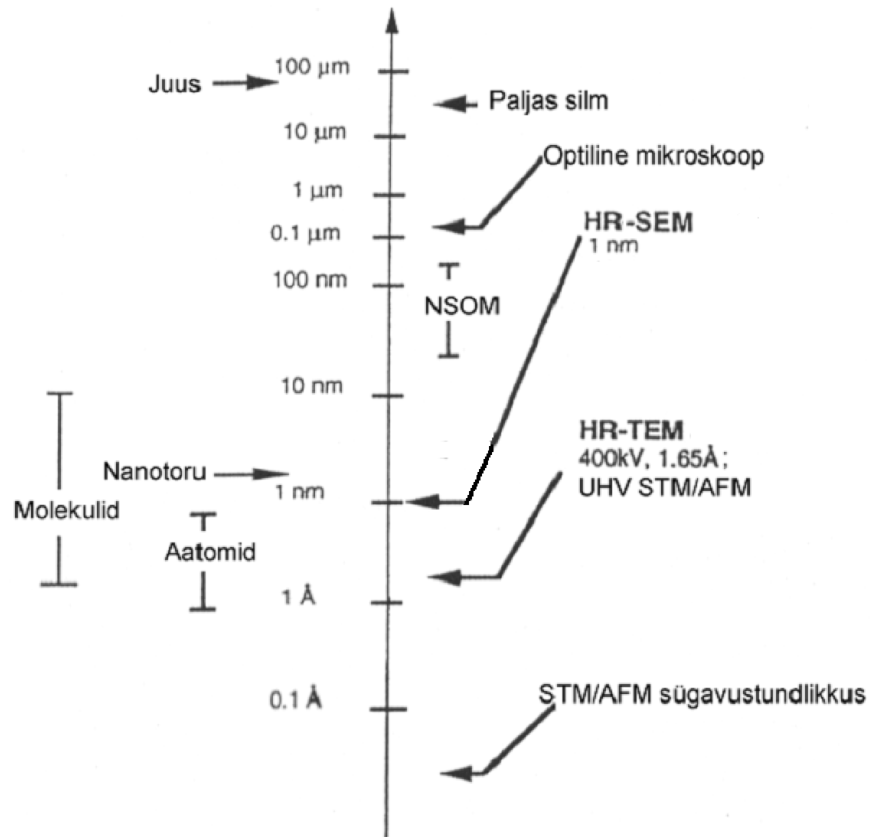
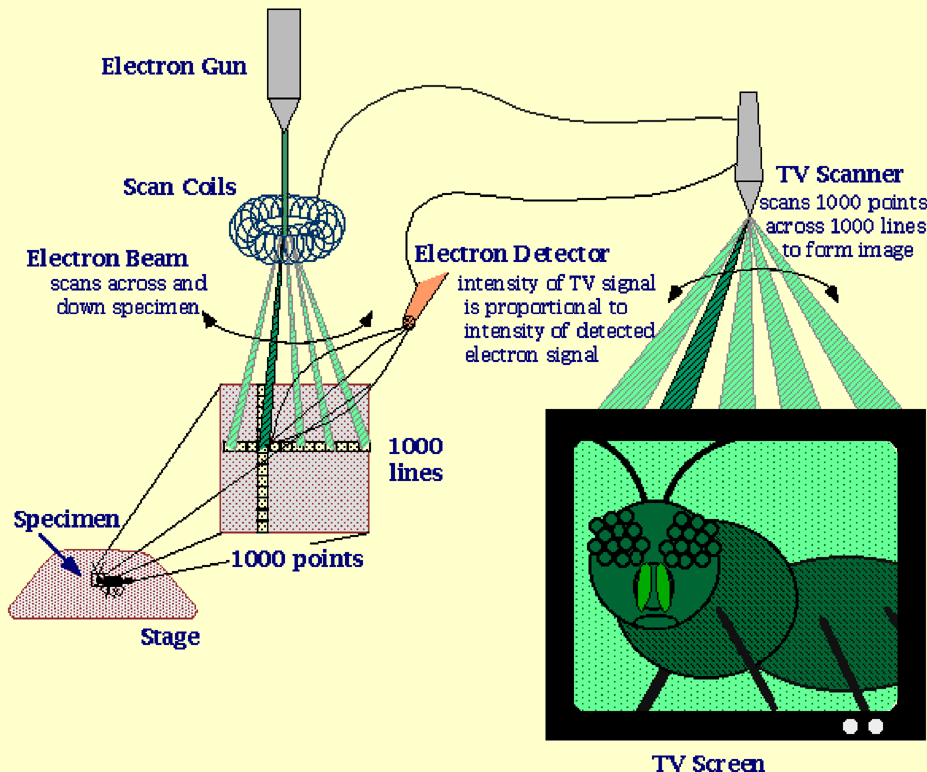
FKFE, anorgaanilise keemia õppetool

Millest tuleb juttu

- Skaneeriva elektronmikroskoobi tööpõhimõte.
- Sekundaarelektronide signaal.
- Peegeldunud elektronide signaal.
- Röntgenkiirguse signaal.
- Teised signaali liigid.
- Kaasaegse SEM suundumused.


Mikroskoopia skaalad
Juus
100 µm
Paljas silm
10 µm
Optiline mikroskoop
1 µm
0.1 µm
HR-SEM
1 nm
100 nm
NSOM
10 nm
HR-TEM
400kV, 1.65Å;
UHV STM/AFM
Nanotoru
1 nm
Molekulid
Aatomid
1 Å
0.1 Å
STM/AFM sügavustundlikkus

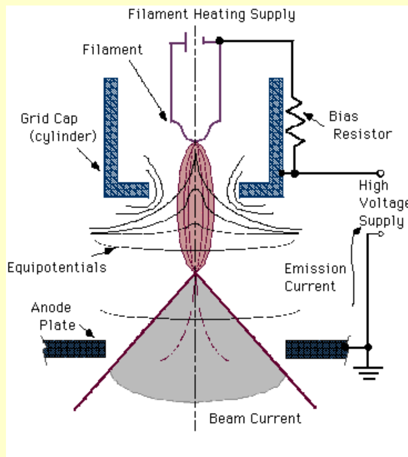
Elektronkiire skaneerimine

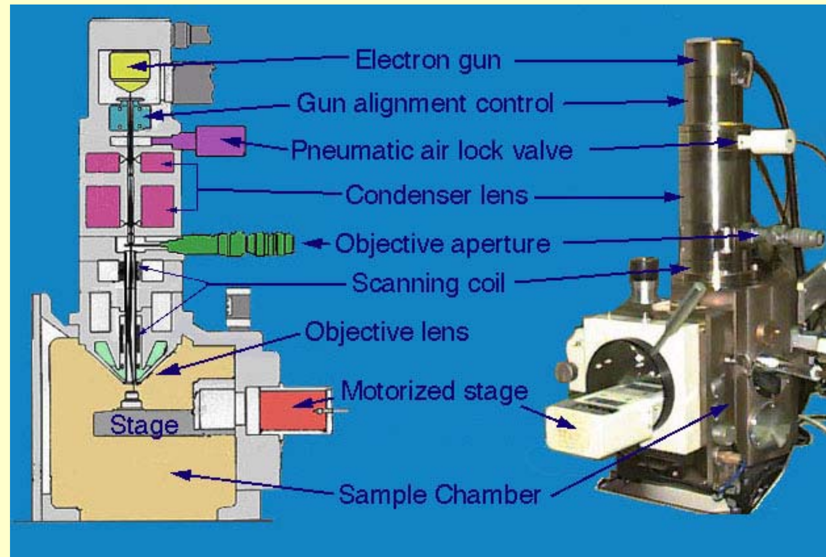


- Elektronsondi formeerimise moodul
- 2 sünkroonselt skaneerivat elektronkiirt
- kujutise punkt-punktiline ülekandmine

Elektron- ja ioonmikroskoopides teostatakse skaneerimine elektri-/magnetvälja abil.

…veidi lähemalt





Elektronallika kujutise läbimõõt:

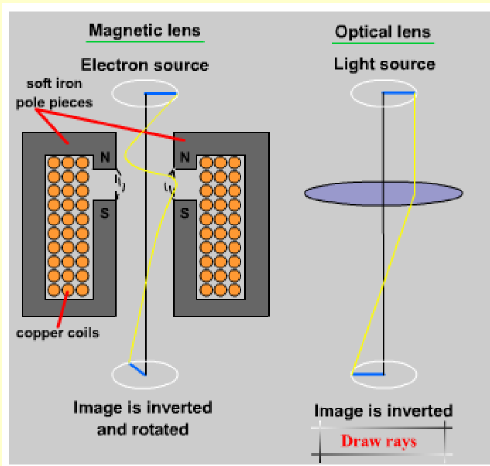
$$d_g = \frac{2}{\pi}\sqrt{\frac{i}{\beta}}\frac{1}{\alpha} \quad ,$$

kus, i- emisiooni vool, β- allika heledus.

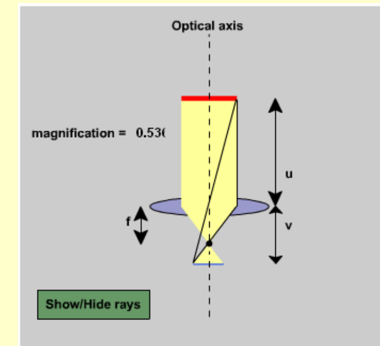
Elektronvoolu tihedus, mida emiteerib katood:

$$J_c = CT^2 e^{-e\Phi}/kT$$

kus, T- katoodi temperatuur, e- elektroni laeng, Φ- väljumistöö, k- Boltzmani konstant. Väljumistöö, Φ on 2.5 ja 4 eV, vastavalt LaB_6 ja W katoodi materjalidele.



Elektronläätsed



Õhukese läätse suurendus:

$$M = \frac{v}{u}$$

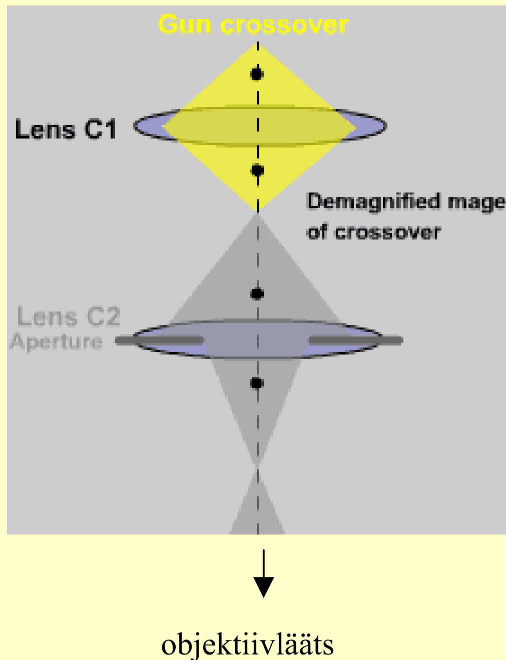
Fookuskaugus, f:

$$\frac{1}{f} = \frac{1}{u} + \frac{1}{v}$$

Kui mähise keerdusid läbib elektrivool, tekib tugev magnetväli, mis väljutatakse elektronoptilisse kanalisse läätsemagneti "ava" kaudu. See telgsümmeetriline väli töötab analoogiliselt õhukese koondava optilise läätsega, tuues teljest kaugemal olevad elektronid tagasi fookusesse. Sealjuures liiguvad teatud energiani, E_0, kiirendatud elektronid keeruka 3D trajektooriga läbi fookuse, projekteerides elektronide allika vähendatud kujutise vahetasandile, telje suhtes pööratuna. Selle pöörde suurus sõltub läätse tugevusest. Läätse fookuskaugust saab muuta läätse läbinud voolu tugevuse muutmisega.

http://www.matter.org.uk/tem/lenses/electromagnetic_lenses.htm

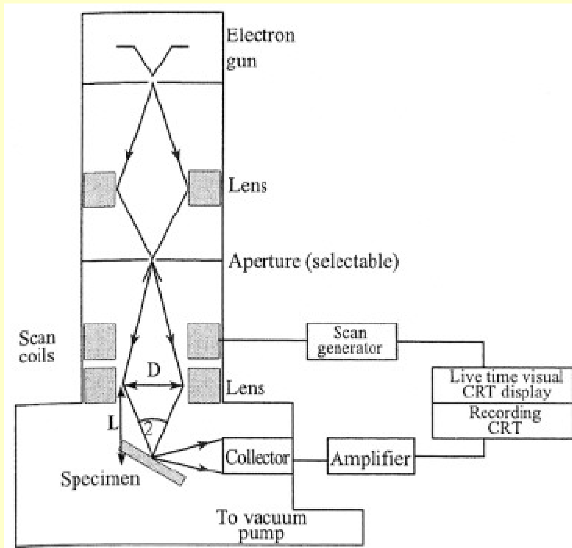
…elektronläätsed - kondensorläätsed

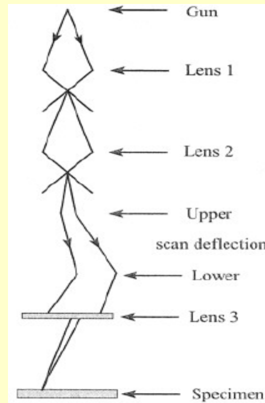


Kahekordne kondensorsüsteem koosneb 2-st kondensorlääät-sest, milline on kasutusel nii SEM, kui TEM elektronoptikas. Selle süsteemi ülesanne on kontrollida elektronsondi parameetreid: voolu suurust, sondi diameetrit ja konvergentsust (koonduvust).

Süsteemis ergastatakse kahte läätse korraga ning see töötab analoogiliselt 2-e järjestikuse õhukese optilise läätsega, millel põhimõtteliselt võivad olla erinevad fookuskaugused. Teise läätse sees on tavaliselt sondi piirav kondensorläätse ava.

…elektronläätsed

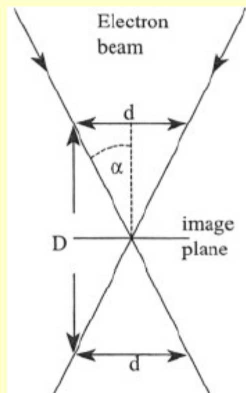




Kui TEM puhul suunab kondensorsüsteem elektronkiire objektile, siis SEM puhul on kondensorsüsteemi ja objekti vahel veel objektiivlääts, mis kontrollib sondi fokuseeringut objekti tasandil ning suurendab sondi konvergentsust.

Elektronkujutise hägusus on määratud voolutiheduse jaotusega sondi täpis objekti pinnal (optilise Airy ketta analoog). Olgu selle täpi läbimõõt *d*, siis kehtib seos:

$$(D/2)\tan\alpha = d/2$$



Kus, D- teljesuunaline nihe, mis väikeste α väärtuste puhul ligineb $D \sim d/\alpha$. Tavaliselt $\alpha \sim 10$ mrad. Suurus *D* võrdub sügavusteravusega, kui *d* ei ületa antud suurendusel oodatavat lahutusvõimet.

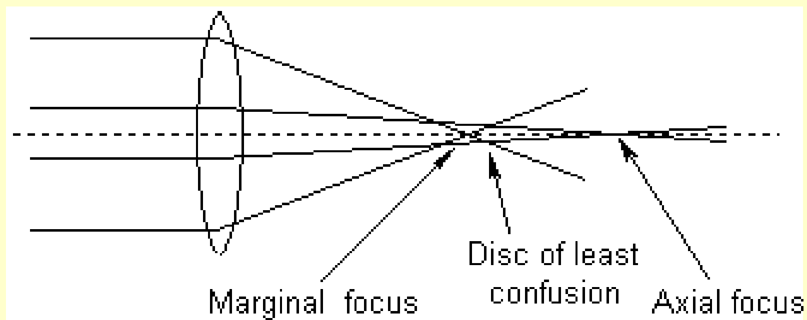
Sügavusteravus ja suurendus

Table 6.1. Depth of field and resolution of the SEM (final aperture 5×10^{-3} rad) compared with that achieved with the optical light microscope (Flewitt and Wild (1985)).

		Depth of field	
Magnification	Resolution	SEM	Optical
20	5 µm	1 mm	5 µm
100	1 µm	200 µm	2 µm
200	500 µm	100 µm	0.7 µm
1000	100 nm	20 µm	–
5000	20 nm	4 µm	–
10000	10 nm	2 µm	–

Suurendust kontrollitakse SEM puhul elektronsondi skaneerimisulatusega objektil!

Aberratsioonid elektronläätses



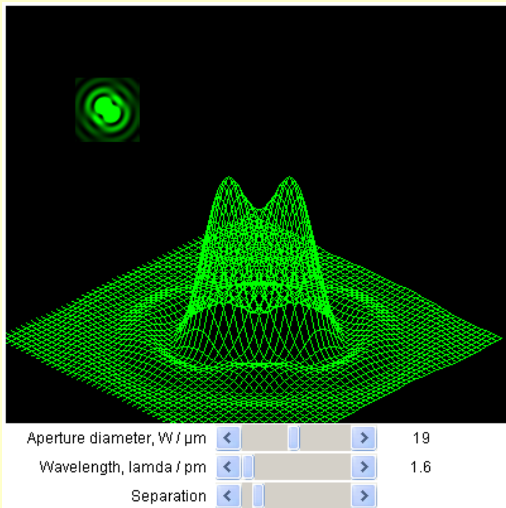
Sfääriline aberratsioon, $C_s \propto E_0$, f, α^3, kus: f- fookuskaugus. Sfäärilisest aberratsioonist tingitud e-sondi laienemine:

$$d_s = 0.5 C_s \alpha^3$$

…difraktsioon

Elektronide laineomadused põhjustavad elektronkiirte difraktsiooni avadel (vt. simulatsioonipilt; Raighley kriteerium). Sellest põhjustatud e-sondi laienemine on $\propto 1.5\ E_0^{-1/2}\ \alpha^{-1}$, ehk:

$$d_d = 1.22\frac{\lambda}{\alpha}$$

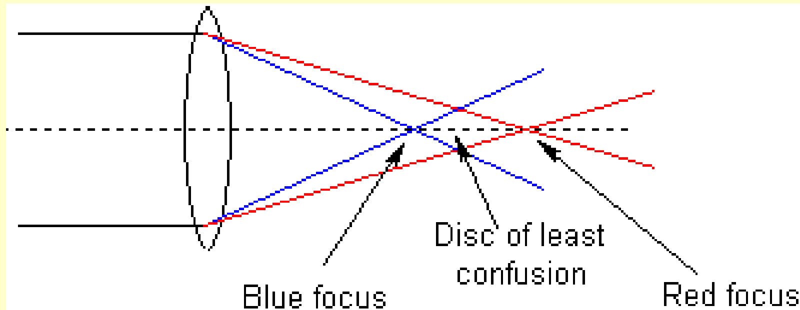


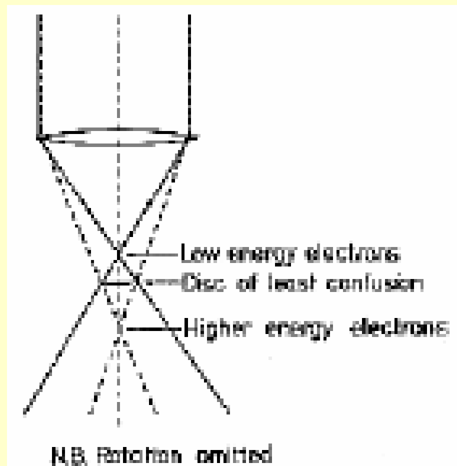
Kogu e-sondi laienemine avaldub:

$$d_t = \sqrt{d_s^2 + d_g^2 + d_d^2}.$$

http://www.matter.org.uk/tem/lenses/electromagnetic_lenses.htm

Kromaatiline aberratsioon



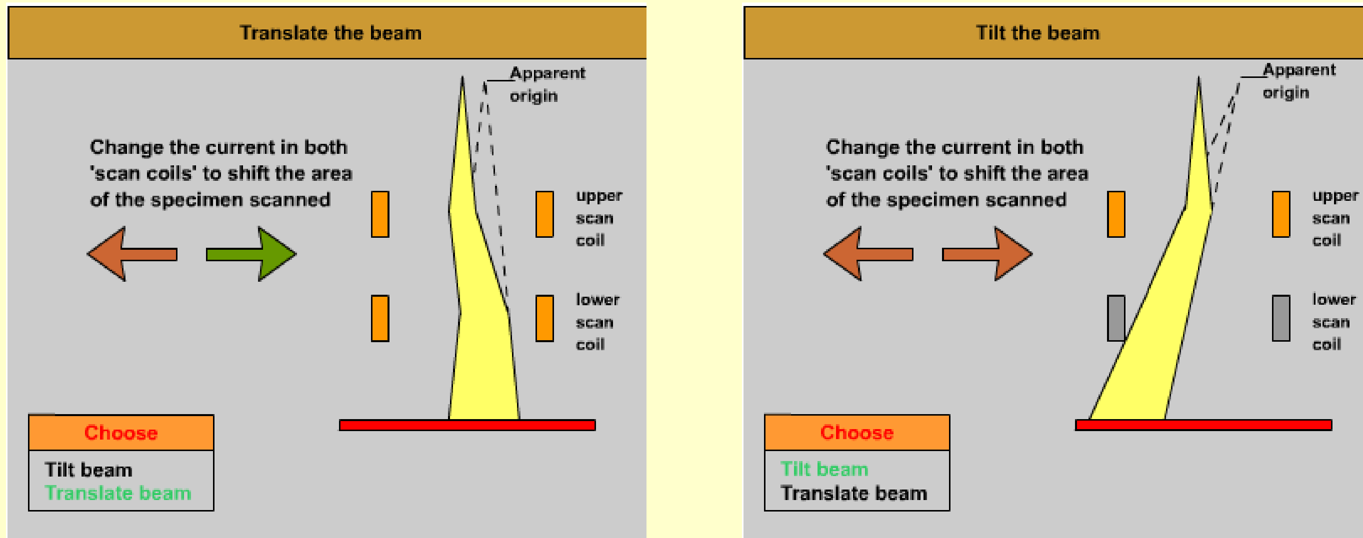


Kromaatiline aberratsioon: $d_\lambda = (\Delta E/E_0)\, C_\lambda\, \alpha$, kus C_λ- kromaatilise aberratsiooni koefitsient, mis sõltub lineaarselt läätse fookuskaugusest, f.

Lõplik sondi diameeter on seega:

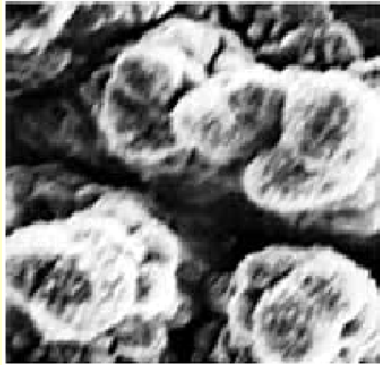
$$d = (d_t^2 + d_\lambda^2)^{1/2}.$$

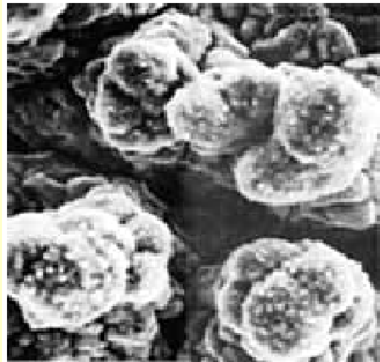
Skaneerimine

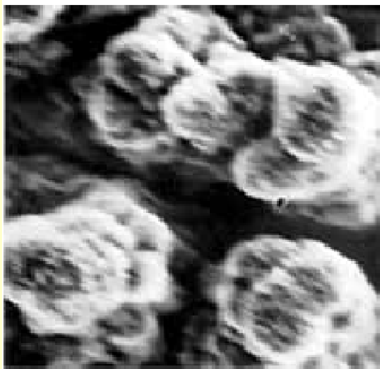


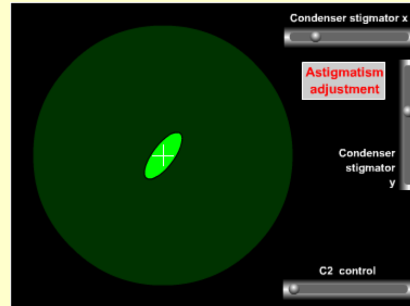
Kahekordne skaneerimismähis (Smith, 1956) lubab elektronkiirt nihutada optilise telje suhtes (skaneerida), ilma et muutuks kiire kaldenurk või siis kiirt kallutada, nii et ei muutuks kiire asukoht objektil.

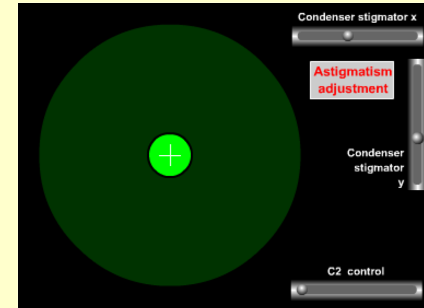
Stigmaator

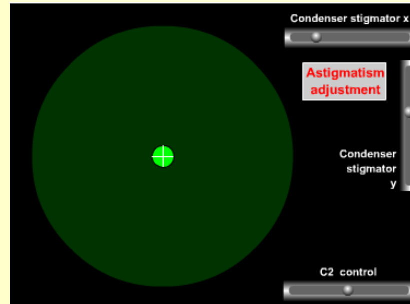


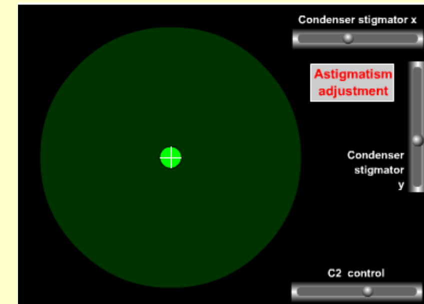


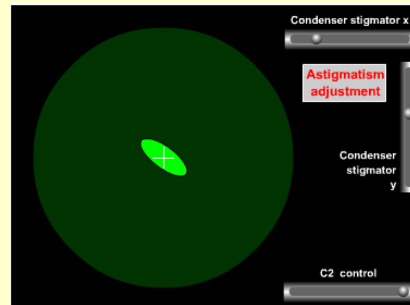


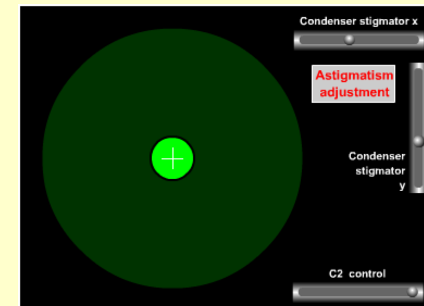




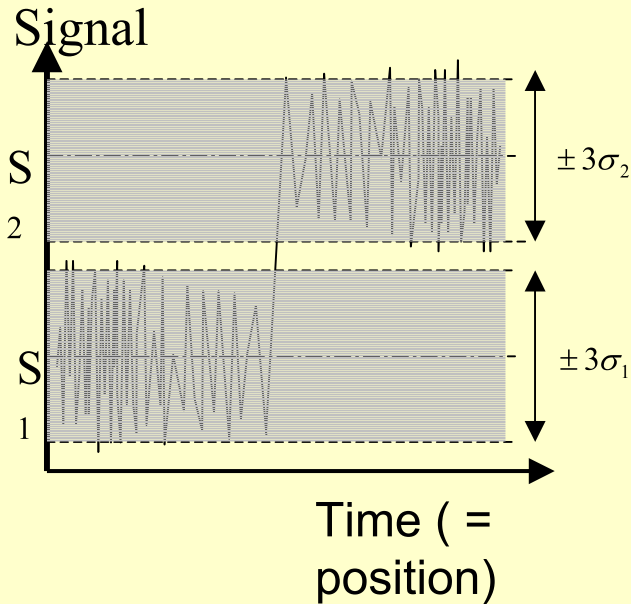








Mürad SEM kujutistes



Loendusstatistika: $\sigma = \sqrt{n}$

99% andmeid asub vahemikus ± 3σ.

Kontrast on defineeritud seostega:

$$C = \frac{\Delta S}{S_1}$$

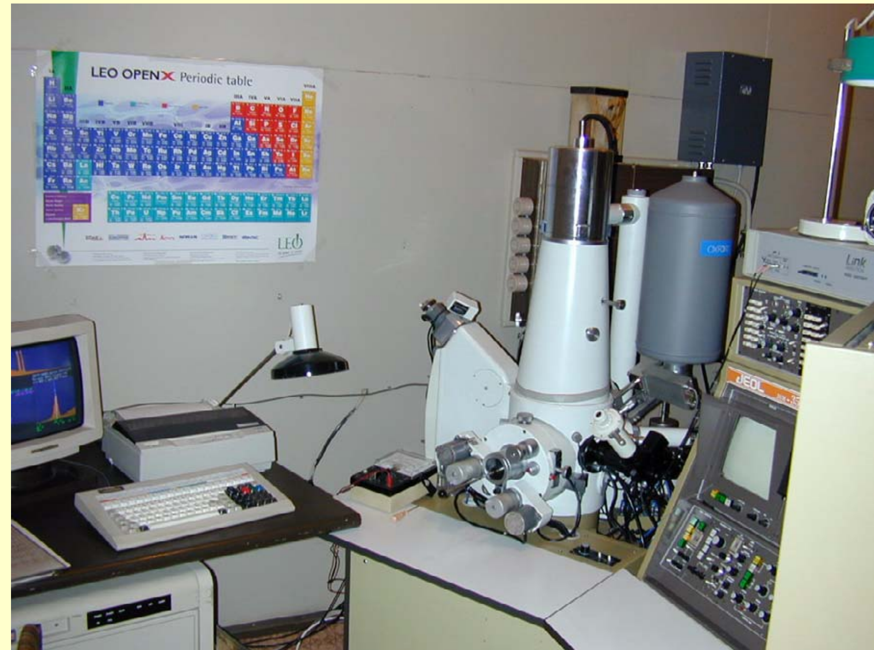
$$\Rightarrow C \geq 6\left(\frac{\sigma_s}{S}\right)$$

$$S_1 + 3\sigma_1 \leq S_2 - 3\sigma_2$$

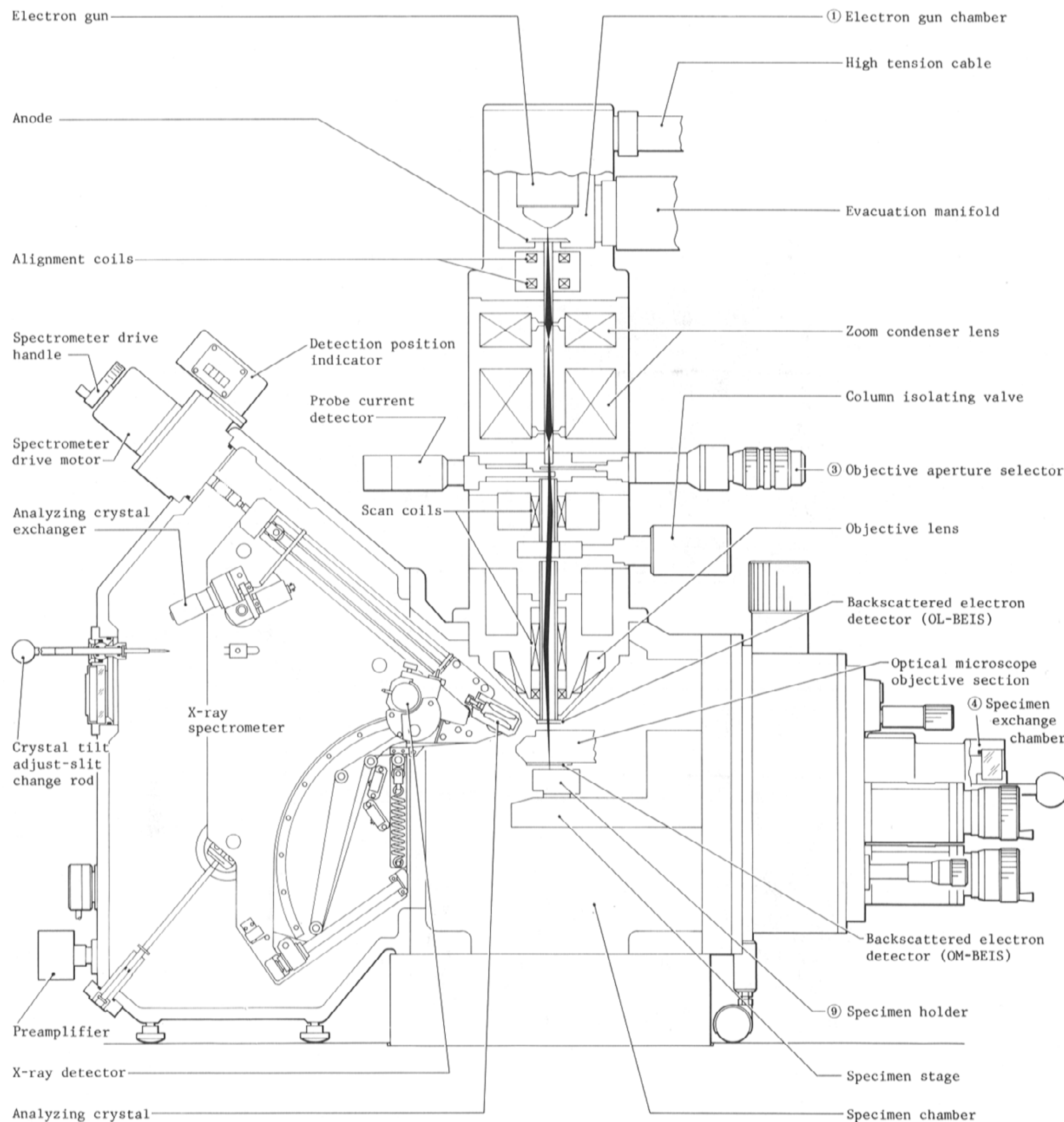
SEM puhul on mürasid (σ) võimalik vähendada suurendades signaali kogumise aega pikselis!

Skaneerivad elektronmikroskoobid TÜ-s

JXA-840A + 2xWDS+EDS



JSM-35CF+WDS+EDS

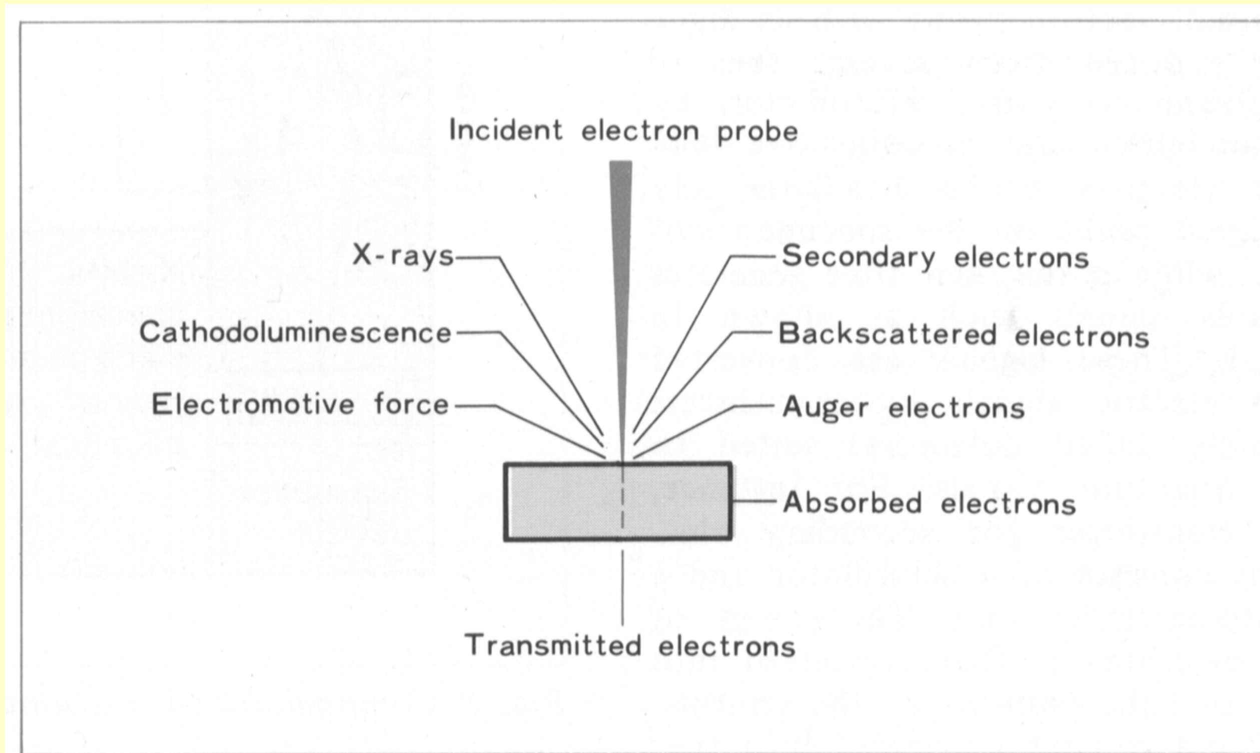


JSM-840 seestpoolt

Põhiosad:

- •Elektronkahur
- •Elektronoptika
- •Objektikamber + -laud + lüüsikamber
- •Detektorid + X-spektromeetrid (WDS, EDS)
- •Vaakum-süsteem

Elektronmikroskoobi signaalid

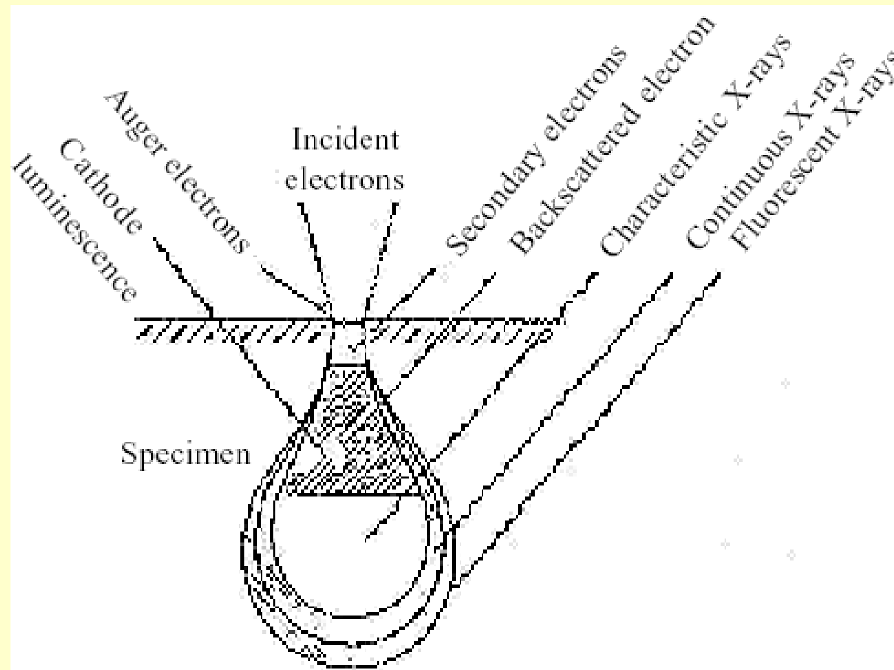


Interaktsiooni produktid

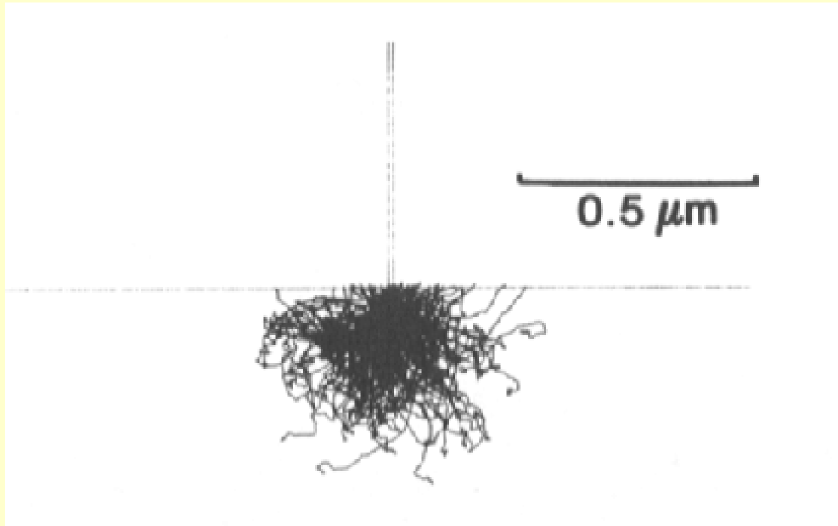
SE- 10-20 nm BE- 100-300 nm X-ray- 1-10 µm

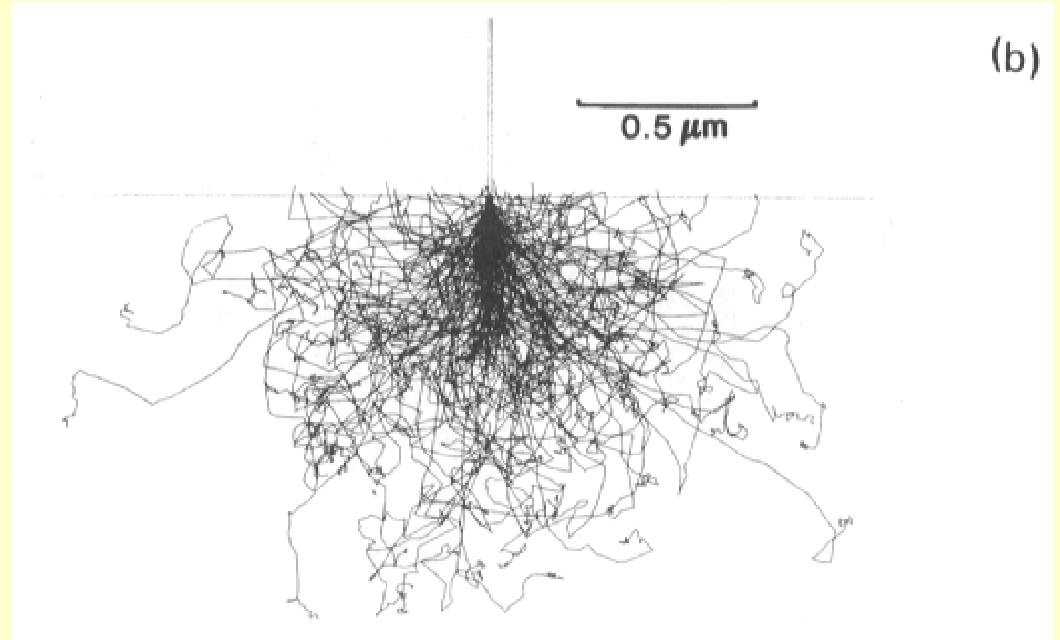
AE- 1-2 nm

CL- 1-5 µm



Elektronide hajumine objektis



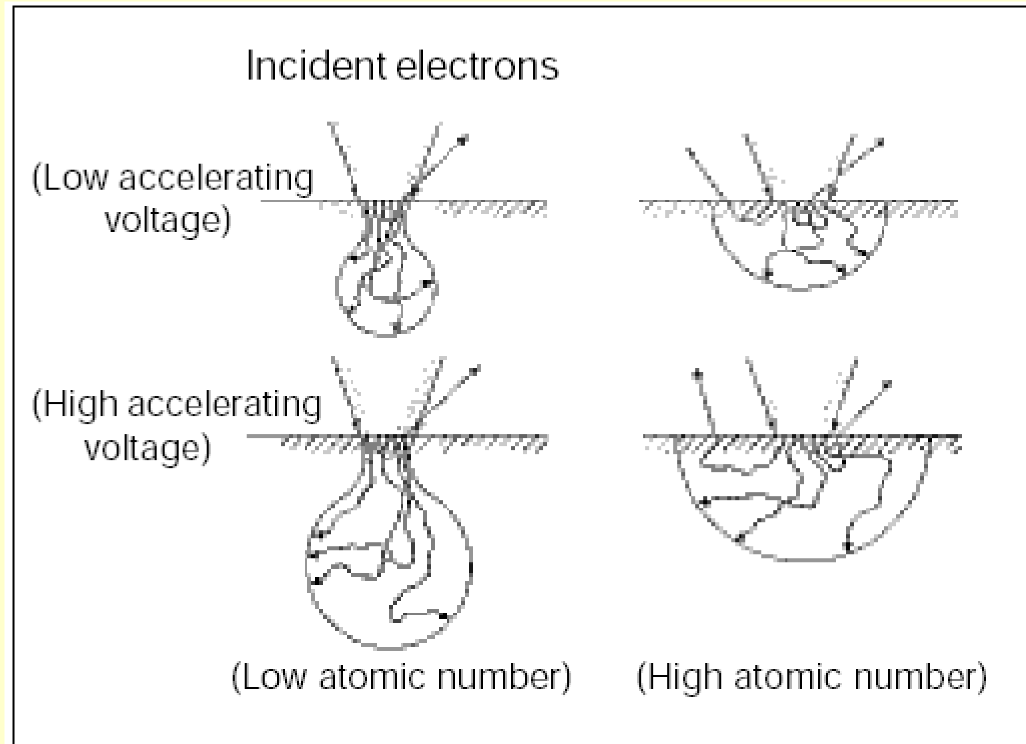


E_0=10 kV

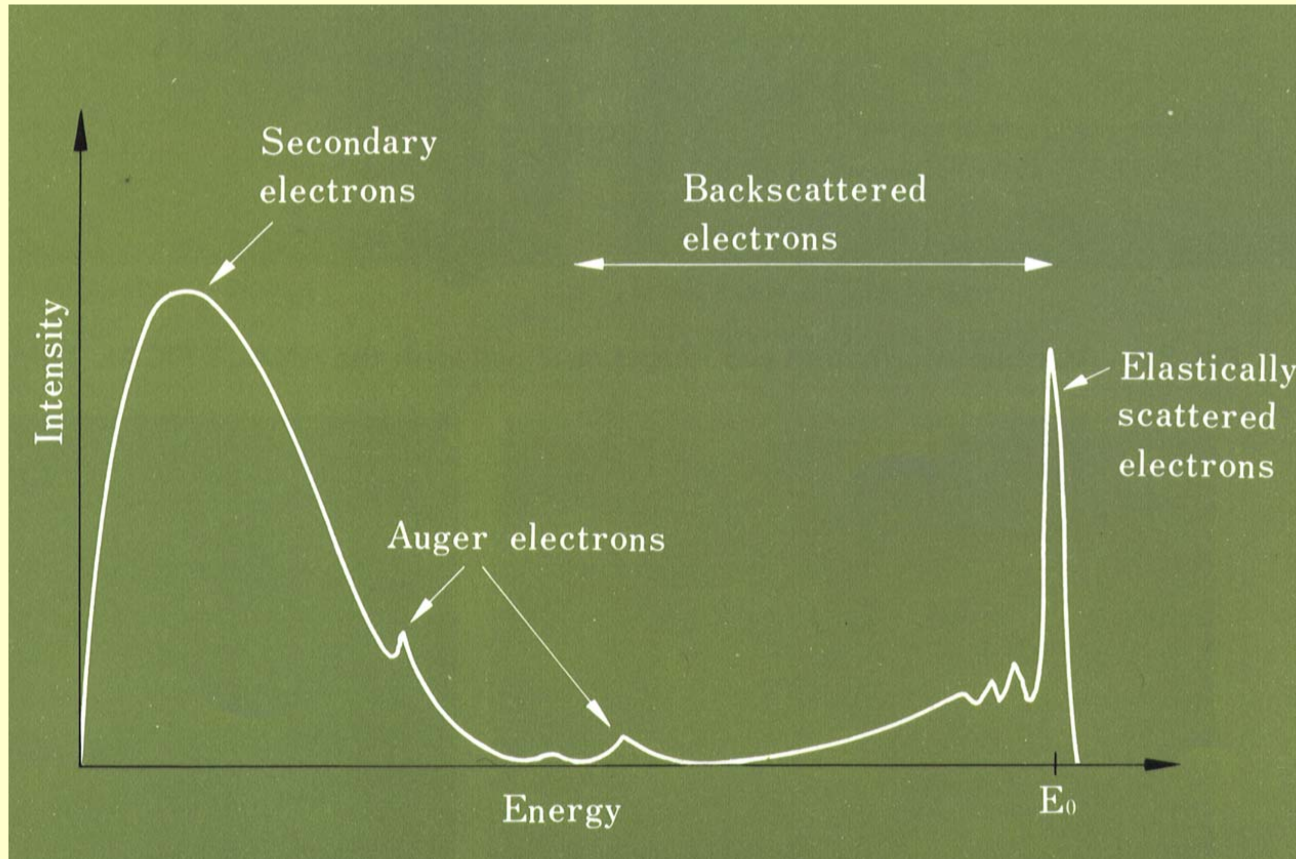
E_0=20 kV

Fe^{26}, M-C simulatsioon

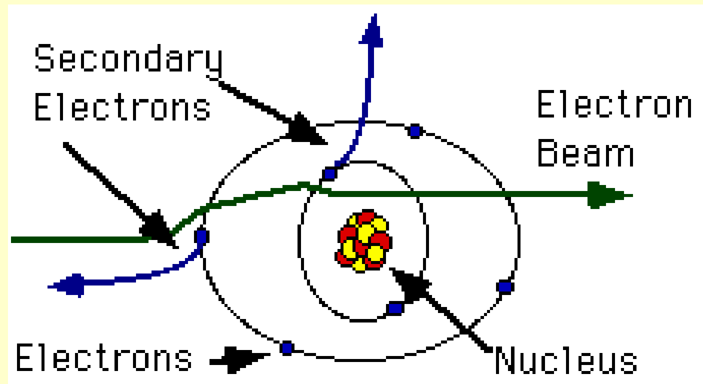
...interaktsioon

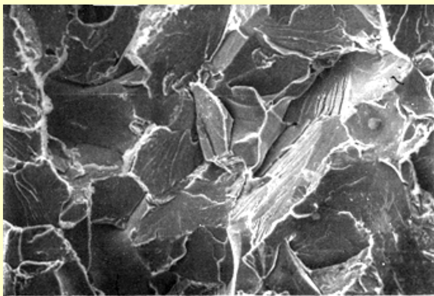


Elektronide energeetiline spekter



Sekundaarelektronid (SE)

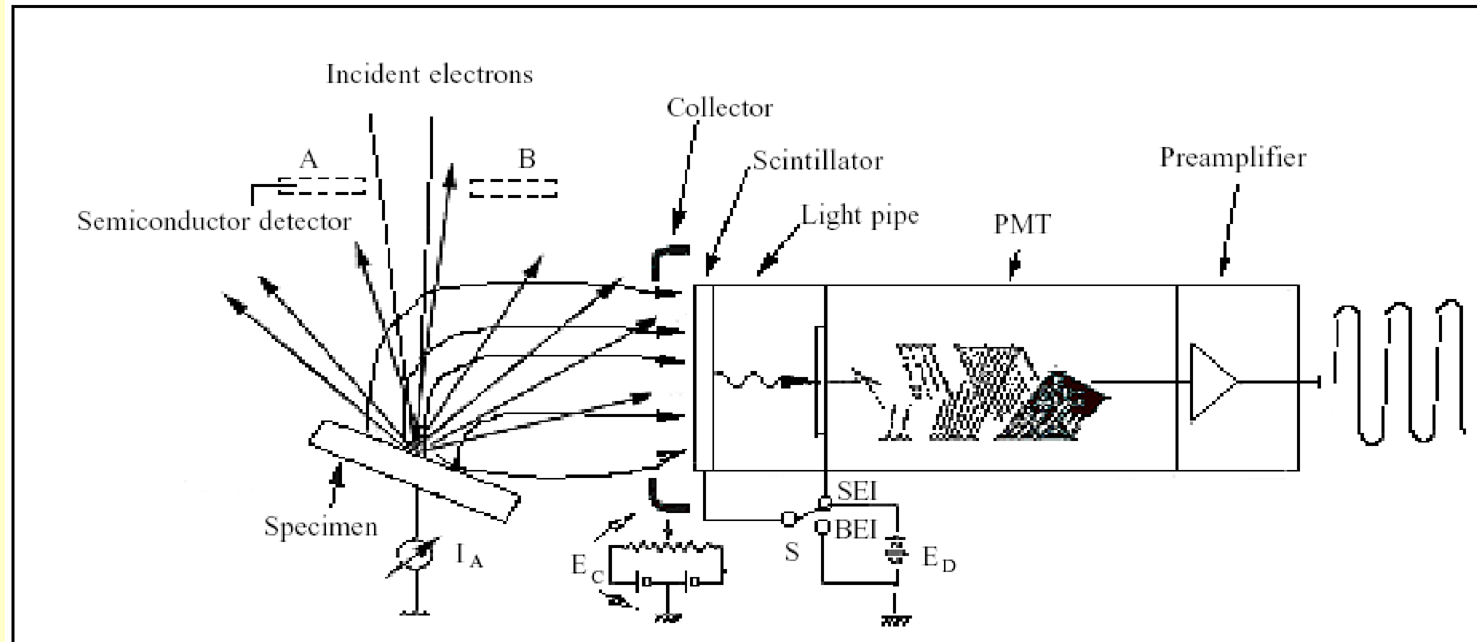




Metallobjekti murdepind, SEI

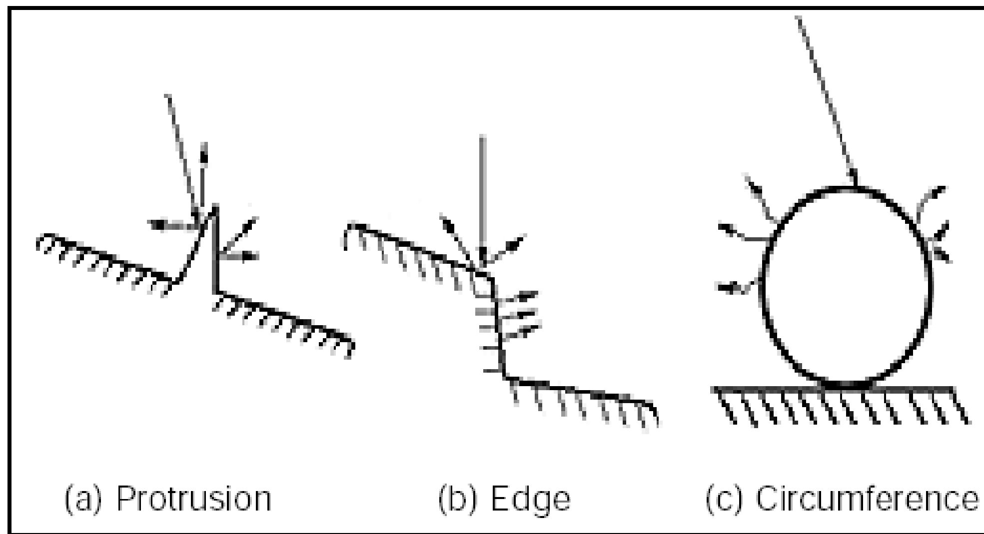
Sometimes beam electrons interact with the electrons present in the atom rather than the nucleus. Since all electrons are negatively charged, the beam electrons will repel the electrons present in the sample. This interaction causes the beam electrons to slow down as it repels the specimen electrons, The repulsion may be so great that the specimen electrons are pushed out of the atom, and exit the surface of the sample, these are called secondary electrons. Unlike the BSE, the secondary electrons are moving very slowly when they leave the sample. Since they are moving so slowly, and are negatively charged, they can be attracted to a detector which has a positive charge on it. This attraction force allows you to pull in electrons from a wide area and from around corners in much the same way that a vacuum pulls in dust particles. The ability to pull in electrons from around corners is what gives secondary electron images a 3-dimensional look.

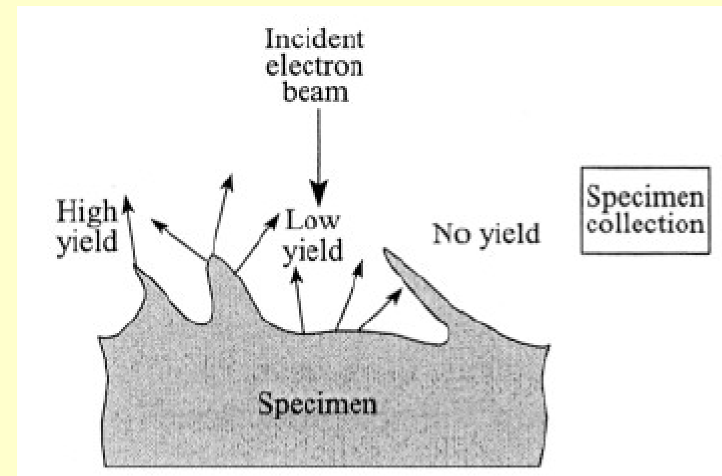
Sekundaarelektronide detekteerimine



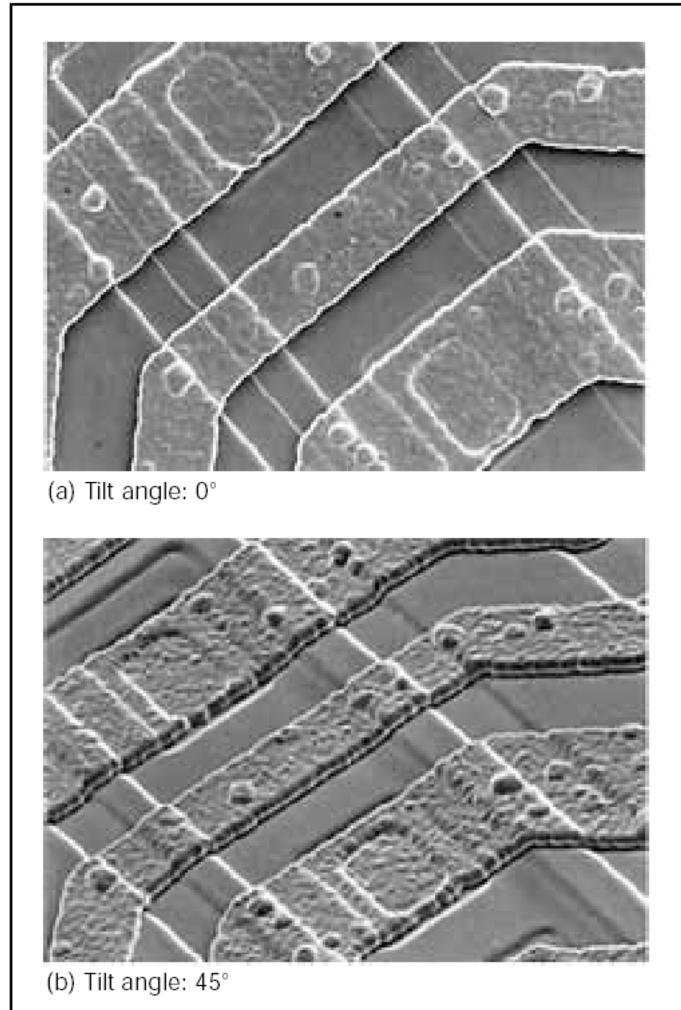
Everhart - Thornley SEI detektor

SEI kontrasti mehhanismid, topograafia





...SEI kontrasti mehhanismid, topograafia



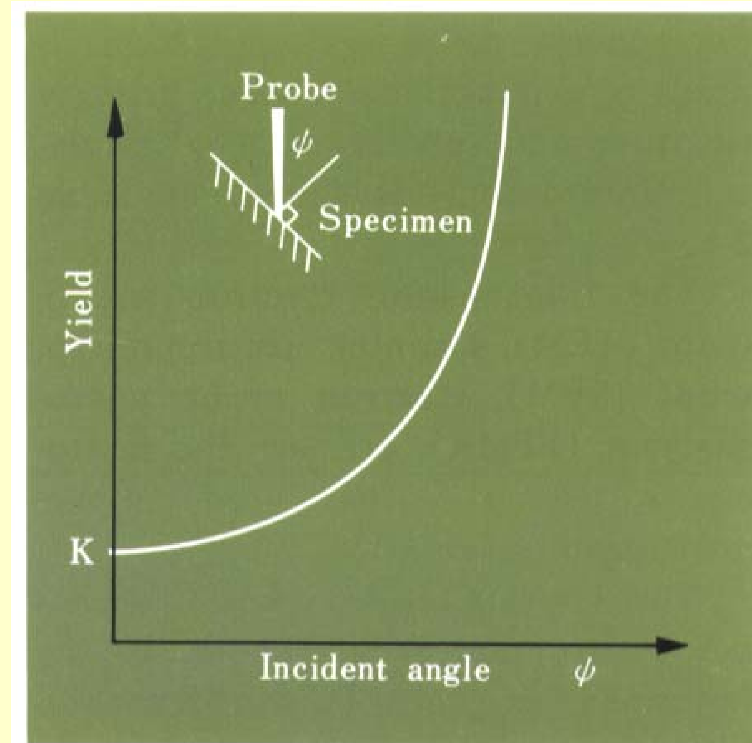
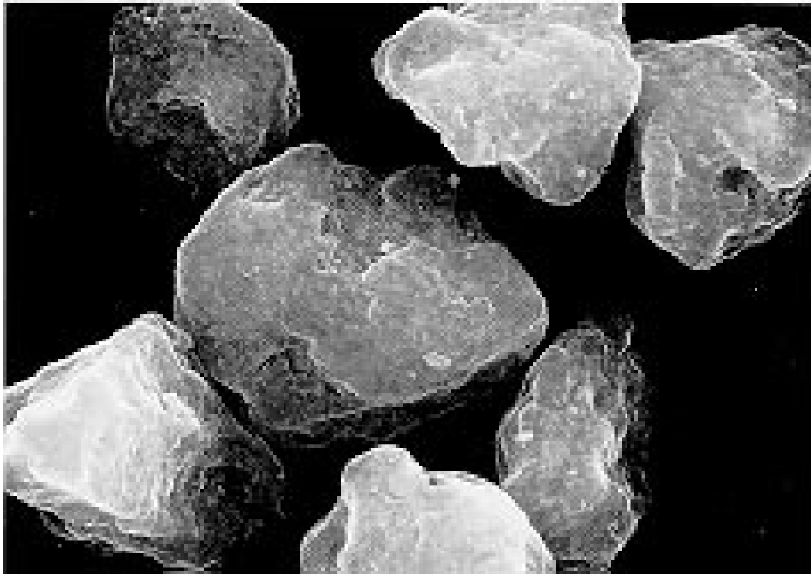
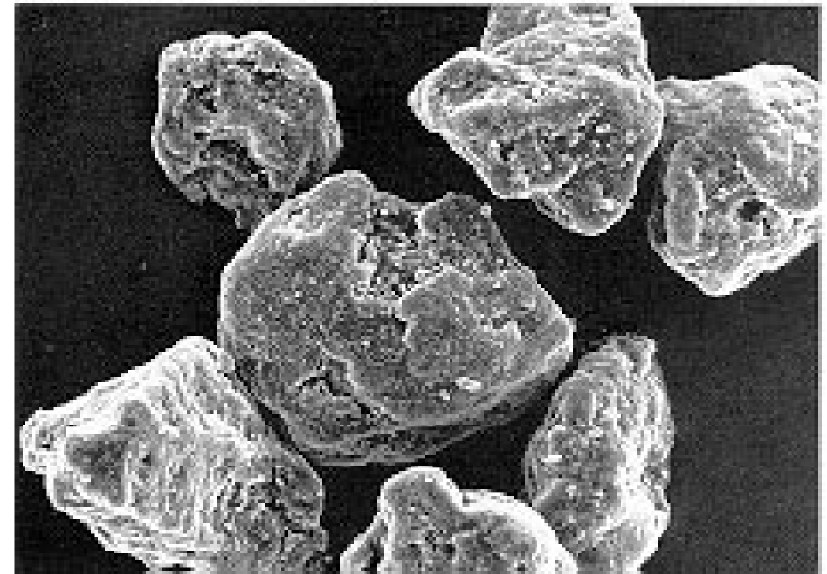


Fig. 1-5 Relation between secondary electron yield and incident angle of an electron probe.

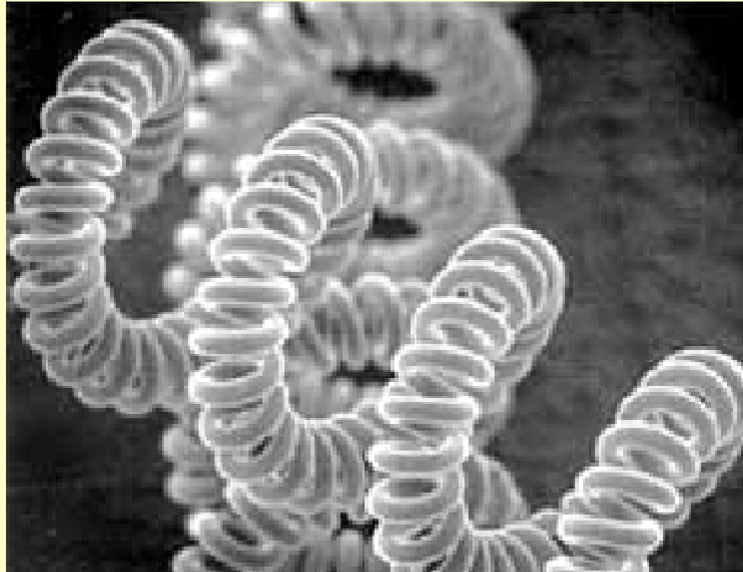
SEI kontrasti mehhanismid, E_0 mõju



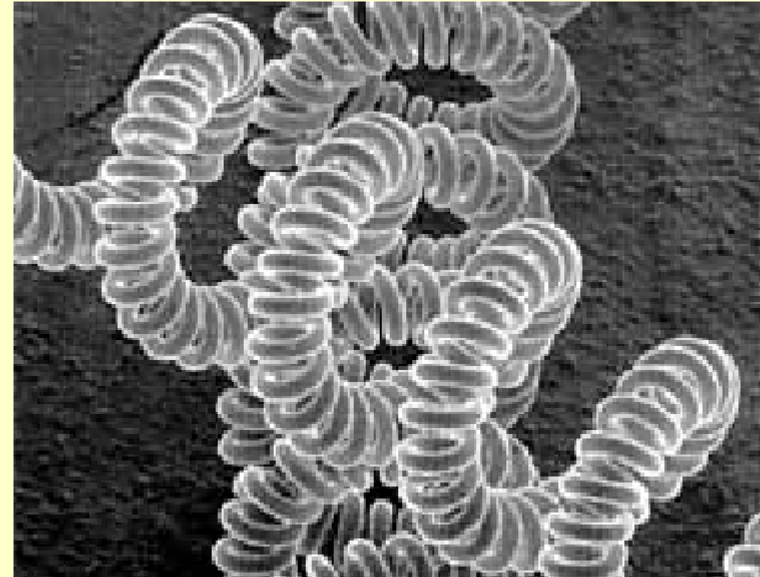


Objekt: tooneri tahmaosakesed; jälgi pinna kontrasti ja laadumise muutust olenevalt kiirendavast pingest (JEOL Ltd. pilt)

SEI-pildi sügavusteravus

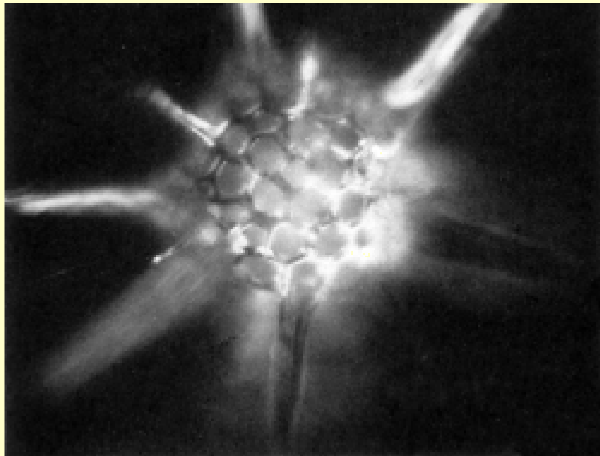


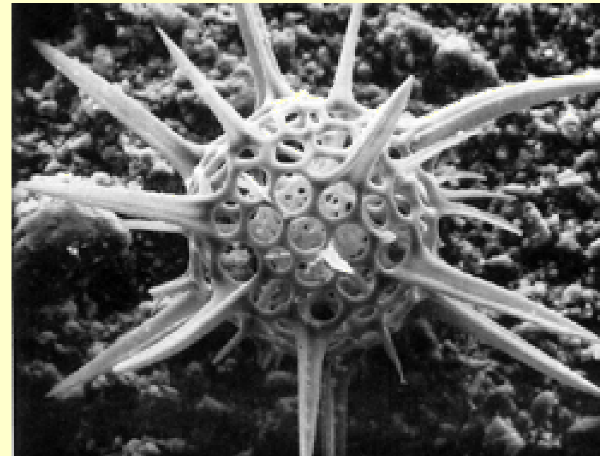
OL aperature diameter: 600μm WD: 10mm



OL aperture diameter: 100μm WD: 38mm

…SEM ja OM-pildi sügavusteravuse võrdlus

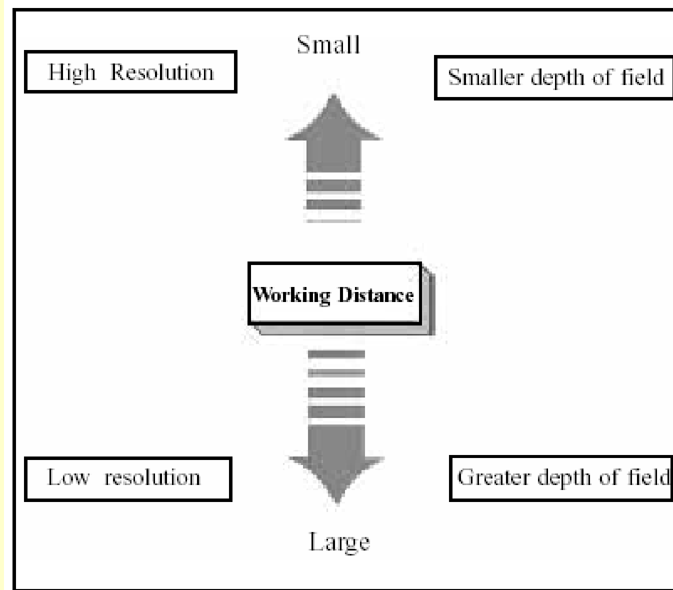


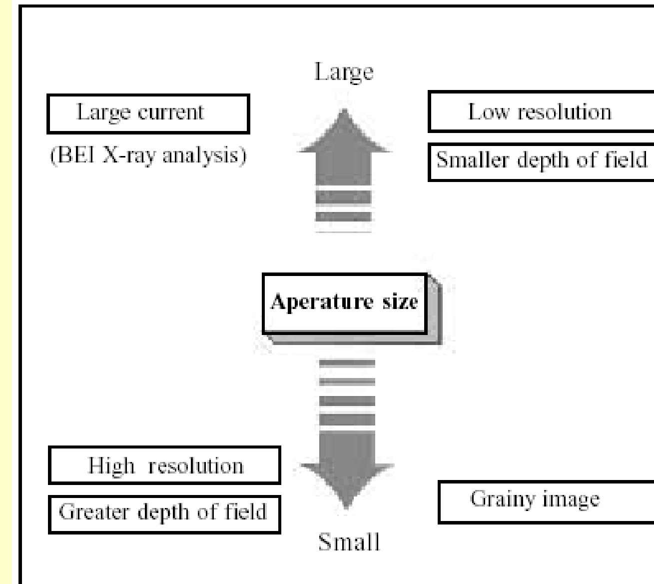


OM kujutis (vasakul) ja sama objekti SEM-kujutis (paremal)

http://www.reed-electronics.com/tmworld/

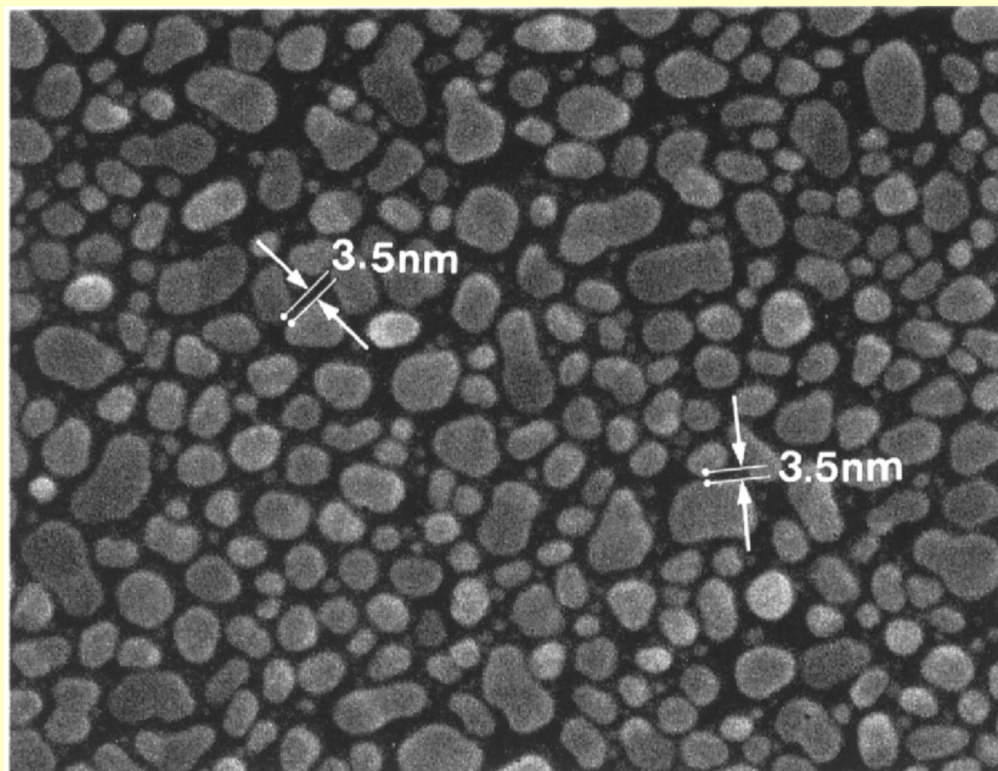
…SEI-pildi sügavusteravus





Töökõrguse (vasakul) ja OL apertuurava (paremal) mõju SEM kujutise omadustele

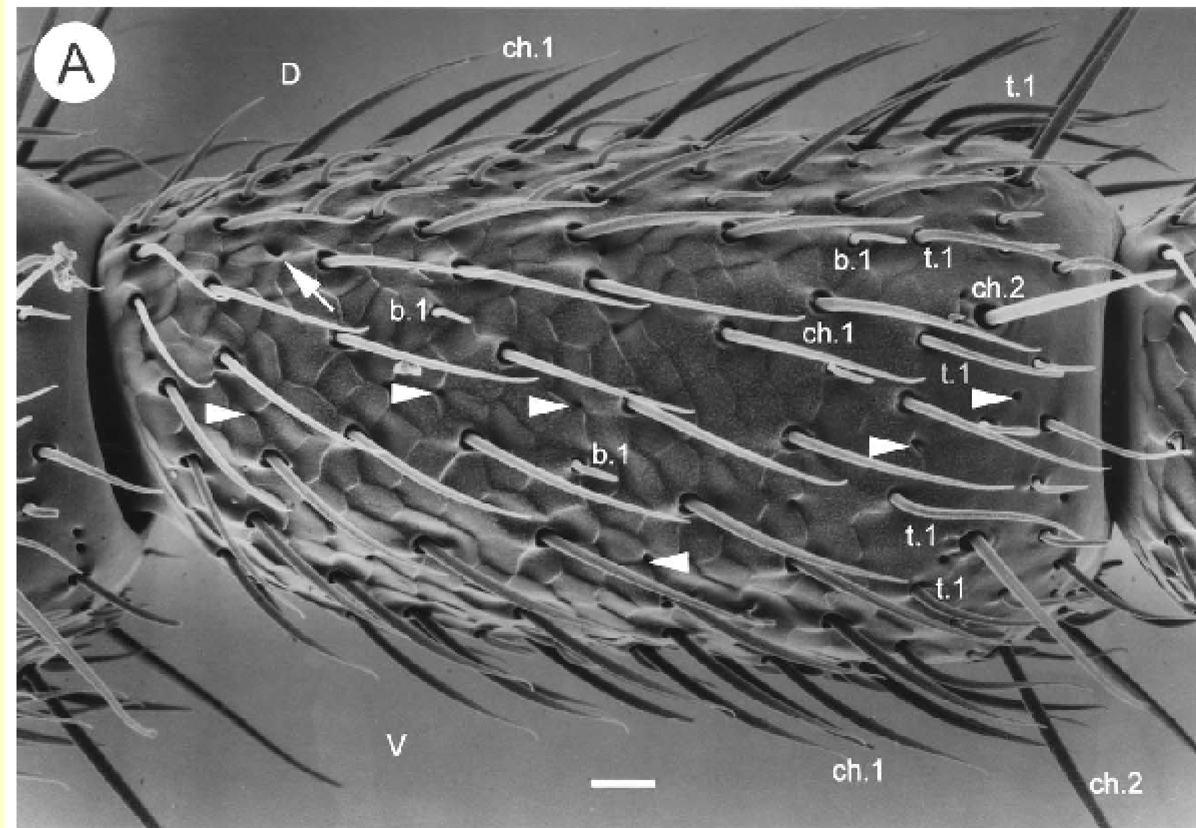
SEI lahutusvõime testpilt



Secondary electron image of evaporated gold particles.

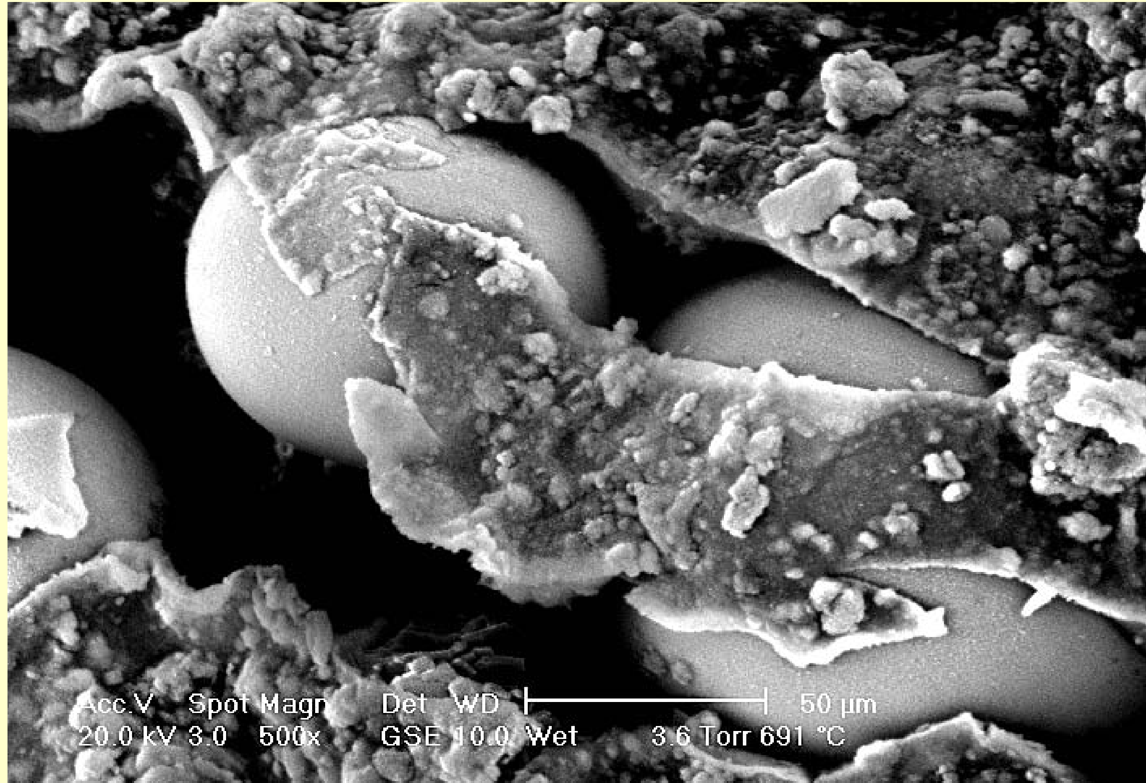
"Standardne" SEM; punkt-punkti lahutusvõime; JEOL Ltd.

Näited SEM rakendustest...SEI-pilt; zoloogia



Objekt: *B. Properans*'i tundla lüli; mõõtjoon=10um. Jälgi suurt sügavusteravust

...SEI-pilt; materjaliteadus



Objekt: traat; T=691ºC, p=3.6 Torri; JEOL Ltd.

...SEI-pilt; nanotehnoloogia

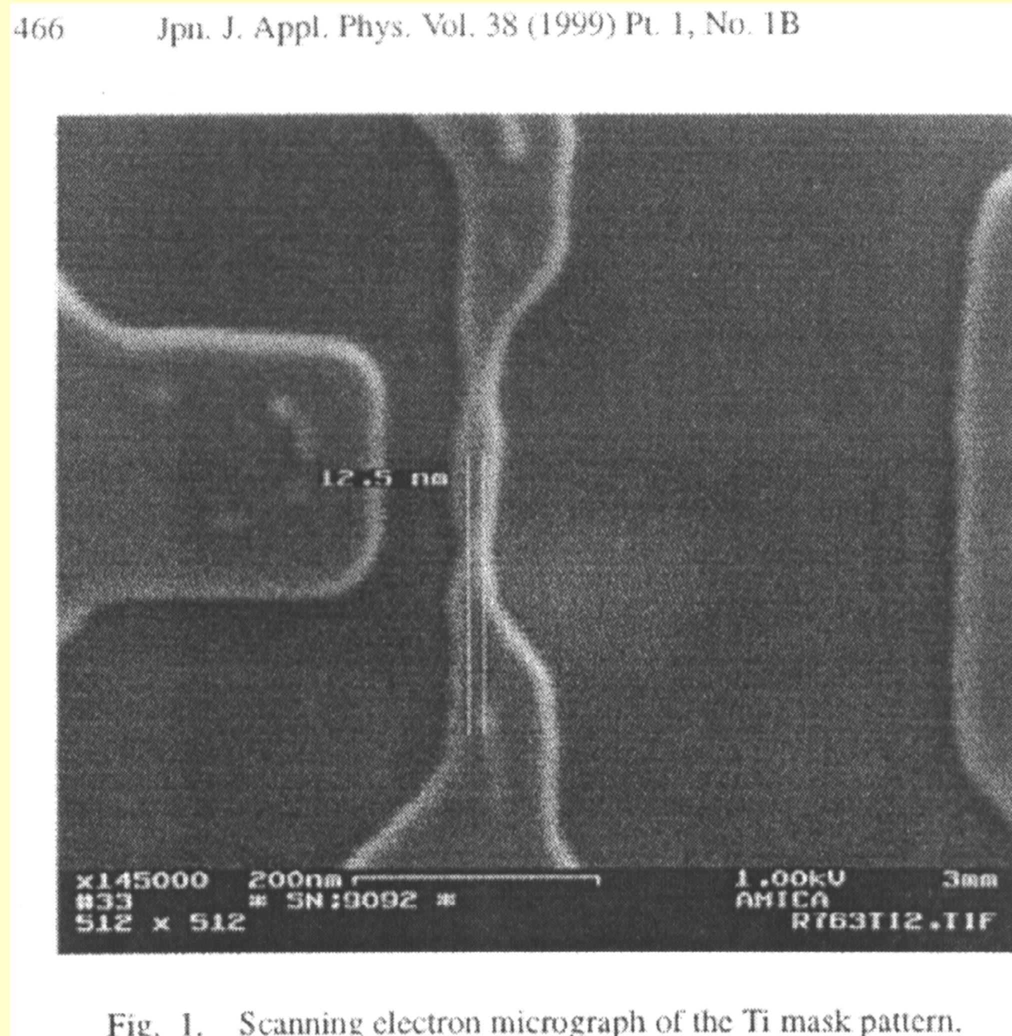
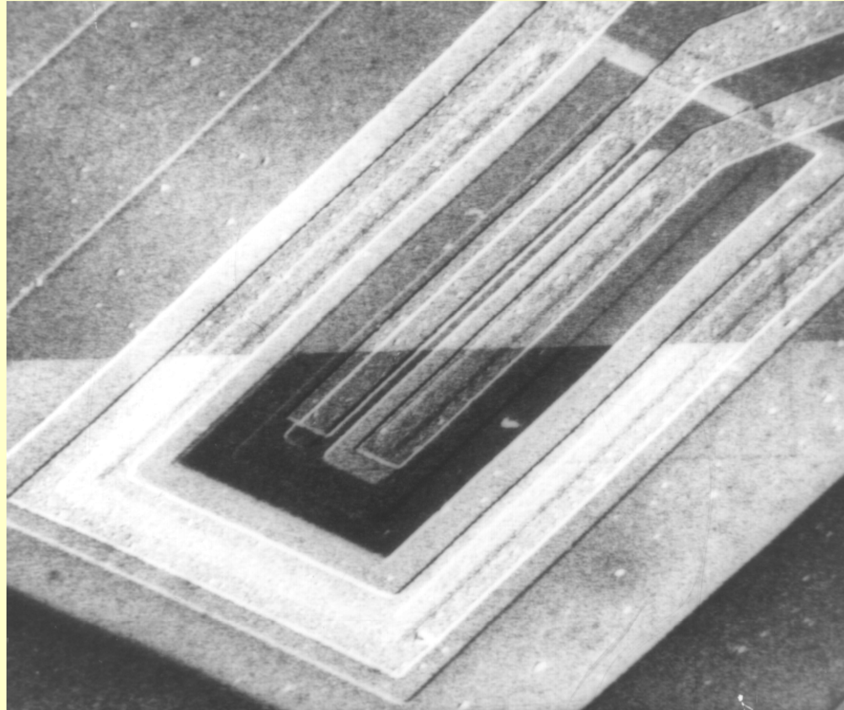


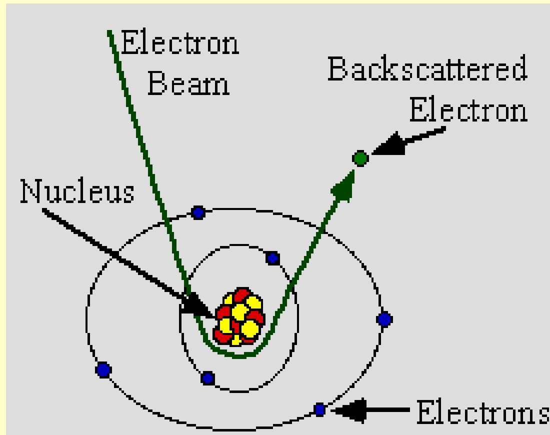
Fig. 1. Scanning electron micrograph of the Ti mask pattern.

...SEI-pilt; elektroonikatööstus



Objekt: IC, Tallinna HP tehas; potentsiaali kontrast (tumedam ala on positiivsema potentsiaaliga)

Peegeldunud elektronid (BE)



Al ja Cu sulam: heledamad kohad on Cu-rikas ja tumedamad Al-rikas faas; BEI

When the electron beam strikes the sample some of the electrons will interact with the nucleus of the atom in much the same way a space craft will interact with the gravity of a planet. The negatively-charged electron will be attracted to the positive nucleus but if the angle is just right instead of being captured by the "gravitational pull" of the nucleus it will circle the nucleus and come back out of the sample without slowing down. These electrons are called backscattered electrons because they come back out of the sample. Because they are moving so fast, they travel in straight lines. In order to form an image with BSE (backscattered electrons), a detector is placed in their path. When they hit the detector a signal is produced which is used to form the TV image.

All the elements have different sized nuclei. As the size of the atom nucleus increases, the number of BSE increases. Thus, BSE can be used to get an image that showed the different elements present in a sample.

Sekundaar- ja peegeldunud elektronide saagis

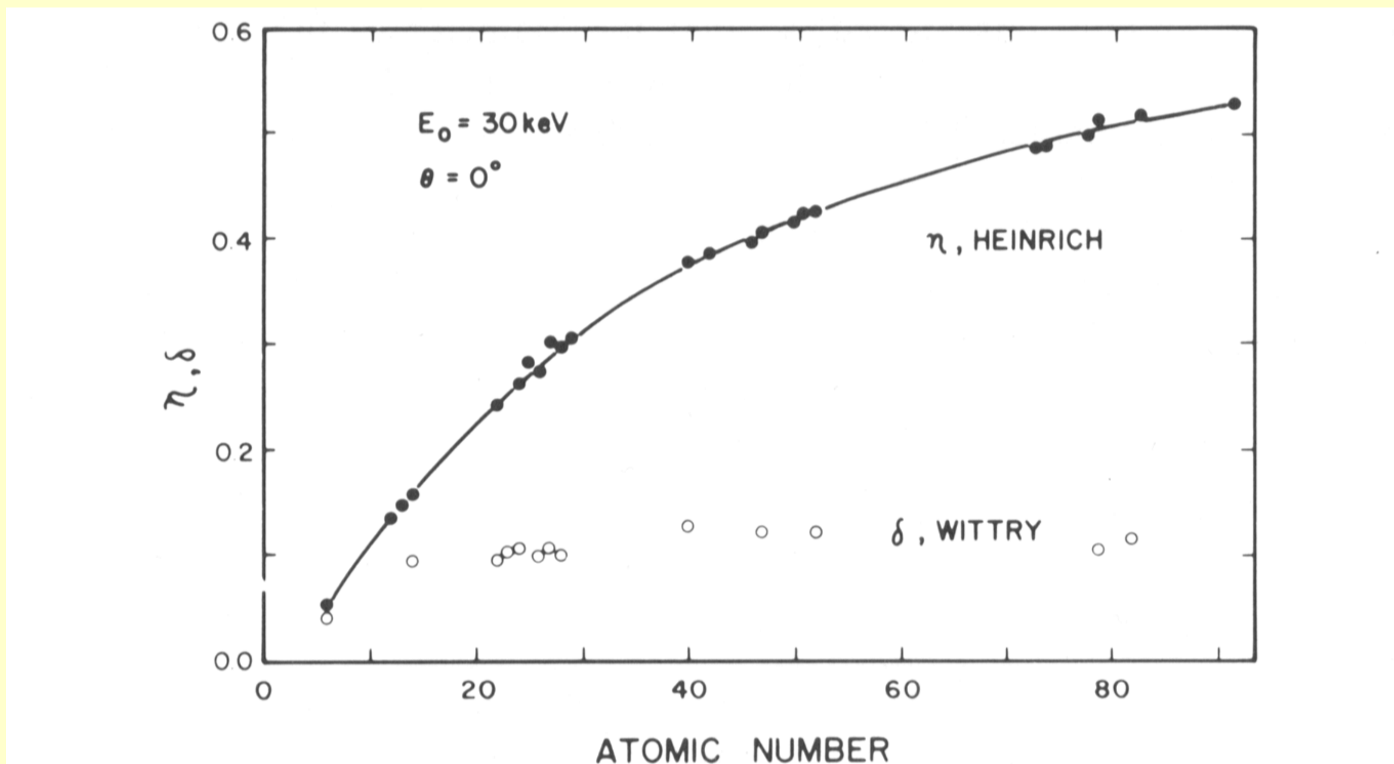
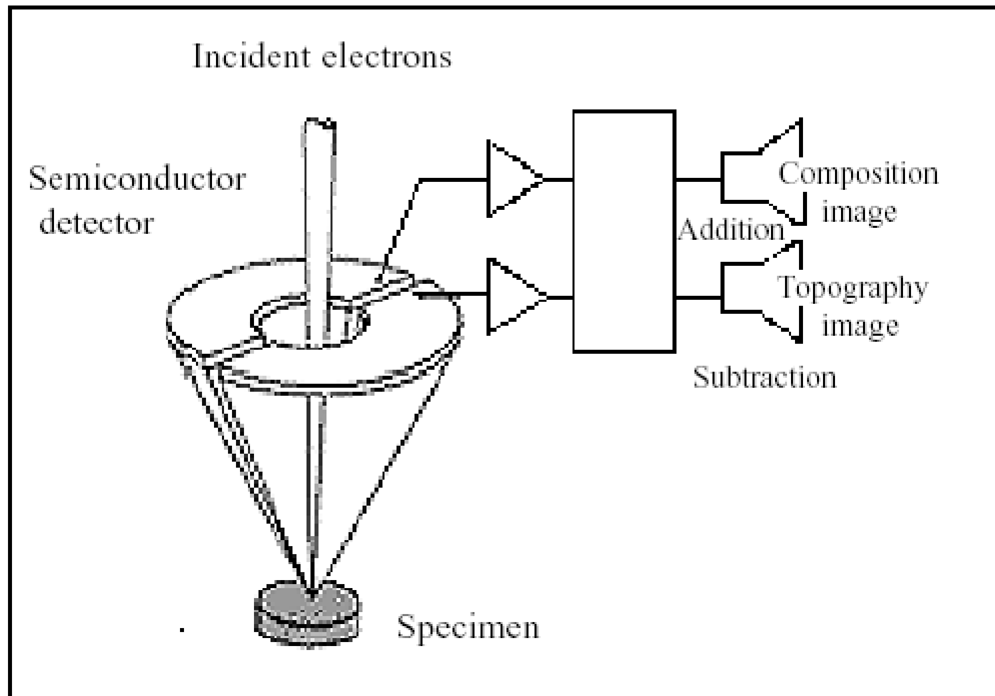
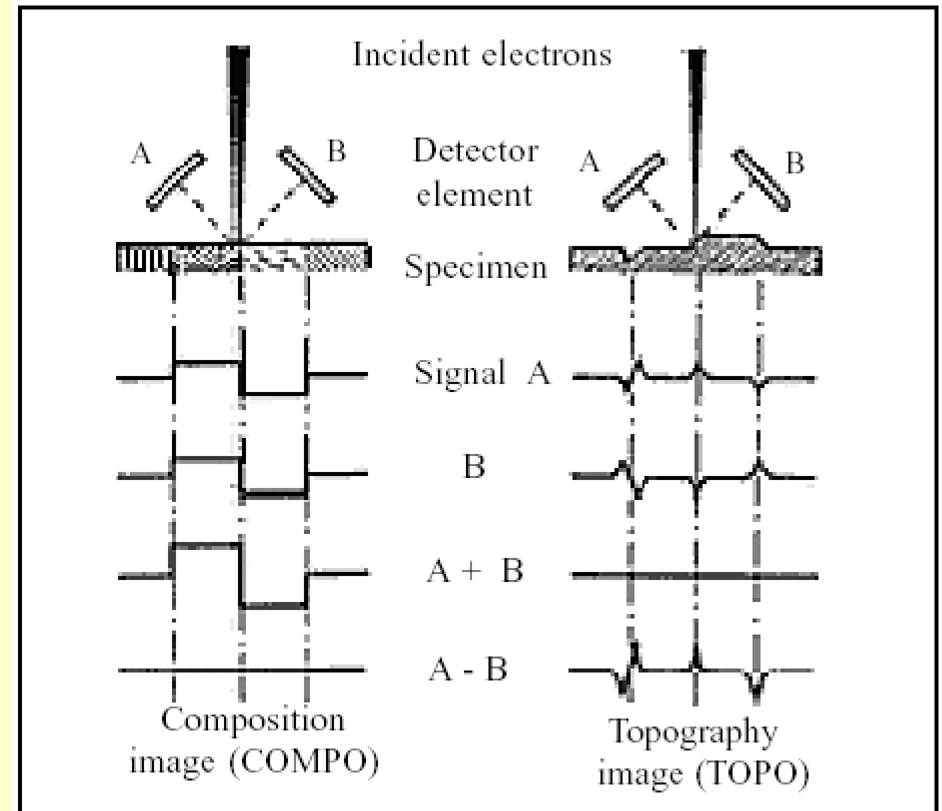


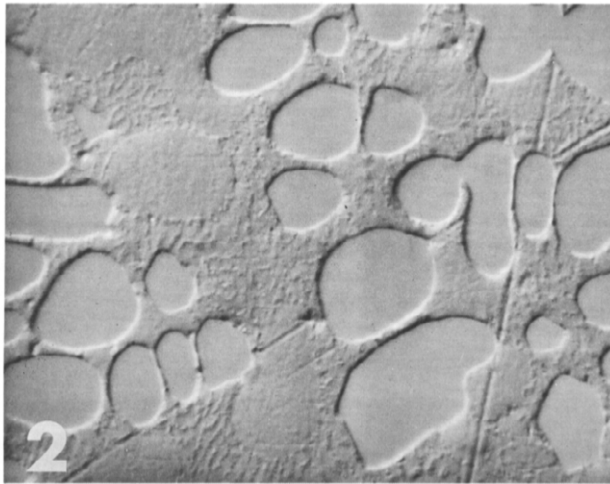
Figure 3.29. Comparison of backscattered electron coefficients and secondary-electron coefficients as a function of atomic number (Wittry, 1966; Heinrich, 1966).

Peegeldunud elektronide detektor

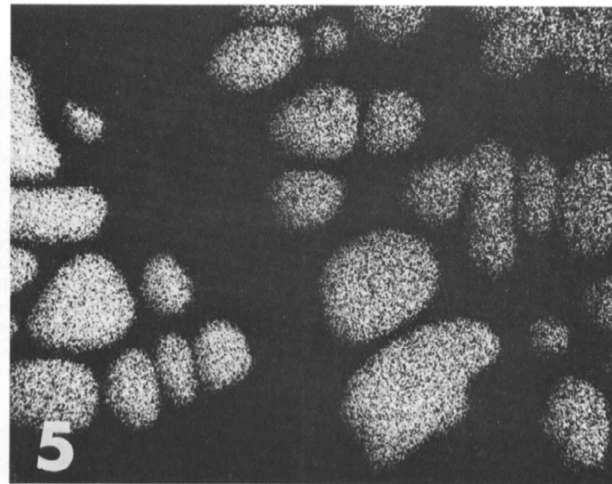




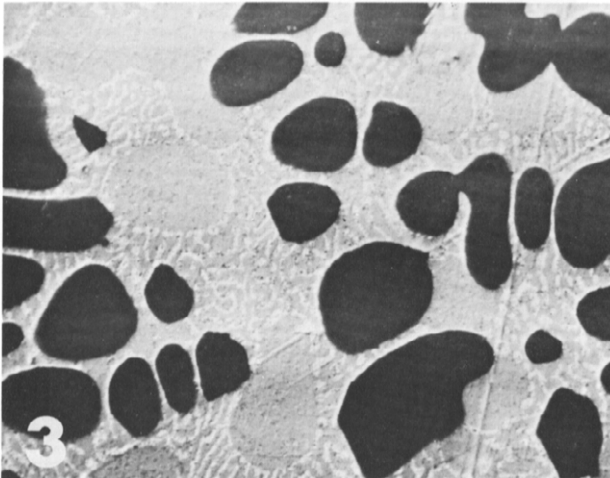
BE-pilt



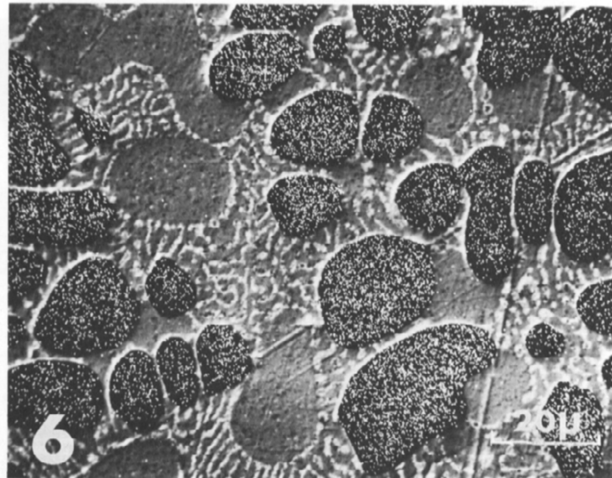
(2) *Backscattered electron image (Topography)*



(5) *Al-Kα X-ray image*



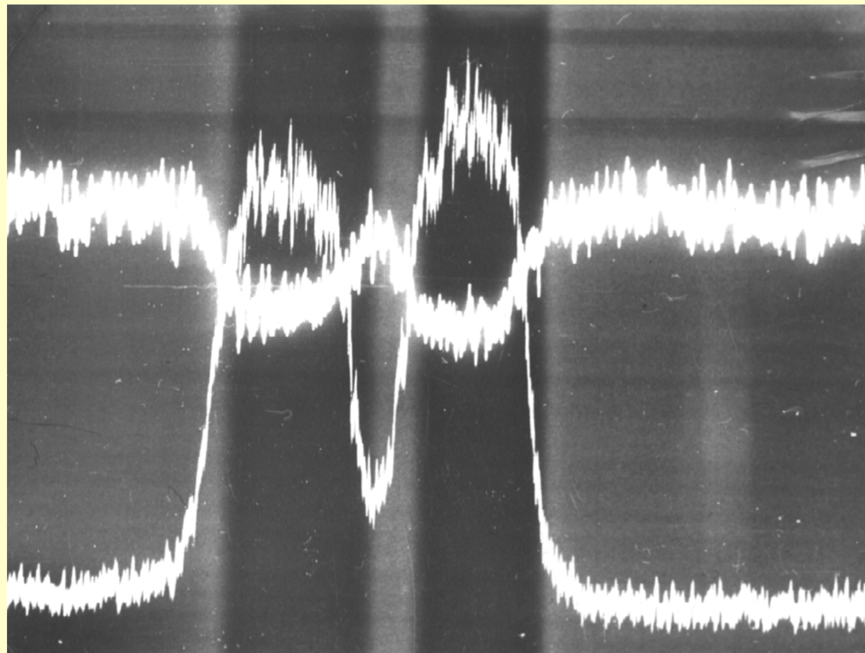
(3) *Backscattered electron image (Composition)*



(6) *Al-Kα X-ray image superimposed on Composition* ×760

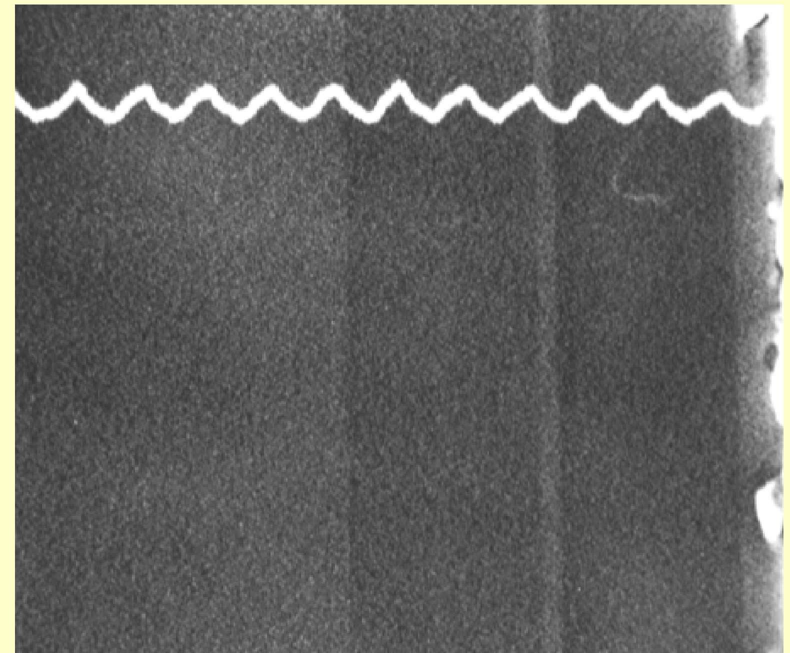
(JEOL Ltd.)

BE-pildid; pooljuhtide tehnoloogia



Ga K_α

Al K_α



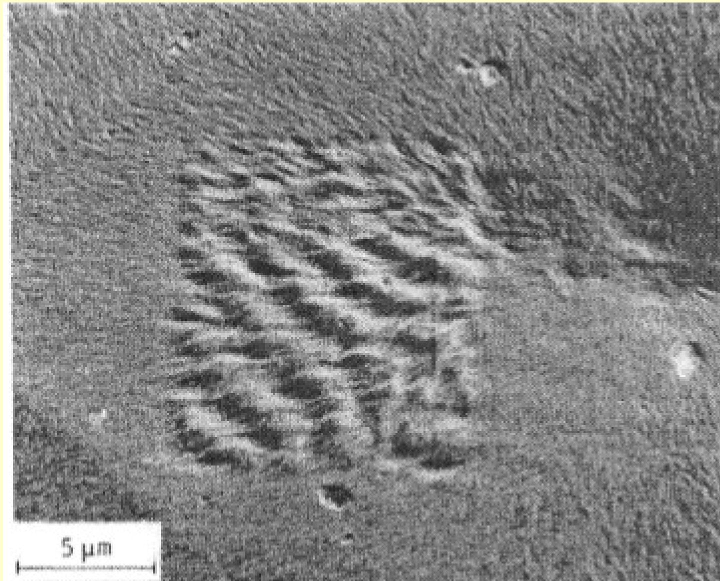
GaAs AlGaAs AlGaAs GaAs

GaAs

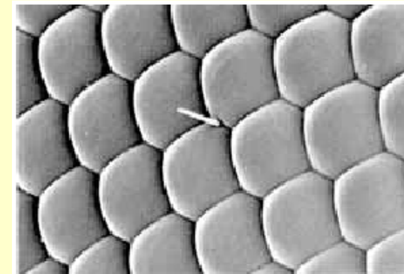
AlGaAsSb AlGaAsSb

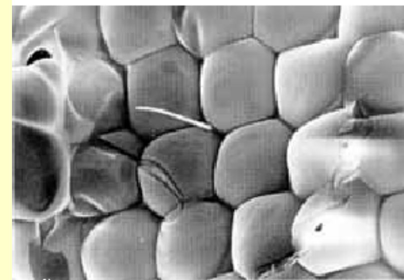
GaSb GaSb GaSb

Objekti kahjustamine



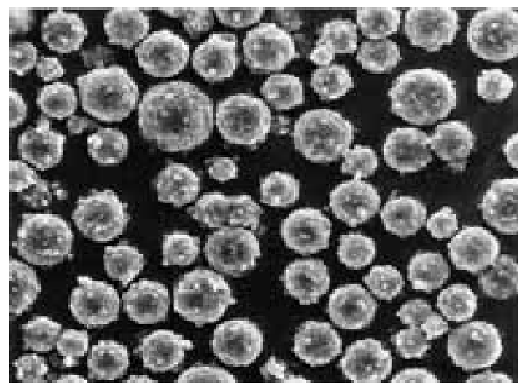
Polümeer

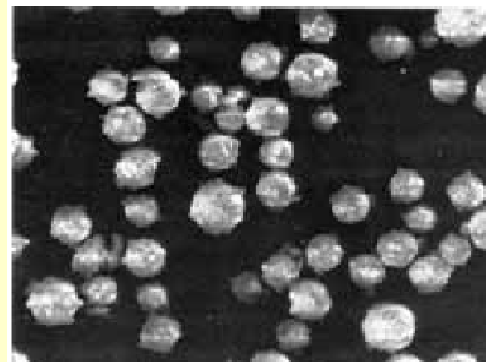

(a) Undamaged specimen

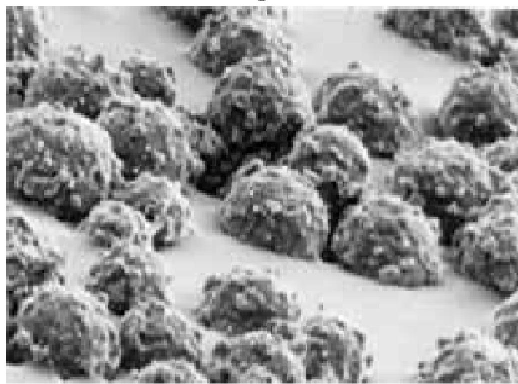

(b) Damaged specimen

Kärbse silm

Objektide prepareerimine



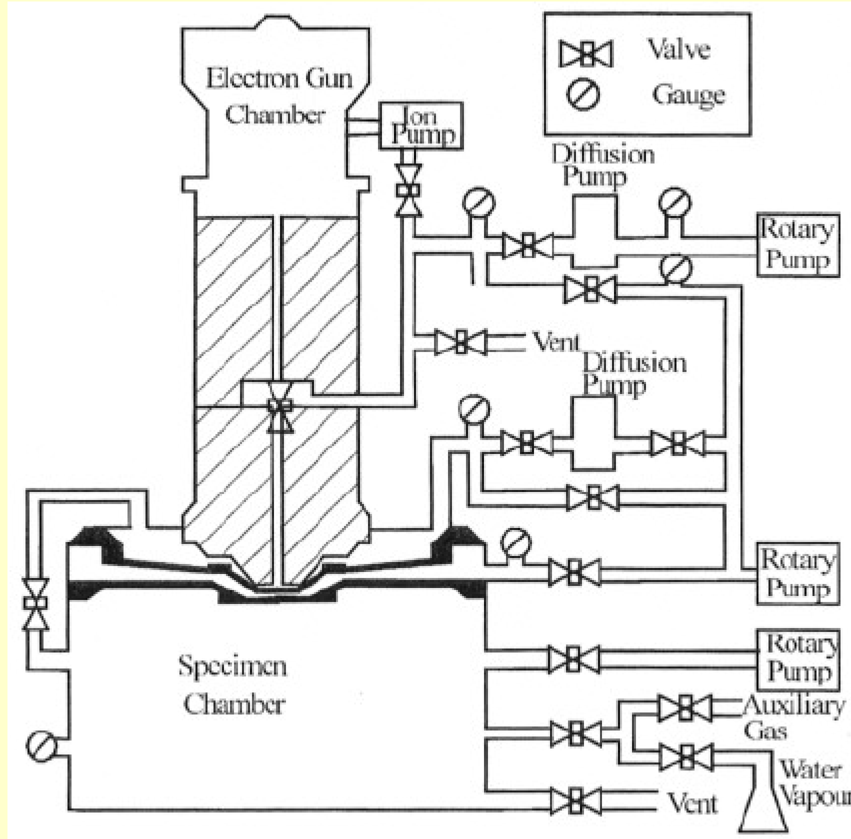




Tooneri tahmaosakesed: 15 (ülemine) ja 5 keV (alumine) rida, SEI kujutised. Prepareeeritud 2-e poolsele teibile, kerge vajutusega (vasakul) ja orgaanilise lahusti abil (paremal). NB! Laadumine parempoolsetel piltidel.

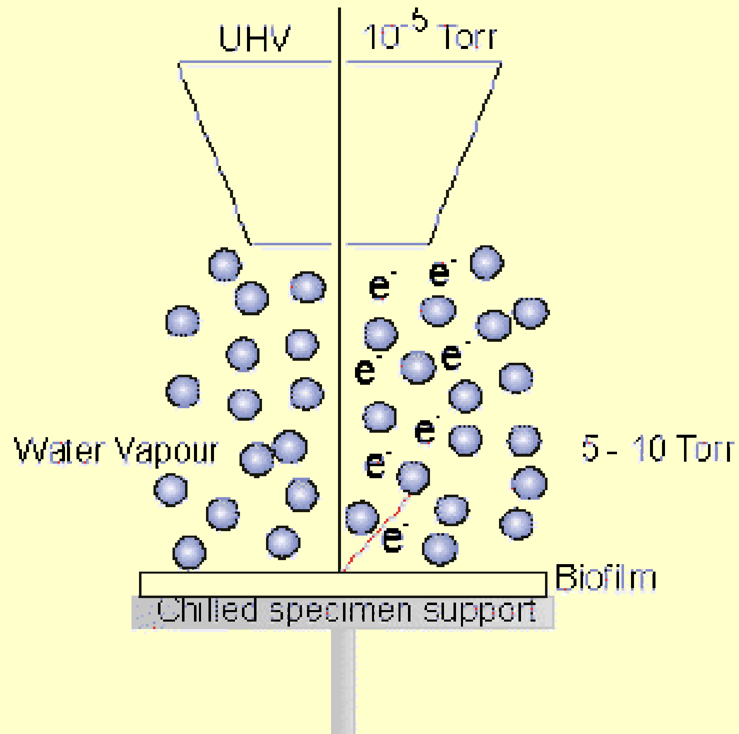
Kaasaegse elektronmikroskoopia suundumused

Muudetava keskkonnaga (madalarõhuline) SEM



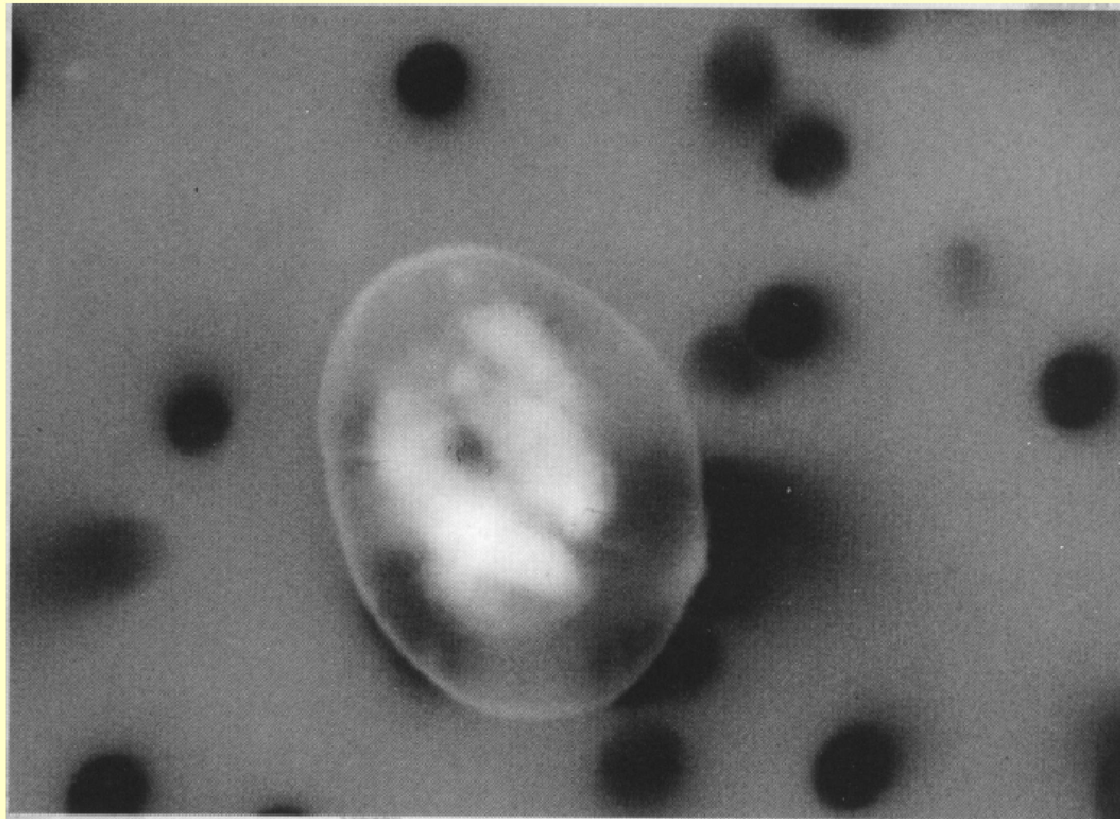
Ingl. k. Environmental SEM (ESEM)

…madalarõhuline SEM



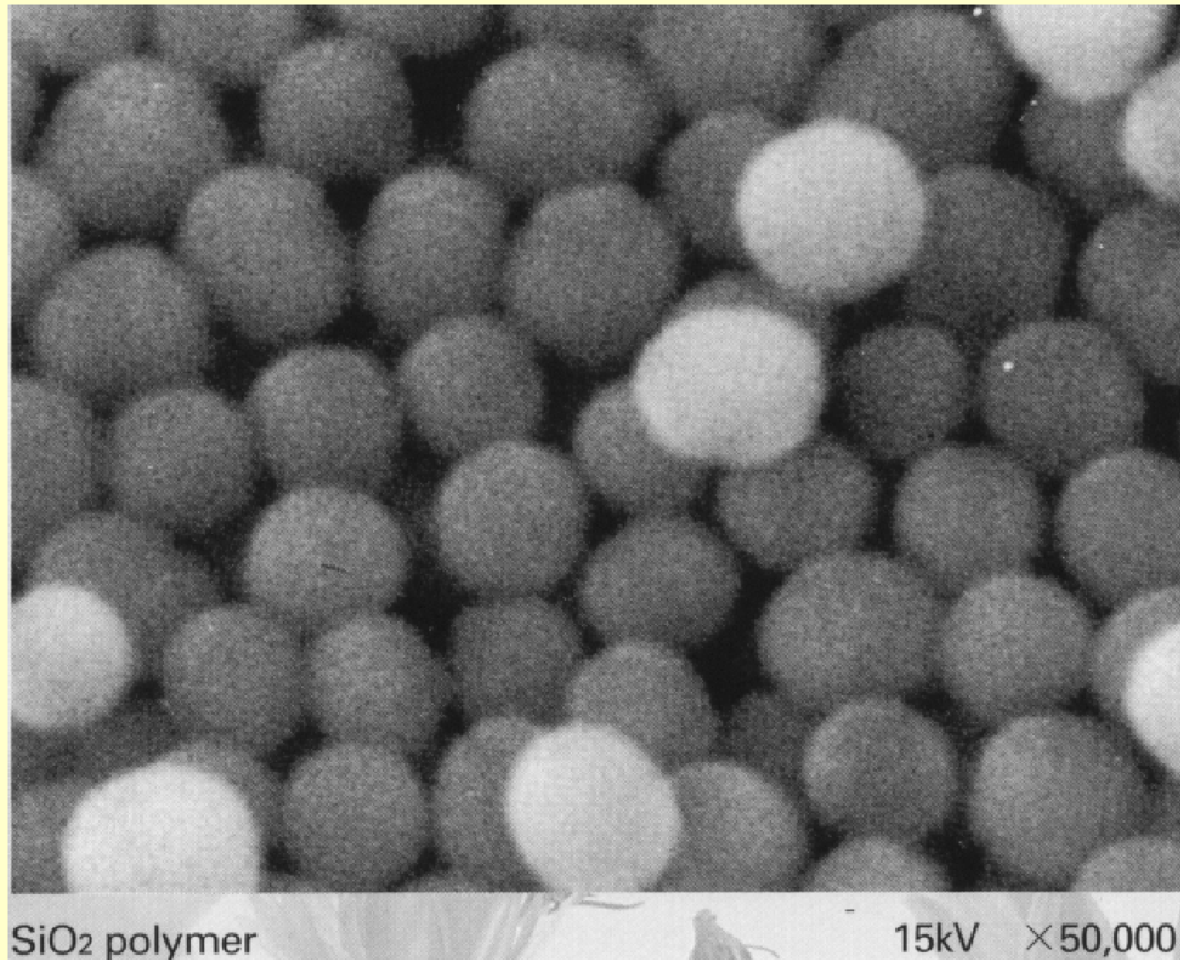
A primary electron beam hits the specimen, which emits secondary electrons. These secondary electrons are attracted to the positively charged detector electrode and as they travel through the gaseous environment, collisions occur between electrons and gas particles, resulting in emission of more electrons and ionisation of the gas molecules. This increase in the number of electrons effectively amplifies the original secondary electron signal. The positively charged gas ions are attracted to the negatively biased specimen and offset charging effects. As the number of secondary electrons varies, the amplification effect of the gas varies. If a large number of electrons are emitted from a position on the specimen during a scan, there is a high signal. If only a small amount of electrons are emitted the signal is less intense. The difference in signal intensity from different locations on the specimen allows an image to be formed. The gas itself can be altered to suit the sample under study, and may be, for example, water vapour, air, argon or nitrogen (Li et al., 1995).

...madalarõhuline SEM

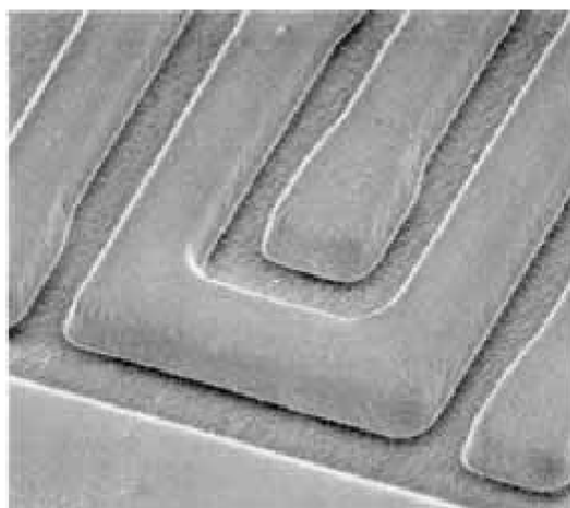


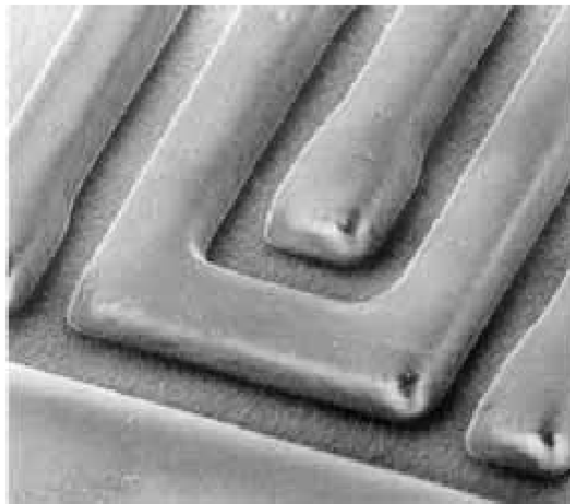
Cryptosporidium parvum oocysts (protozoan) 20kV ×10,000
in river water Freeze-dried in LV mode (fixed with formalin)
Specimen courtesy of Tokyo Metropolitan Research Laboratory of Public Health

...madalarõhuline SEM



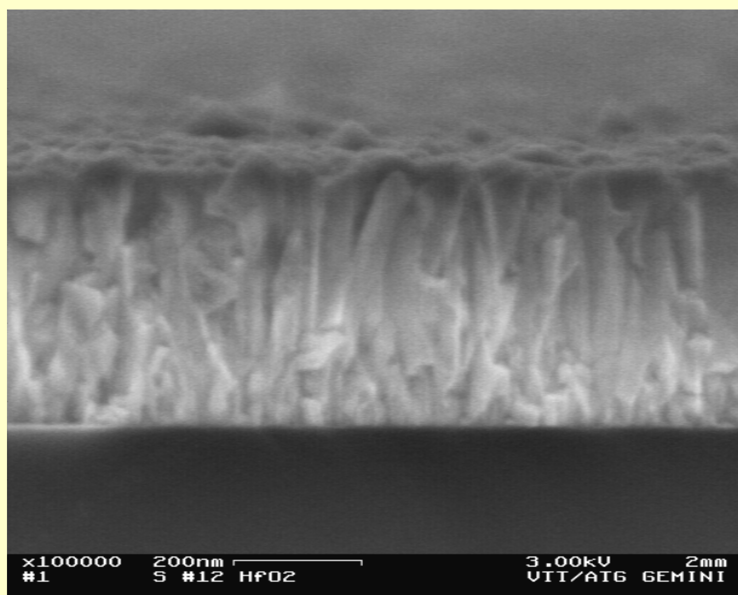
Madalapingeline SEM - Shottky katood

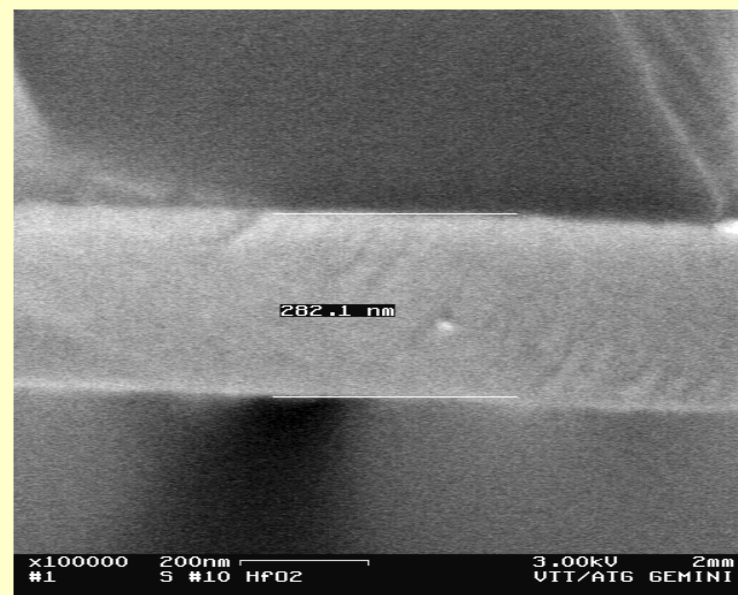

(a) 1.0 kV x3,200


(b) 1.3 kV x3,200

Specimen: Resist.
Charge-up can be prevented by properly selecting the accelerating voltage.

Kõrglahutusega mikroskoopia





Kristalliline (vasakul) ja amorfne (paremal) HfO_2 kile Si-alusel;
HR-SEM pilt, 200000x suurendus

SEM tootjad

A & J Scientific Ltd.

CamScan USA Inc.

Electron Beam Technology Ltd.

FEI Company

FEI Company

HKL Technology A/S.

HKL Technology Inc.

International Equipment Trading Ltd.

Carl Zeiss SMT AG

Millbrook Instruments Ltd.

Nanomotion Ltd.

Obducat CamScan Ltd.

Omicron NanoTechnology GmbH

Omniprobe, Inc.

Raith GmbH

Target-Messtechnik

TESCAN, s.r.o.

University of Brighton

Zyvex Corporation