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Basics and Applications of TEM for beginners

Hydrogen Boride Research Center

and

Tsukuba Institute for Advanced Research (TIAR)

University of Tsukuba

TEM HF5000 Hitachi

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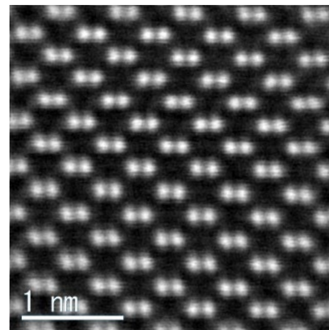
version 1, July 2026

Checked by Prof. Yoshikazu Ito



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For Better Understanding of This Lecture for Beginners

Scope of this lecture

- What are the differences between TEM and STEM?
  → Noticing the differences in the mechanisms of image formation.

- What are the signals for EDS, EELS, and EFTEM (EFI)?
  → Noticing the mechanisms of excitations.

- What are the differences between image, diffraction, and spectrum?
  → Noticing the information of them correspond to real space, reciprocal space and energy space, respectively.

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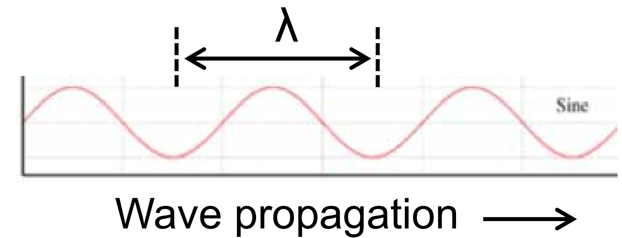
1. Overview of Transmission Electron Microscopes (TEM)

1-1) Resolution of TEMs

Why do we need TEMs?

TEMs allow us not only to observe but also to analyze objects at a much higher resolution than optical microscopes.



Resolution of lens

Electron microscopes, like optical microscopes, use waves and lenses to magnify objects. However, their resolutions are very limited.

Resolution: The minimum distance (δ) at which two adjacent objects can be distinguished. The smaller the δ, the higher the resolution. The resolution of a lens is given by:

$\boldsymbol{\delta \approx 0.61\lambda}$ (λ: wavelength)

purple, blue, green, yellow, orange, red

380 λ(nm): 780

Wavelength of optical light

Resolution of optical microscopes

For green light (λ = 550 nm), the best resolution of an optical microscope is approximately

$\delta \approx 300$ nm

Resolution of electron microscopes

Electrons accelerated at high voltage behave as waves with a very short wavelength:

λ = 0.00251 nm (for electrons accelerated at 200 kV)

State-of-the-art TEMs can achieve resolutions of ~0.05 nm. However, the electron source, specimen, and recording media must be placed under vacuum in the microscopes.

Wavelength of electron beam

Acceleration V (kV)	Wavelength (nm)
100	0.00370
200 (common)	0.00251
1000	0.000872

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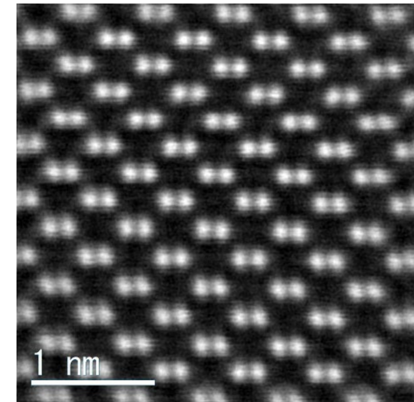
1. Overview of Transmission Electron Microscopes (TEM)

1-2) Basic Functions of TEM

The basic functions of TEM, observation and analysis, can be correlated each other in high space and energy resolution at a sub-nanometer size area.



Lattice image of a Si specimen
Captured by HB center's HF5000

Super magnifying glass

Magnifies specimens by more than one million times. With a resolution of ~0.1 nm, individual atoms or atomic columns can be distinguished.

Electron diffraction observation

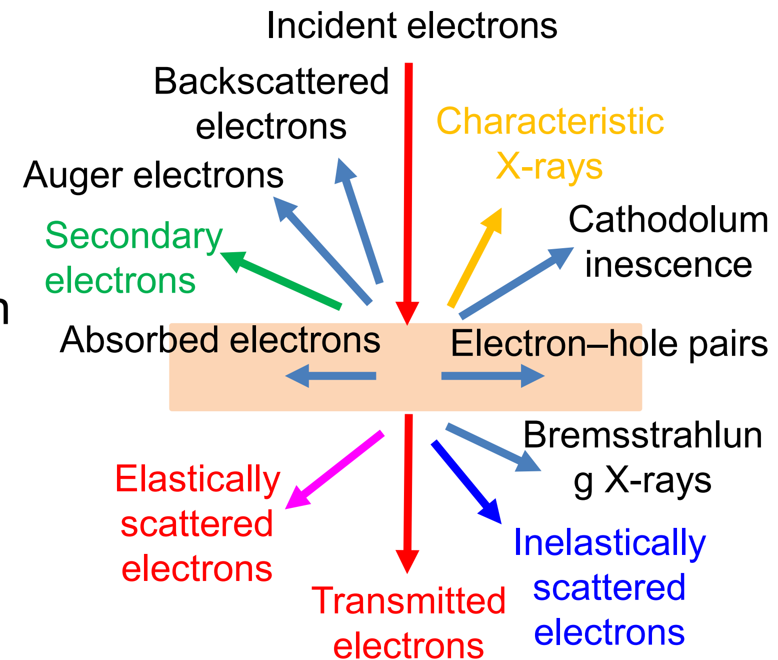
Used to observe crystal structures (in reciprocal space) of specimens.

Specimen analysis

By using secondary signals excited from the specimen, the composition and chemical states can be analyzed at the nano- or sub-nanometer scale.

Signals typically used for the analysis in TEM:

Transmitted electrons
elastically scattered electrons
inelastically scattered electrons
characteristic X-rays



Interactions between incident electrons and specimens

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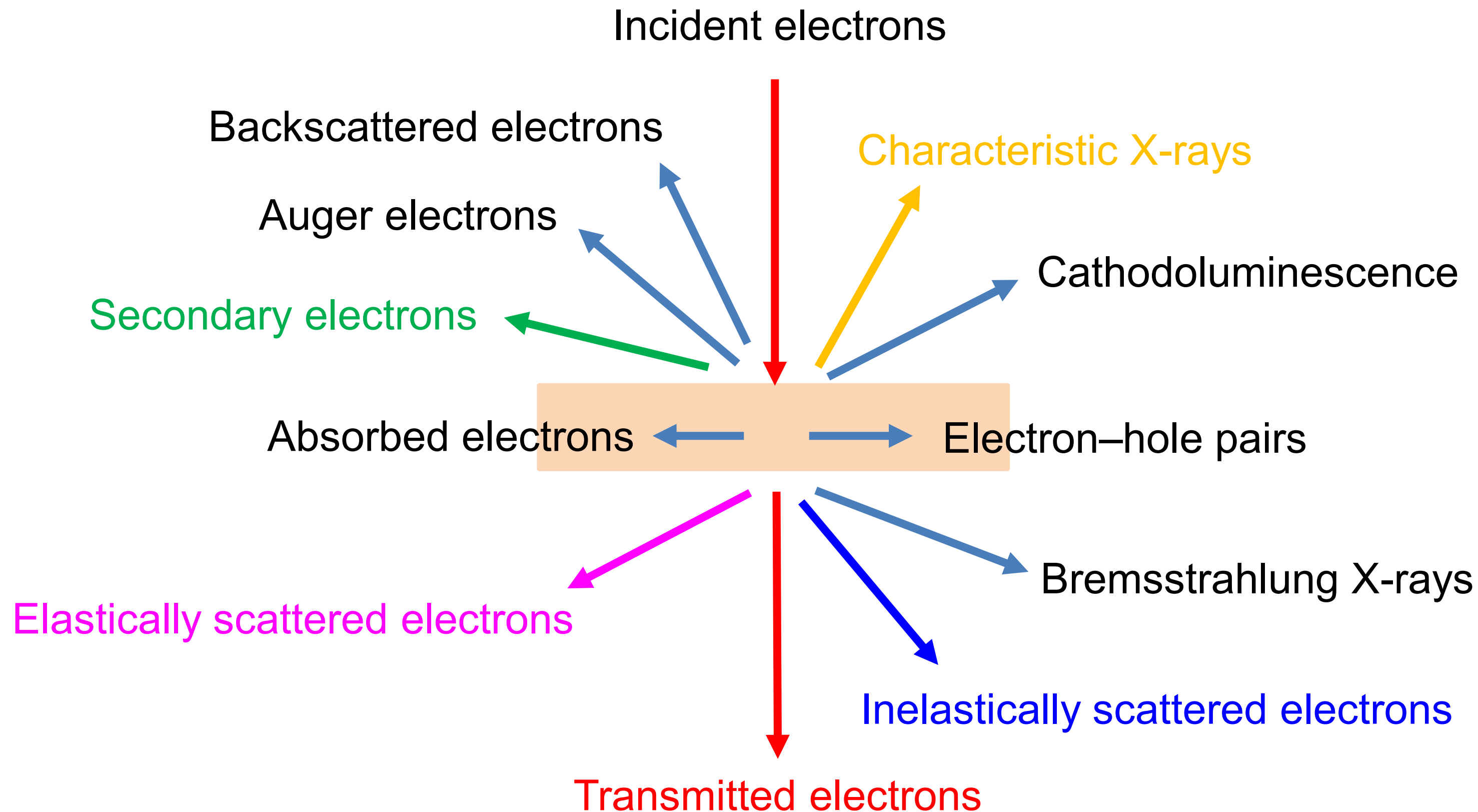
ZOOM UP: Interactions between incident electrons and specimens

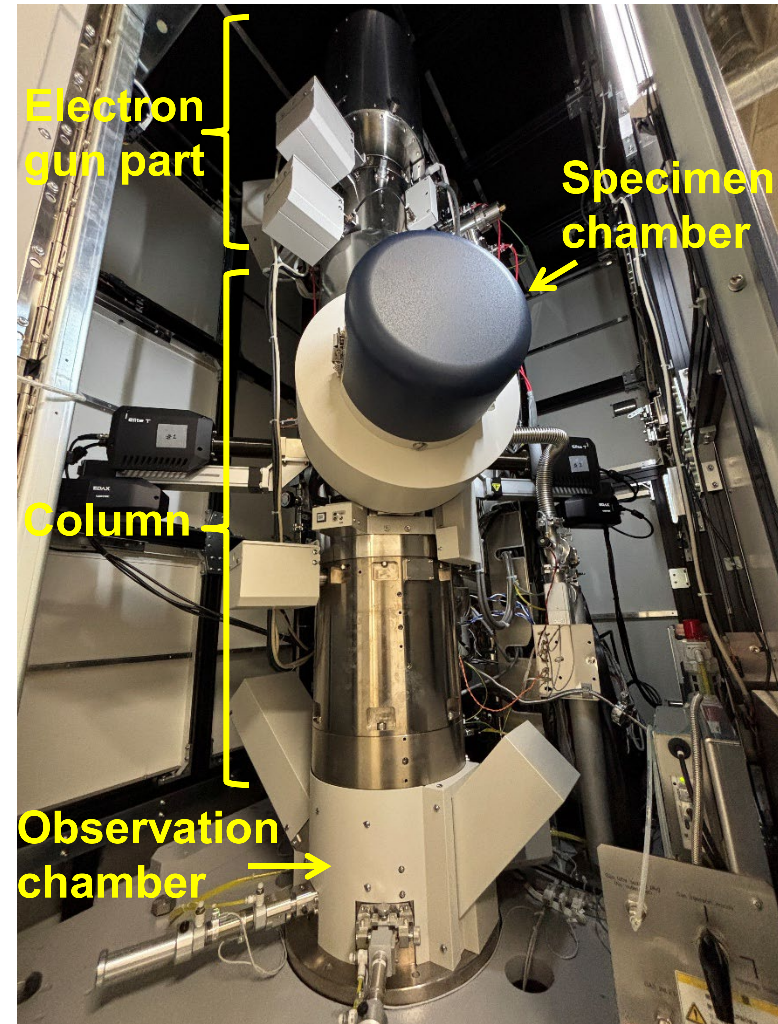


The signals in **red, orange, green, blue** are usually used in a commercial TEM for imaging and analysis.

2. Basic Structure of a TEM

2-1) Specification of a Typical TEM, HF5000

Model : HF5000
Electron Source : Cold field-emission gun
Accel. Voltage : 60, 80, or 200 kV
Spherical Aberration Correction:
Illumination system Cs corrector
Resolution : 0.102 nm (TEM)
0.078 nm (STEM)
Imaging Modes: Transmission (TEM),
Scanning transmission (STEM)
Scanning electron imaging
Image Recording : CCD camera
Analysis Functions: EDS, EELS, EFTEM
Specimen holders : Single-tilt holder
double-tilt holder
heating holder



TEM, HITACHI HF5000

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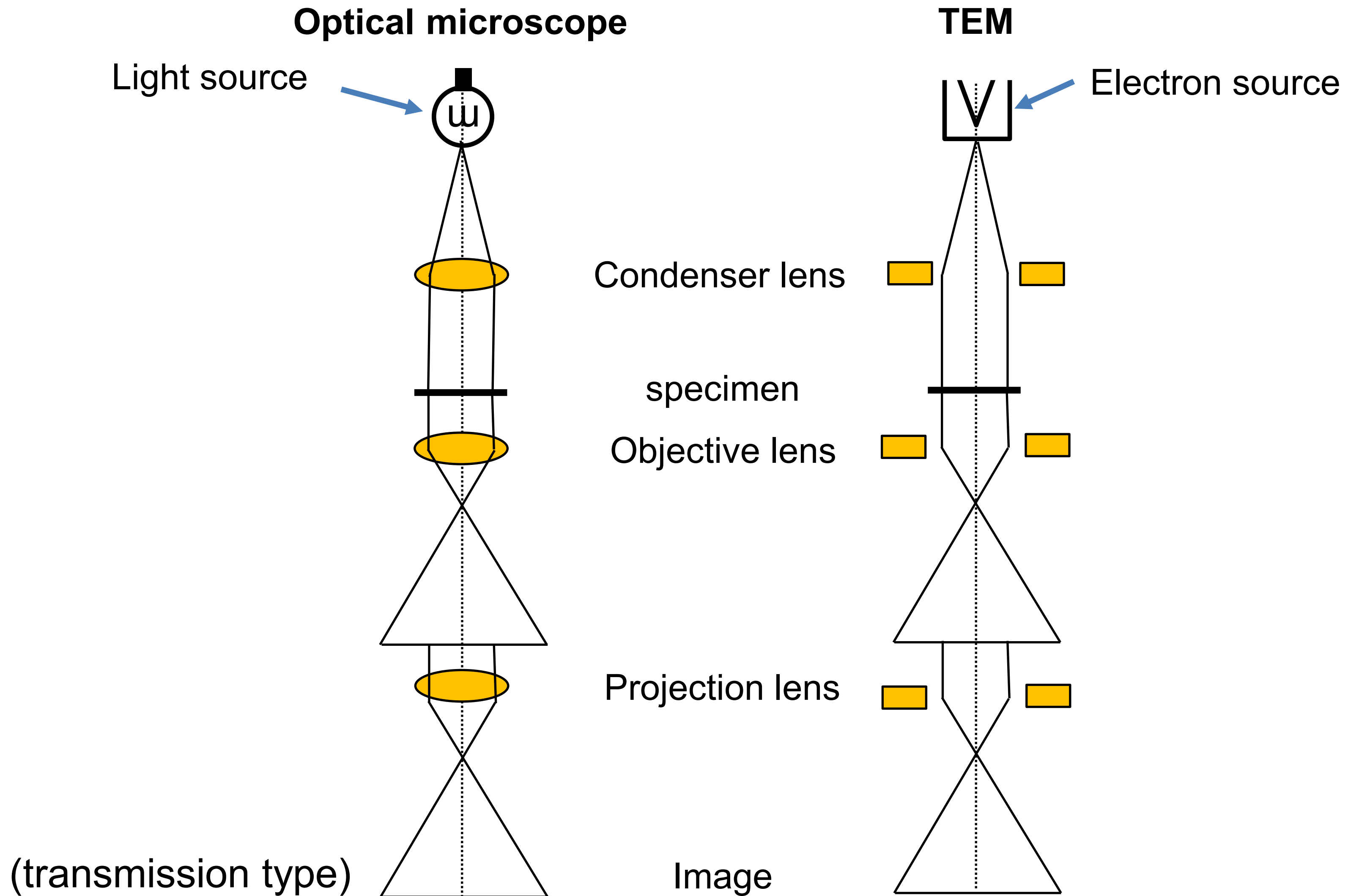
2. Basic Structure of a TEM

2-2) Comparison of TEMs with Optical Microscopes

The basic structure of them is similar.



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2. Basic Structure of a TEM

2-3) Main Components of a TEM

Electron gun & acceleration tube
Generate an electron beam at the acceleration voltage.

Condenser lens
Forms a parallel or convergent electron beam.

Objective lens
Forms a magnified image or a diffraction pattern (DP) of the specimen.

Intermediate & projection lenses
Further magnify the image or DP.

Condenser lens aperture
Controls the beam intensity and beam size.

Objective lens aperture
Selects the direct beam or diffracted beams to form bright-field (BF) or dark-field (DF) images, etc.

Selected-area aperture
Selects a specific area to form a DP.

Specimen holder
Holds and positions the specimen in the TEM (i.e. sample holder)

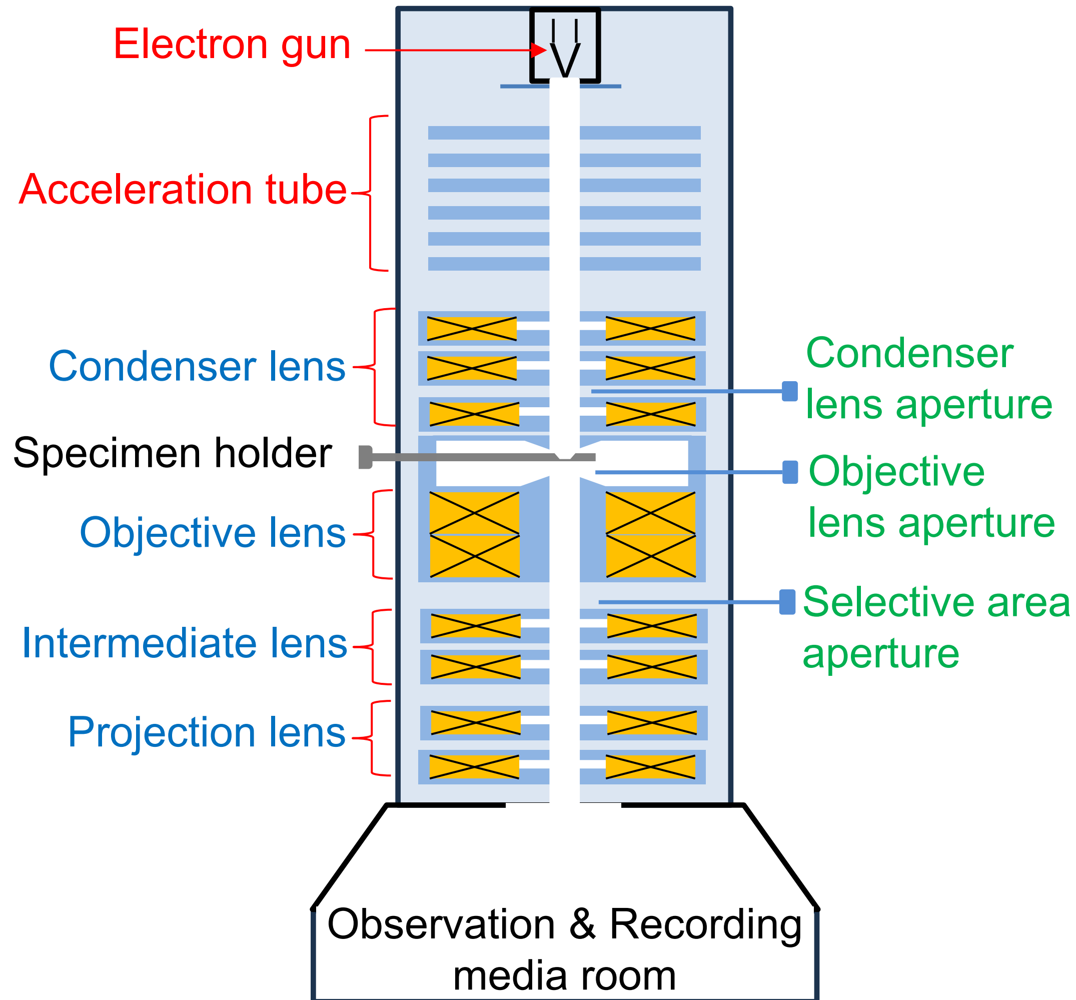

Illustration of the structure and main components of a TEM

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3. TEM Specimens and Specimen Holders

Basic requirements for a TEM specimen
Sufficiently thin (typically 1 nm ~100 nm, depending on material and observation purpose), electrically conductive, thermally conductive (i.e. withstand electron irradiation).

Observable specimens
In principle, any materials (sufficiently thin and relatively stable under electron irradiation) can be observed.
Any shapes (nanoparticles, nanowires, and nanosheets etc) can be observed.
Bulk materials must be sliced by mechanical polishing, chemical polishing, or ion milling.

TEM specimen holders and size
Typical specimen holders can accommodate specimens within a diameter of 3 mm and a thickness of about 0.2 mm
Standard holders (single-tilt, double-tilt) and special holders (heating, cooling, electrical biasing, environmental, etc.)

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3. TEM Specimens and Specimen Holders

TEM Specimen Preparation Methods

- **Methods**
  Dispersion, FIB, Ar ion milling, electro-polishing, etc.
- **Dispersion method**
  For nanoscale specimens (~100 nm or smaller, or sub-micrometer, depending on material), specimens are dispersed in a solvent and drop-casted onto a TEM grid.
  Grids with holey support films are suitable for nanofibers or nanosheets.
  Grids with non-holey support films are suitable for nanoparticles or powders of tens-of-nanometer, or smaller sizes.
  The dispersion process are same for any specimen's shape or compositions but must not release gas inside the TEM and have ferromagnetic and scattered characters.
- **FIB method**
  A thin lamella is fabricated from bulk material using accelerated Ga ions.
  Low-energy Ar ion milling is effective for removing Ga contamination and surface damage.
- **Ar ion milling and electro-polishing method**
  under preparation

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4. Observation Modes on TEM

4-1) Scattering of Electrons

Electrons interact with the Coulomb fields of atomic nuclei and the surrounding electrons, resulting in deflection of their trajectories or energy loss. This scattering produces image contrast and the signals for material analysis.

Scattering cross section:

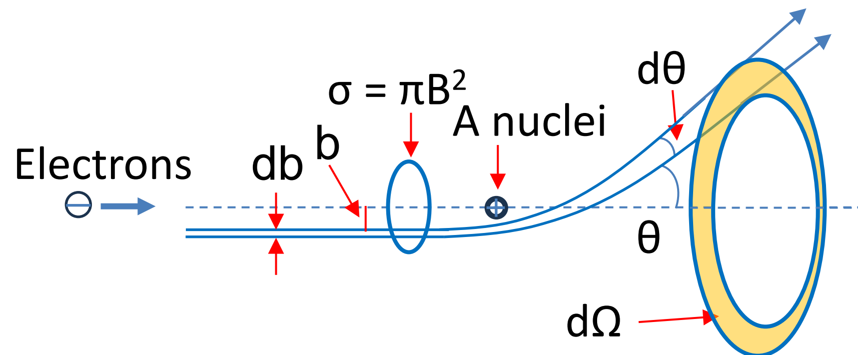
If the electrons incident to a nuclei within the area of $\sigma = \pi B^2$, they are scattered by the nuclei and outside the area, they are not scattered, then σ is called as scattering cross section of a nuclei to incident electrons.

The electrons coming into the band between radius b and b+db are scattered out between angle θ and θ+dθ. The measured electrons in dΩ equals to Idσ (I, the incident electron density) and is proportional to dΩ (with a factor). Namely,

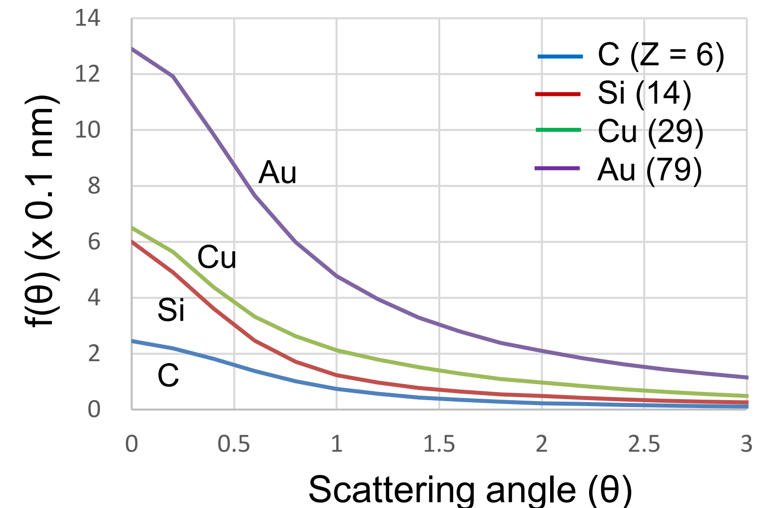
$$I d\sigma = I \left| f(\theta) \right|^2 d\Omega$$

f(θ) is called as atomic scattering factor of electron, which depends on the target atom species, energy of electron, and scattering angle (θ). Lower accelerating voltages and larger atomic numbers (Z) result in larger values of f(θ).

Illustration of scattering cross section of electrons scattered by a nuclei (based on Rutherford scattering model)



Relation of atomic scattering factor of electron to atom species and scattering angle.



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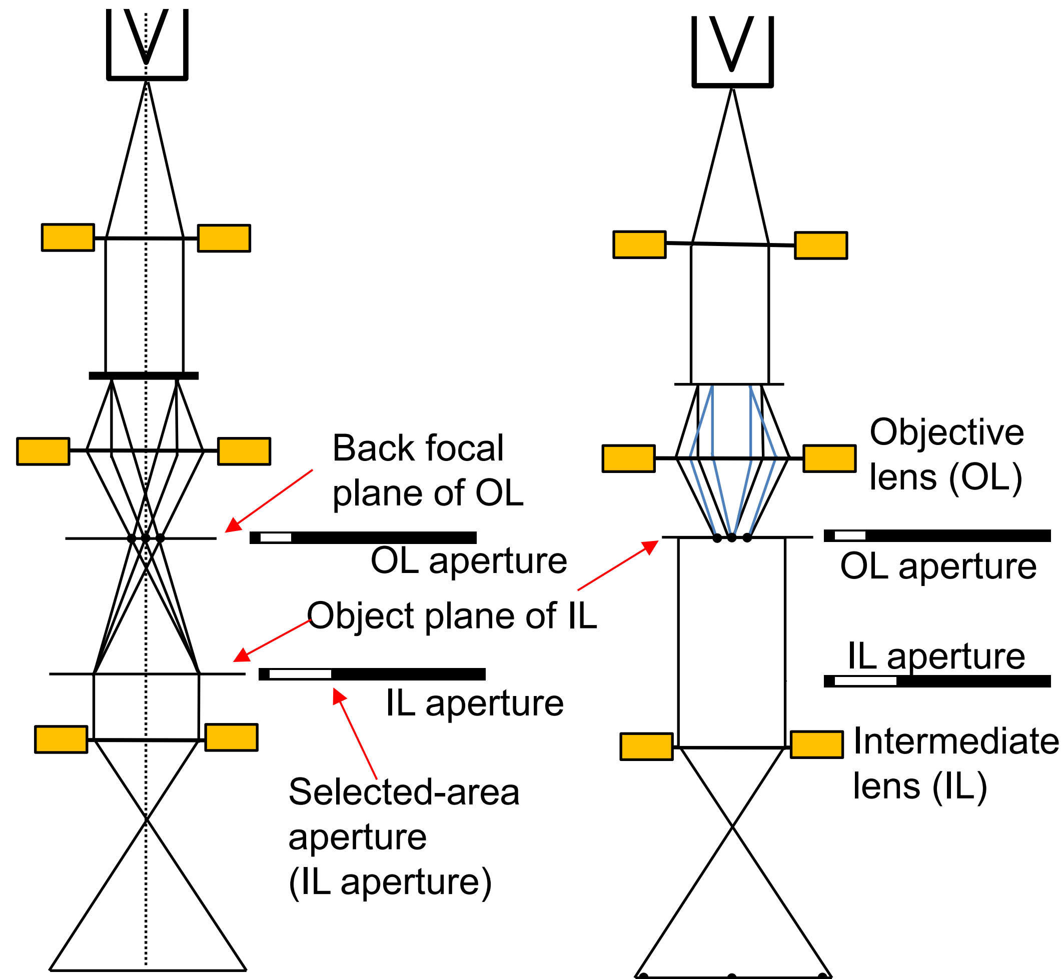
4-2) Image and Diffraction Pattern Observation

Observation modes
- TEM: imaging (BF, DF) and diffraction
- STEM: HAADF, LAADF, BF, etc.

Image and diffraction observations in TEM
- Switching between imaging and diffraction modes is achieved by changing the current of the intermediate lens.

Movable apertures
- Objective lens (OL) aperture controls image contrast and enables bright-field (BF) and dark-field (DF) imaging.
- Selected-area aperture Selects a specific region of the specimen for selected-area electron diffraction (SAED).

Image observation mode Diffraction observation mode



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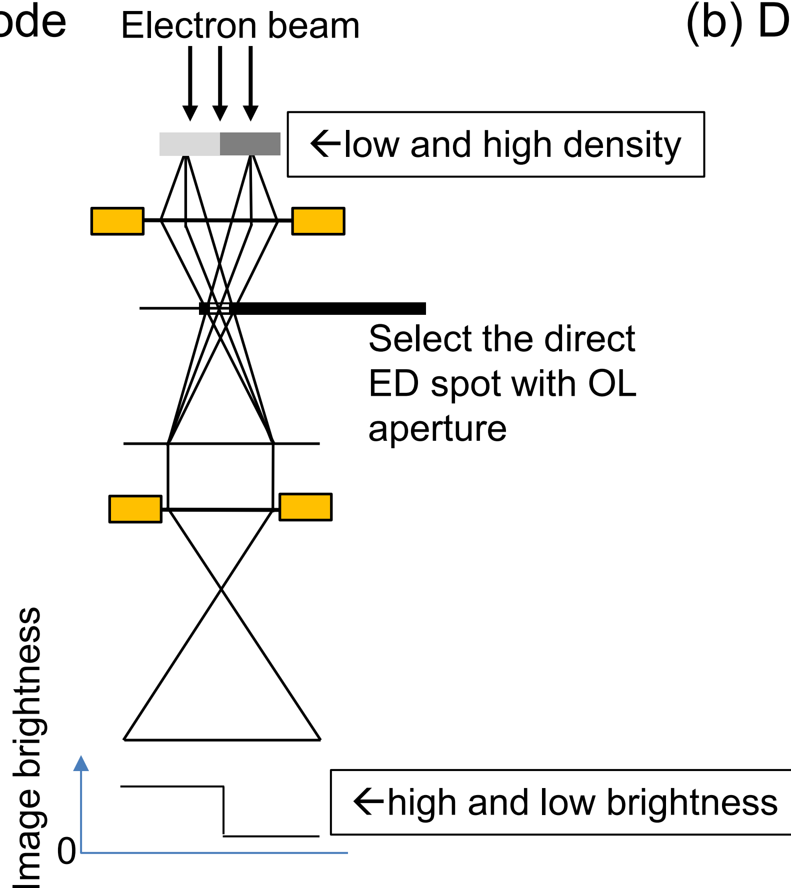
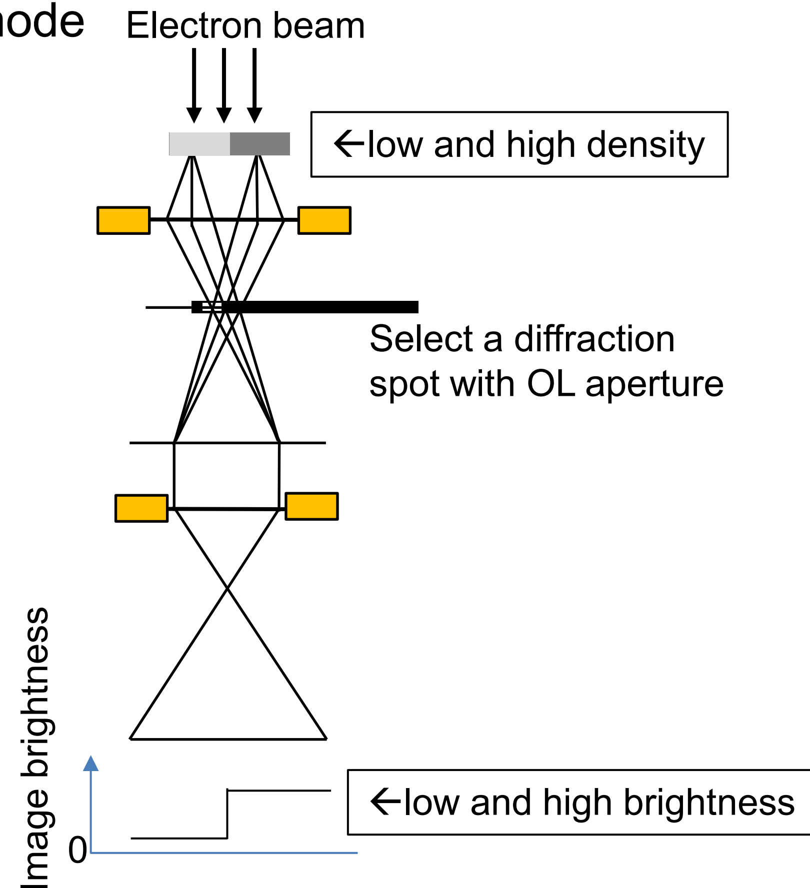
4. Observation Modes on TEM

4-3) Bright-Field (BF) and Dark-Field (DF) Images

BF images are formed using unscattered or weakly scattered transmitted electrons. Regions that are thinner or composed of lighter elements appear brighter.

DF images are formed using scattered electrons. Image contrast is generally opposite to that of the BF image.





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Examples of BF and DF Images

4-3) Bright-Field (BF) and Dark-Field (DF) Images

By using the diffraction spot (c) from Si crystal, Si crystal part on the DF image (b) shows brighter contrast than other parts (SiO_2, etc.) and reversal contrast compared to the Si part in the BF image (a).

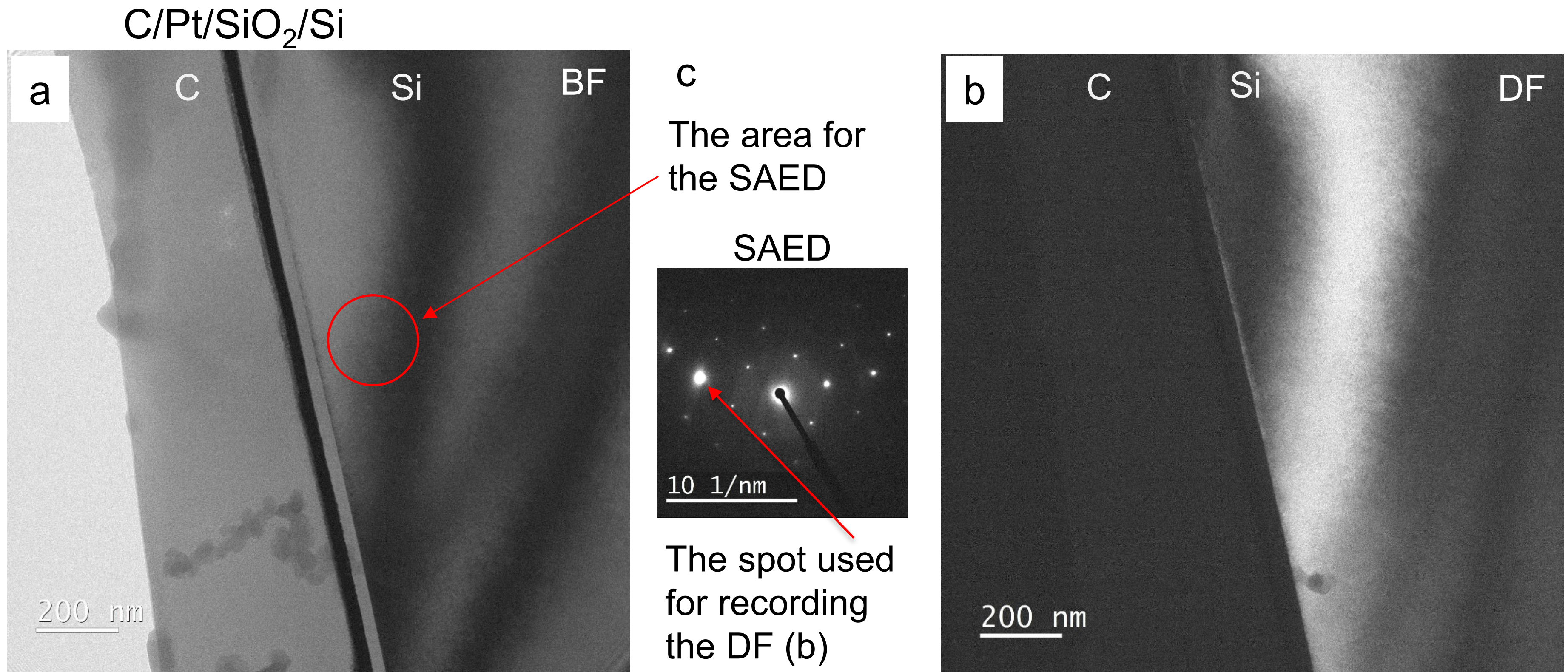

Figure. BF (a), DF (b) images, and SAED pattern (c) of a specimen with a $C/Pt/SiO_2/Si$ multilayer structure

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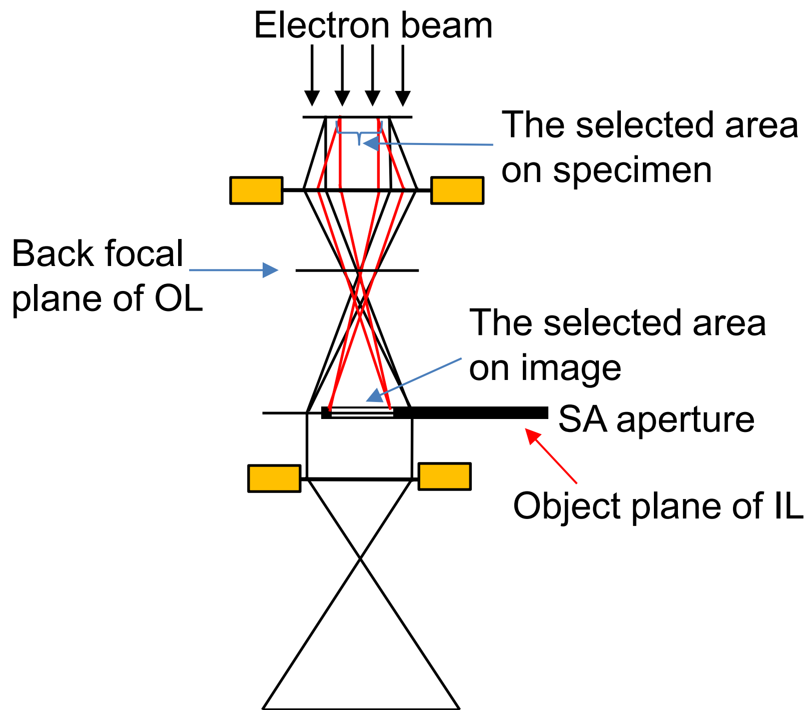
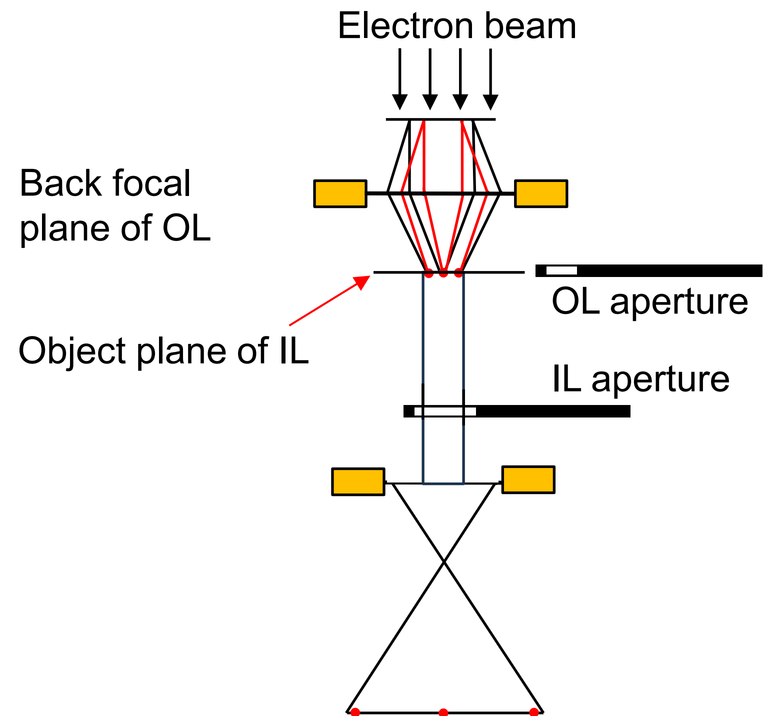
4-4) Selected-Area Electron Diffraction (SAED)

Using a selected-area (SA) aperture (a) located at the image plane of OL lens, the electron beam passing through the intermediate lens is restricted to a selected area of the specimen. As a result, the diffraction pattern (b) magnified by the intermediate lens originates only from this selected area, yielding a selected-area electron diffraction (SAED) pattern.

(a) Selecting an area by the selected-area aperture



(b) Diffraction observation by changing object plane of IL to back focal plane of OBL



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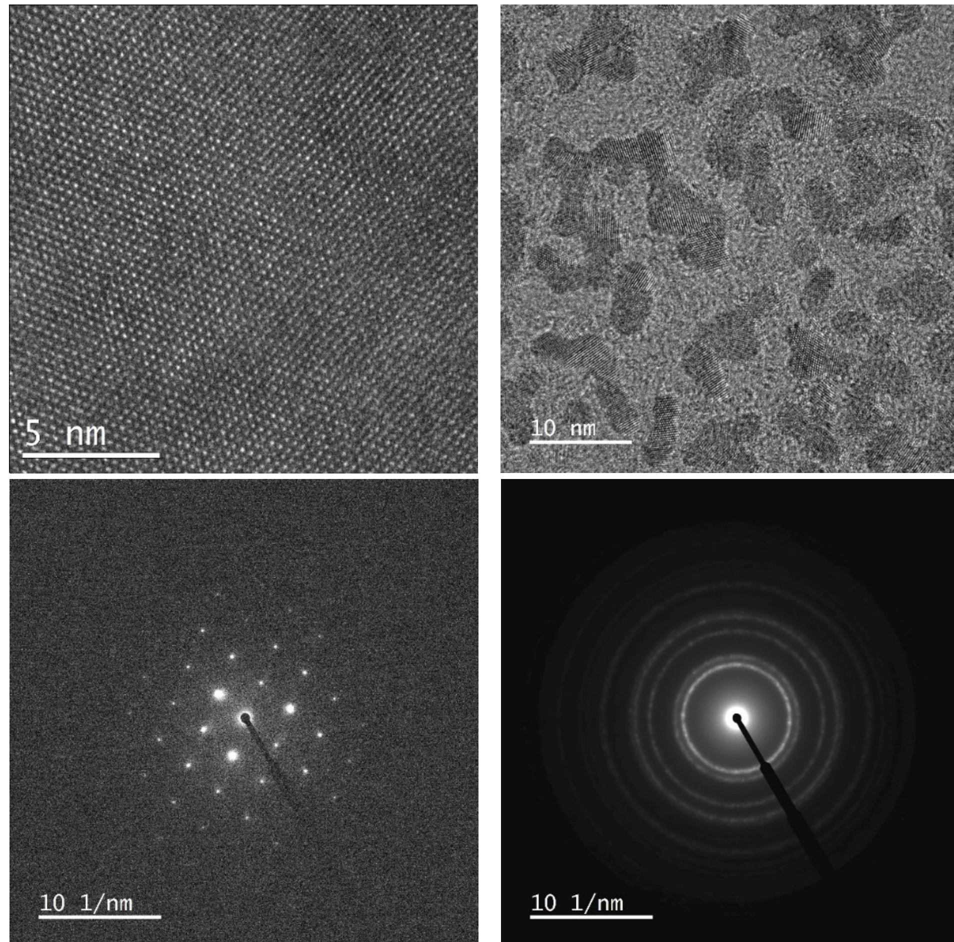
Examples of Diffraction Patterns

The observed area of a single crystal specimen is in the same orientation, therefore, the DP shows separate but regularly arranged spots.

For a polycrystalline specimen, due to random orientations of the grains, the diffraction spots form diffraction rings.

Single crystal of Si Polycrystalline Pt sample



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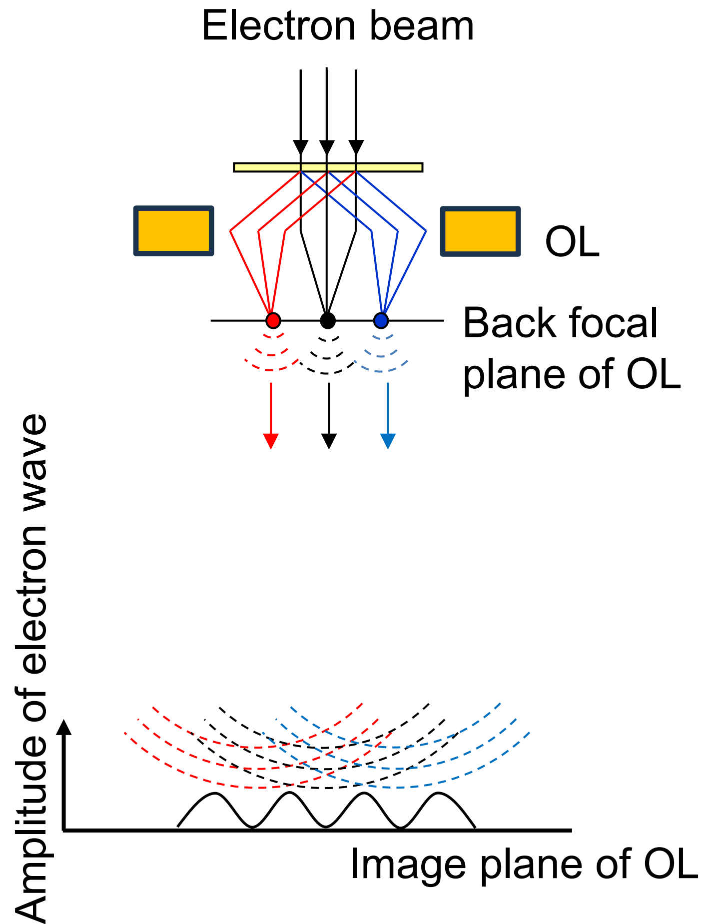
4-5) High-Resolution TEM Image (HRTEM)

An HRTEM image, which reflects the lattice or atomic arrangement of a crystalline specimen, is a **phase-contrast image** formed by the interference of multiple electron waves from different directions.

Bragg-diffracted electron beams form a diffraction pattern at the **back focal plane of the objective lens (OL)**. An OL aperture is inserted to allow several diffracted beams to pass.

The diffraction spots can be regarded as point electron sources. These waves propagate and interfere at the image plane of the OL, forming an HRTEM image. The image is further magnified by subsequent lenses and observed on a screen or CCD camera. The amplitude and phase of electron waves depend on specimen thickness and lens aberrations such as astigmatism and defocus. Therefore, bright (or dark) features in an HRTEM image do not always correspond directly to atomic column positions. In principle, correct interpretation of HRTEM images requires image simulation.

Formation of an HRTEM image



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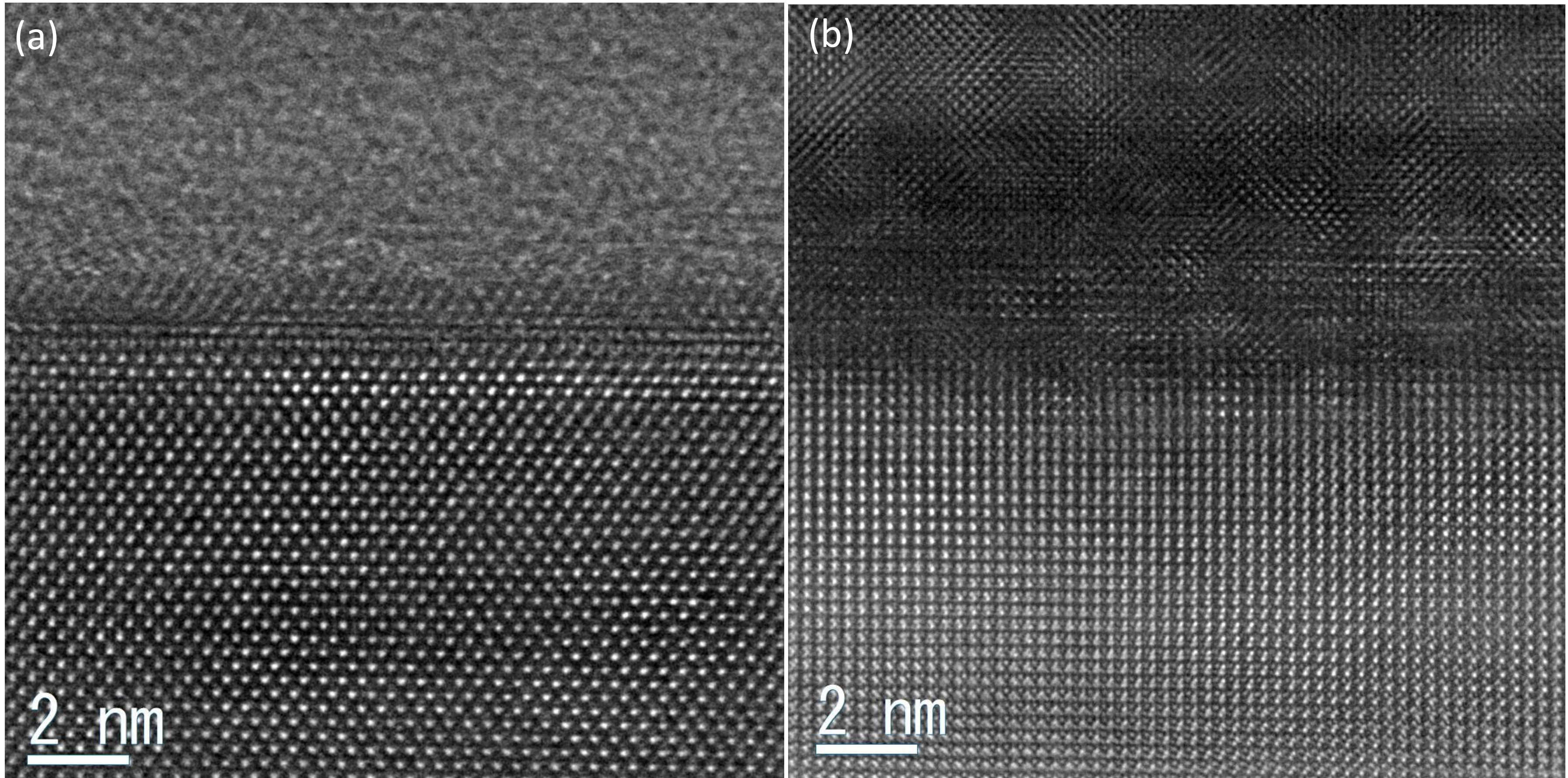
Examples of HRTEM Images

Atom rows or lattice of a crystalline specimen can be observed directly. Local structures, such as surface, interfaces, and second phases can be identified.



HRTEM images of crystalline specimens. (a): Cross section image of a Si crystal substrate near the surface. (b): Cross section of the interface of $SrTiO_3/Mn_4N$.

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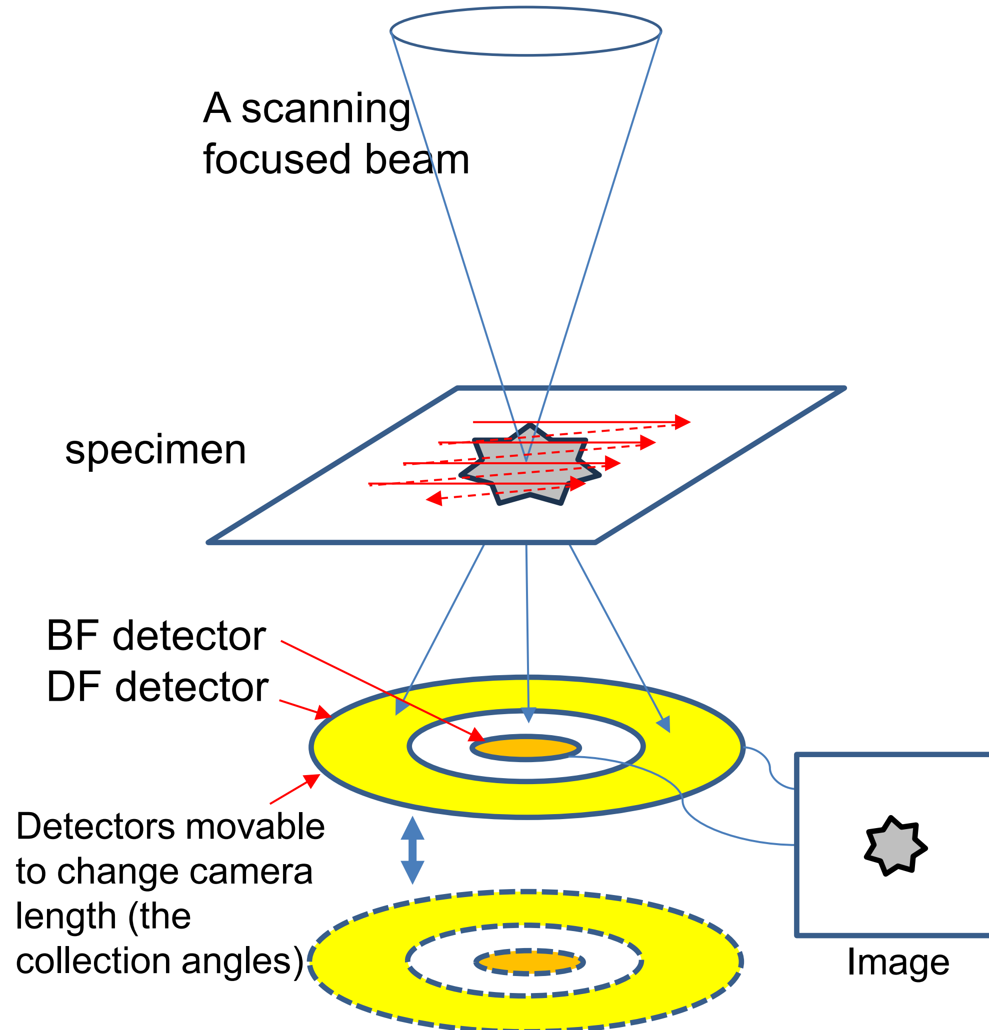
4. Observation Modes on TEM

4-6) Scanning Transmission Electron Microscopy (STEM)

A finely focused electron beam scans across the specimen, and the transmitted or scattered electrons are collected to form images.



STEM Bright-Field (BF)
Image formed using electrons that are unscattered or only weakly scattered.

STEM Dark-Field (DF)
Image formed using scattered electrons.

STEM LAADF (Low-Angle Annular Dark-Field)
Contrast largely depends on diffraction effects.

STEM HAADF (High-Angle Annular Dark-Field)
Contrast mainly depends on the atomic number (approximately proportional to Z^2) and/or the specimen thickness.

Because lens defocus does not cause contrast reversal, STEM images are generally easier to interpret the position of atoms or atom rows than conventional TEM images.

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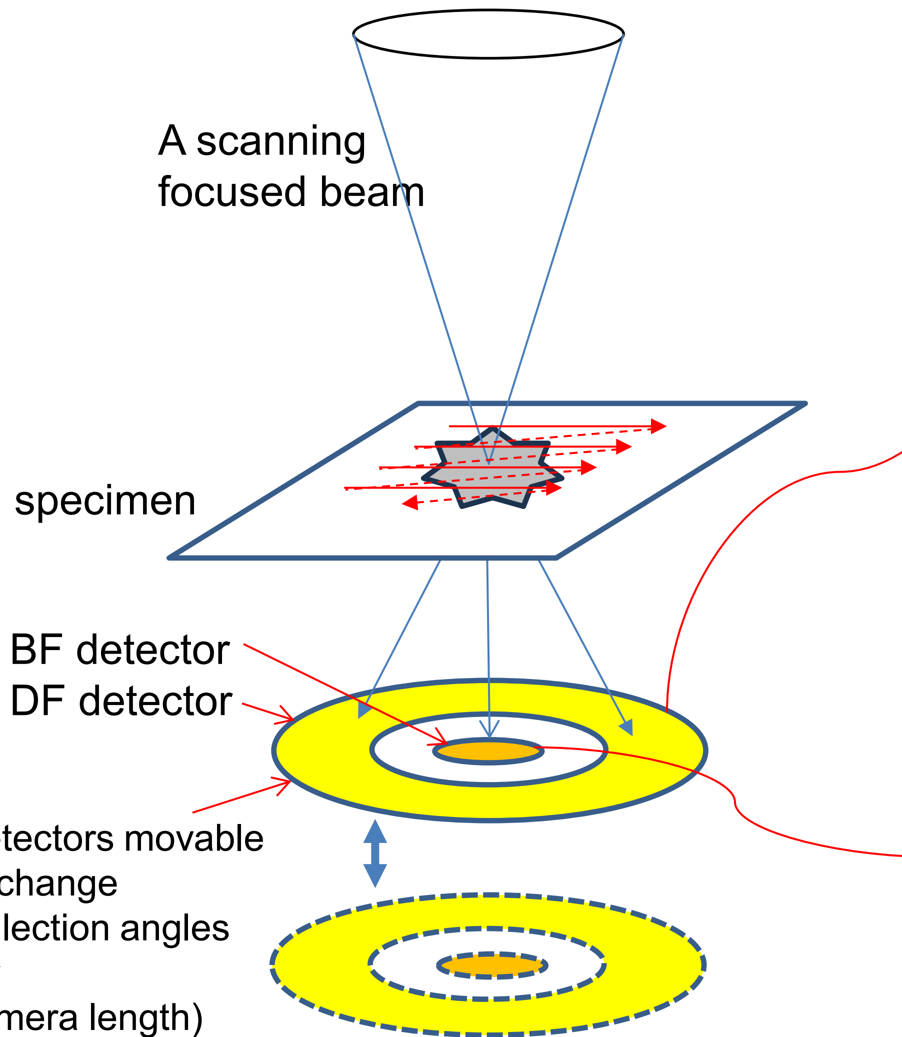
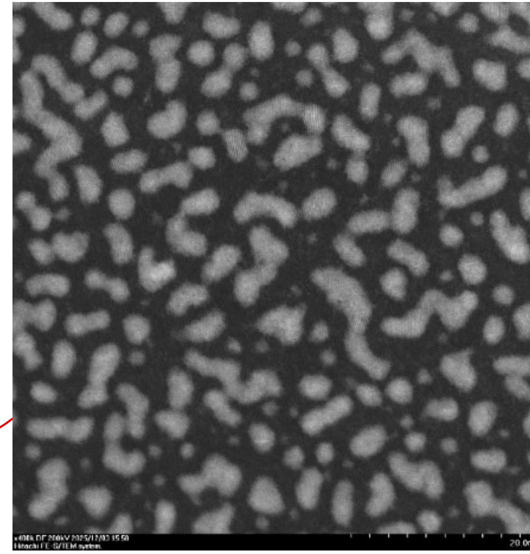
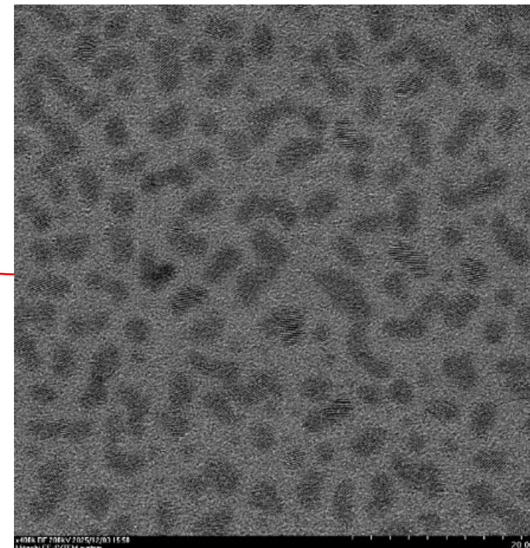
Examples of STEM-DF and -BF Images



Pt nano-particles



STEM DF:
Image with scattered electrons



STEM BF:
Image with electrons almost not being scattered. The contrast is basically reverse to that of the DF.

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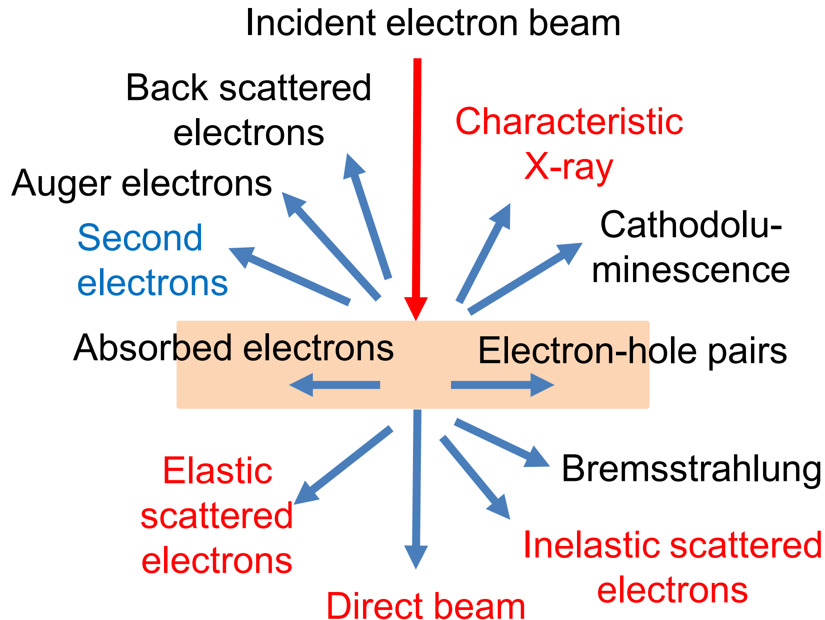
5. Analytical Capabilities of TEM

5-1) Signals for Material Analysis

Interaction of incident electrons with a specimen



Among the various signals, **characteristic X-rays** and **inelastically scattered electrons** are most commonly used for specimen analysis.

EDS
Energy-Dispersive X-ray Spectroscopy
Characteristic X-rays emitted from the specimen are measured for elemental analysis.

EELS
Electron Energy-Loss Spectroscopy
The energy losses of inelastically scattered electrons are measured to analyze the composition and chemical states of elements of a specimen.

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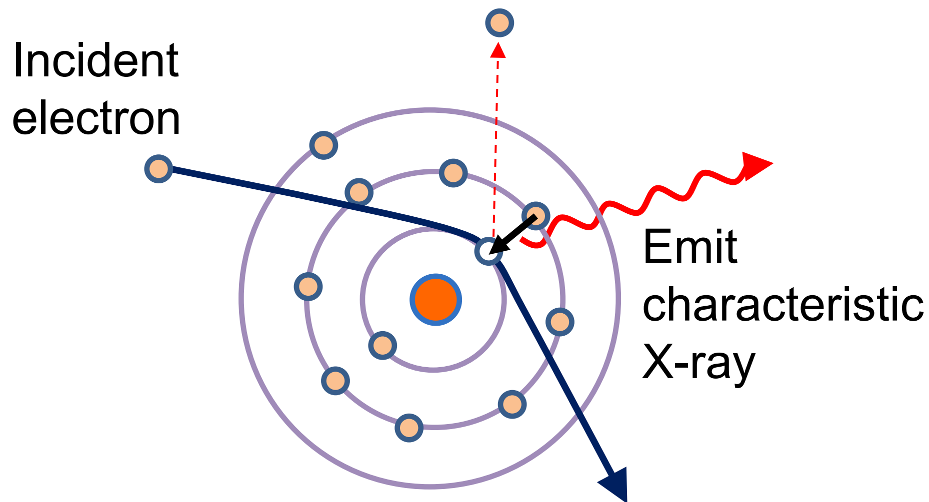
The Signals for EDS and EELS

A case of inelastic electron scattering



EDS

When an electron in a higher energy level fills a hole in a lower energy level, a characteristic X-ray is released.
These X-rays are analyzed using EDS.

EELS

An incident electron loses part of its energy through inelastic scattering.
The energy-loss electrons are analyzed using EELS.

Character comparison

EDS: The energies of characteristic X-rays are specific to each element
→ **elemental analysis**. (suitable for heavy elements)

EELS: The energy losses are characteristic of both the element and its chemical state
→ **elemental and chemical-state analysis**. (suitable for light elements)
Target elements: from Li to Fe or Ni

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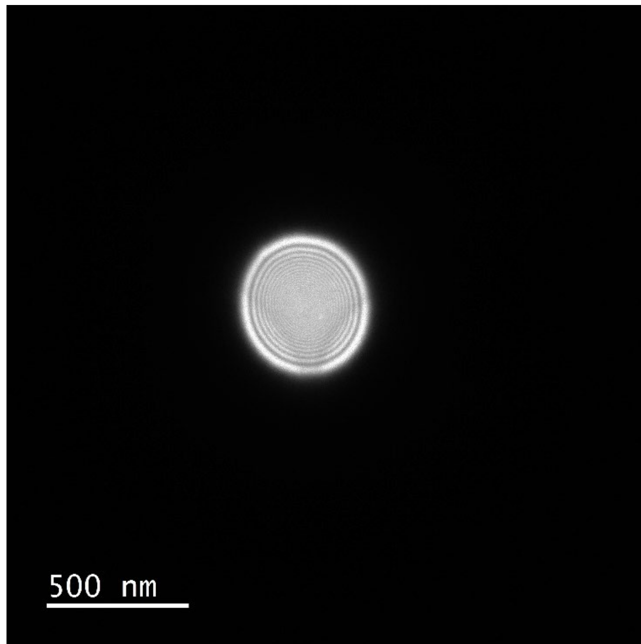
5-2) EDS

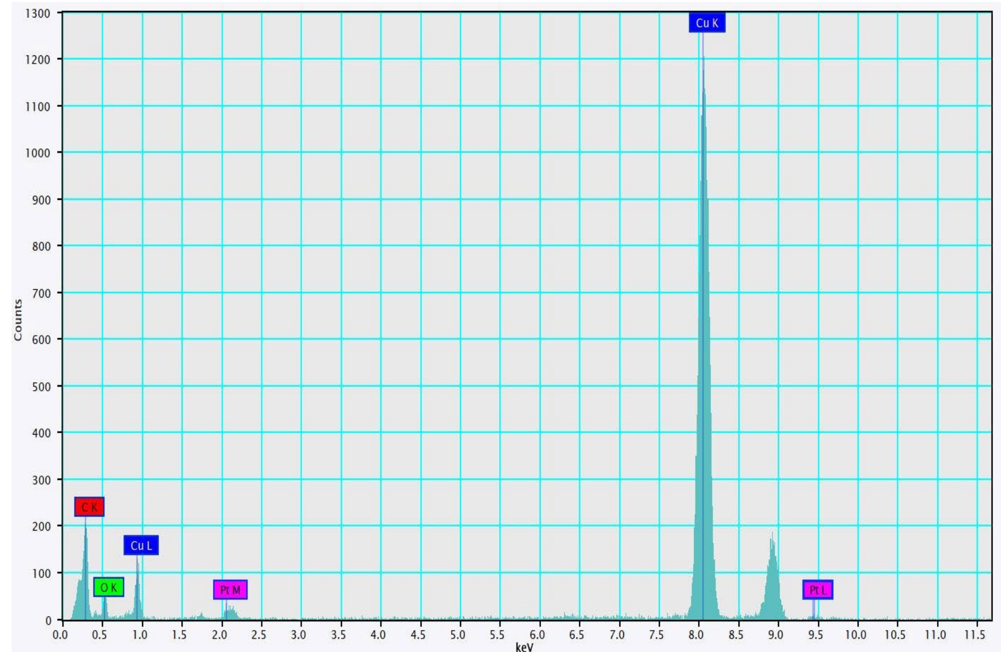
- **Measurement Modes for EDS**

TEM image mode

The electron beam was focused onto a point or an area, and an EDS spectrum was acquired. It is a quick way to measure the composition of a specimen.

An example EDS spectrum measured on TEM mode.
(a) The area to be measured was selected by the electron beam. (b) The measured spectrum. (Specimen: Pt nanoparticles on microgrid supported by a Cu mesh)





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- **Measurement Modes for EDS**

STEM mode

As the electron beam is scanned over an area, an EDS spectrum is acquired at each pixel. A typical data format is a **spectrum image (SI),** a three-dimensional (3-D) dataset (X, Y, ΔE).

3-D information of an SI

STEM mode

The electron beam scanning on an area

EDS detector

X-ray

Specimen

A scanning electron beam

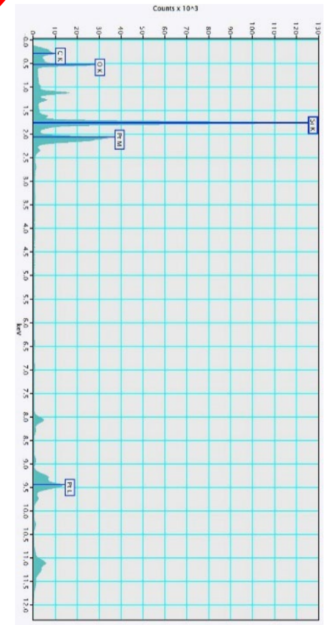
X

Y

E of X-ray

Energy of X-ray photon

A spectrum is collected from each pixel



For a specimen transparent to electron beam, X-ray signals are from the electron path in the specimen.

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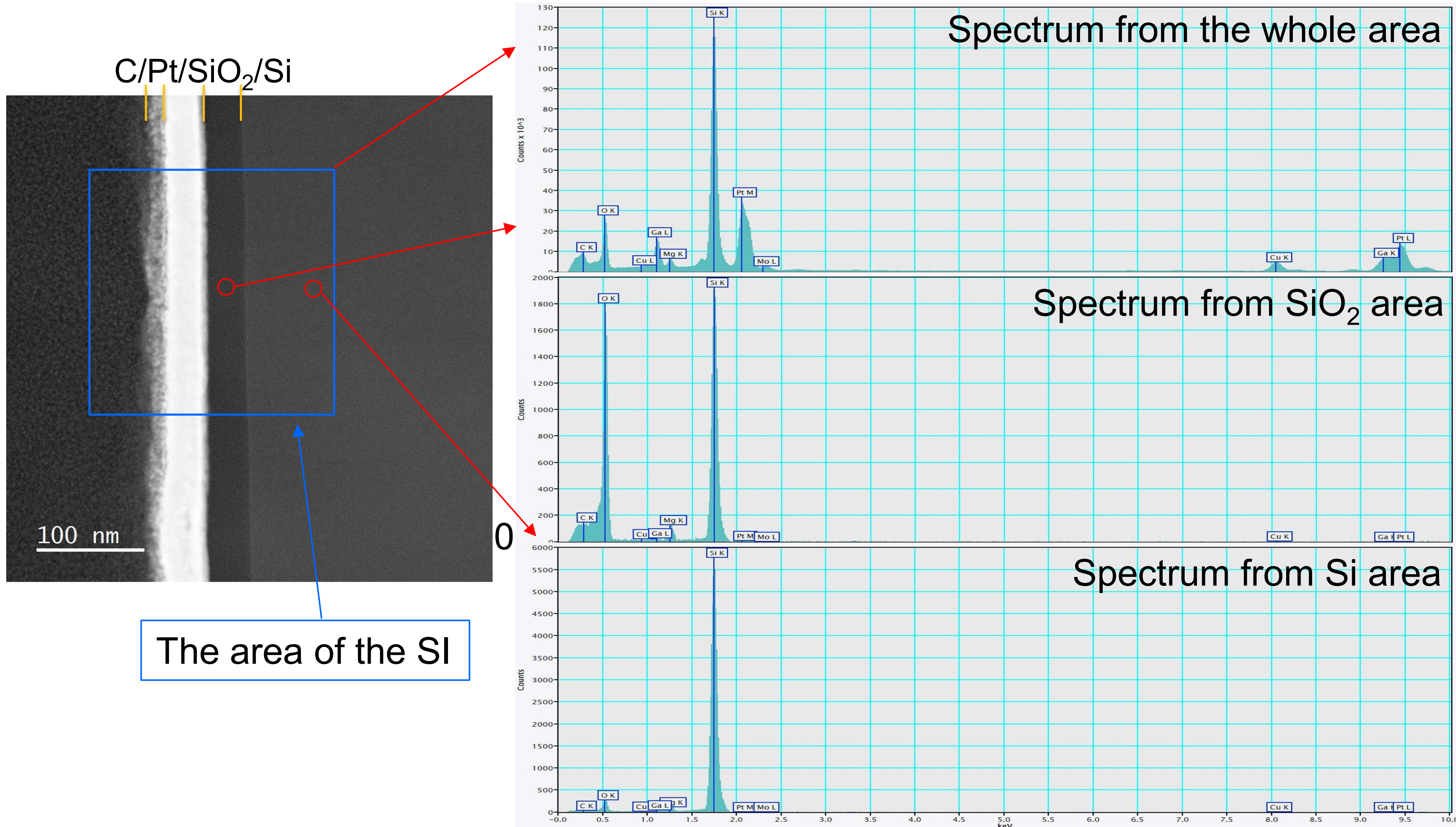
Example of an EDS-SI

Spectra and composition analysis:

Spectra extracted from the whole area and sub-areas of the SI. The compositions at different areas can be identified.



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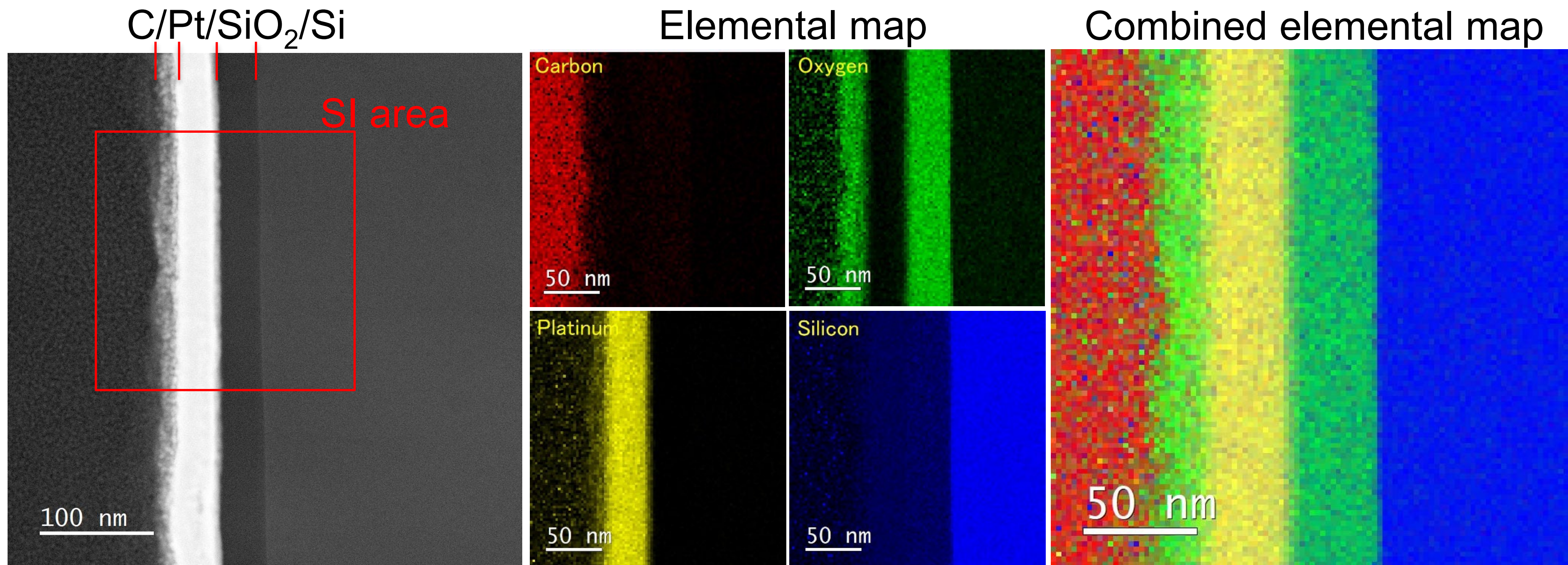
Example of an EDS-SI

Elemental map

STEM-HAADF and elemental distribution maps of a specimen with C/Pt/SiO_2/Si multilayer structure. The elemental distributions of the specimen are visualized.



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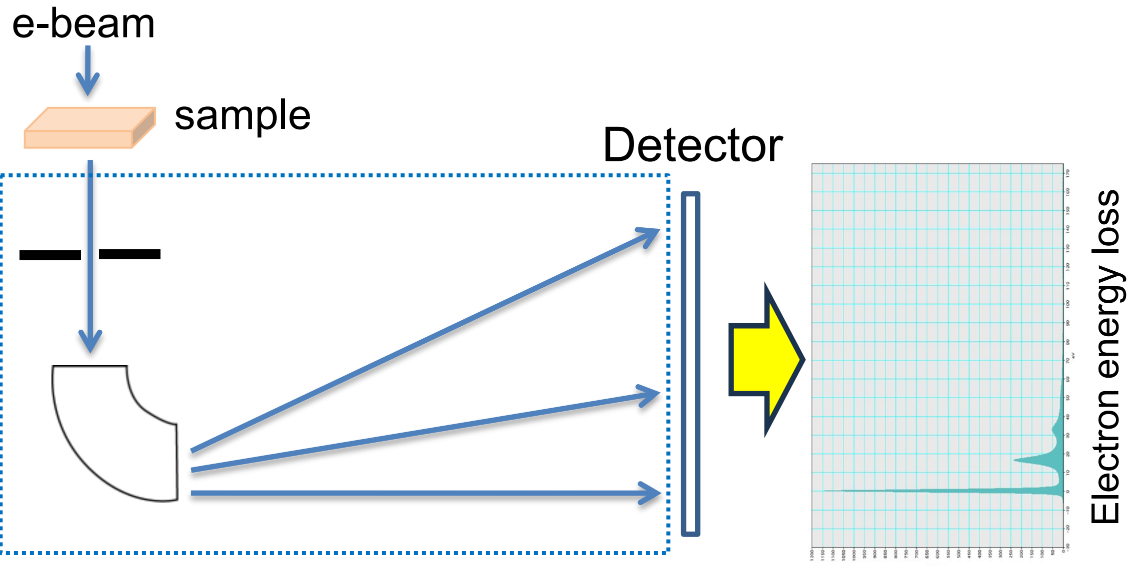
5-3) EELS and Energy Filtered TEM (EFTEM, or EFI (Energy Filtered Imaging)

- EELS spectrometer



Spectrometer
Incident electrons are bended about 90 degrees and dispersed with energy.

A Gatan spectrometer mounted to a TEM

An EELS spectrum is obtained with the detector.

EELS shows the energy and their intensity of energy loss electrons.
Energy filtered image: to form an image using the electrons lost definite energies.

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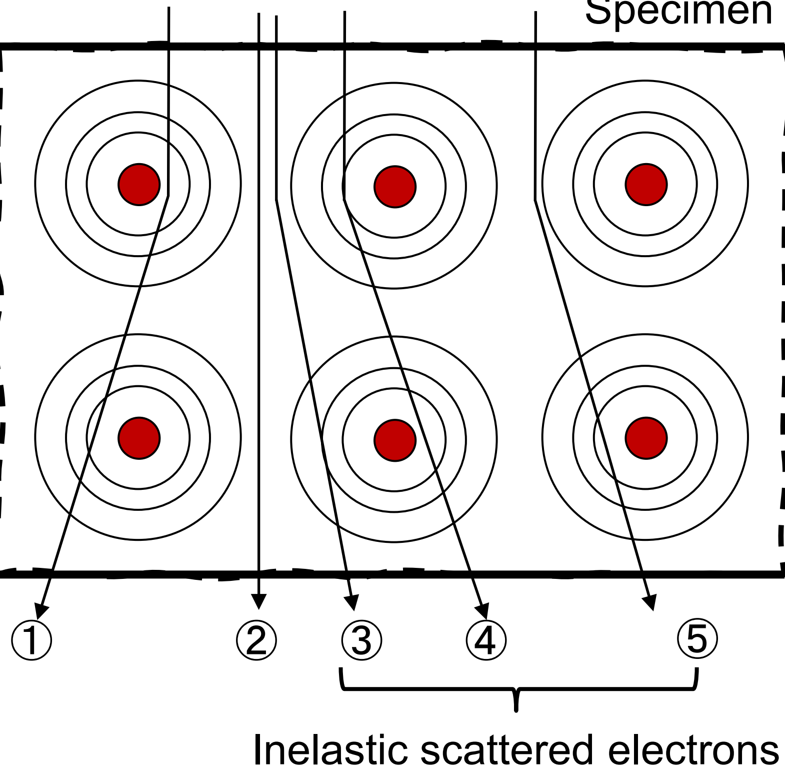
Energy Losses of Incident Electrons and Their Mechanisms



①Elastic scattered electron
②Transmit electron
③Plasmon loss electron
④Core loss electron
⑤Bremsstrahlung loss electron

Inelastic scattering events (referred to as excitations in EELS) and their typical energy losses:

- **Phonon excitation (lattice vibrations)**: ~0.1 eV or less
- **Free-electron excitation (secondary electron emission)**: ~50 eV or less
- **Bremsstrahlung emission**: up to the incident electron energy
- **Valence-electron excitation (interband transitions)***: ~10 eV or less
- **Plasmon excitation (plasmon loss)***: ~30 eV
- **Core-electron excitation (core loss)***: ~13 eV & above

* Signals typically analyzed by EELS

Single and multiple scattering

- **Single scattering**:

Only one excitation event occurs for an incident electron. The energy loss equals the energy for that excitation.

- **Multiple scattering****:

Two or more excitation events occur for a single electron. The total energy loss is the sum of individual losses. Forms a cumulative background that degrades the signal-to-noise ratio and complicates EELS interpretation.

As specimen thickness increases, the probability of multiple scattering increases. Therefore, specimens thinner than approximately **one electron mean free path are desirable.

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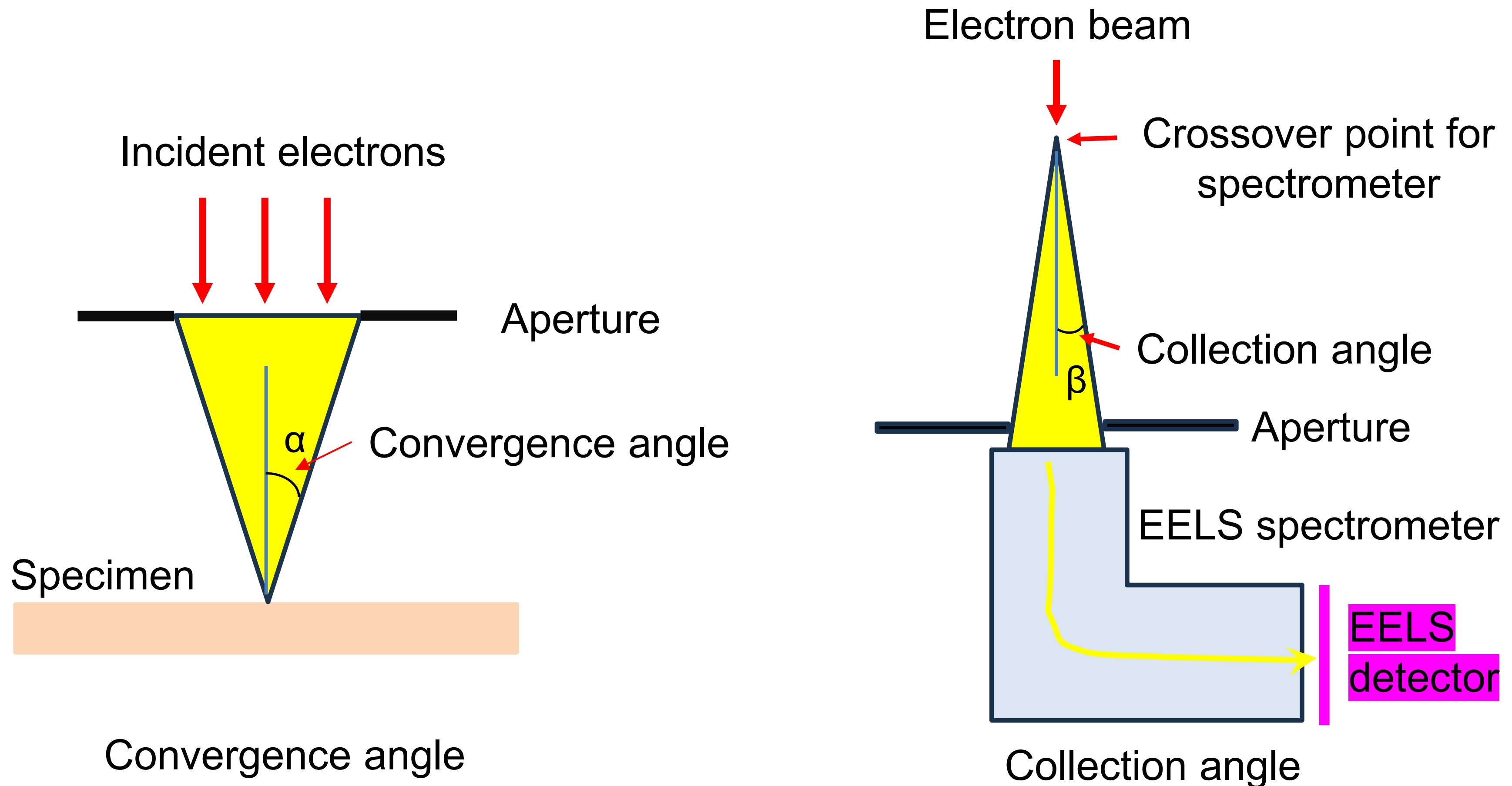
Convergence and Collection Angles

The values of the two angles are necessary for quantitative analysis of EELS results. Usually, half value of two different angles is used in analysis and in literatures.



Reference：the α and β may be documented in the measured results dependent on the TEM and GMS（Gatan microscopy suite, DigitalMicrograph）, if not, measurement of on your sample is necessary.

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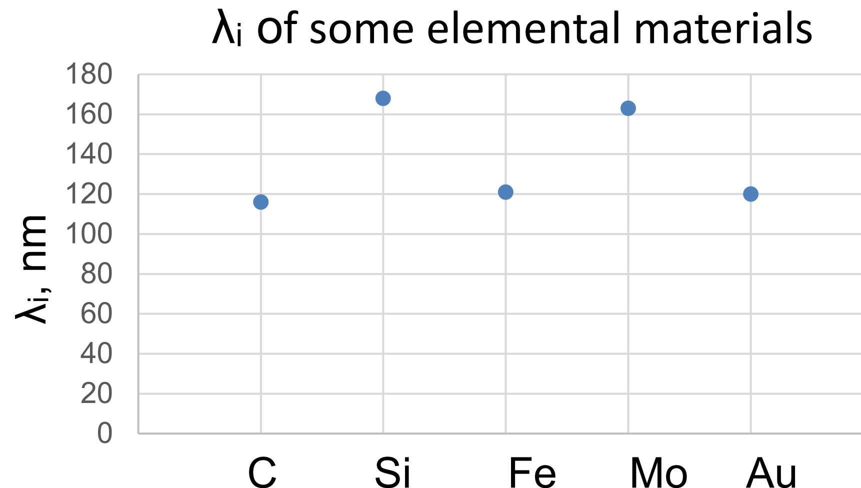
Electron Mean Free Path (λ_i)

The electron mean free path (λ_i) is the average distance of an electron travels before undergoing scattering. λ_i depends on the type of scattering, accelerating voltage, material, and scattering angle in the specimen. Plasmon scattering and core-loss scattering are two major inelastic scattering processes and are readily identifiable in EELS.

λ_i of several elemental materials are graphed. Most of the elemental materials have λ_i of ~100 nm. For achieving a mainly single-scattering condition, a rough value of the thickness of a specimen is less than about 100 nm.



*: E_0=200kV, α = 20 mrad, β = 20 mrad.

*: λ_i reflects the plasmon and signal

(Reference: Egerton R.F. Electron Energy-Loss Spectroscopy in the Electron Microscope (2011))

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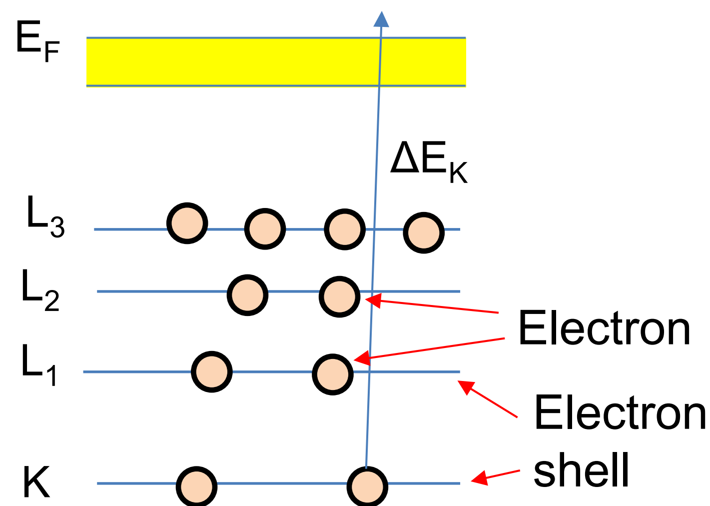
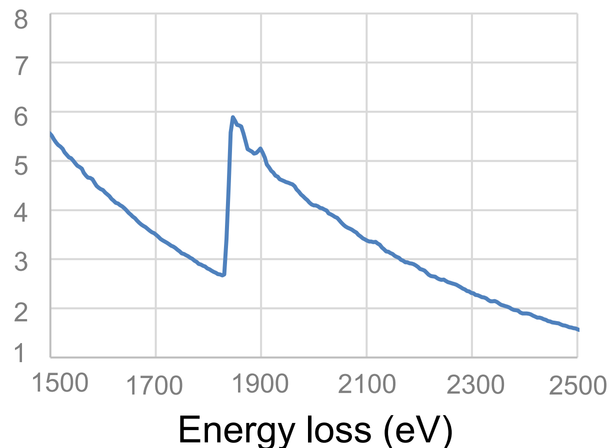
Inner-Shell Excitation and EELS Signals

Example: Silicon (Si)

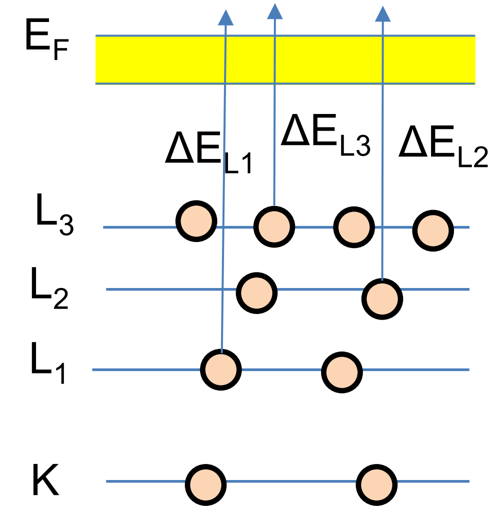
ΔE: The energy difference between two electronic states involved in the excitation.

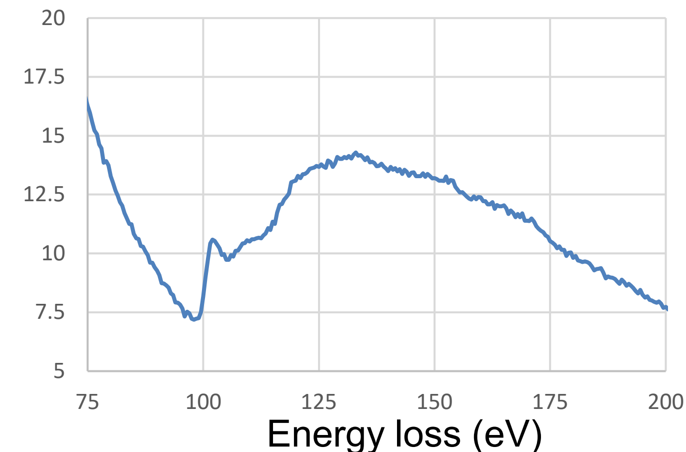
This energy difference corresponds to the energy loss of the incident electron.





Excitation of K-shell electrons of Si and the corresponding EELS signal.





Excitation of L-shell electrons of Si and the corresponding EELS signal.

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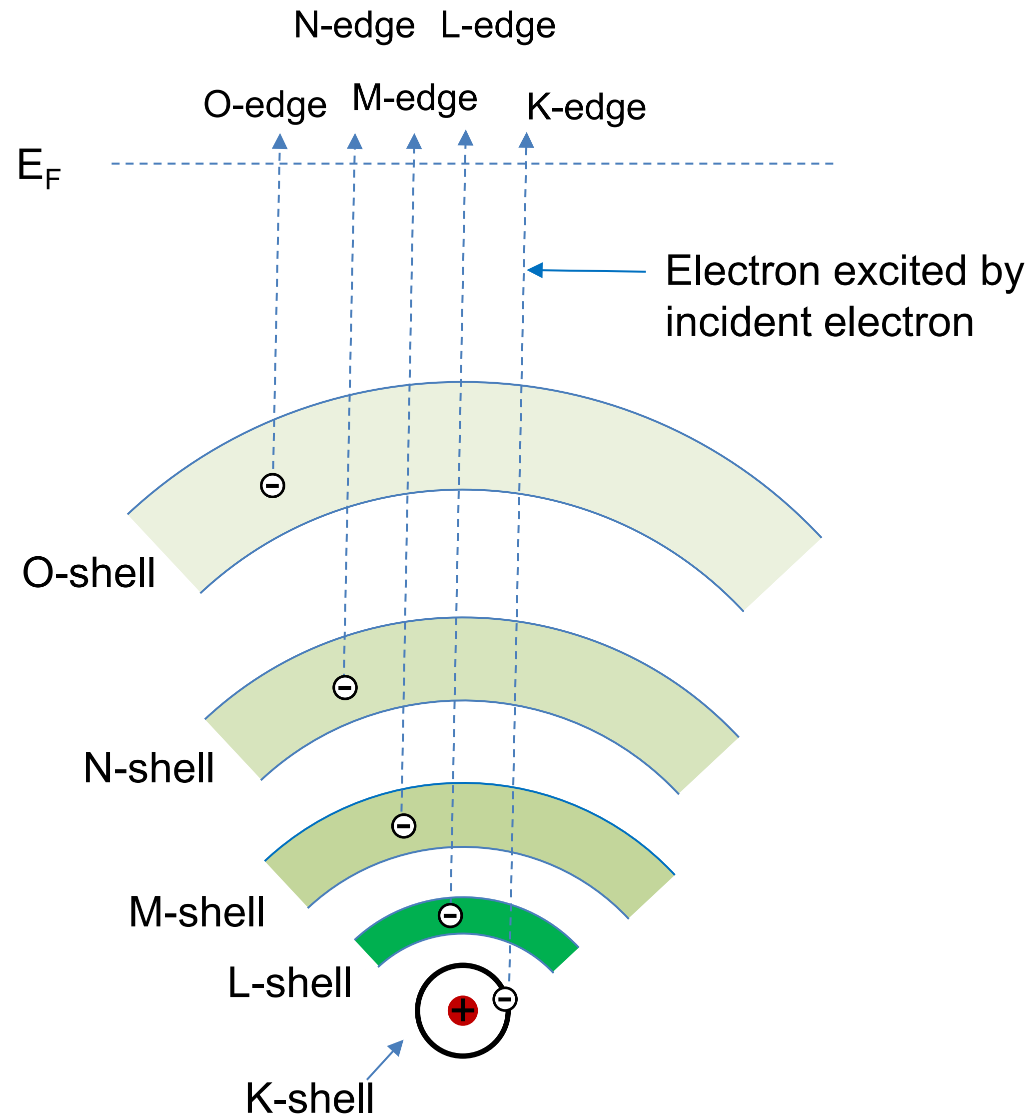
The Excitation of Inner Shell Electrons and the Signal of EELS

Energy loss edges in a general case.

For heavier elements (e.g. Z ≥ 35 (Br), the edges of shallower shells (M or higher) are usually used for EELS measurement, because the signals of L-, or K-edges are too weak to be effectively detected.



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Specimen Preparation for EELS Measurements

For reliable EELS results, several aspects of specimen preparation should be considered.

Specimen thickness

The specimen should be sufficiently thin, preferably less than λ_i.
Only a single plasmon peak should be visible in the low-loss region.

Electrical and thermal conductivity

Poor electrical conductivity can cause charge-up, specimen drift, and contaminations.
Poor thermal conductivity may increase beam damage.
A thin carbon coating (~2 nm) on both sides of the specimen can effectively improve both conductivity.

Contamination control of specimen

Contaminations reduce signal-to-noise ratio and may make measurements impossible.
Baking and vacuum storage of the specimen prior to observation are effective for reducing contamination.
Electron-beam showering or plasmon cleaning can suppress contamination, if applicable.

Reduction of beam damage

Adjusting the accelerating voltage and other measurement conditions are necessary.

Reduction of specimen drift

Use a stable supporting film/mesh and firmly attach the specimen to the specimen holder.

Sensitivity to elements of EELS

EELS working at a lower accelerating voltage (e.g. 80 kV compared with 200 kV) principally has higher sensitivities to elements for an appropriate specimen.

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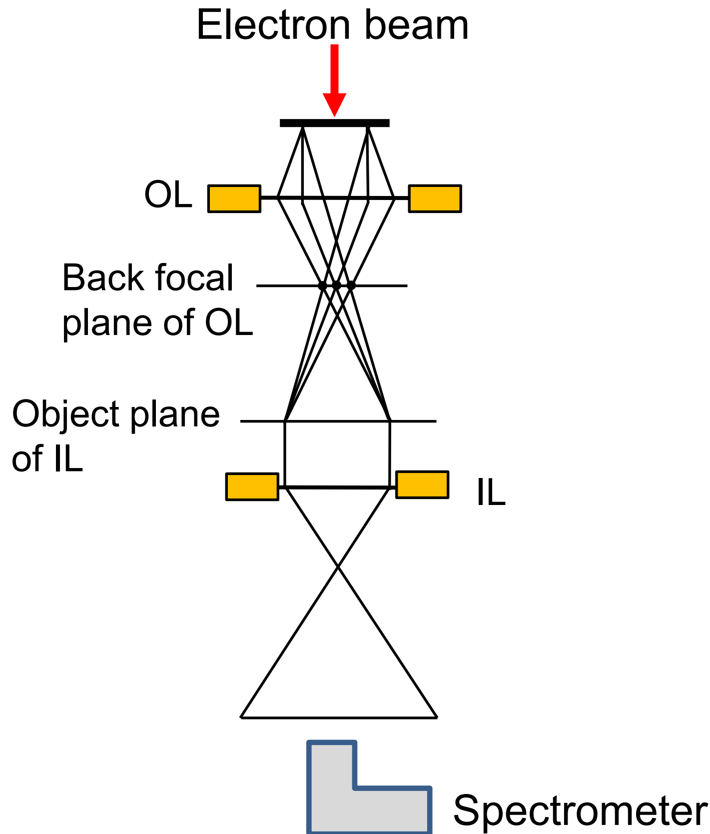
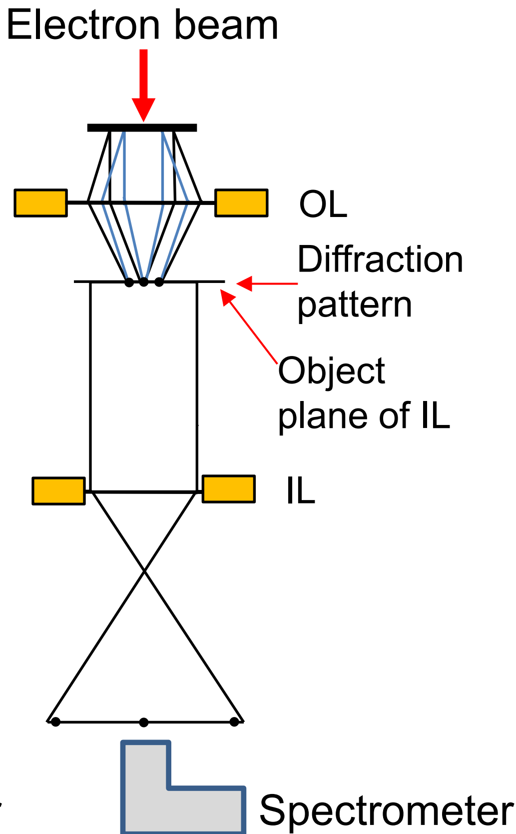
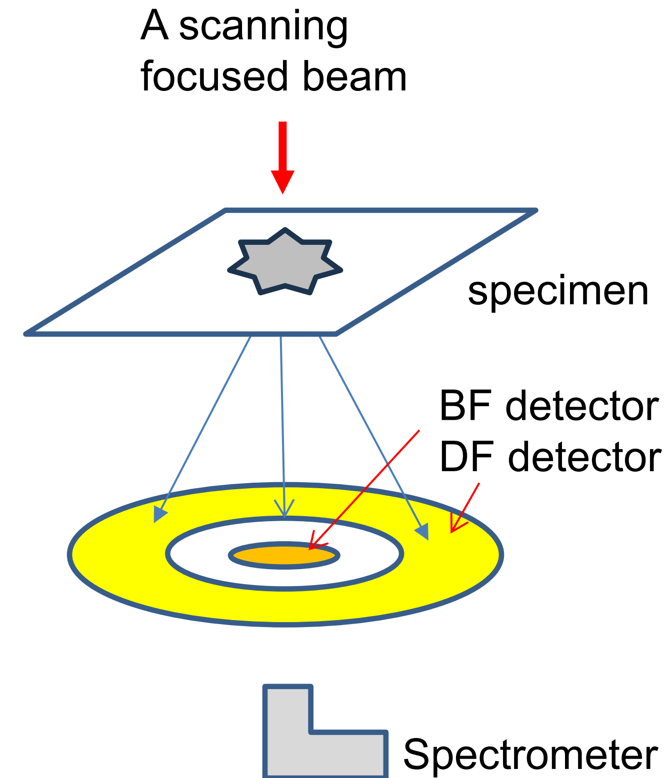
Different Measurement Modes of EELS (STEM EELS is mainly introduced in this section)







α: ~0 mrad
β: Decided by the size of OL aperture

α: ~0 mrad
β: Decided by camera length

α: Decided by the size of CL aperture
β: Decided by camera length

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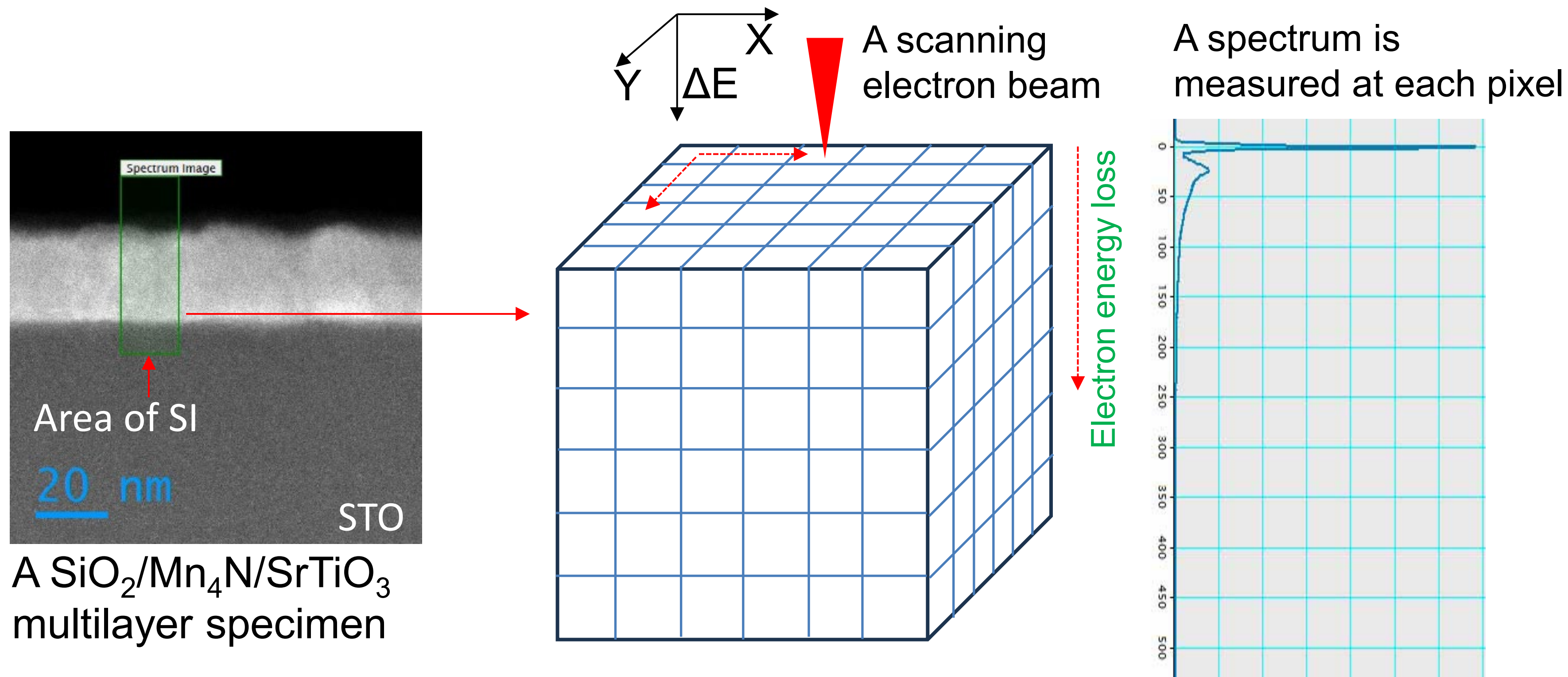
Spectrum Image (SI)

A spectrum image (SI) is also a typical data format in **STEM-EELS** mode. The electron beam scans across a selected area of the specimen, and an EELS spectrum is acquired at each pixel. As a result, a **three-dimensional (3-D) dataset** (X, Y, ΔE) is obtained.

3-D information of an SI



A $SiO_2/Mn_4N/SrTiO_3$ multilayer specimen

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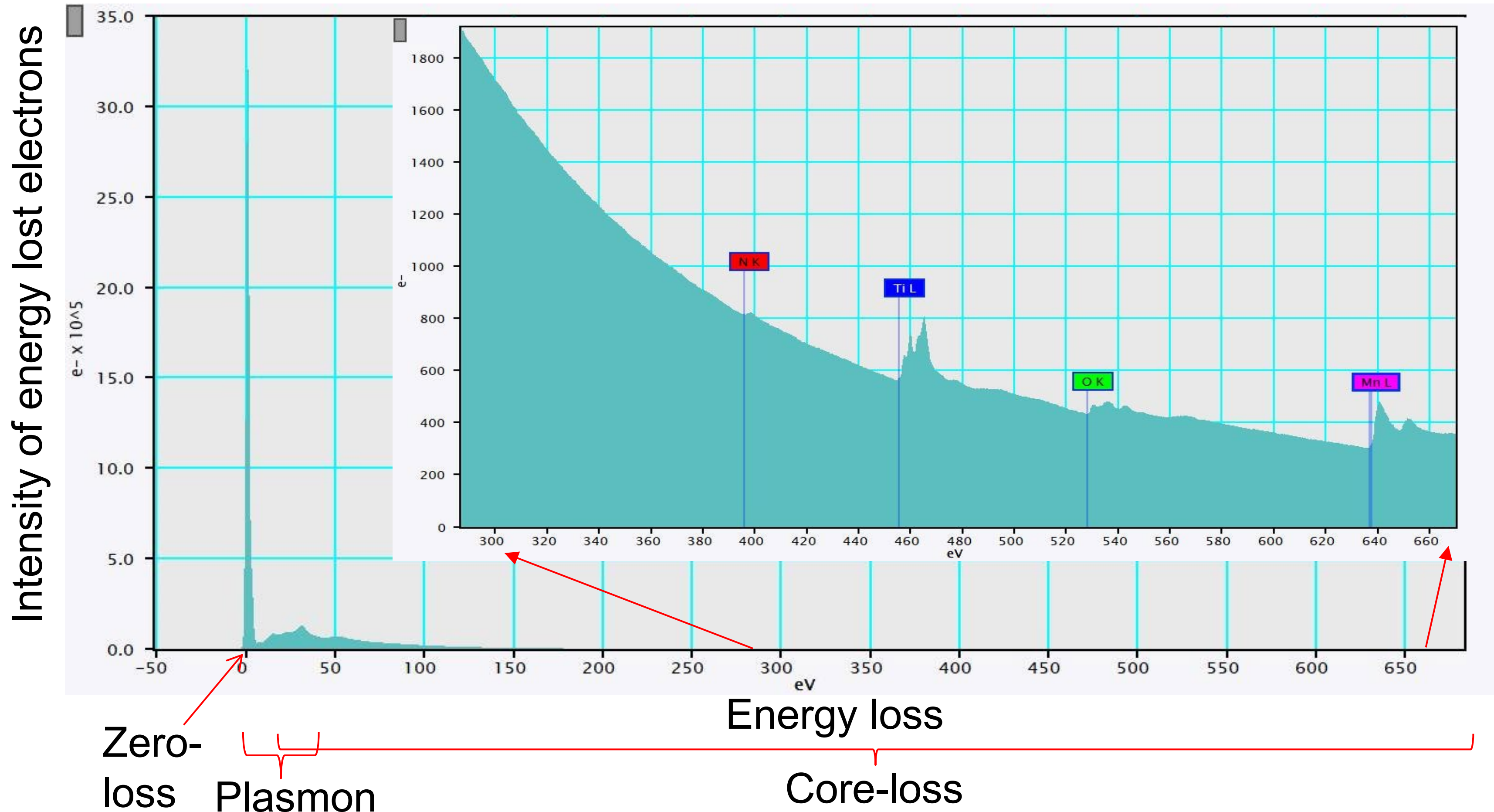
Information from EELS Spectra (1)

The energy (eV) and intensity of plasmon-loss and core-loss edges of a specimen

An example of EELS spectrum: Energy loss ranges on a spectrum



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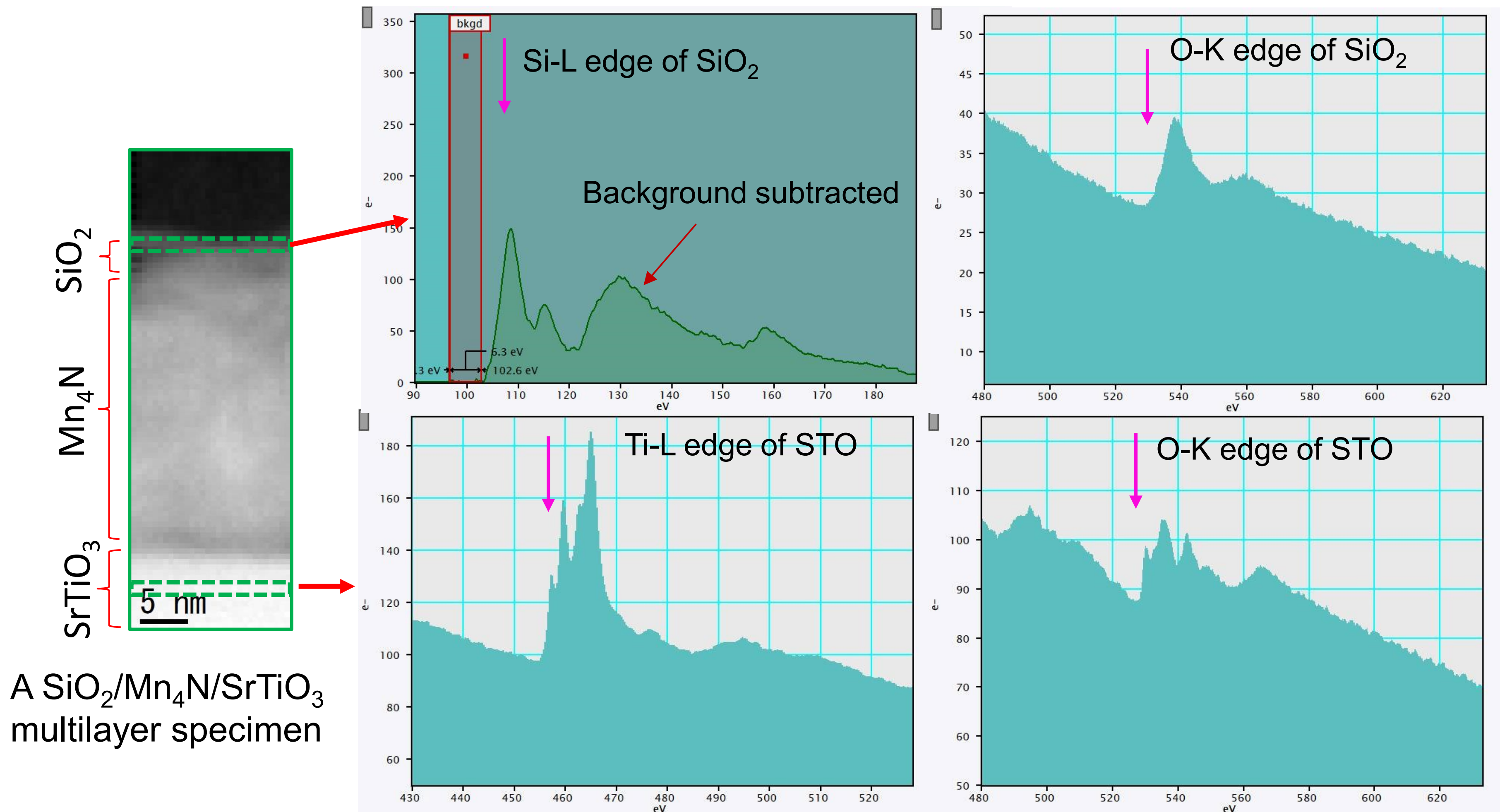
Information from EELS Spectra (2)

Identify elements and their chemical states with core-loss edges

Examples of EELS spectra: Core-loss edge of a $SiO_2/Mn_4N/SrTiO_3$ multilayer specimen.
Oxygen has different chemical shifts in SiO_2 and STO



A $SiO_2/Mn_4N/SrTiO_3$ multilayer specimen

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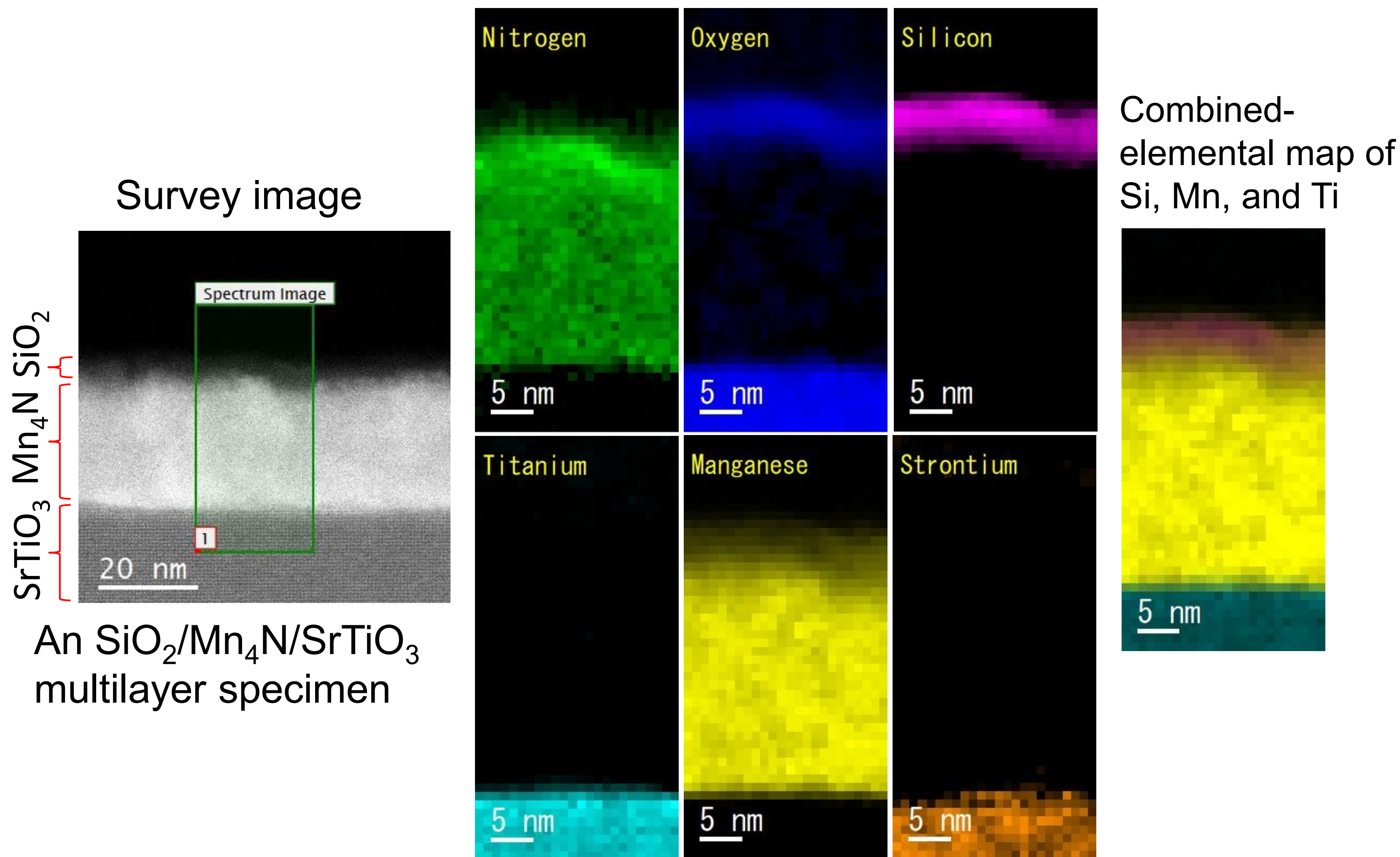
Information from EELS Spectra (3)

- Distribution and composition of elements

An example of measurement: distribution of elements and their composition



An $SiO_2/Mn_4N/SrTiO_3$ multilayer specimen

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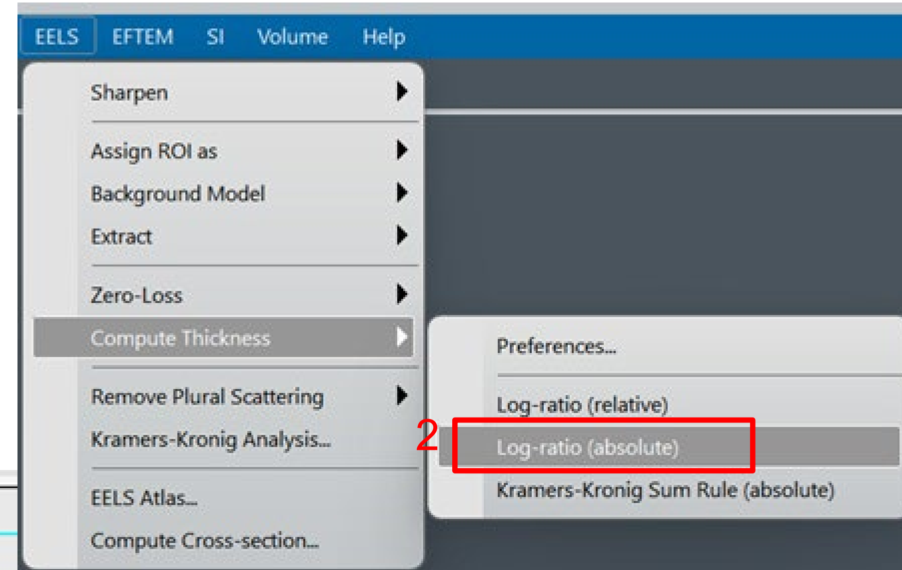
Information from EELS Spectra (4)

- Measuring Specimen Thickness

Using an EELS spectrum that includes energy losses from 0 to approximately **150 eV or higher**, the thickness of a specimen can be measured.

Procedure

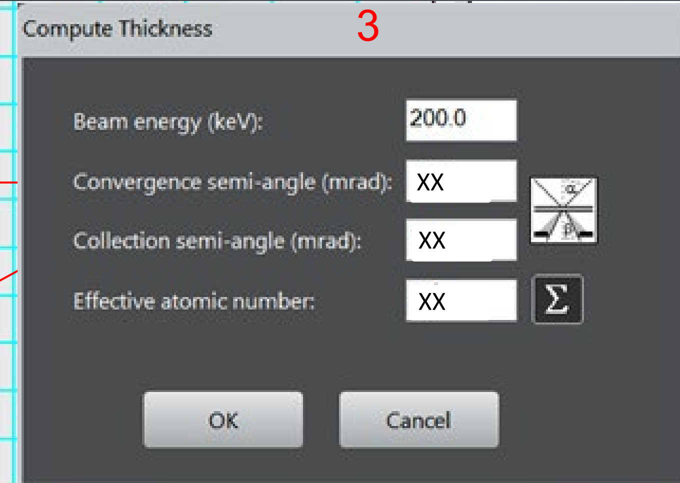
1. Acquire an EELS spectrum and calibrate the energy scale.
2. Select "menu": **EELS → Compute Thickness**.
3. Input the experimental parameters and click **OK**.



1

Decided by condenser Lens aperture

Decided by camera L. & entrance aperture



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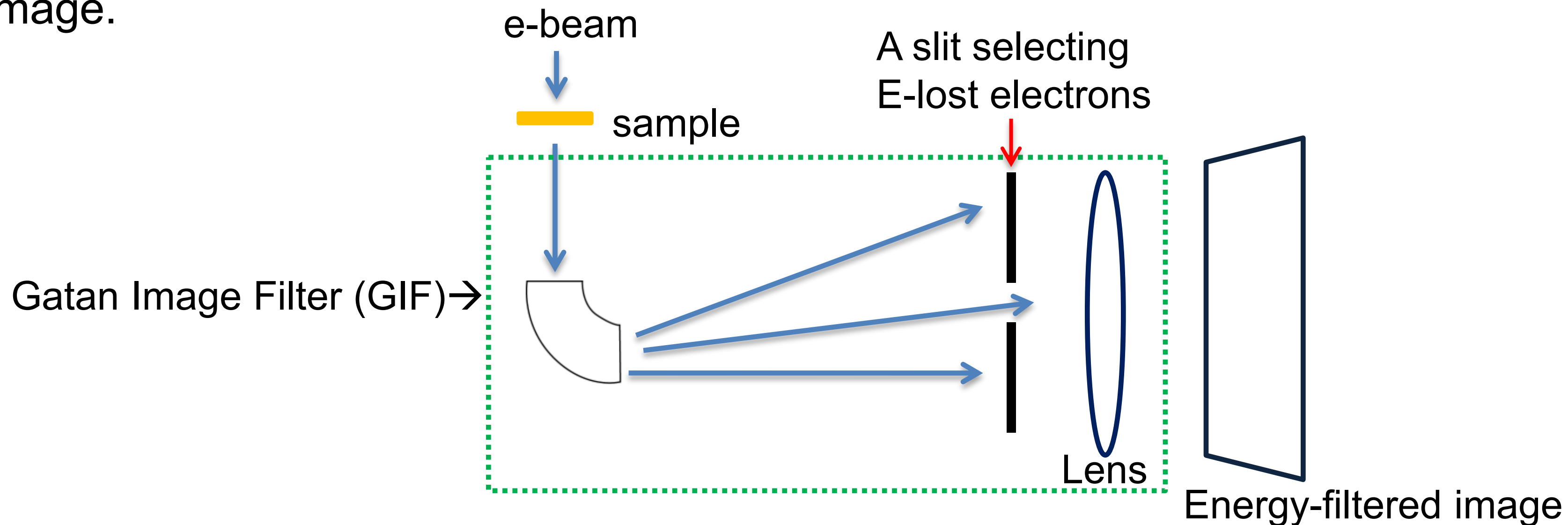
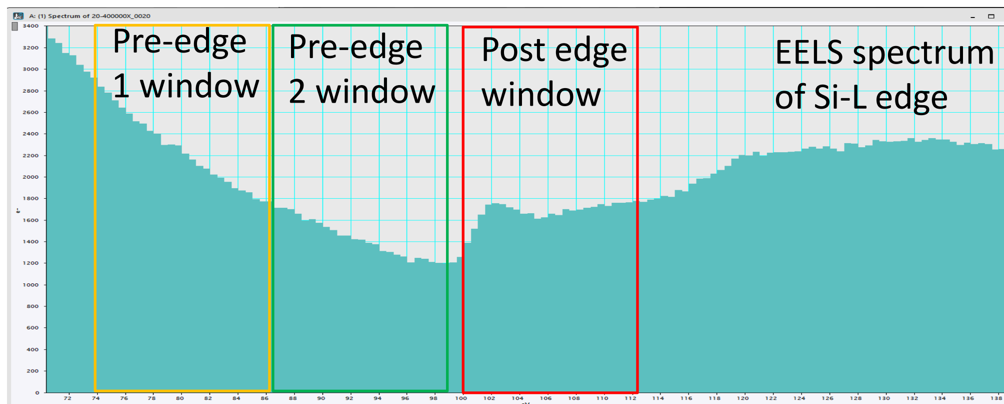
Energy Filtered TEM (EFTEM, or Energy-Filtered Image (EFI))

An energy-filtered image (EFI) is obtained using a **Gatan Image Filter (GIF)** with the TEM set to **image mode**. By inserting an energy-selecting aperture (slit), electrons within a specific energy-loss range are selected. These electrons propagate through the lens system and form an image, which is referred to as an energy-filtered image.





Example

A **Si elemental map** is obtained with the 3-window method. Three images are obtained with signals in the separate windows, then the elemental map is treated with the images.

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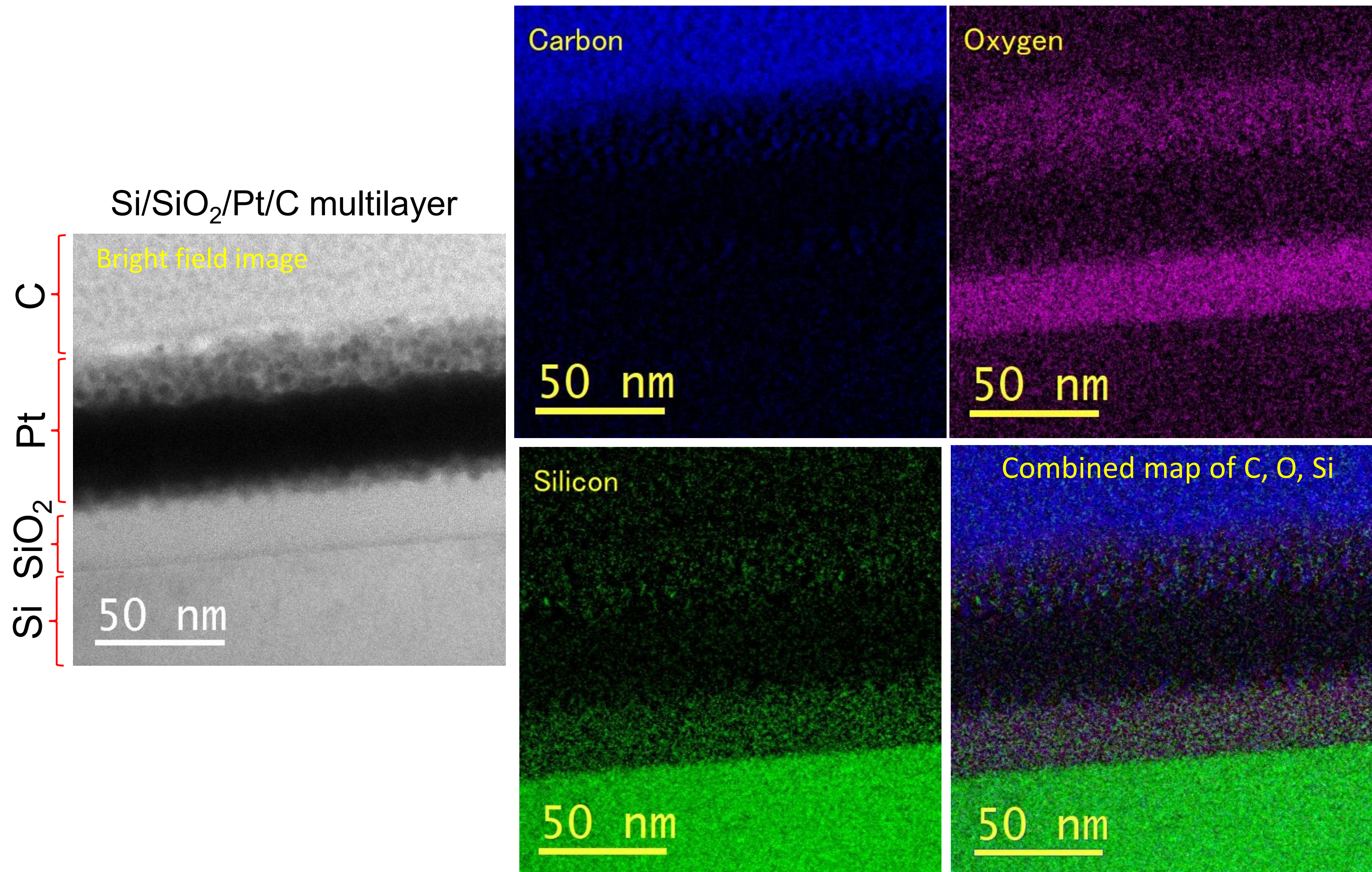
Examples of Elemental Maps with EFTEM



TEM and energy-filtered images (EFI) of a Si/SiO_2/Pt/C multilayer specimen. The distribution and co-existence areas of elements are visualized.

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5-4) Summery of Analytical Capabilities of TEM

The feature of elemental analysis methods in TEM
The appropriate analysis method depends on what information you want for your specimen.

Method	Space resolution	Sensitivity to light element	Chemical state	Quantitati ve	Simplicity of operation
EDS	△	△	×	○	○
EELS	○	○	○	○	△
EFTEM	△	○	△	△	△

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6. Summery and References

A Quick guide for possible measurement with different techniques of TEM/STEM

Aim	Recommended technique	Reason
Phase or structure identification	SAED, HRTEM, STEM	The information of crystal structure and lattice image are obtainable
Elemental analysis	EDS, EELS, EFTEM	Signals relate to element
Atomic contrast image	STEM-HAADF	Contrast highly relates to Z
Observing light elements	BF, STEM-ABF, EFTEM	HAADF is weak for light elements
Chemical analysis	EELS	Chemical shift can be measured
Measuring thickness	EELS	Good quantitative

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References

A free access multi-simulation program:
Recipro https://yseto.net/soft/recipro
Diffraction pattern, HRTEM image, etc. can be simulated.
There may be other useful information also on the internet.

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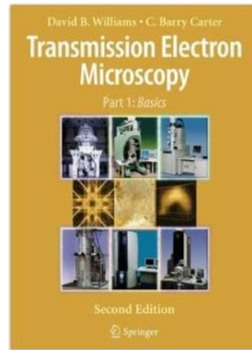
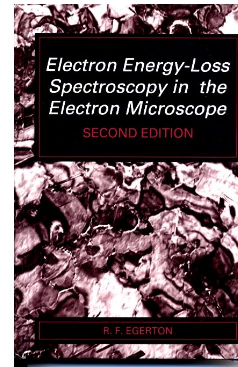
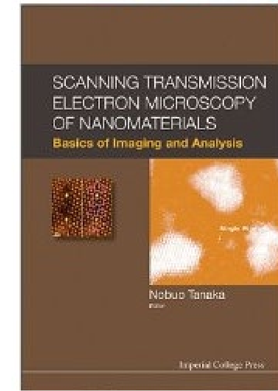
Books to Reference



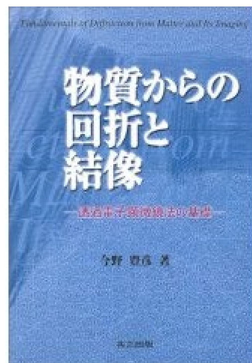
Williams & Carter:
On all aspects of TEM/ STEM



R. F. Egerton：
One of the most comprehensive books on EELS

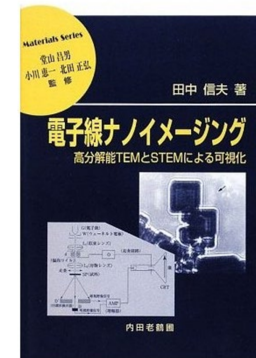


Editor. N. Tanaka



今野豊彦著：
電子顕微鏡の結像の原理が基礎から詳しく書かれている．式の導出も丁寧でわかりやすい．

進藤大輔・及川哲夫著：
EELSとEDSを中心にその原理と応用について平易に解説している
.



田中信夫 著：
弱位相物体近似を基に，収差とコントラストについて詳しく書かれている．

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Acknowledgments

本事業は、筑波大学高等研究院およびオープンファシリティー推進機構の共用機器である透過型電子顕微鏡を利用して実施されました。
本データの一部は、筑波大学数理物質系末益崇教授と末益研究室の学生から提供された試料を用いて、教材とさせていただきました。末益教授並びに末益研究室の学生に感謝申し上げます。

A part of this work was conducted using a transmission electron microscope at the Tsukuba Institute for Advanced Research (TIAR) and the Organization for Open Facility Initiatives, University of Tsukuba.
Some of the data presented here was provided by Professor Takashi Sueyoshi and students of the Sueyoshi Laboratory at the University of Tsukuba, and was used as teaching material. We would like to express our gratitude to Professor Sueyoshi and the students of the Sueyoshi Laboratory.
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The end

Thank you very much!