Moderní experimentální metody
Elektronová spektroskopie a mikroskopie IV

Elektronová mikroskopie
- Transmisní
- Rastrovací
- Běžné přidružené analytické metody
    - EDS, WDS
    - EELS
- Elektronová difrakce
    - EBSD
    - LEED
    - RHEED

History of electron microscopy

- First transmission electron microscopy
  Ernst Ruska 1931

- First Czechoslovak electron miscoscope
  – Armin Delong late 1940’s
- Scanning electron microscopy
  – Zvorykin 1942

- Aberation corrected electron microscopy
  – atomic resolution
  – Ondrej Krivanek 1970s

Basic Microscope Classifications

condenser aperture
objective aperture
selected area aperture
electron source
first condenser lens
second condenser lens
objective condenser lens
microcondenser lens
specimen (thin)
objective imaging lens
diffraction lens
intermediate lens
first projector lens
second projector lens
projector chamber
fluorescent screen

(Illustration of a TEM shown)

Charged particle microscope

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Comparing Microscopes

	LIGHT MICROSCOPE	ELECTRON MICROSCOPE
The source of illumination	The ambient light source is light for the microscope	Electrons are used to "see" – light is replaced by an electron gun built into the column
The lens type	Glass lenses	Electromagnetic lenses
Magnification method	Magnification is changed by moving the lens	Focal length is charged by changing the current through the lens coil
Viewing the sample	Eyepiece (ocular)	Fluorescent screen or digital camera
Use of vacuum	No vacuum	Entire electron path from gun to camera must be under vacuum

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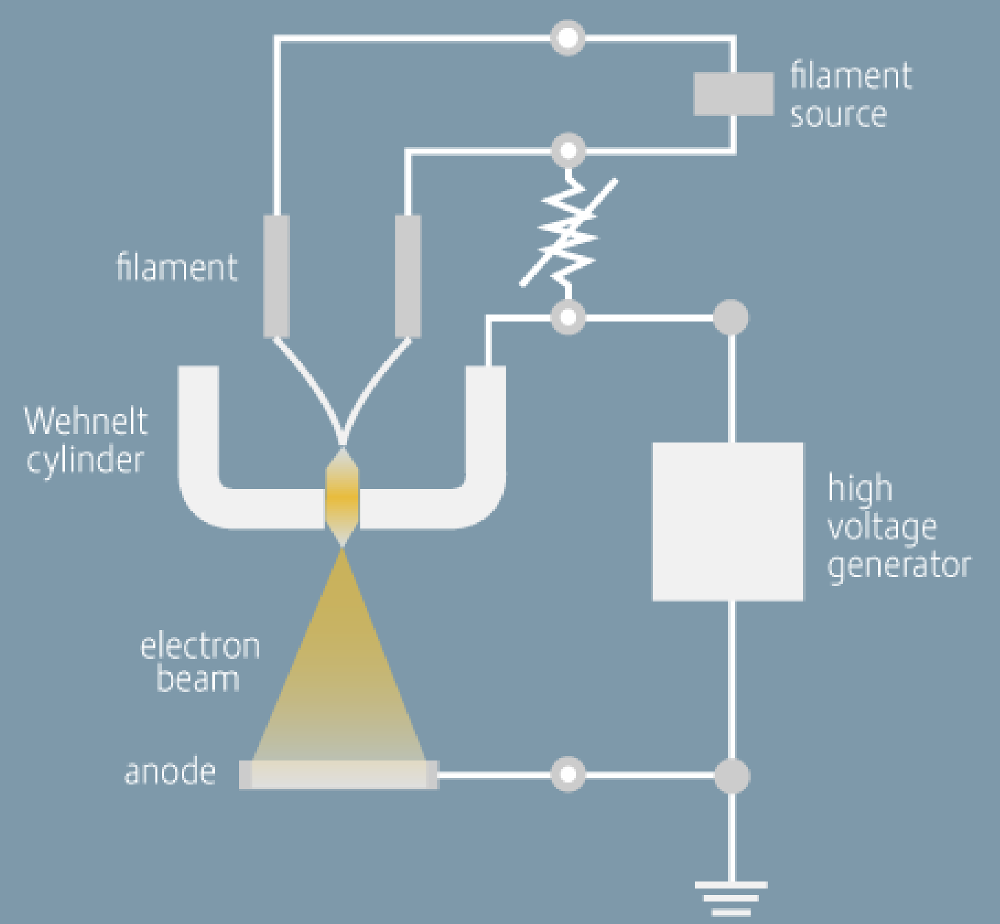
CORE TECHNOLOGY: The Electron Gun

- Three main sources of electrons:
  - Tungsten
  - LaB6 (lanthanum hexaboride)
  - Field Emission Gun (FEG)
- Different costs and benefits of each
- Each selected primarily for their brightness



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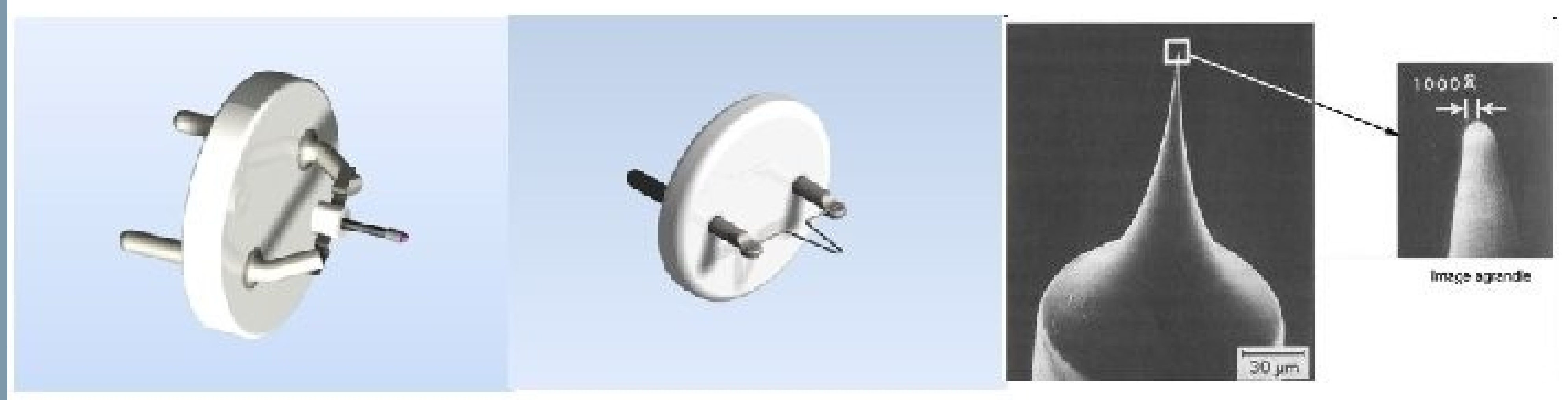
Electron guns

- With field emission guns we get a smaller spot and higher current densities compared to thermionic guns
- Vacuum requirements are tougher for a field emission guns



Single crystal of LaB_6 | Tungsten wire | Field emission tip

CORE TECHNOLOGY: Electromagnetic Lenses

electron beam

C

C

C

C

P

P

P

P

C electrical coil

P soft iron pole piece

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What is a Transmission Electron Microscope?

electron source

condenser system

specimen (thin)

objective lens

projector lens

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Electron microscopy

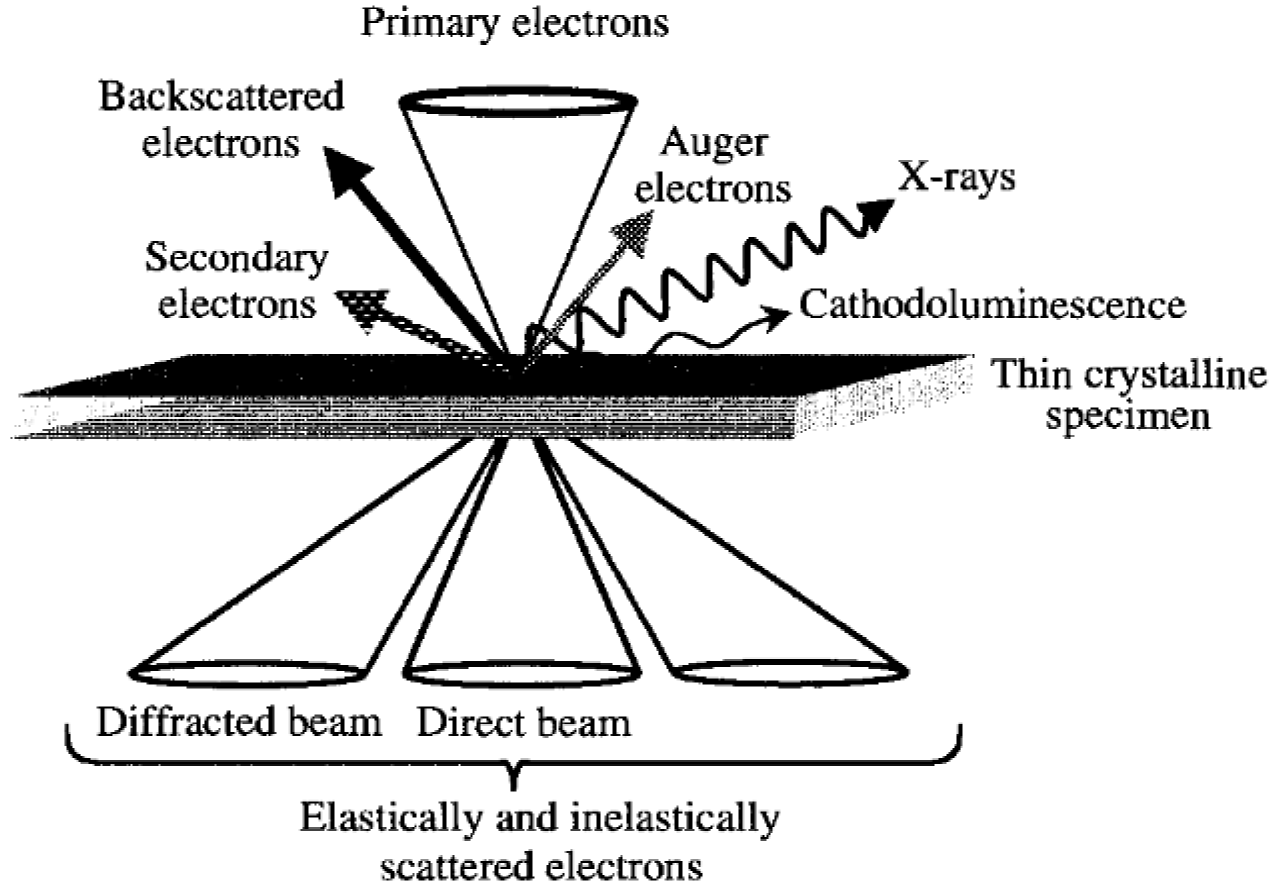


Fig. 2.31. Schematic diagram of the interactions between high-energy electrons and matter in TEM.

Electron microscopy

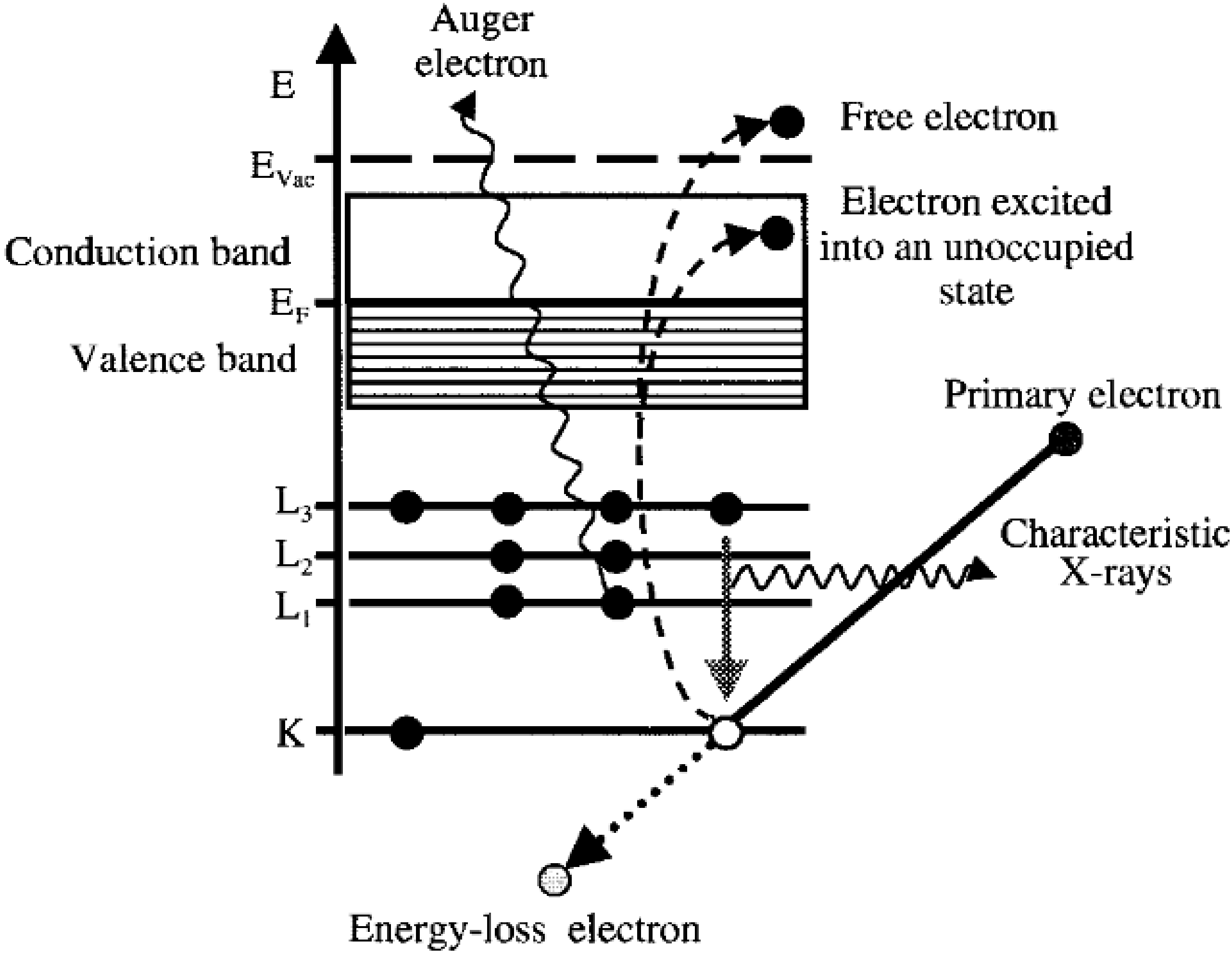
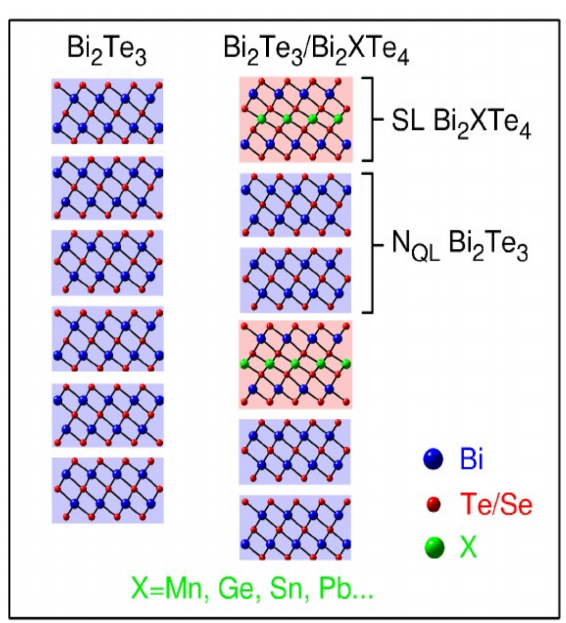


Fig. 2.32. Atomistic view of interaction processes between incident high-energy electrons and electrons of an individual atom.

JKU Linz, Graz

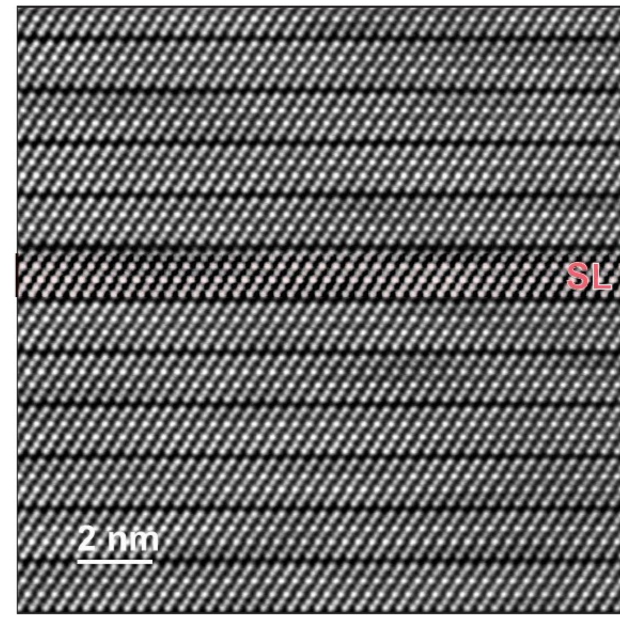
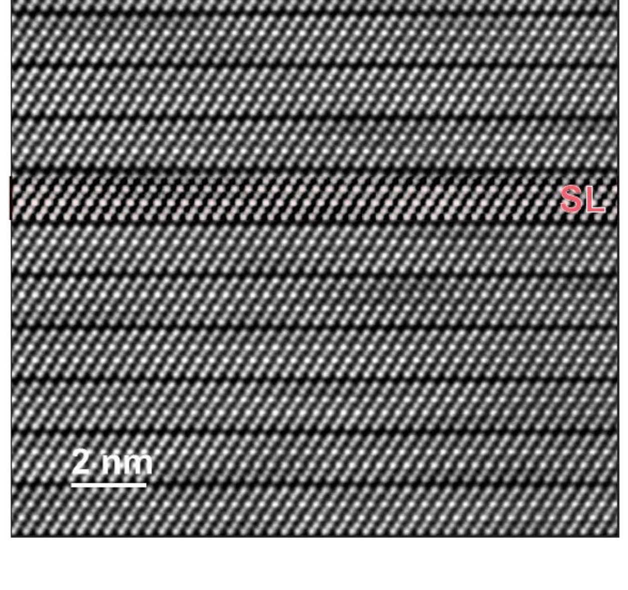
10% Mn Bi_2Te_3

HAADF STEM

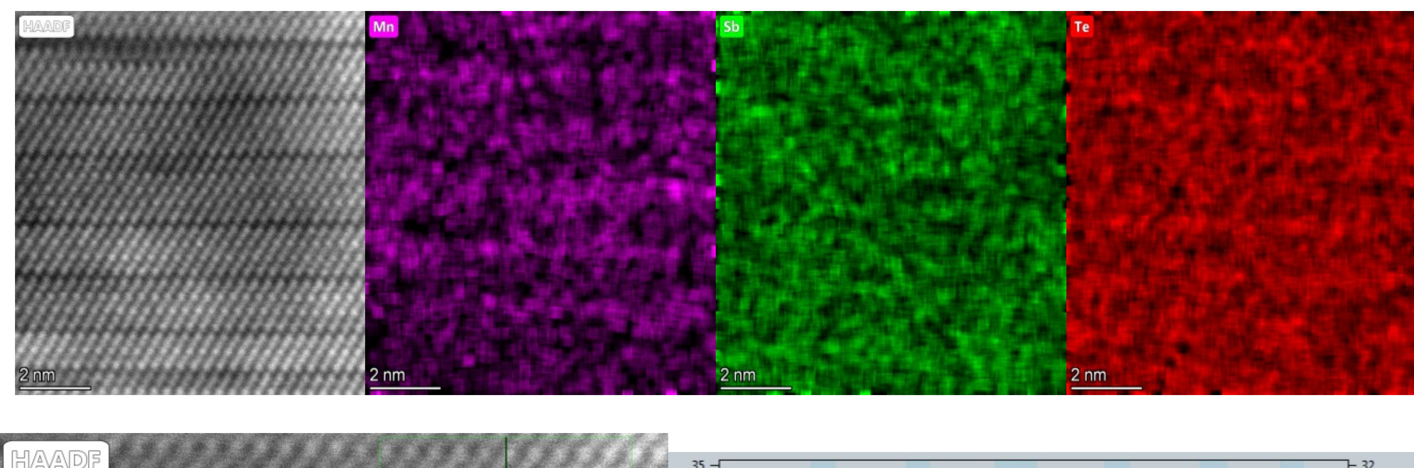


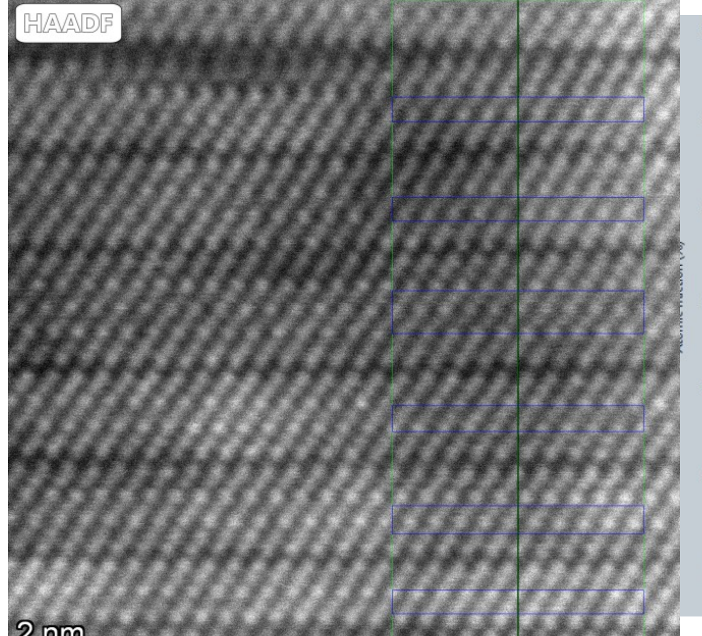
CEITEC Nano

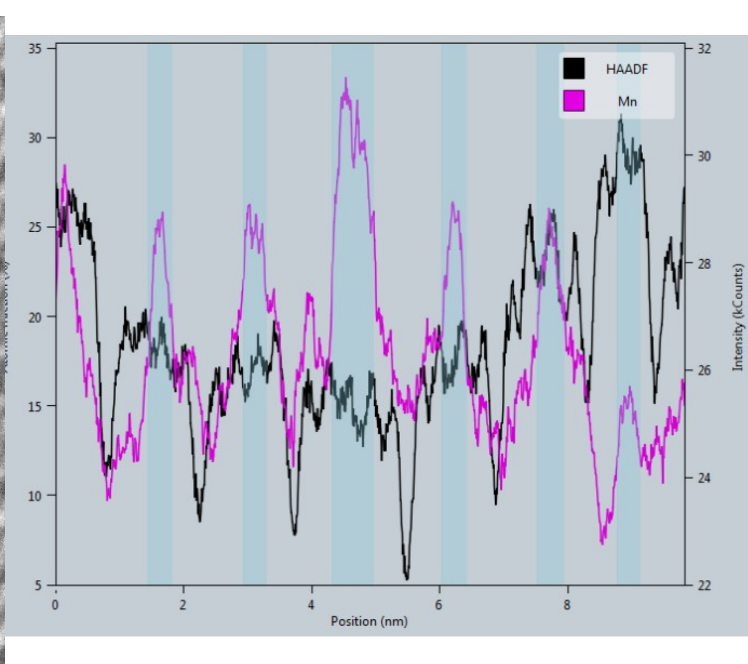
$MnSb_2Te_4$

HAADF STEM



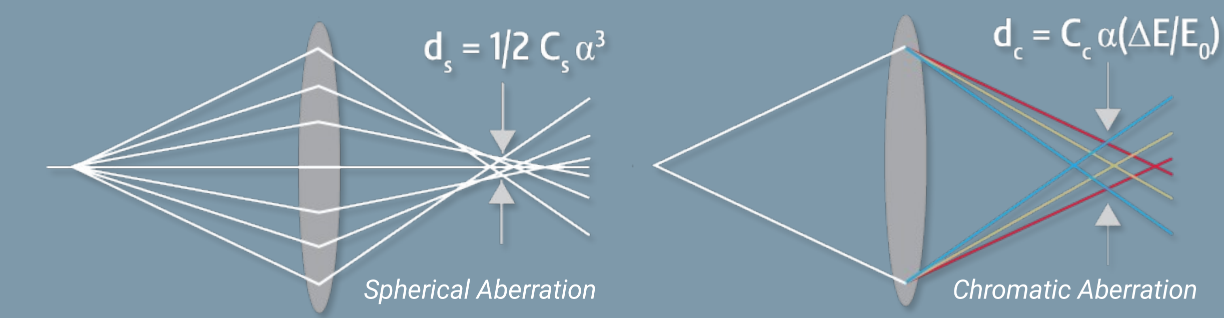




Defect nine atomic planes

Most probably $Mn_2Sb_2Te_5$

TEM Aberration Correction



- Chromatic aberration is distortion that occurs when there is a failure of a lens to focus all colors (wavelengths) to the same convergence point.
  - Correcting the aberration is necessary, otherwise the resulting image would be blurry and delocalized, a form of aberration where periodic structures appear to extend beyond their physical boundaries.
  - Recent improvements in aberration correction have resulted in significantly-improved image quality and sample information.
- Spherical aberration occurs when parallel light rays that pass through the central region of the lens focus farther away than the light rays that pass through the edges of the lens.
  - Result is multiple focal points and a blurred image.

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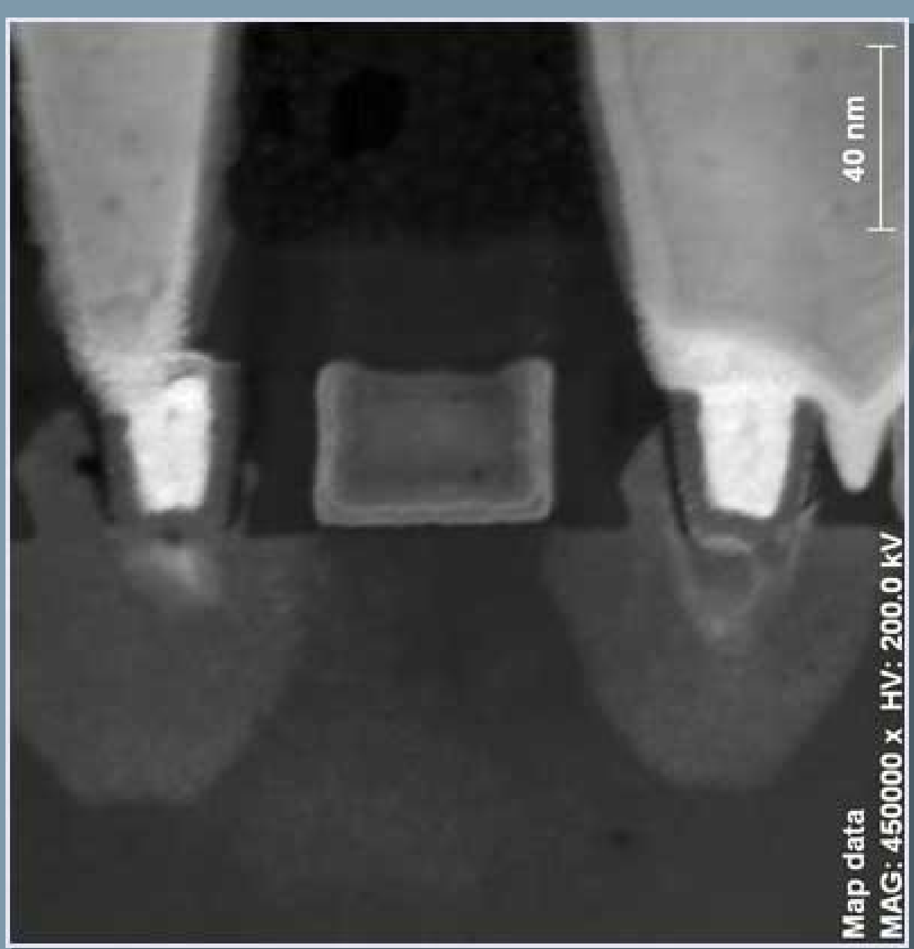
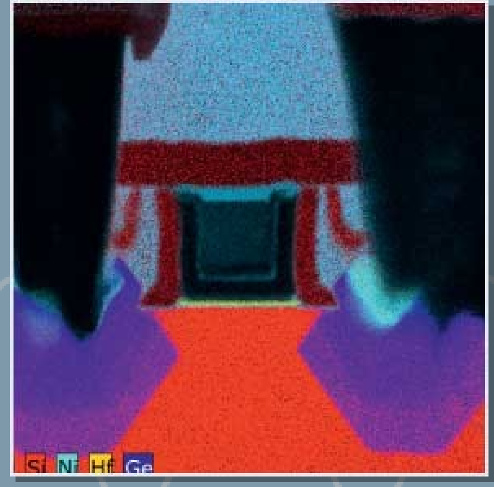
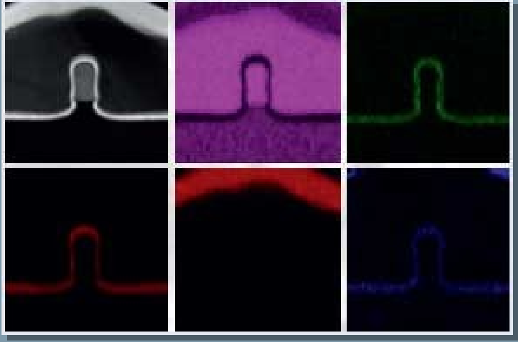
What is Scanning Transmission Electron Microscopy?



STEM image of a 32nm semiconductor device



Elemental map of a 45 nm PMOS transistor structure



EDX map of semiconductor device

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What is a Scanning Electron Microscope?

electron source

electron beam
impact area

vacuum

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Comparing SEM and TEM

	TEM	SEM
Electron Beam	Broad, static beams	Beam focused to fine point; sample is scanned line by line
Voltages Needed	TEM voltage ranges from 60-300,000 volts	Accelerating voltage much lower; not necessary to penetrate the specimen
Interaction of the beam electrons	Specimen must be very thin	Wide range of specimens allowed; simplifies sample preparation
Imaging	Electrons must pass through and be transmitted by the specimen	Information needed is collected near the surface of the specimen
Image Rendering	Transmitted electrons are collectively focused by the objective lens and magnified to create a real image	Beam is scanned along the surface of the sample to build up the image

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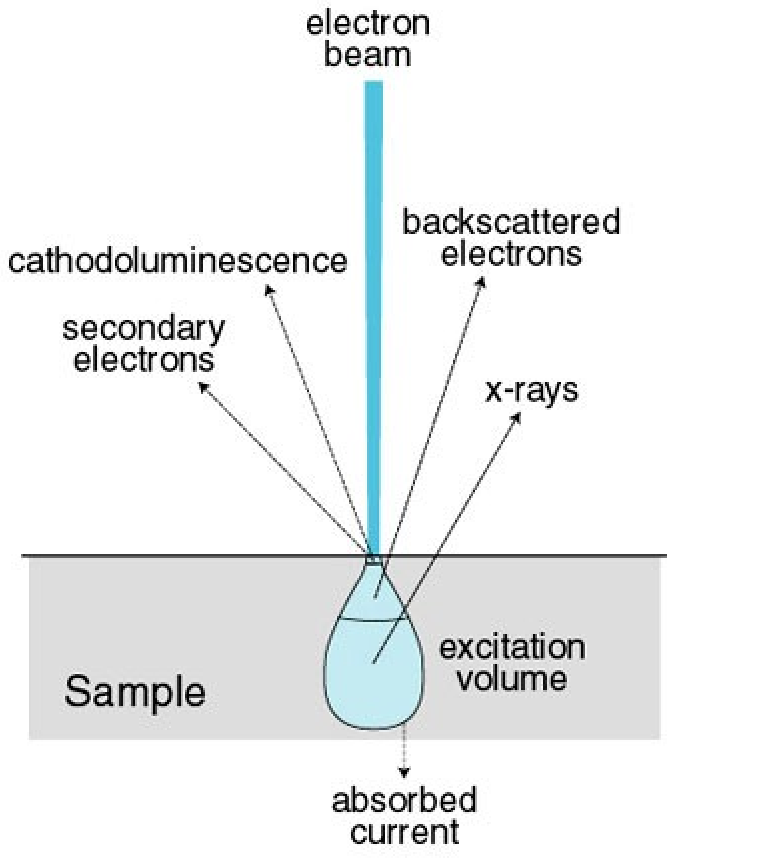
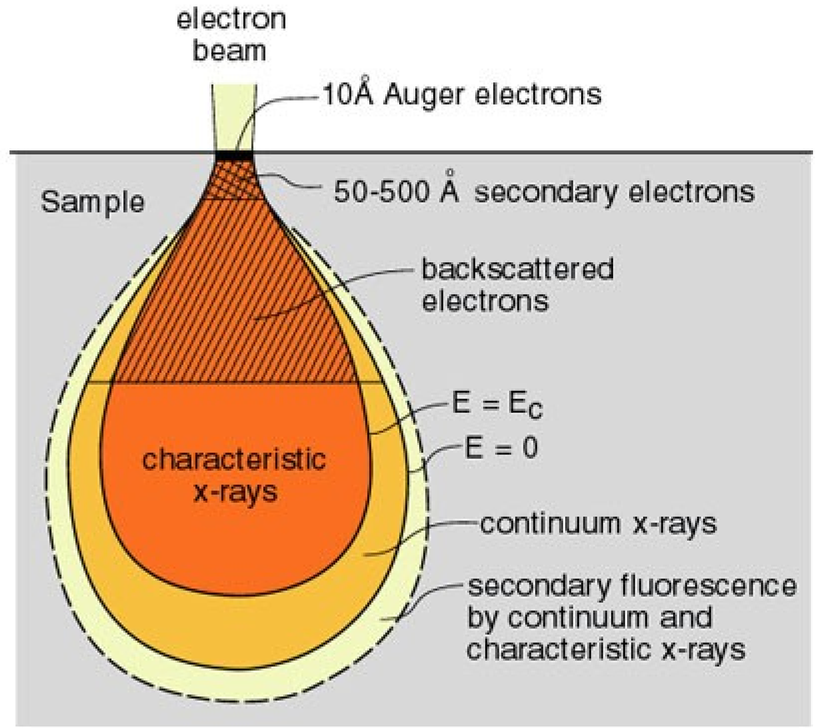
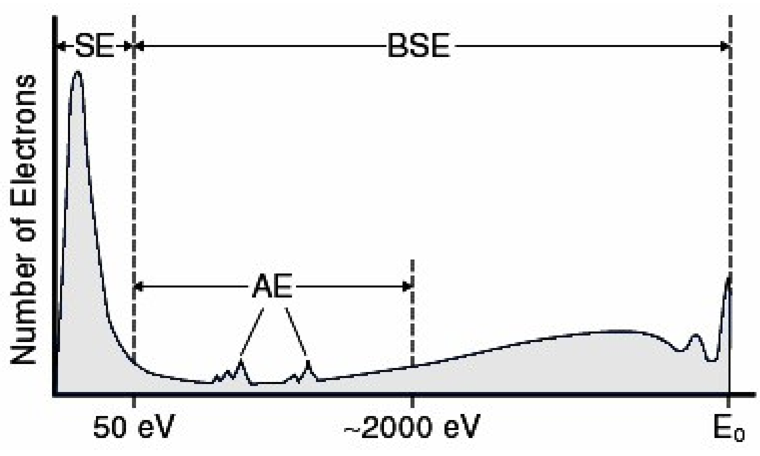
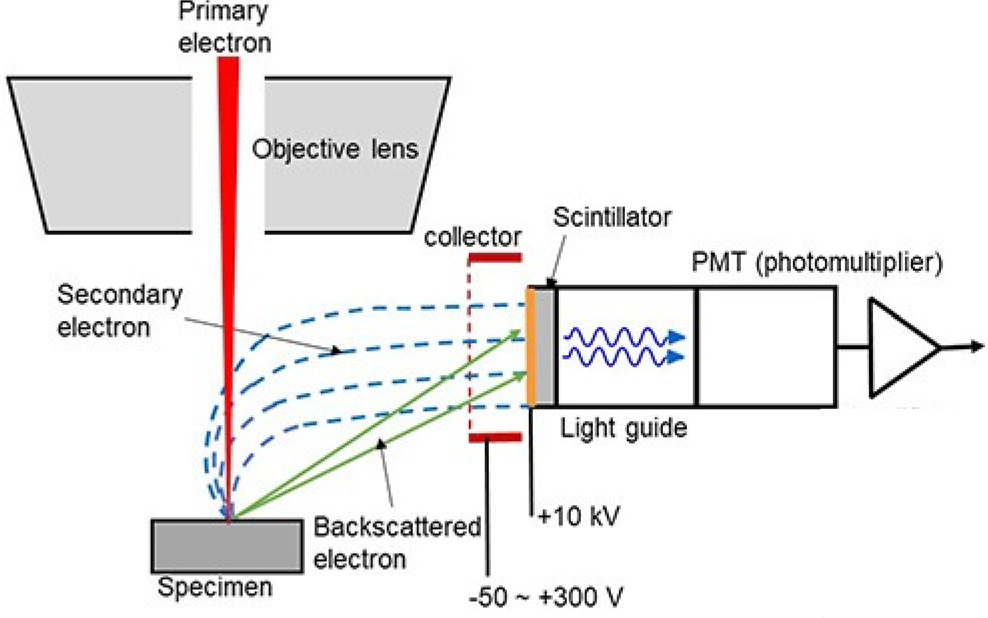
Electron microscopy

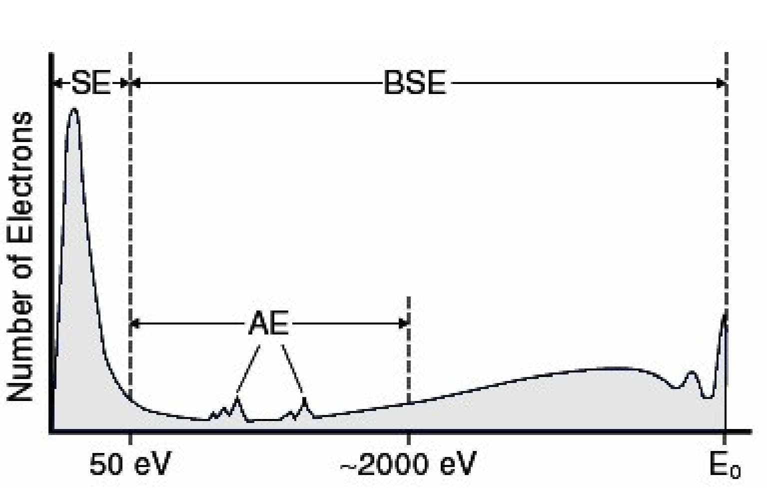






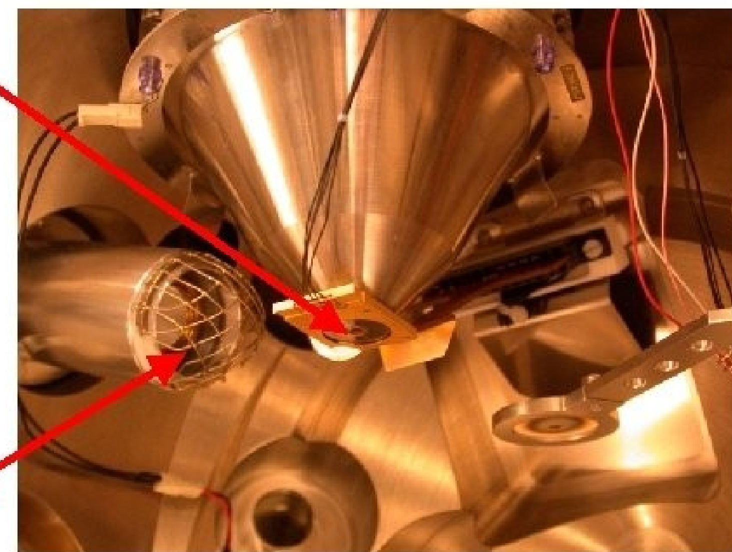
Electron microscopy





Detectors

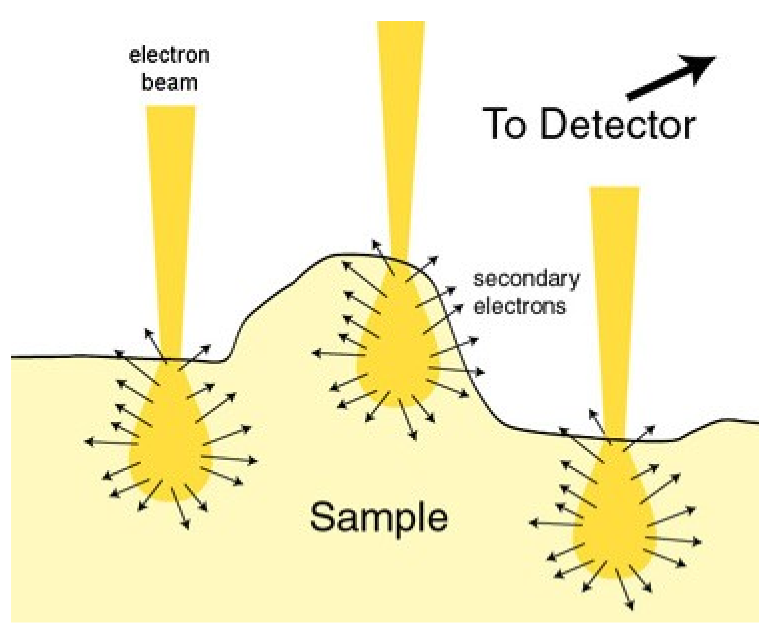
Backscattered electron detector:
(Solid-State Detector)



Secondary electron detector:
(Everhart-Thornley)

Image: Anders W. B. Skilbred, UiO

Electron microscopy



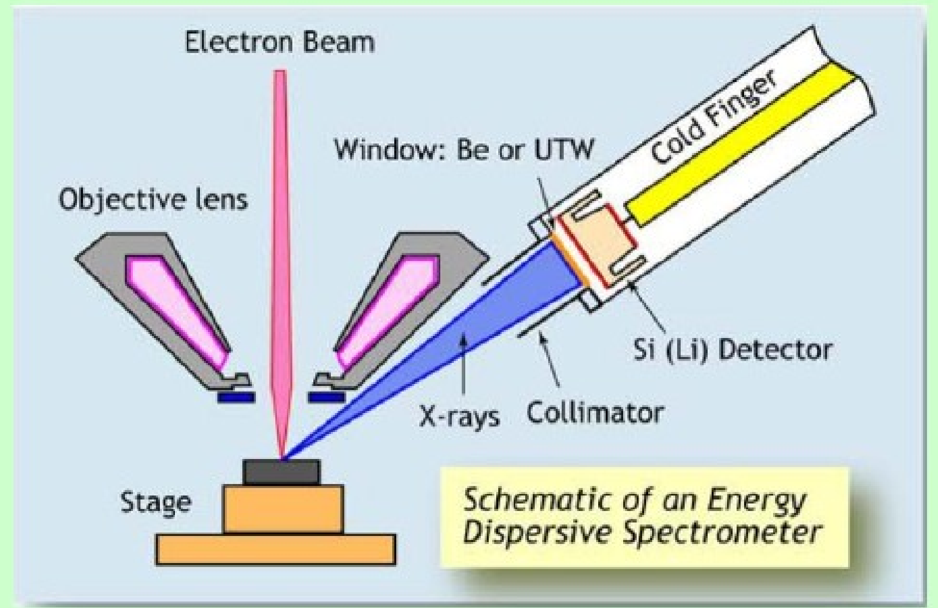
Sekundární elektrony – topografický kontrast

Zpětně odražené – chElectron microscopyický kontrast

Electron microscopy

EDS detector

filament
grid
anode
first ('condensing') lens
aperture
second ('objective') lens
LN2 dewar
viewing microscope
scan coils
Si(Li) detector
crystal
electron detector(s)
sample
isolation valve
detector
diffusion pump
sample current microammeter
to backing pump

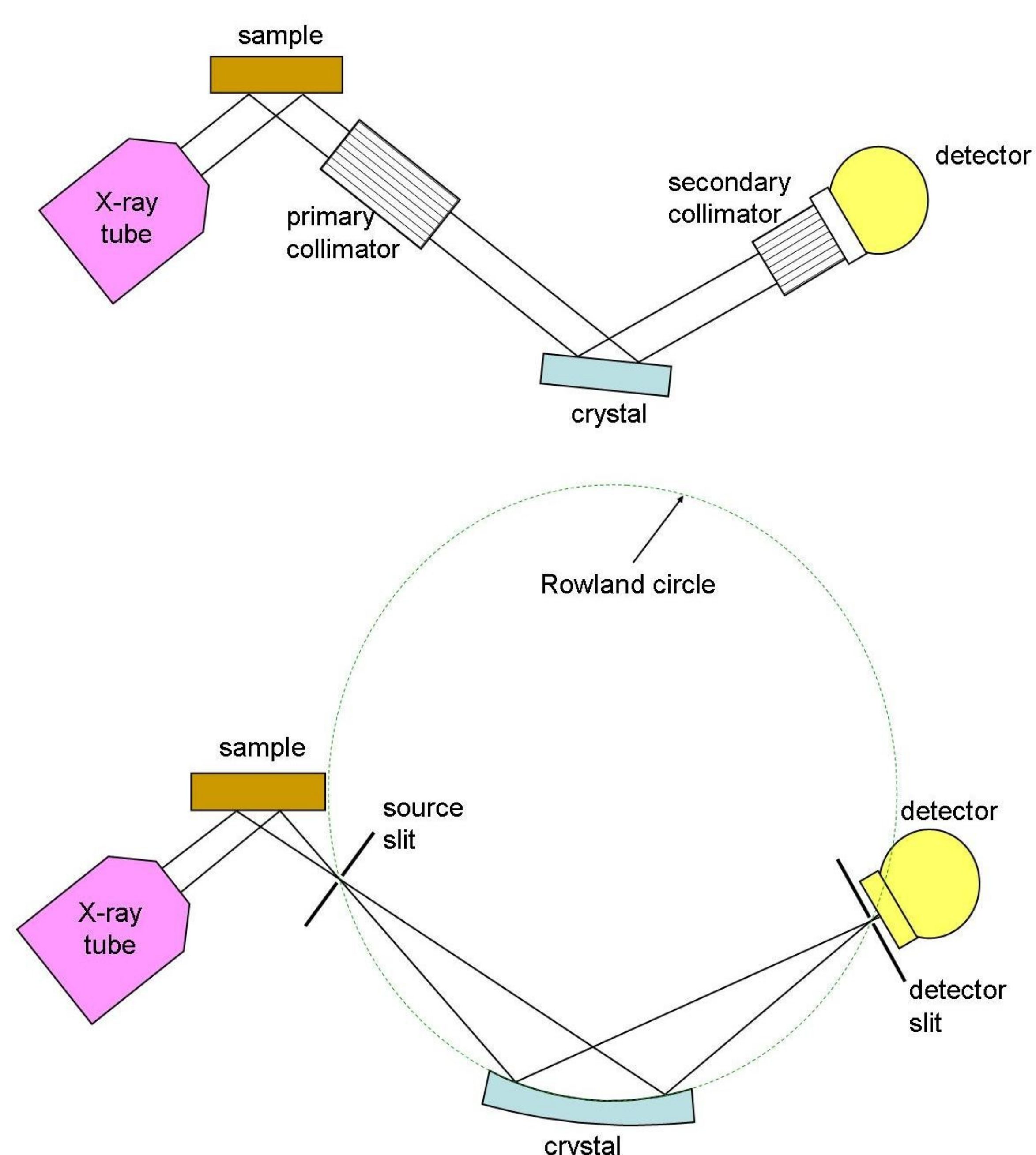


SEM combined with Energy dispersive spectrometer (EDS) and Wavelength Dispersive Spectrometer (WDS).

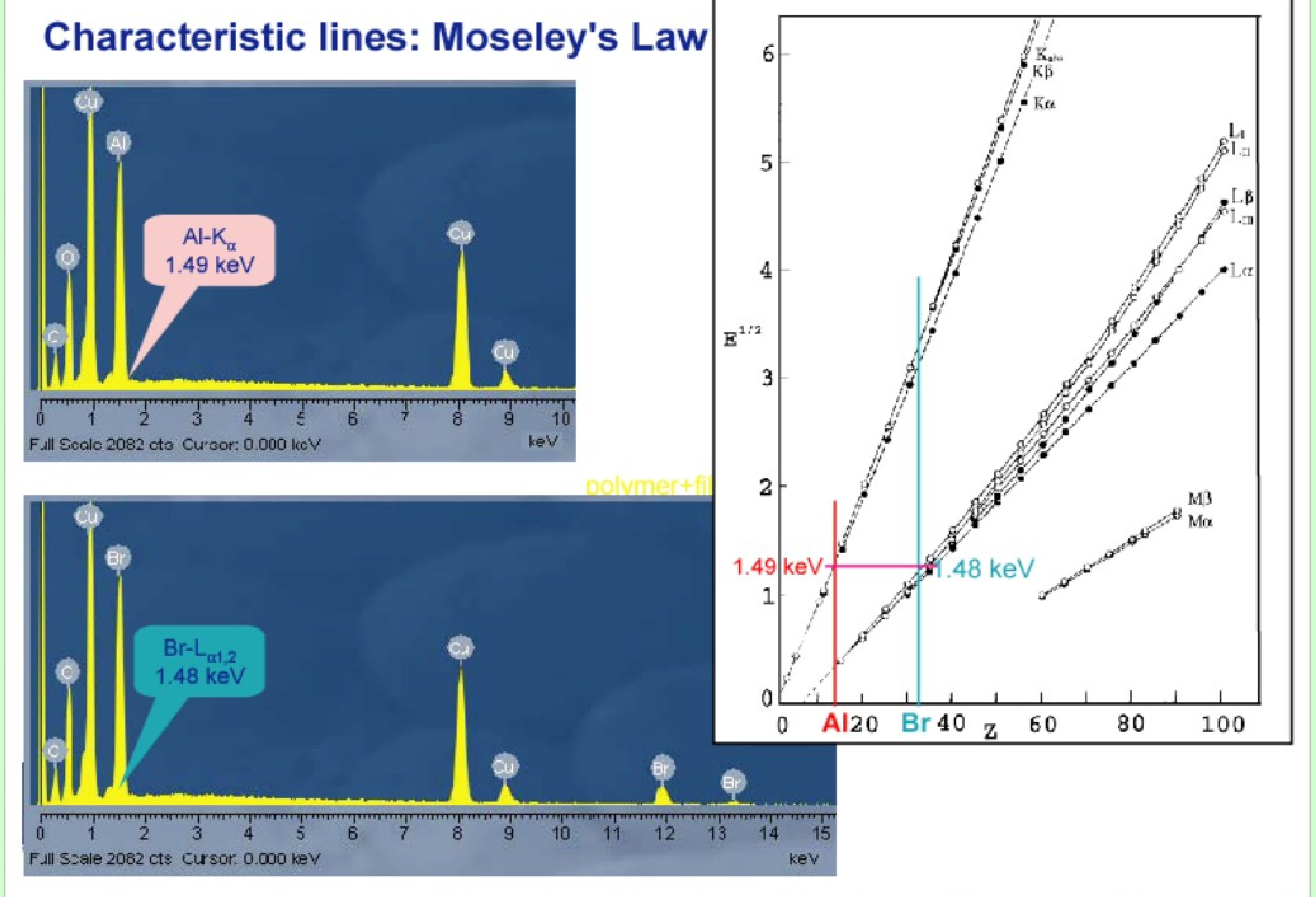
WDS spectrometers

Electron microscopy

WDS:
better energy resolution
better precission
longer time



Electron microscopy



Electron microscopy

Schematic of an Electron Microprobe with a Wavelength Dispersive Spectrometer

Filament
Wehnelt cap
Electron gun
Anode
Condenser lenses
Optical microscope & CCD camera
Objective lens aperture
Probe current detector
Scan coils
Crystal
OM objective lens
Objective lens
Secondary electron detector
Wavelength dispersive spectrometer
X-ray detector
Stage

Energy Resolution of EDS vs WDS

EDS
Au Pt Nb
1.8
2.0
2.2
2.4

WDS
Pt Mα
Au Mα
Pt Mβ
Au Mβ
Nb Lα
Nb Lβ
2.00
2.05
2.10
2.15
2.20
2.25
2.30
X-ray Energy (keV)

What is a Focused Ion Beam? (FIB)

Platinum Nano-Wire

Physical Failure Analysis

FIB-cut in steel v2a FE by 1nA to 1B milling-002 steel

wafer imaged with a plasma FIB

5µm

V400ACE

FEI V400ACE Focused Ion Beam

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EELS

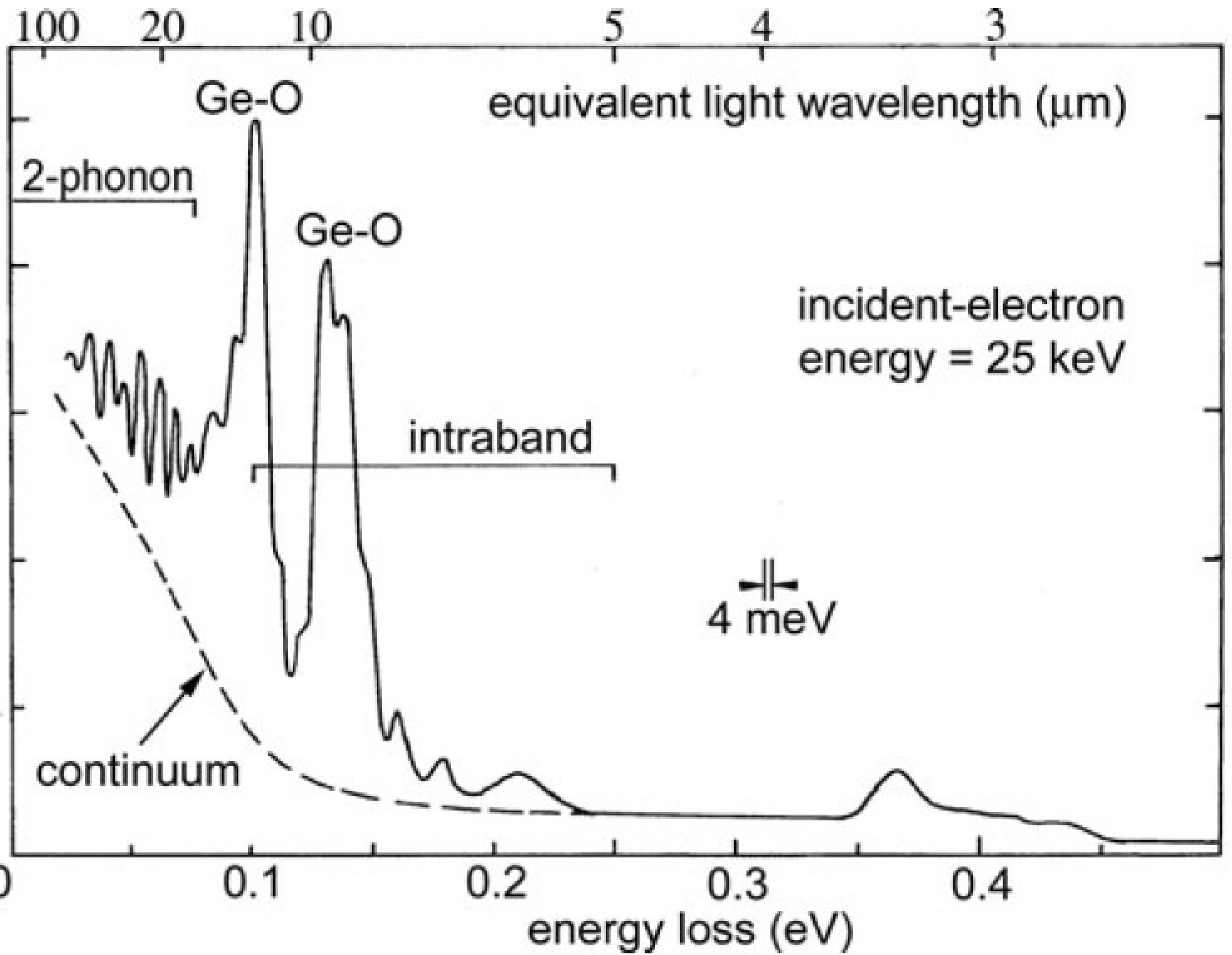


Fig. 1.9 Energy-loss spectrum of a 25-nm germanium film showing phonon and vibrational modes, as well as intraband electronic transitions (Schröder, 1972)

Electron microscopy

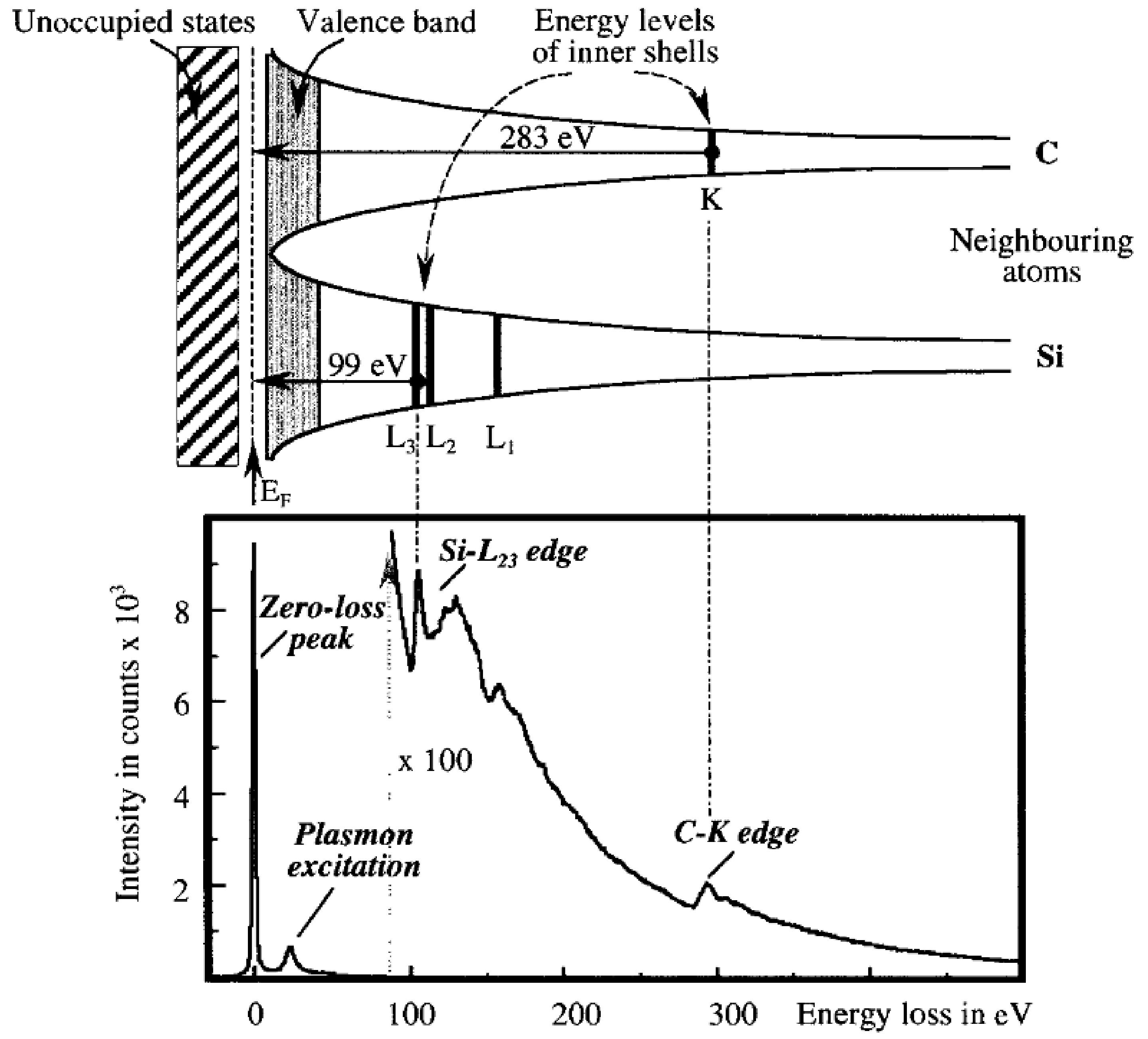
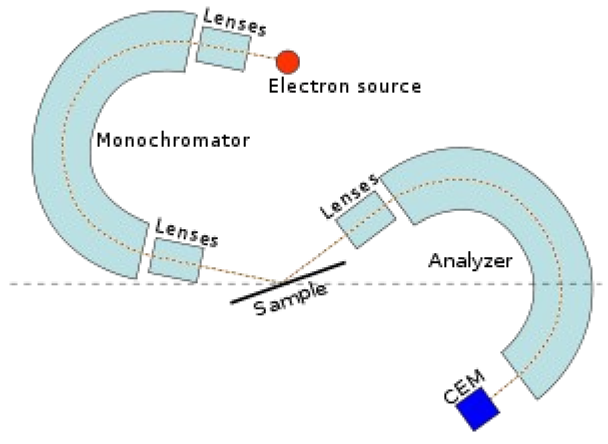
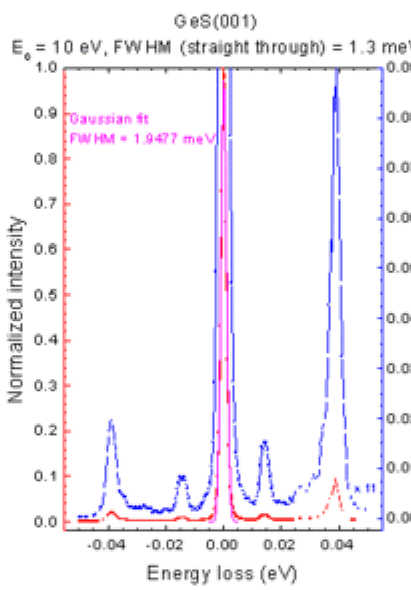
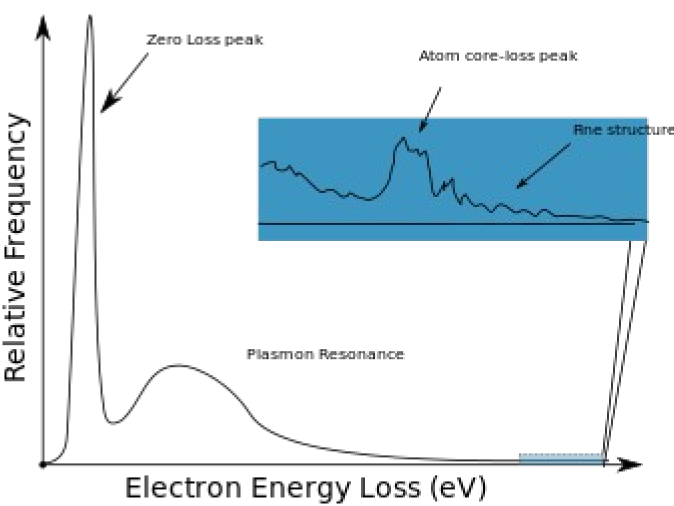


Fig. 2.35. Typical EEL spectrum and corresponding energy-level scheme.

EELS







EELS

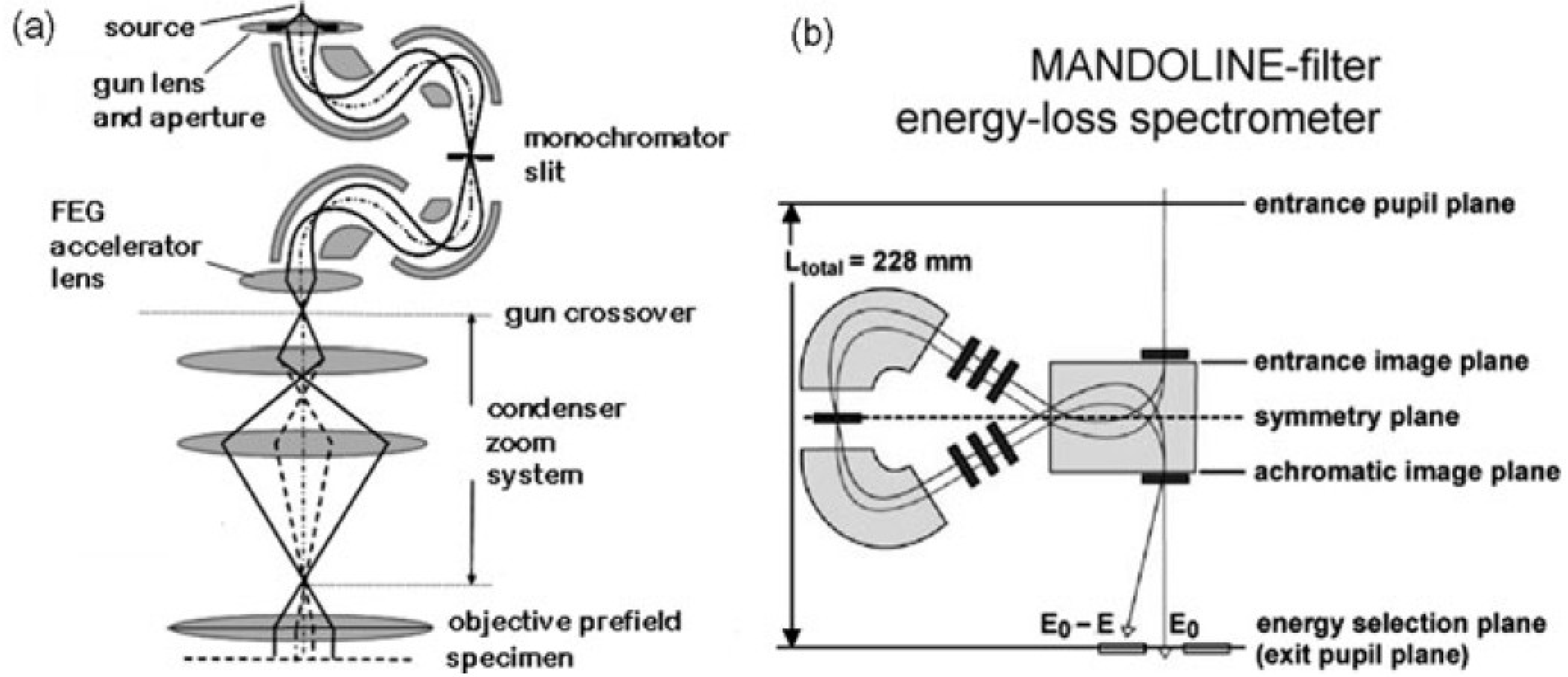
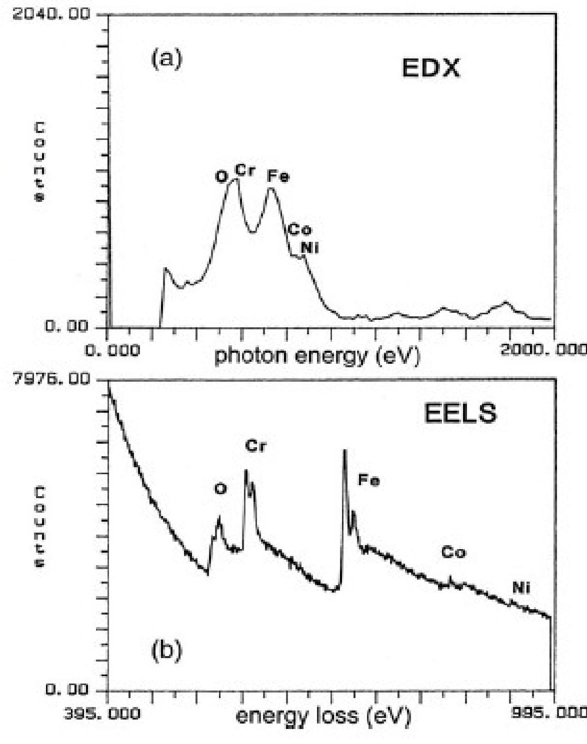


Fig. 2.32 Components of the Zeiss SESAM instrument: (**a**) electrostatic omega filter (dispersion $\approx$ 12 μm/eV at the midplane slit) and (**b**) MANDOLINE filter, whose dispersion at the energy-selecting plane exceeds 6 μm/eV

EELS

Fig. 1.11 (**a**) X-ray emission spectrum recorded from an oxidized region of stainless steel, showing overlap of the oxygen *K*-peak with the *L*-peaks of chromium and iron. (**b**) Ionization edges are more clearly resolved in the energy-loss spectrum, as a result of the better energy resolution of the electron spectrometer (Zaluzec et al., 1984). From Zaluzec et al. (1984), copyright San Francisco Press, Inc., with permission



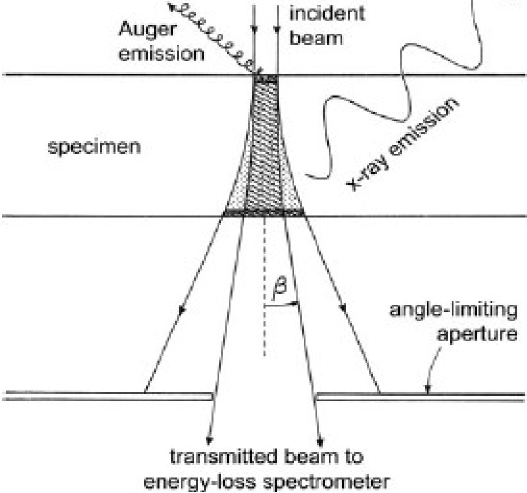


Fig. 1.12 Spreading of an electron beam within a thin specimen. X-rays are emitted from the *dotted region*, whereas the energy-loss spectrum is recorded from the *hatched region*, the spectrometer entrance aperture having a collimating effect. Auger electrons are emitted within a small depth adjacent to each surface

EELS

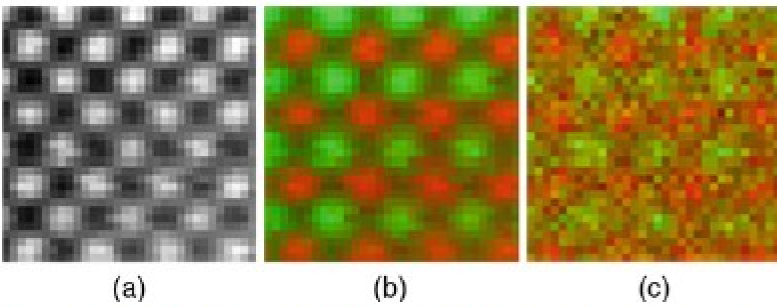


Fig. 1.14 (**a**) HAADF-STEM image of a [100]-projected GaAs specimen, showing bright Ga and As atomic columns. (**b**) EELS image showing the Ga-L_{23} intensity in *green* and As-L_{23} intensity in *red*. (**c**) EDX spectroscopy image with Ga-Kα intensity in *green* and As-Kα intensity in *red*. Courtesy of M. Watanabe

EELS

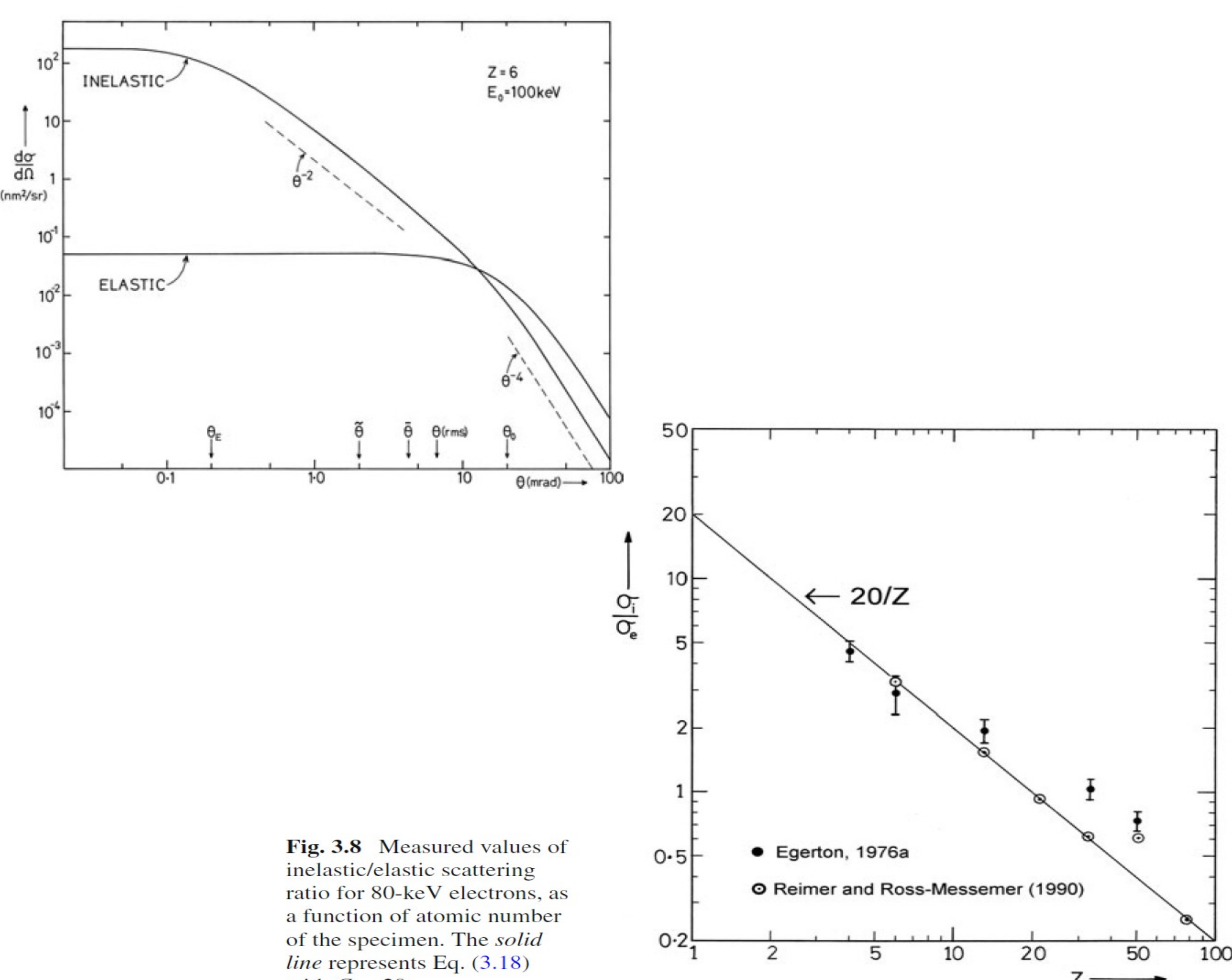


Fig. 3.7 Angular dependence of the differential cross sections for elastic and inelastic scattering of 100-keV electrons from a carbon atom, calculated using the Lenz model (Eqs. (3.50), (3.7), and (3.15)). Shown along the horizontal axis are (*from left to right*) the characteristic, median, mean, root-mean-square and effective cutoff angles for total inelastic scattering, evaluated using Eqs. (3.53), (3.54), (3.55), and (3.56)

Fig. 3.8 Measured values of inelastic/elastic scattering ratio for 80-keV electrons, as a function of atomic number of the specimen. The *solid line* represents Eq. (3.18) with $C = 20$

EELS

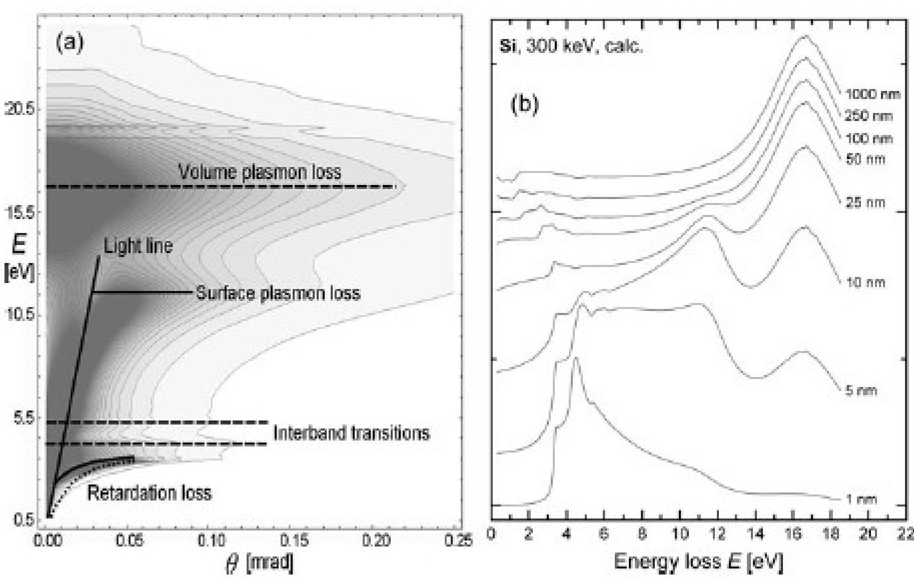
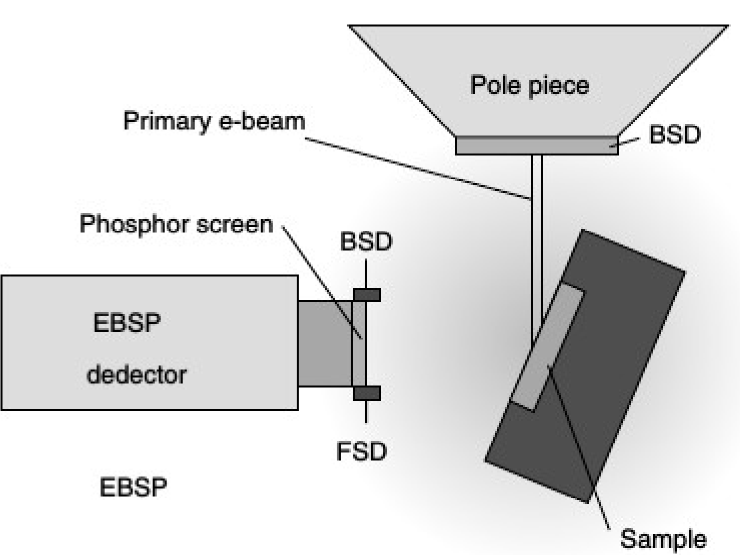
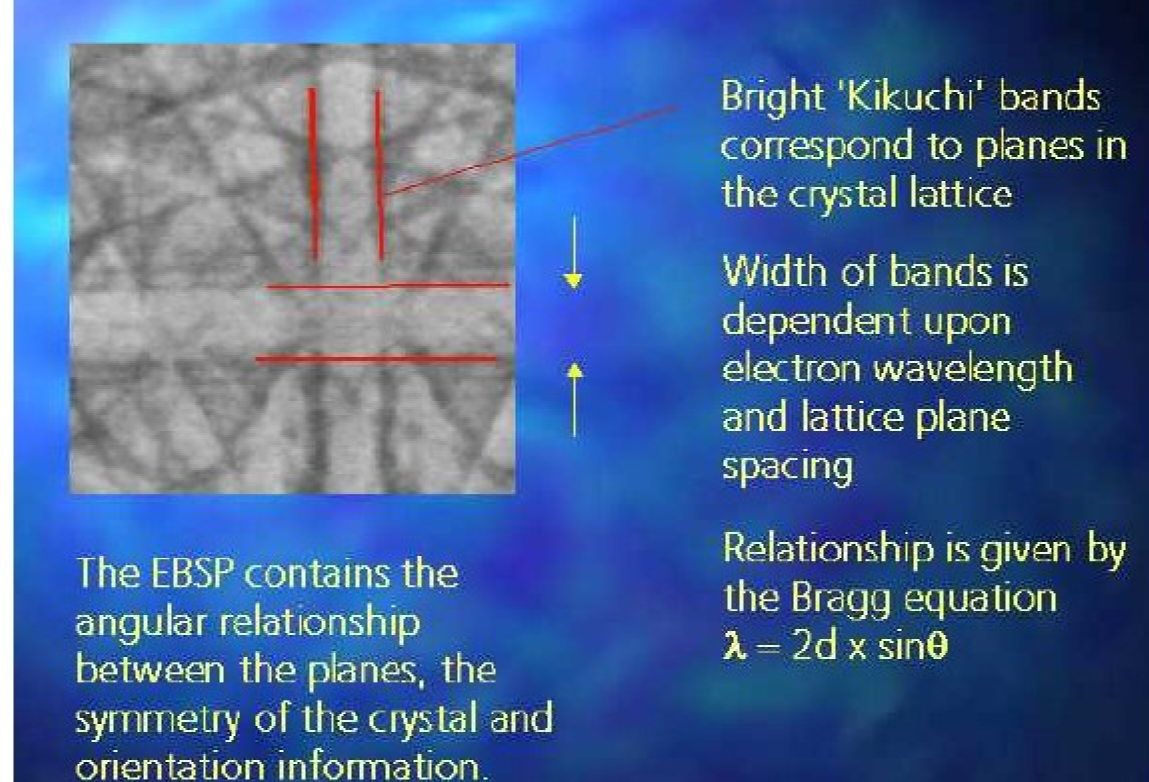


Fig. 3.26 (**a**) Schematic and contour plot (*gray background*) showing the calculated energy loss and angular dependence of intensity for a 50-nm Si specimen and 300-keV incident electrons. (**b**) Energy-loss spectra calculated from Eq. (3.84) for 2.1-mrad collection semi-angle, 300-keV incident electrons, and various thicknesses of silicon, assuming no surface-oxide later. The intensities are normalized and the spectra displaced vertically for clarity. Reproduced from Erni and Browning (2008), copyright Elsevier. A computer program for evaluating Eq. (3.84) is described in Appendix B9

EBSD



EBSD



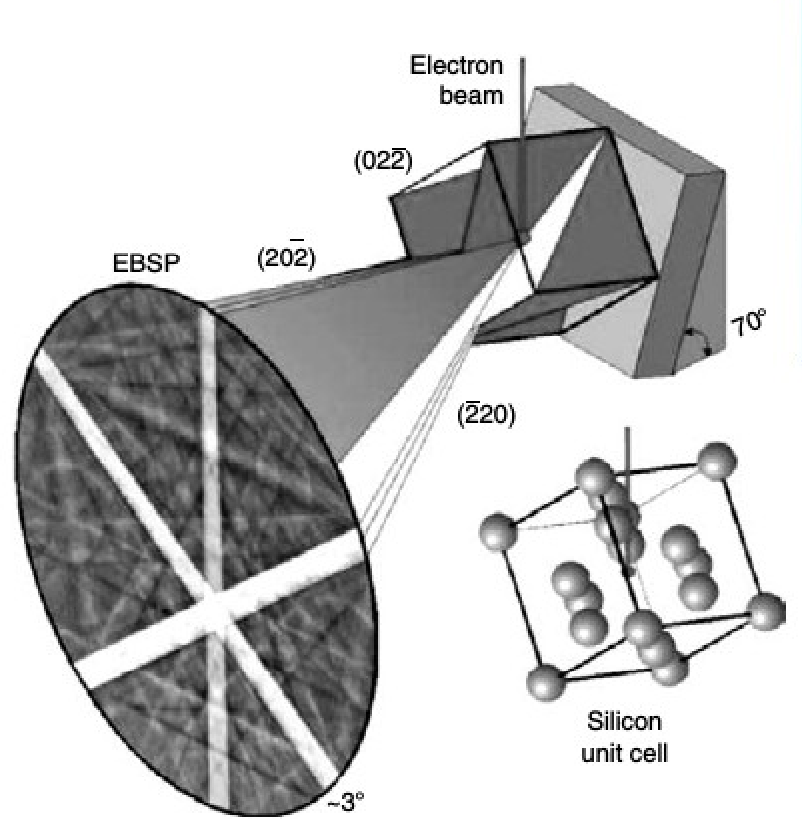


FIGURE 2.8. Electron interaction with crystalline material. (Adapted from [13].)

EBSD

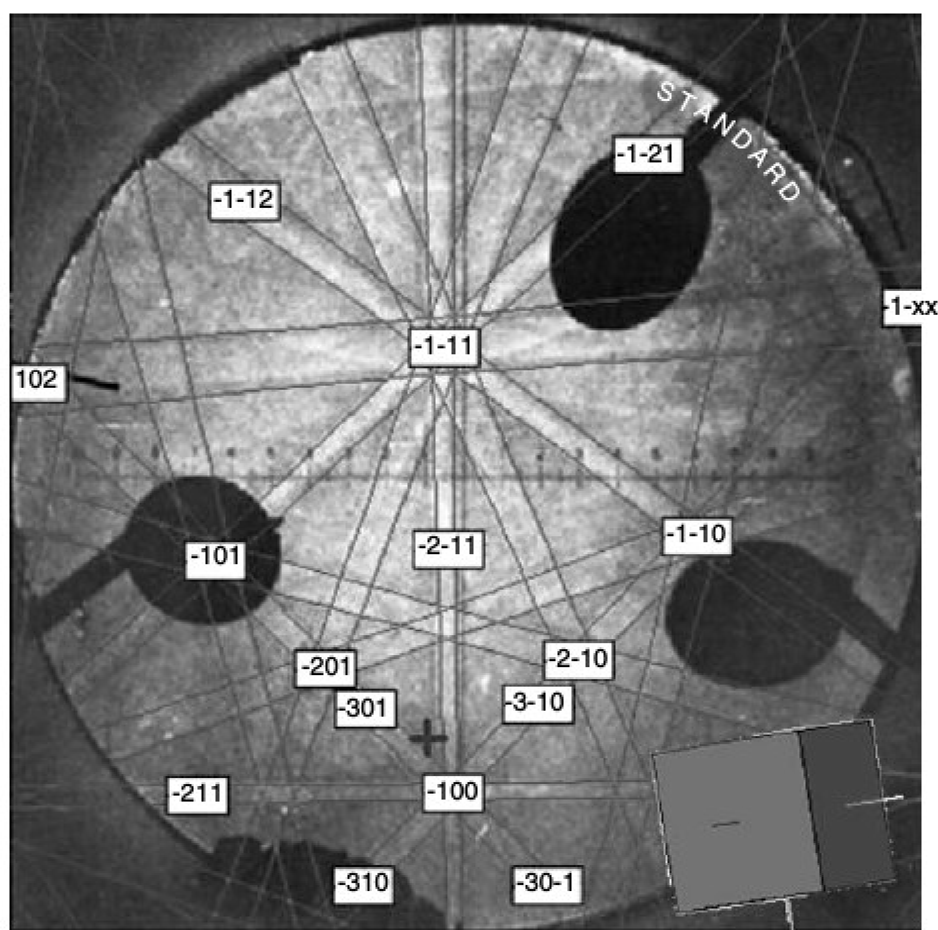
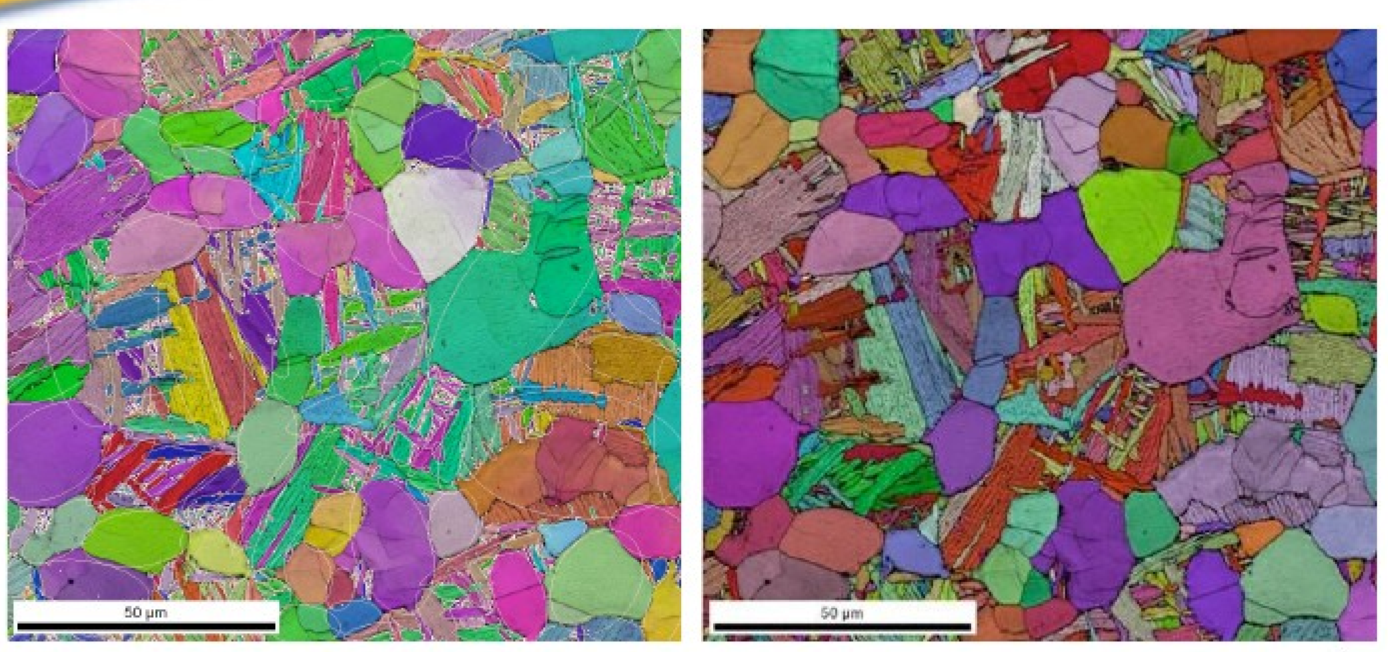


FIGURE 2.6. Venables' early EBSP indexed by modern software. The cross shows the computed location of the pattern center. Graphic inset shows approximate orientation of the crystal with respect to the sample surface.

EBSD

Titanium Grain Structure



$10\bar{1}0$

0001 $2\bar{1}\bar{1}0$

Once the grain shape is known, the orientation of the major axis can also be examined to understand growth mechanisms.

EDAX TSL

Electron diffraction

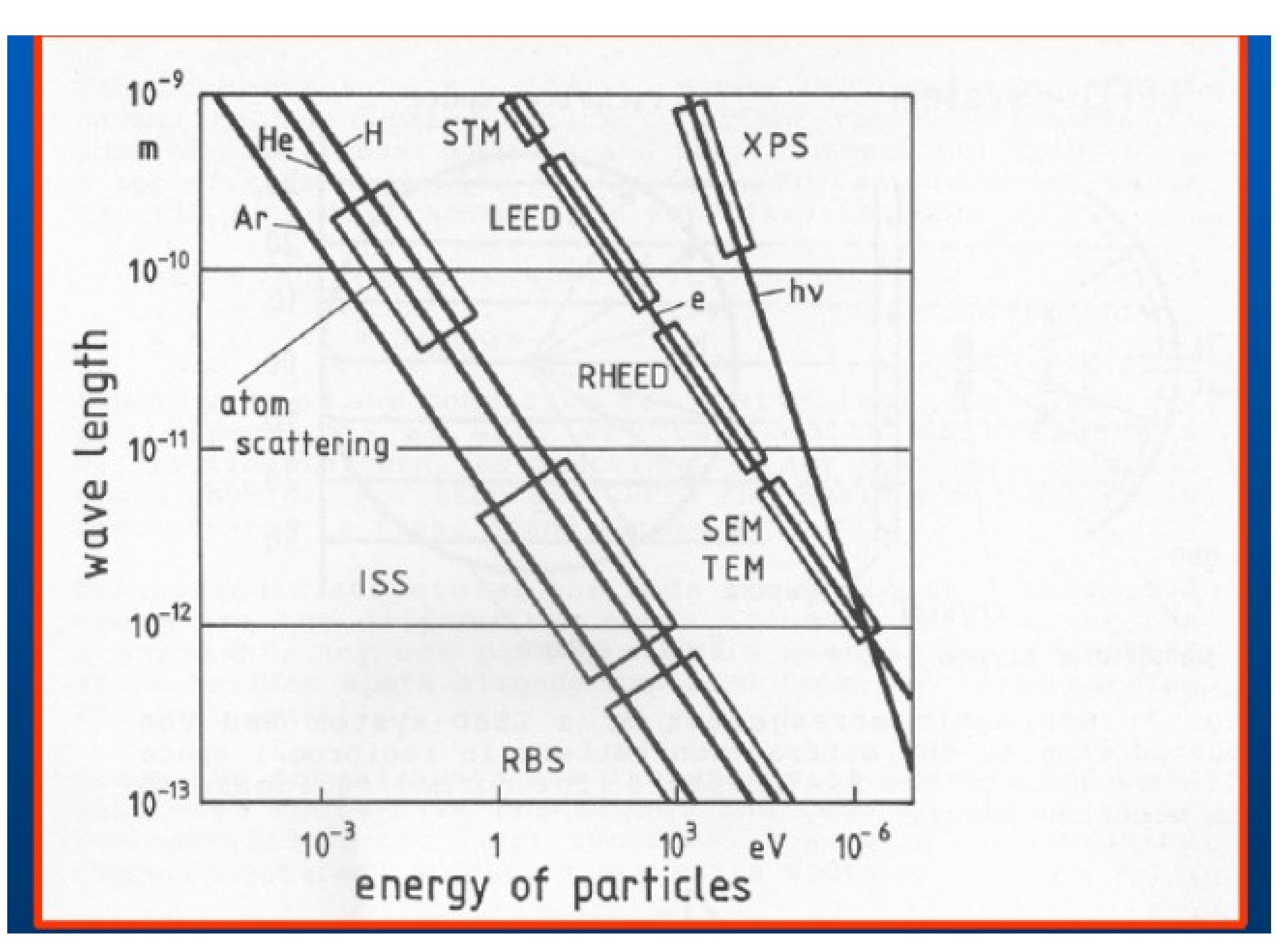
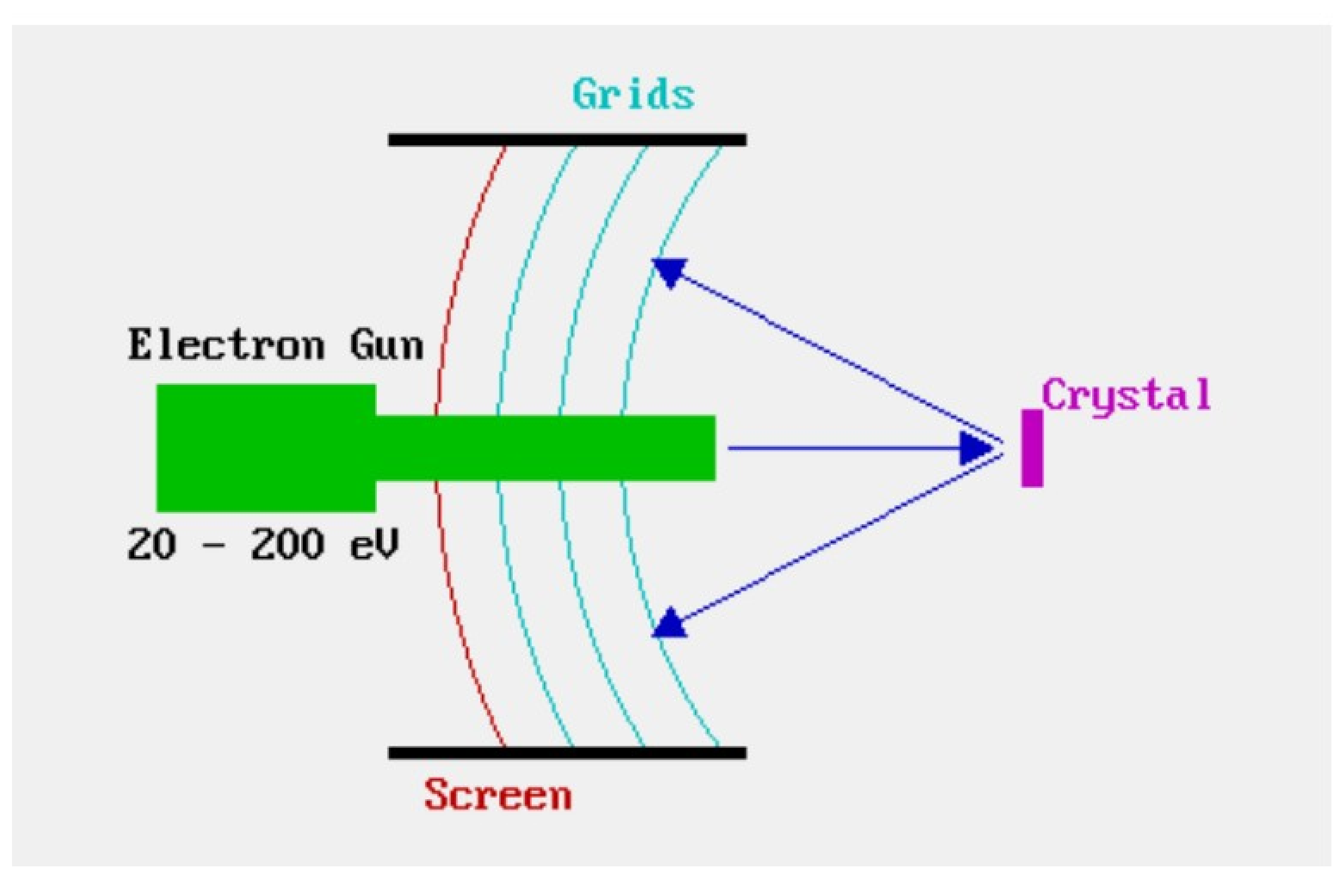


Schéma LEED

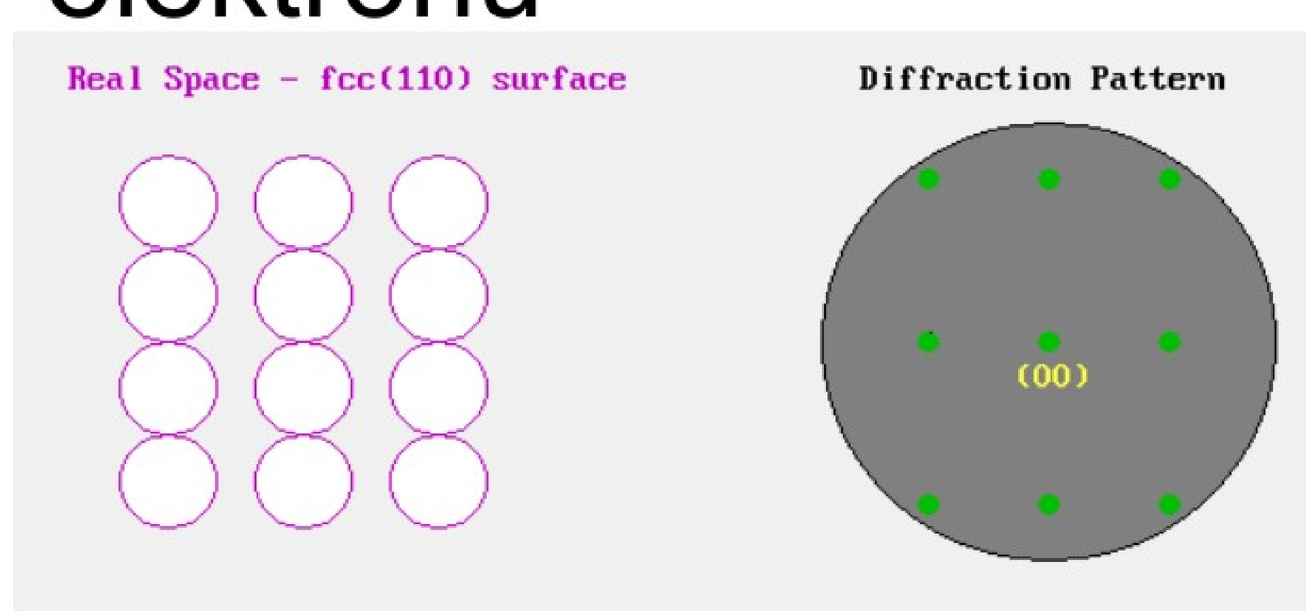


LEED

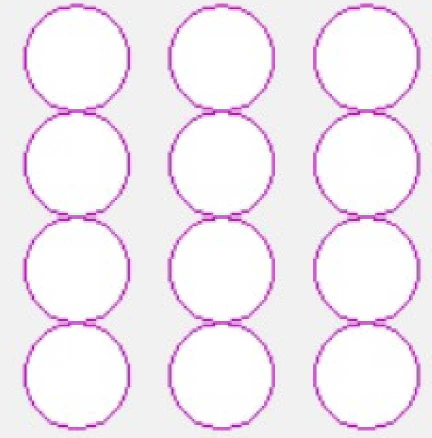
- Typické energie 20 eV až 200 eV, ve směru normály povrchu
- Kvalitativně:
  - Z pozice spotů lze zjistit také velikost, symetrii, atd. absorbátů na povrchu, lze samozřejmě analyzovat i jen samotný substrát
- Kvantitativně:
  - Přesné pozice atomů lze zjistit z měření intenzit, závislosti I-V, porovnáním s teorií a modely
  - Profil intenzit jednotlivých spotů

Co se stane pokud zvýšíme energii elektronů

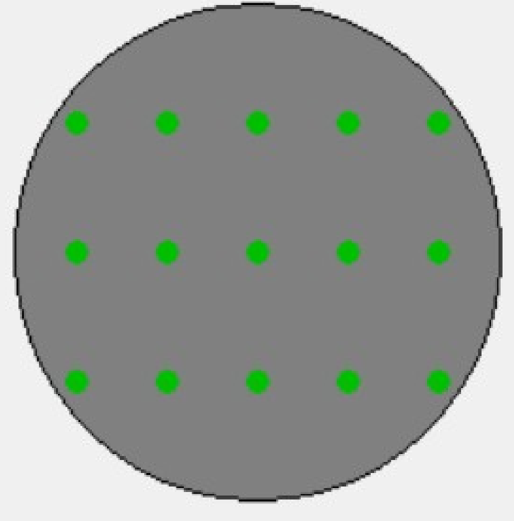
- Nízká
- Zde jen n = 1 pro úhly θ, viz podmínka pro vznik maxim.
-
-
-
- Vyšší
  - (dvojnásobná)



Real Space - fcc(110) surface

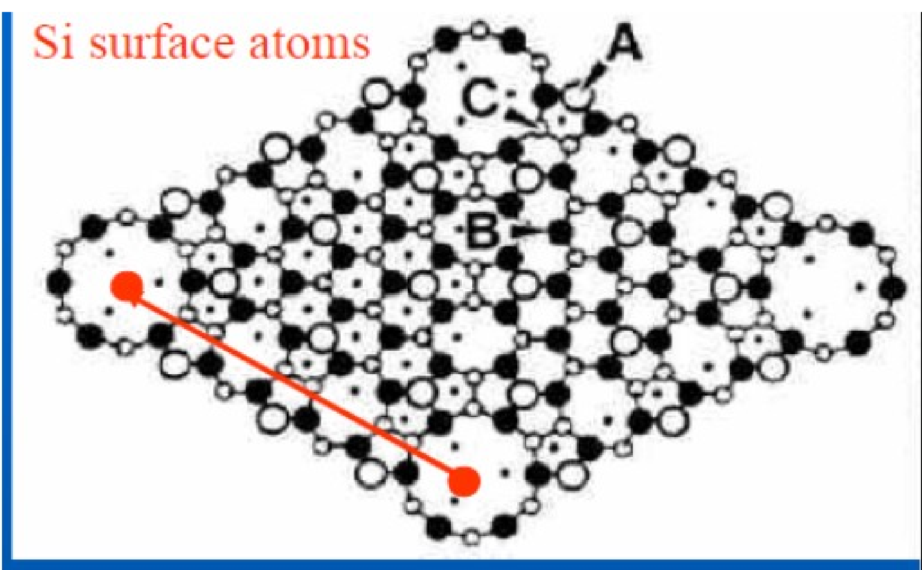


Diffraction Pattern



Electron diffraction

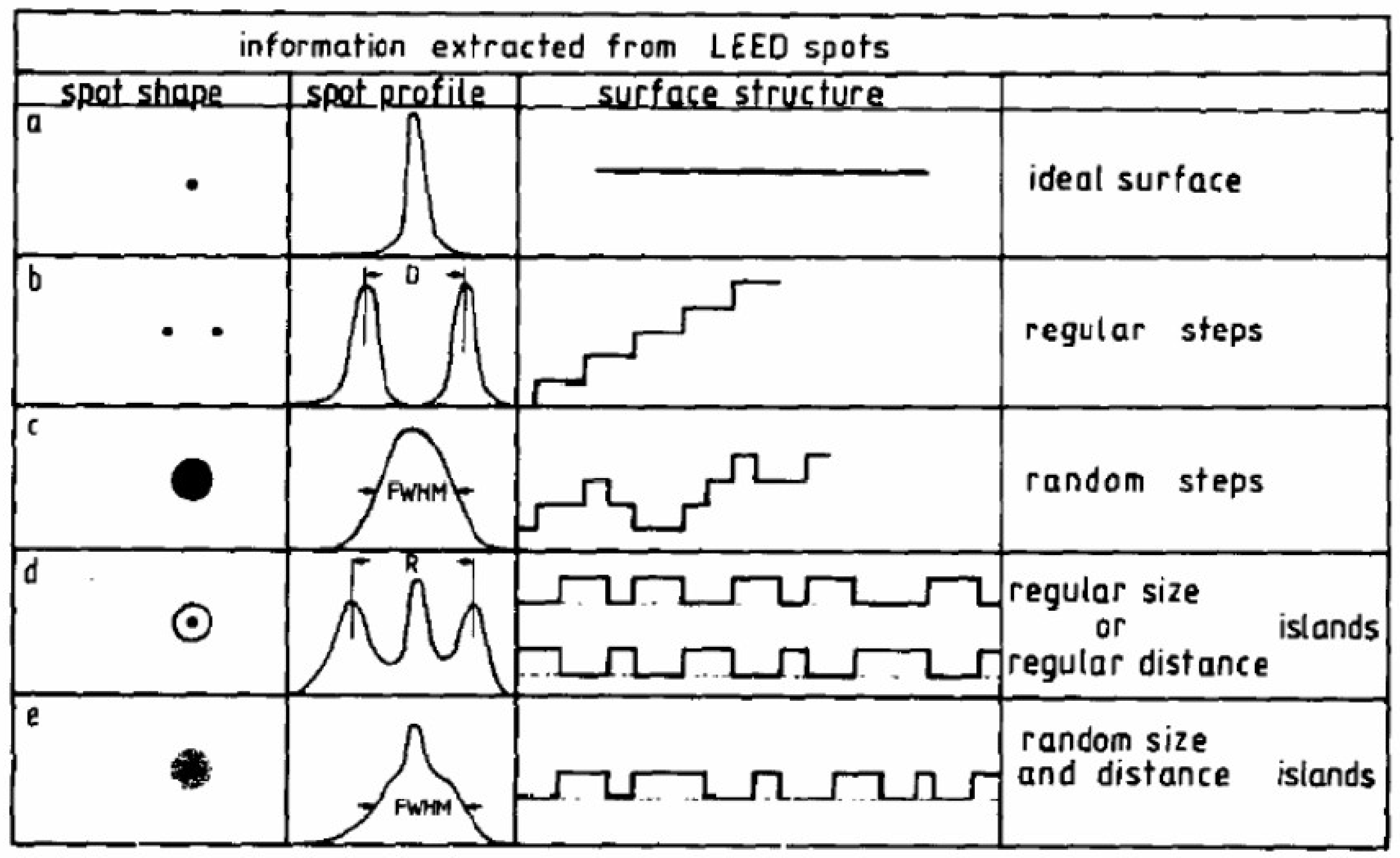
Si 7x7



35 eV

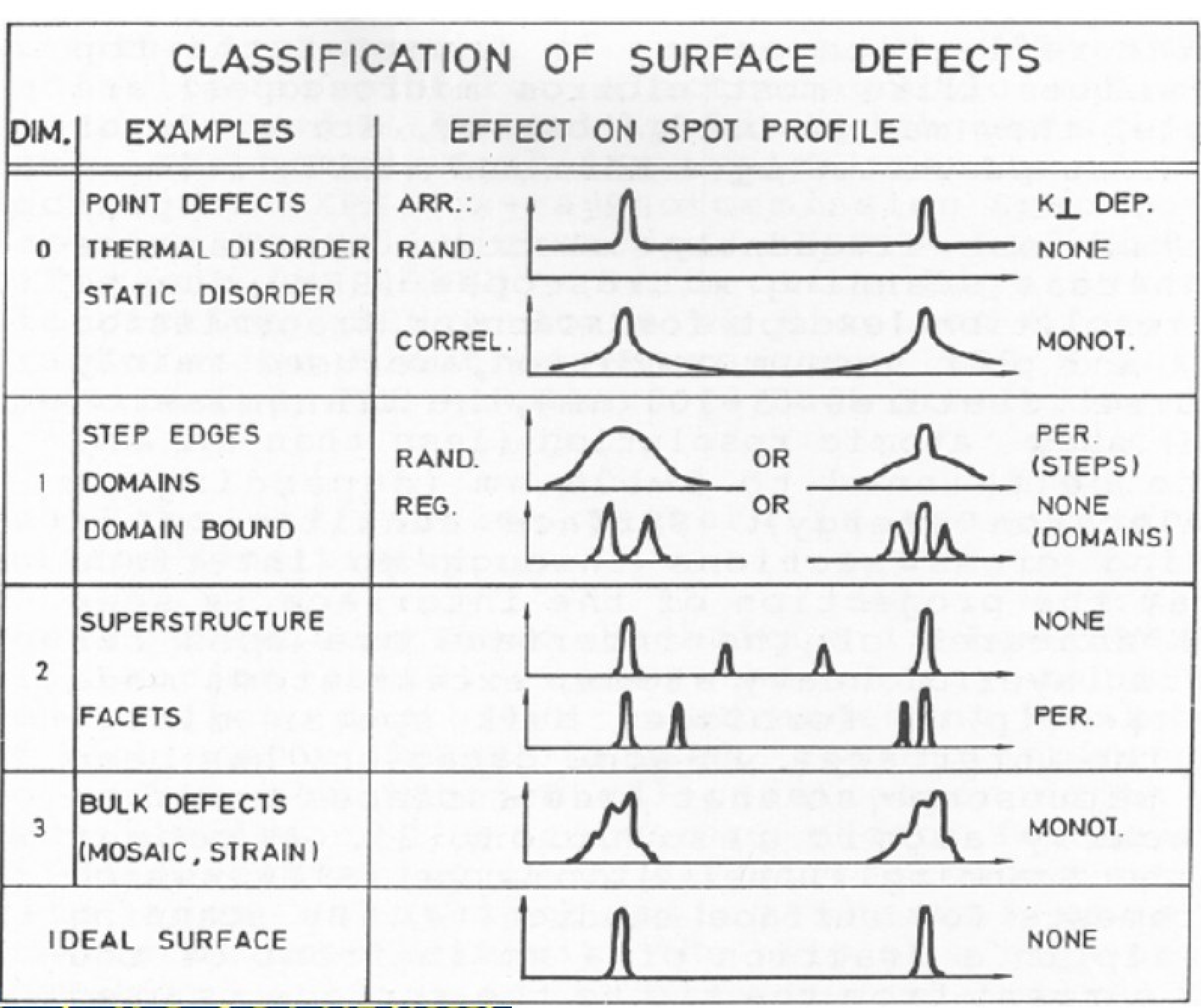
65 eV

Electron diffraction



Gronwald and Henzler M. Surf. Sci. 117 (1982)

Electron diffraction



CLASSIFICATION OF SURFACE DEFECTS

DIM.	EXAMPLES	EFFECT ON SPOT PROFILE	
0	POINT DEFECTS THERMAL DISORDER STATIC DISORDER	ARR.: RAND.	K⊥ DEP. NONE
		CORREL.	MONOT.
1	STEP EDGES DOMAINS DOMAIN BOUND	RAND. OR	PER. (STEPS)
		REG. OR	NONE (DOMAINS)
2	SUPERSTRUCTURE		NONE
	FACETS		PER.
3	BULK DEFECTS (MOSAIC, STRAIN)		MONOT.
	IDEAL SURFACE		NONE

Gronwald and Henzler M. Surf. Sci. 117 (1982)

Electron diffraction

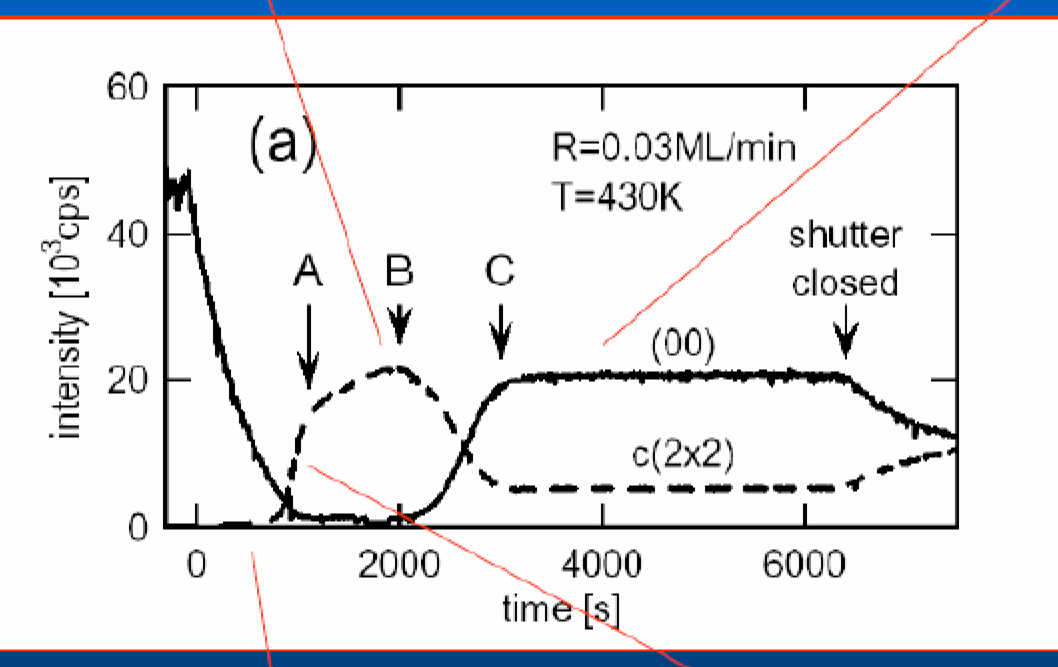
Simultaneous Inspection of Several Diffraction Spots

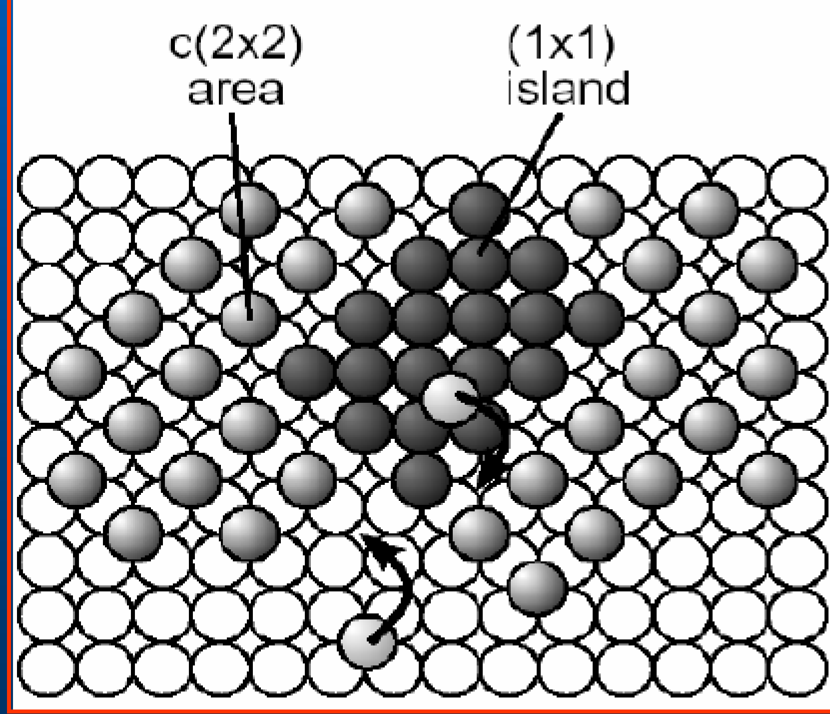
Mg/Ag(100)

perfect order - c(2x2)

adsorption – desorption equilibrium

c(2x2) has a higher bonding energy than (1x1) pseudomorphic layer





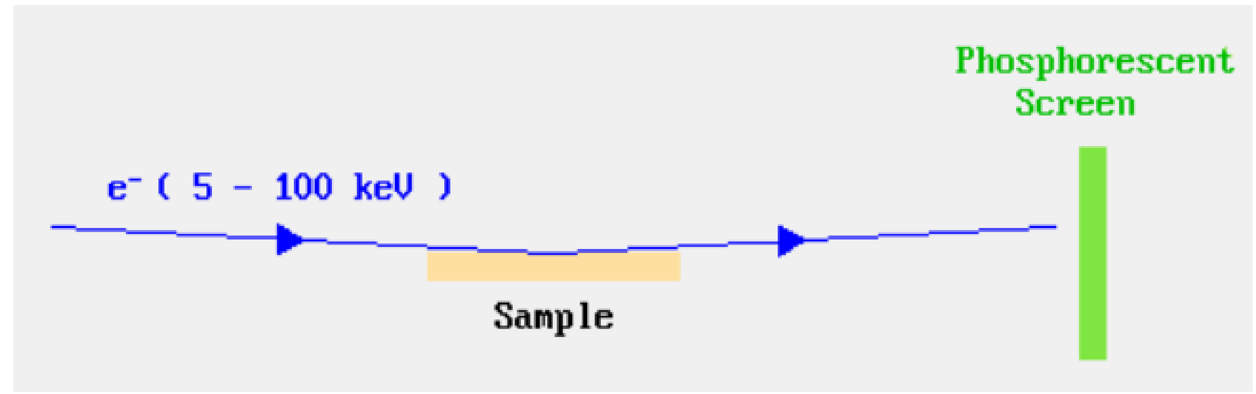
delay in c(2x2) formation due to surface alloying

demixing – c(2x2)

K. Tegenkamp, M. Michailov, H. Pfnuer: Appl. Surf. Sci., 151, 40 (1999)
NATO – ASI series, vol. 360, (1997)

RHEED

- Typické energie 5 keV až 100 keV,
- Charakteristiky
  - Dopředný rozptyl
  - Úhel rozptylu



Electron diffraction

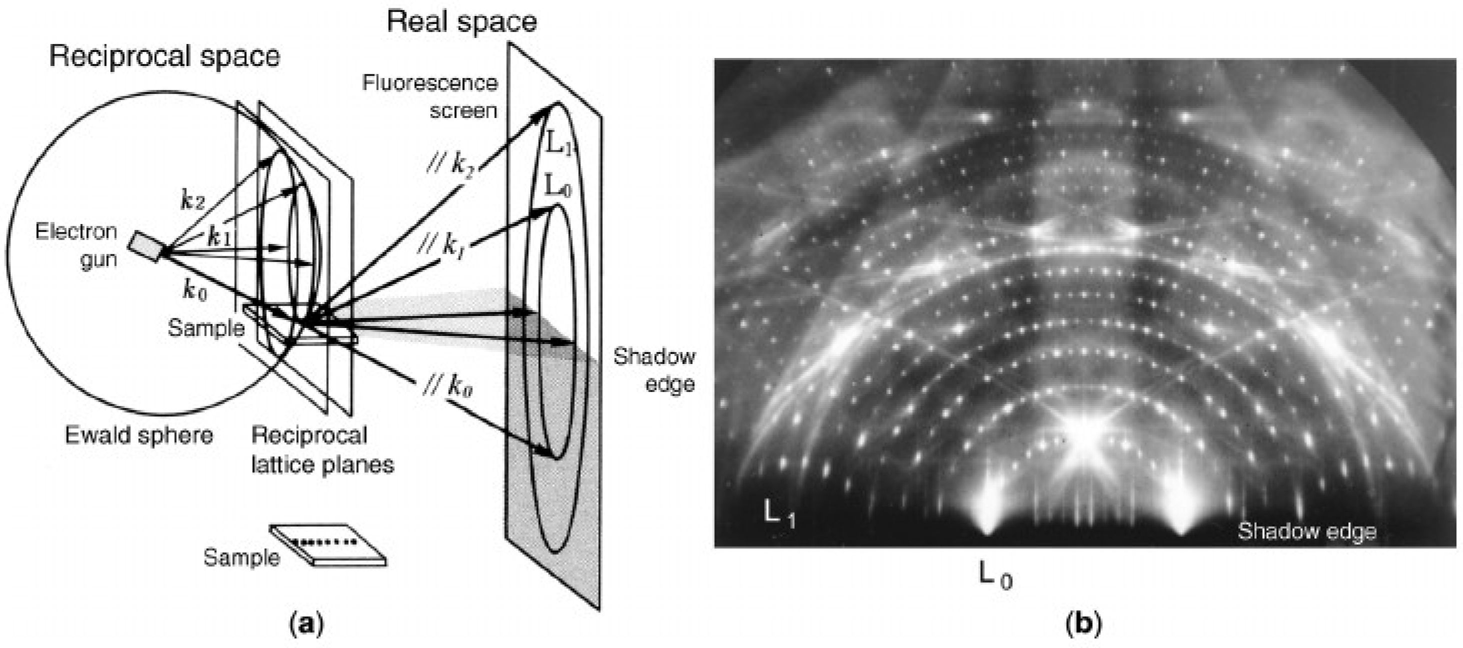


Figure 2. (**a**) Direct space and reciprocal space of RHEED. (**b**) RHEED pattern taken from a Si(1 1 1)–(7 × 7) reconstructed surface. The electron beam was 15 keV in energy with $[11\bar{2}]$ incidence in azimuth direction and about 3° in glancing angle θ_g to attain a surface-wave resonance condition.

Electron diffraction

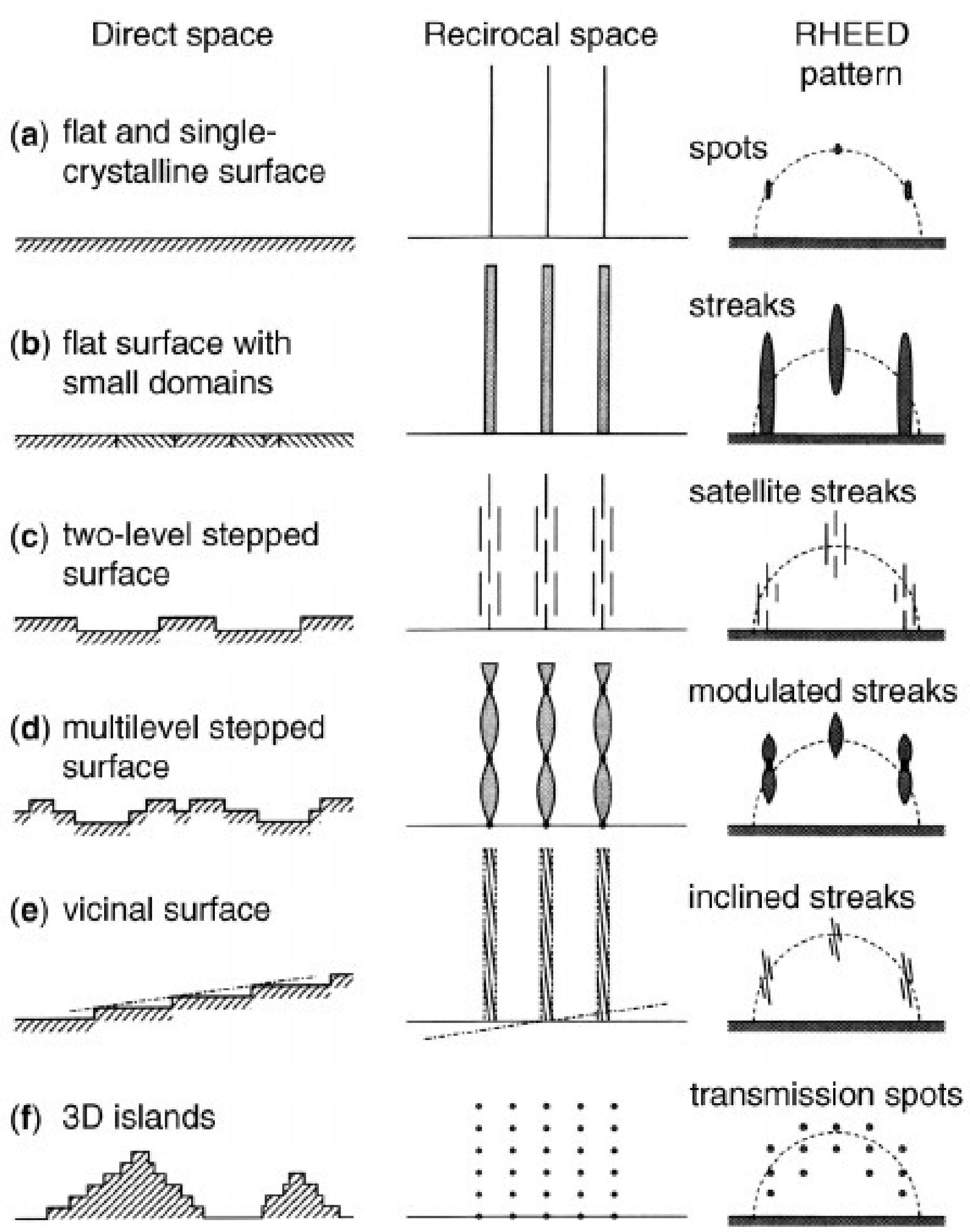


Figure 3. Schematics of various kinds of realistic surfaces, in real-space morphology, in reciprocal space, and their RHEED patterns (courtesy by Yoshimi Horio).

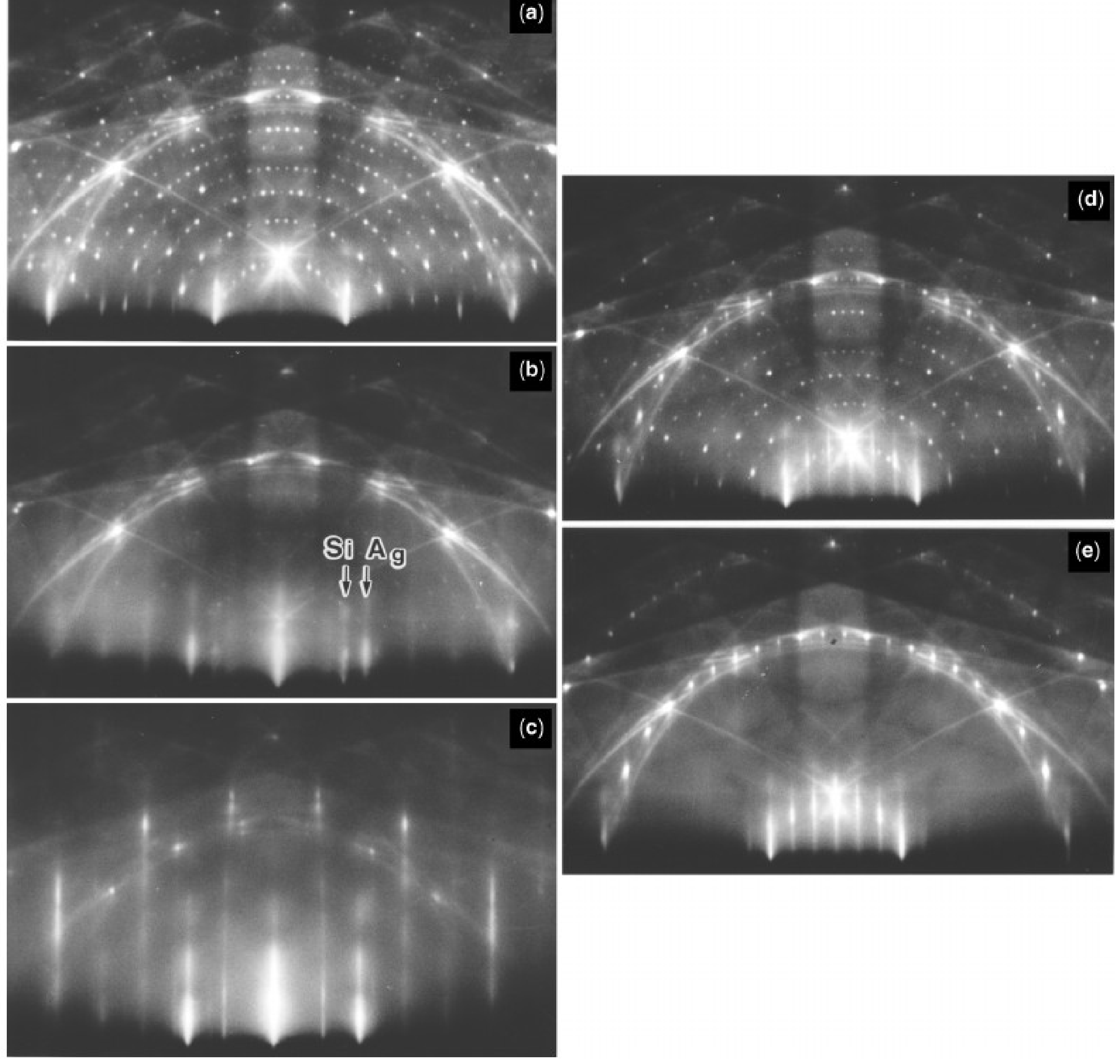


Figure 4. RHEED patterns observed during Ag deposition onto Si(1 1 1) –(7 × 7) reconstructed surface (**a**) at room temperature (**b**)(**c**) and at 440°C (**d**) (**e**). The amount of Ag deposited is (b) 1.5 ML, (c) 3.0 ML, (d) 0.5 ML, and (e) 1.0 ML. ML (monolayer) means a single-atomic layer. Reprinted from (Hasegawa et al., 1987), Copyright 1987, with permission from Elsevier.

Electron diffraction

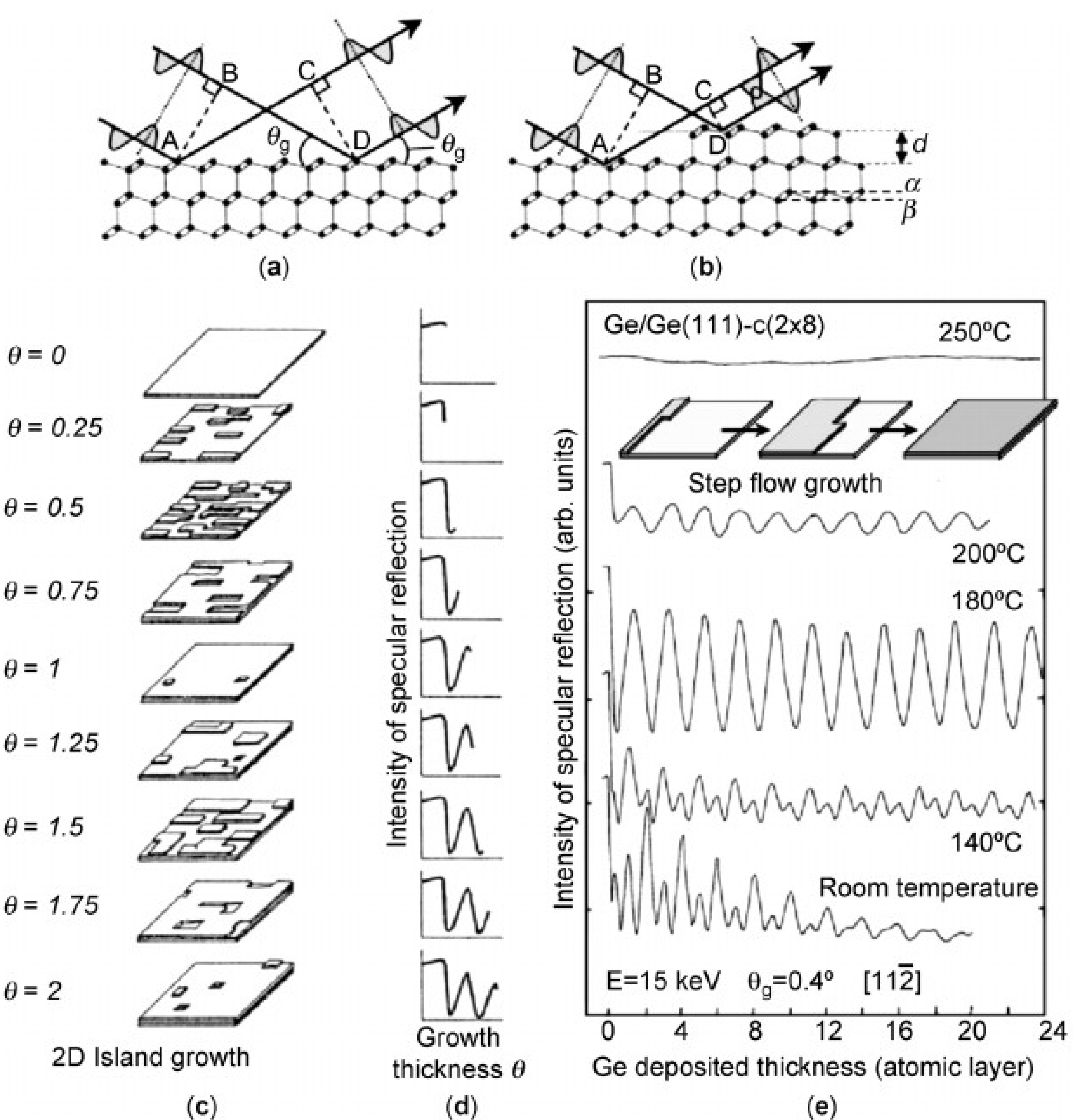


Figure 10. RHEED intensity oscillation and crystal growth monitor. (**a**) and (**b**) Specular beam from a flat surface and a stepped surface, respectively. (**c**) Illustrations showing a 2D-island growth style and (**d**) the intensity change of specular spot during the growth, reprinted from (Joyce et al., 1986), Copyright 1986, with permission from Elsevier. (**e**) Experimental data of RHEED intensity oscillations during Ge homoepitaxy on Ge(1 1 1) surface (Fukutani et al., 1992).

Table 1.1 Imaging and analysis techniques employing electron, ion, and photon beams, with estimates of the achievable spatial resolution

Incident beam	Detected signal	Examples	Resolution (nm)
Electron	Electron	Electron microscopy (TEM, STEM)	0.1
		Electron diffraction (SAED, CBED)	10–1000
		Electron energy-loss spectroscopy (EELS)	<1
		Auger electron spectroscopy (AES)	~2
	Photon	X-ray emission spectroscopy (XES)	2–10
		Cathodoluminescence (CL)	
Ion	Ion	Rutherford backscattering spectroscopy (RBS)	1000
		Secondary ion mass spectrometry (SIMS)	50
		Local electrode atom probe (LEAP)	0.1
	Photon	Proton-induced x-ray emission (PIXE)	500
Photon	Photon	X-ray diffraction (XRD)	30
		X-ray absorption spectroscopy (XAS)	20
		X-ray fluorescence spectroscopy (XRF)	
	Electron	X-ray photoelectron spectroscopy (XPS)	5–10
		Ultraviolet photoelectron spectroscopy (UPS)	1000
		Photoelectron microscopy (PEM or PEEM)	0.5
	Ion	Laser microprobe mass analysis (LAMMA)	1000