CHARACTERIZATION OF NANOSTRUCTURES

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The entire presentation is available at: http://web.iitd.ac.in/~anandh/Nanotech2007.ppt
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  *Daisuke Shindo and Kenji. Hiraga*
  Springer-Verlag, Tokyo, 1998.

- Experimental High-Resolution Electron Microscopy
  *John C.H. Spence*

- High-Resolution Electron Microscopy
  *John C.H. Spence*
When do we see anything? – Contrast
How do we see fine detail? – Resolution
How can we see better? – The TEM
What is meant by ‘High-Resolution’?
What things are there to be ‘seen’? – Imaging *(as we go along)*
HRTEM
Nanostructures by HRTEM: Image Gallery

HRTEM = High-Resolution Transmission Electron Microscopy
How can we define nanostructures in a general way?
What do we want to see in nanostructured materials?
Why do we want to see these details?
TEM: High-resolution phase contrast image

High-resolution micrograph from a Lu-Mg doped Si$_3$N$_4$ sample showing the presence of an Intergranular Glassy Film (IGF).
Atomic-resolution scanning transmission electron microscope (STEM) images of an intergranular glassy film (IGF) in La-doped $\beta$-Si$_3$N$_4$:

a) High-angle annular dark-field (HAADF-STEM)
b) Bright-field (BF-STEM)

“Observation of rare-earth segregation in silicon nitride ceramics at subnanometre dimensions”
HAADF-STEM images of the interface between the IGF and the prismatic surface of an $\beta$-Si$_3$N$_4$ grain. The $\beta$-Si$_3$N$_4$ lattice structure is superimposed on the images. **a)** La atoms are observed as the bright spots (denoted by red arrows) at the edge of the IGF. The positions of La atoms are shifted from that of Si atoms based on the extension of the $\beta$-Si$_3$N$_4$ lattice structure; these expected positions are shown by open green circles. **b)** reconstructed image of a, showing the La segregation sites more clearly. The predicted La segregation sites obtained by the first-principles calculations are shown by the open white circles.

“Observation of rare-earth segregation in silicon nitride ceramics at subnanometre dimensions”
TEM: Diffuse Dark Field Image

Diffuse Dark Field image from a grain boundary in SrTiO$_3$
When do we see anything? – Contrast

We see something if
- Light enters our eyes
- There is contrast in the image

We have strong or weak contrast*

$$C = \frac{I_1 - I_2}{I_2} = \frac{\Delta I}{I_2} > 5-10 \% \Rightarrow \text{we see}$$

- Amplitude contrast
- Phase contrast ~ Fringes

* But not bright or dark contrast (these terms refer to intensity and not to contrast)
In most of the situations both type of contrasts contribute to an image—although one will tend to dominate.
How do we see fine details? - *resolution*

- Resolution of human eyes \( \sim (0.1 - 0.2) \text{ mm} \)
- Highest useful magnification is governed by the resolution
- Raleigh criterion is used for the definition of resolution

**Optical Microscope**

\[
\delta = \frac{0.61 \lambda}{\mu \sin \beta}
\]

\[ \delta \sim \frac{\lambda}{2} \]

*Green light*

\( \lambda = 550 \text{ nm} \)

\( \delta \sim 300 \text{ nm} \)

\( \rightarrow 1000 \text{ atomic diameters} \)

**TEM**

\[
\lambda \sim \frac{1.22}{E^{1/2}}
\]

100 keV electrons \( \rightarrow \lambda = 4 \text{ pm} \)

- \( \delta \rightarrow \) Smallest distance that can be resolved
- \( \lambda \rightarrow \) Wavelength of radiation
- \( \beta \rightarrow \) Semi-angle of collection
- \( \mu \rightarrow \) Refractive index of viewing medium
Magnification without detail in image does not help!
Rayleigh criterion

- Not a fundamental rule but a practical definition.
- Figure of merit in terms of the eyes ability to distinguish separate images of two self-luminous incoherent point sources.
- A single point source will not be imaged as a point even if aberrations are absent.
- Any physical limit in the path of the rays (*outer boundary of the lens / aperture*) will lead to diffraction effects.
- Diffraction $\rightarrow$ point is imaged as a disc (*Airy disc*).
What is meant by High-Resolution?

**In general**

- **High Spatial Resolution**
  - microscope’s ability to process high spatial frequencies

- **High Energy Resolution**
  - spectrometer’s ability to resolve two closely positioned energy peaks

- **High Chemical Resolution**
  - ability to detect small quantities of a given chemical species

Additionally, one would like to differentiate two structures adopted by a material of a given composition
  - *This is essentially related to the above-mentioned points*
Phase contrast (lattice fringe) image
but not ‘high’-resolution

Phase contrast image of as-cast Mg_{37}Zn_{38}Y_{25} alloy
showing a 18 R modulated phase
Picture taken in a JEOL 2000FX microscope
with W filament. Resolution ~ 2.8Å

High-resolution
but not a lattice fringe image

Fourier Filtered Fresnel Contrast Image from Si_{3}N_{4} – grain boundary having an IGF
Picture taken in a JEOL JEM 4000 EX, with LaB_{6} filament. Resolution ~ 1.8Å
How can we see better? – *The TEM*
Absorbed Electrons → Electron-Hole Pairs

Incident High-kV Beam

Direct Beam

SPECIMEN

Secondary Electrons (SE)

Characteristic X-rays

Visible Light

Bremsstrahlung X-rays

Inelastically Scattered Electrons

Elastically Scattered Electrons

Backscattered Electrons (BSE)

Auger Electrons

Backscattered Electrons (BSE)

Elastically Scattered Electrons

Direct Beam

Electron-Hole Pairs

Absorbed Electrons ← SPECIMEN
Incident electron beam direction

Incident electron beam With Uniform intensity

Thin specimen

Scattered electrons with varying intensity

Image

Incident electron beam direction

Scattering by the specimen changes the spatial and Angular distribution of the electrons

Thin specimen

Diffraction pattern

Forward scattered beam directions
Elastic scattering is usually coherent if specimen is thin & crystalline.
In TEM elastic scattering is predominant in (1 - 10)° in the forward direction.
Elastically scattered electrons at large angles (> 10°) can be incoherent.
Inelastic scattering → almost always incoherent.
Most of scattered electrons < ±5° (< about 3° they are coherent).

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Specimen

- Thin → Coherent forward scattering
- Thick → Incoherent back scattering
Coherent incident beam

Incoherent elastic backscattered electrons

Secondary electrons from within the specimen

Thin specimen

Coherent elastic scattered electrons

Incoherent elastic forward scattered electrons

Direct beam

Incoherent inelastic scattered electrons

Incoherent elastic backscattered electrons

Coherent incident beam

Incoherent elastic backscattered electrons

Secondary electrons from within the specimen

Bulk specimen
Interaction cross section ($\sigma$)

- The chance of a electron undergoing any interaction with an atom is determined by an interaction cross section.

Probability that a electron scattering event will occur = \( \frac{\sigma \text{ (in area units)}}{\text{Actual area of atom}} \)

\[ \sigma_T = \sigma_{\text{elastic}} + \sigma_{\text{inelastic}} \]

\[ \sigma = \pi r^2 \]

\[ r_{\text{elastic}} = \frac{Ze}{V\theta} \] (approximate) neglects screening effects

- $r \rightarrow$ effective radius of a scattering centre
- $Z \rightarrow$ atomic number
- $\theta \rightarrow$ Scattering angle (greater than this)
- $V \rightarrow$ potential of incoming electron
- $e \rightarrow$ charge in esu (4.803 x 10^{-10} esu)

**probability of scattering by the specimen** $= P = Q_T t = \frac{N_0 \sigma_T (\rho t)}{A}$

- Transition metals 100 keV electrons (Mean free path ($\lambda_{MFP}$))

\[ \lambda_{MFP} = \frac{1}{\theta} = \frac{A}{N_0 \sigma_T \rho} \]

\[ \sigma_{\text{elastic}} \approx 10^{-22} \text{ m}^2 \]

\[ \sigma_{\text{inelastic}} \approx \left( 10^{-22} - 10^{-26} \right) \text{ m}^2 \]

\[ \lambda_{MFP} \approx 10s \text{ of nm} \]
Elastic Scattering

A. Scattering by electron cloud

B. Scattering by nucleus (Rutherford scattering)

At high angles Rutherford scattered electrons become incoherent

- Forward scattered
  - Z contrast imaging $\rightarrow$ atomic resolution microanalysis

- Back scattered
  - In TEM this signal is small
    - Image of beam entrance surface (Z + topography) $\rightarrow$ used in SEM

- A, B processes may not be truly elastic (B may produce x-rays)
- Higher the angle that an electron emerges $\rightarrow$ more the chance that it would have been inelastically scattered
Change in atomic scattering factor with scattering angle

\[ f(\theta) = \frac{\sin \theta}{\lambda} \]
Variation of Rutherford cross section with scattering angle

Beam Energy = 100 KeV

- Basis for HAADF
  - High-angle scattering is incoherent
  - $\Rightarrow$ image contrast is due to Z and not orientation of the specimen

Decreases orders of magnitude!
Inelastic Scattering (Energy Loss)

- Bremsstrahlung
- X-ray
- Characteristic x-rays
- Secondary electrons
- Collective interactions / Oscillations
- Electron-hole pairs and cathodoluminescence
- Radiation damage
Incoming electrons (~100 keV)

Vacuum

Conduction Band

Valence Band

Energy levels

$E_{L_3}$ $E_{L_2}$ $E_{L_1}$ $E_K$

Nucleus

Energy Loss electrons

Characteristic x-rays (10^{-16}s later)

Characteristic x-rays
Secondary electrons

Topography in SEM
(resolution using FEG ~ 1nm at 30 keV)
+
STEM (high-resolution)

- Slow SE
  - ~50 eV

- Fast SE
  - (50-200 keV)

100s ev - few keV
Auger electrons

FSE are unavoidable and undesirable
- One does not form images with them as they can come from deep within the specimen
- May degrade the quality of spectroscopic data

For surface chemistry

Up to 50% Energy loss
Semiconductors

Conduction band

Valence band

Band gap

Electron-hole pairs and cathodoluminescence

$e^-$

Semiconductors

Hv cathodoluminescence (CL)

spectroscopy

Bias

Electron beam induced current (EBIC)

Charge collection microscopy

OR
Cross sections for the various scattering processes

- **Plasmons**
- **Elastic scatter**
- **Ionization**
- **Secondary Electrons**

Incident beam energy (keV) →

Aluminium

Cross sections for the various scattering processes

- **Plasmons**
- **Elastic scatter**
- **Ionization**
- **Secondary Electrons**
### Electron properties as a function of accelerating voltage

<table>
<thead>
<tr>
<th>Accelerating voltage (kV)</th>
<th>Nonrelativistic wavelength (nm)</th>
<th>Relativistic wavelength (nm)</th>
<th>Mass (x (m_0))</th>
<th>Velocity (x (10^8) m/s)</th>
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<tr>
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</tbody>
</table>

Resolution in the TEM

Electron Source $\lambda = f(\text{Voltage})$, High coherency $\rightarrow$ FEG

Imaging System

- Electron Source
- Imaging System
\[
\beta = \frac{\lambda}{\beta}
\]

Can’t increase as lens aberrations ↑ with \( \beta \)

\( \beta \) Can be increased by a larger lens aperture but electron lenses are not good

Note: Electron sources are coherent ⇒ can’t use Rayleigh criterion.
Resolution

Electron Source
\[ \lambda = f(\text{Voltage}), \text{High coherency} \rightarrow \text{FEG} \]

Imaging System

Lenses
• Lens defects affect the resolution but give us increased ‘Depth of Focus’ & ‘Depth of Field’
• Can suffer from 10 kinds of aberration

Spherical Aberration

Chromatic Aberration

Astigmatism

Apertures
Control
• Beam current and Convergence
• Cutoff paraxial rays which suffer aberration
• Image contrast
• Type of image (BF / DF)
• Select areas from which we want diffraction patterns (SAD)

Control the quality of
• Images
• Diffraction patterns
• Analytical signals
The current best electromagnetic lens is like using a coke bottle for a magnifying lens.

If the lens of our eyes was as good as the best electromagnetic lens available then we would be legally blind.

Lenses aberrations

Spherical Aberration
\[ r_{\text{sph}} = C_s \beta^3 \]
\[ C_s \sim \text{Focal length} \sim (1-3 \text{ mm}) \]
- Most important in objective lens in TEM & condenser lens in STEM

Chromatic Aberration
\[ r_{\text{chr}} = C_c \frac{\Delta E}{E} \beta \]
- Current high tension supplies are stable to 1 in 10^6 (100 keV beam → 0.1 eV)
- For electrons passed through a sample (50-100 nm thick) → \( \Delta E = (15-25) \text{ eV} \)

Astigmatism
\[ r_{\text{ast}} = \beta \Delta f \]
- Due to non-uniform magnetic field in the path of electrons
- Can be corrected with a stigmator

Maximum difference in focus introduced by astigmatism
The spherical aberration of the lens causes wavefronts from a point object P to be spherical distorted.

⇒ The point is imaged as a disc with a minimum radius in the plane of least confusion.

The disc is larger in the Gaussian image plane.

This figure has been exaggerated in the angular dimension.
Spherical aberration

Assume

Specimen is thin → low chromatic aberration

⇒ $r_{sph}$ limits the resolution

Further ⇒ resolution $\equiv f$(Rayleigh criterion, aberration error)

$$r_{\min}(\beta_{opt}) = 0.91 \left( c_s \lambda^3 \right)^{\frac{1}{4}} \approx \left( c_s \lambda^3 \right)^{\frac{1}{4}}$$

$$\beta_{opt} = 0.77 \left( \frac{\lambda}{c_s} \right)^{\frac{1}{4}} \approx \left( \frac{\lambda}{c_s} \right)^{\frac{1}{4}}$$

- 100 kev electrons
- $\lambda = 0.0037\,nm$
- $c_s = 3\,mm$
- $\beta_{opt} \approx 15mrad$
- $r_{\min}(\beta) \approx 0.3\,nm$
- HREM $\rightarrow r_{\min}(\beta) = 0.15\,nm$

Useful magnification

Take resolution on specimen $\sim 2\,\AA$ → Magnify the resolution of eye $0.2\,mm$

$$M_{useful} = \frac{0.2 \times 10^{-3}}{0.2 \times 10^{-9}} = 10^6$$
Inelastic scattering causes loss of the energy of electrons
Electron-electron interaction
Loss in energy + Change in momentum
Thin specimen required
EELS spectrometer has a very high energy resolution
\[ (FEG \sim 0.3 \text{ eV}), \quad (XEDS \rightarrow \text{resolution} \sim 100\text{eV}) \]
Note: beam energy can be 400 kV
Can be used in forming energy filtered images + diffraction patterns
EELS

Phonon excitation
(~ 0.2 eV, 5-15 mrad)
- Part of the zero loss peak.
- Not resolved.
- Causes specimen to heat up.

Inter of intra band transitions
(5-25 eV, 5-10 mrad)
- Signature of the structure

Plasmon excitation
(~5-25 eV, < 0.1 mrad)
- Half the energy of bulk plasmons
- Transverse waves
\( \lambda_p \sim 100\text{nm} \)

Surface plasmon

Bulk plasmon
- Longitudinal waves

Inner shell ionization
(~30-1000 eV, 1-5 mrad)
COLLECTIVE OSCILLATIONS

PLASMONS

- Collective oscillations of free electrons
  - Most common inelastic interaction
  - Damped out in $< 10^{-15}$ s
  - Localized to $< 10$ nm
  - Predominant in metals (high free electron density)

PHONONS

- Collective oscillations of atoms
  - Can be generated by other inelastic processes. (Auger / X-ray energy)
  - Will heat up the specimen
  - Small energy loss $< 0.1$ eV
  - Phonon scattered electrons $\rightarrow$ to large angle (5 – 15 mrads)
  - Diffuse background
Plasmon excitation

- Longitudinal wave like oscillations of weakly bound electrons
- Rapidly damped (lifetime $\sim 10^{-15}$ sec, localized to $< 10$ nm)
- Dominate in materials with free electrons ($n$) (Li, Na, Mg, Al)
  - But occur in all materials
- $E_p = f(n) \rightarrow$ microanalytical information
- Carry contrast formation, limit image resolution through chromatic aberration
- Can be removed by energy filtering
Inter of intra band transitions

- Change in orbital state of the core electron
- Interactions with molecular orbital → can be used for finger printing
- Secondary electron emission ($< 20\text{eV}$)
  
  *(hence in same energy-loss regime as band transitions)*
- Weakly bound outer-shell electrons control the reaction of a material to an external field → controls the dielectric response (can measure with the signal from the $< \sim 10\text{eV}$ region)
Inner shell ionization

- The other side of the coin of XEDS.
- Small cross section ($\Delta E \uparrow \rightarrow \text{cross section} \downarrow$)
- K, L ($L_1$, $L_2$, $L_3$), M ($M_1$, $M_2$, $M_3$, $M_{4,5}$).
- Combination of ionization loss with plasmon loss can occur.

- There are intensity variations superimposed on the ionization edge
  - ELNES (starting from about 30 eV of the edge and extending ~100s of eV)
  - EXELFS (~50 eV after ELNES)
- ELNES and EXELFS arise due to the ionization process imparting more than critical energy ($E_c$) for ionization
The EELS spectrum

- **Forward scattered (cone of few mrad)**
- **000 spot of DP**
- **Bragg diffracted peak (~20mrad) → rarely enters the spectrometer**
- **Includes energy loss of ~0.3 eV**
- **Includes phonon loses ⇒ EELS does not resolve phonon loses**

Zero loss peak

- FWHM defines the energy resolution
  - $\Delta E \uparrow \Rightarrow$ resolution $\downarrow$
  - $kV \uparrow \Rightarrow$ resolution $\downarrow$

Plasmon peak

Low loss region ~ 50 eV

Energy-loss (eV)

- **ELNES - Energy Loss Near Edge Structure**
- **EXELFS - EXtended Energy Loss Fine Structure**

Coordination, Bonding effects

Density of states, Radial distribution function
Chemical analysis (structural and elemental) using EELS

- Thin specimen is better (plasmon peak intensity < 1/10\textsuperscript{th} zero loss peak)
- Use high $E_0$ (scattering cross-section $\downarrow$, but benefit is in $\downarrow$ of plural scattering + edge signal to noise ratio $\uparrow$)
- Energy resolution limited by electron source

**Resolution**

- **Spatial Resolution**
  - Limited by size of probe (~1nm)
  - Limited by selecting aperture at spectrometer entrance (its effective size at the plane of the specimen)

- **Energy Resolution**
  - 1 eV (incident energy 200-400 eV!)

No visible Grain Boundary

Fourier filtered image

Dislocation structures at the Grain boundary

Si peak at 1839 eV
Sr L$_{2,3}$ peaks

Grain Boundary

$\sim$8° TILT BOUNDARY IN THE SrTiO$_3$ POLYCRYSTAL

VG microscope
EELS microanalysis

Si L edge ELNES changes across a Si-
SiO₂ interface due to change in Si bonding → atomic level images with spectra localized to individual atomic columns

Differences between C edge between graphite and diamond

Change in Cu L edge as Cu metal is oxidized

“Seeing is believing”

- 2D image of 3D specimen
- Diffraction contrast
- Contrast depends on lenses + apparatus
- Absorption contrast is usually small
- Phase contrast related to specimen and not to topography (or “depth”)
High Resolution Microscopy

With Reference to Lattice Fringe Imaging in a TEM
Point spread function

Real space

$f(x, y) = f(r)$

Specimen

Diffraction pattern

Back focal plane

Reciprocal space

$F(u)$

Contrast transfer function

$H(u)$

Image

$g(x, y) = g(r)$

Fourier transform

$G(u)$
Specimen transmission function

\[ g(r) = f(r) \otimes h(r) \]

Point spread function

\[ G(u) = H(u)F(u) \]

Convolution in real space gives multiplication in reciprocal space

Contrast transfer function

\[ H(u) = A(u)E(u)B(u) \]

Contrast transfer function

Aberration function

Envelope function

Aperture function

Specimen transmission function

\[ B(u) = \exp[i\chi(u)] \]

\[ \chi(u) = (\pi \Delta f \lambda u^2) + \left( \frac{\pi C_s \lambda^3 u^4}{2} \right) \]

\[ \chi(u) = f(\Delta f, \lambda, u, C_s) \]

Phase distortion function

Property of lens
- High resolution implies ability to see two closely spaced features in the sample (real ‘r’ space) as distinct.

- This corresponds to high spatial frequencies \(\Rightarrow\) large distances from the optic axis in the diffraction pattern (reciprocal (u) space).

- Rays which pass through the lens at such large distances are bent through a larger angle by the objective lens.

- Due to an imperfect lens (spherical aberration) these rays are not focused at the same point by the lens.

- Point in the sample \(\rightarrow\) disc in the image (spreading of a point in the image).

- Objective lens magnifies the image but confuses the detail (hence the resolution is limited).

- Each point in the final image has contributions from many points in the specimen (no linear relation between the specimen and the image).

- Is a linear relationship possible?
Specimen and approximations

\[ f(x, y) = A(x, y) e^{-i\phi(x,y)} \]

Phase Object approximation

\[ f(x, y) = e^{-i\phi(x,y)} \]

Phase change = f(V_t)

\[ f(x, y) = e^{-i\sigma V_t(x,y)} \]

Including absorption

\[ f(x, y) = e^{[-i\sigma V_t(x,y) - \mu(x,y)]} \]

Weak Phase Object approx.

\[ f(x, y) = 1 - \sigma V_t(x,y) \]

\[ V_t(x, y) = \int V(x, y, z) dz \]

\[ d\phi = \frac{\pi}{\lambda E} V_t(x, y) = \sigma V_t(x, y) \]

WPOA ⇒ For a very thin specimen the amplitude of the transmitted wave function will be linearly related to the projected potential

- \( f(x,y) \rightarrow \) specimen function
- \( \phi \rightarrow \) phase (function of \( V(x,y,z) \))
- \( V(x,y,z) \rightarrow \) specimen potential
- \( V_t(x,y) \rightarrow \) projected potential
- \( \sigma \rightarrow \) Interaction constant (\( =\pi/\lambda E \))
In vacuum: \[ \lambda = \frac{h}{\sqrt{2meE}} \]

In the specimen: \[ \lambda' = \frac{h}{\sqrt{2me(E + V(x, y, z))}} \]

Phase change:
\[ d\phi = \left( \frac{2\pi}{\lambda} \right) dz - \left( \frac{2\pi}{\lambda'} \right) dz = d\phi = \frac{\pi}{\lambda E} V(x, y, z) \, dz \]
\[ d\phi = \frac{\pi}{\lambda E} V_i(x, y) = \sigma V_i(x, y) \quad V_i(x, y) = \int V(x, y, z) \, dz \]

\( \sigma \rightarrow \) Interaction constant (=\( \pi/\lambda E \)) \( \rightarrow \) tends to a constant value as \( V \) increases
(energy of electron proportional to \( E \) & \( \lambda^{-1} \), variables tend to compensate)

\[ e^{-x} = 1 - \frac{x}{1!} + \frac{x^2}{2!} - \frac{x^3}{3!} + \frac{x^4}{4!} - \ldots \]
- POA holds good only for thin specimens
- If specimen is very thin \( \{V_t(x,y) \ll 1\} \rightarrow WPOA \)
- In WPOA \( \{f(x,y) = 1 - i\sigma V_t(x,y)\} \rightarrow \) the amplitude of the transmitted wave will be linearly related to the projected potential of the specimen
\[ g(r) = f(r) \otimes h(r) \]

\[ \Psi(x, y) = [1 - \sigma V_t(x, y)] \otimes h(x, y) \]

\[ \Psi(x, y) = [1 - \sigma V_t(x, y)] \otimes [\cos(x, y) + i \sin(x, y)] \]

\[ I = \Psi \Psi^* \]

Neglecting \( \sigma^2 \)

\[ I = [1 + 2\sigma V_t(x, y)] \otimes \sin(x, y) \]

\[ \Rightarrow \text{Only imaginary part of } B(u) \text{ contributes to intensity} \]

\[ B(u) = \exp[i\chi(u)] \]

\[ B(u) = 2\sin[\chi(u)] \]
Objective lens transfer function

\[ T(u) = A(u)E(u) \cdot 2 \sin[\chi(u)] \]

Approximately

\[ T(u) = 2A(u)\sin[\chi(u)] \]

Or

\[ T_{\text{effective}}(u) = T(u)E_{\text{chromatic}}E_{\text{spatial coherence}} \]

- Incoherent illumination \( T(u) = H(u) \)
- \( T(u) \rightarrow -\text{ve} \Rightarrow +\text{ve} \) phase contrast
  (atoms would appear dark)
- In WPOA \( T(u) \) is sometimes called CTF
- Envelope is like a virtual aperture at the back focal plane
\[ T(u) = 2A(u)\sin[\chi(u)] \]

\[ C_s = 1 \text{ mm}, \quad E_0 = 200 \text{ keV}, \quad \Delta f = 58 \text{ nm} \]
Scherzer defocus and Information Limits

\[ T(u) = 2A(u)\sin[\chi(u)] \]

\[ \Delta f_{\text{Scherzer}} = -\left(\frac{4}{3} \frac{C_s \lambda}{\Delta f}\right) \]

\[ u_{\text{Scherzer}} = 1.51 \frac{C_s}{\lambda} \left(\frac{\lambda}{4}\right)^{\frac{3}{4}} \]

\[ r_{\text{Scherzer}} = 0.66 \frac{C_s}{\lambda} \left(\frac{\lambda}{4}\right)^{\frac{3}{4}} \]

Instrumental resolution limit → can use nearly intuitive arguments to interpret the contrast

Instrumental resolution limit

Damping envelope
Computer simulation (a) and electron micrograph (b) of a Cu Cubeoctahedron consisting of seven shells (1415 atoms) in [001] orientation.

Thickness dependency of lattice images of a Si crystal (200 kV, $\Delta f = 65$ nm). Thickness changes from 1 nm (a) to 86 nm (r) in steps of 5 nm.

Defocus dependency of lattice images of a Si crystal (200 kV, $t = 6$ nm). Thickness changes from $-20$ nm (a) to 90 nm (l) in steps of 10 nm.

HRTEM - some tips

- In lattice fringe imaging mode:
  - Things which look like ‘atoms’ are not atoms!
  - Use image simulation to confirm what you see
  - Do not interpret details below the resolution limit of the microscope
- Spherical aberration correctors for illumination as well as imaging lens systems,
- Electron beam monochromators reducing the energy spread of the electron beam to < 0.1 eV
- High-energy resolution spectrometers (Sub-eV mandoline filter for EELS)
- Imaging energy filters
- Advanced specimen holders for in-situ experiments
- High-sensitivity and high-dynamic-range detectors for images, diffraction patterns, electron energy loss spectra (EELS) and energy dispersive X-ray spectra (EDXS)
- Advanced methods → Focal series reconstruction
  → Electron Holography
  → In-focus ‘Fresnel’ reconstruction
- It is possible to record phase contrast images with point resolutions of well below 0.1 nm, or alternatively, scan electron probes of size < 0.1 nm across the specimen, recording high-angle scattering or high-energy resolution spectra from areas as small as a single atomic column.
High-resolution (a) amplitude and (b) phase images of the aberration-corrected object wave reconstructed from an electron hologram of [110] Si, obtained at 300 kV on a CM 30 FEG TEM. The characteristic Si dumbbell structure is visible only after aberration correction.

Exit face wave reconstructed profiles, of the imaginary part of the inner potential, using zero-defocus Fresnel fringe images.
HRTEM micrographs of MWNT:
(a) 5-walled, diameter = 6.7 nm;
(b) 2-walled, diameter = 5.5 nm;
(c) seven-walled, diameter = 6.5 nm (hollow diameter = 2.2 nm).

HRTEM image of a BN multi-walled nanotube (inner shell distance of ~ 0.33 nm).
Cross section of TiN/NbN nanolayered coatings: (a) Conventional TEM micrograph with SAD patterns, (b) HRTEM of {200} lattice fringes.

High-resolution micrograph of Si$_3$N$_4$ – TiN composite prepared by CVD.


Gas atomized Al-Si powders:
(a) HREM, (b) SEM, (c) BFI.

HREM of ZrO$_2$ showing formation of nanotwins.

HREM image of nanocrystalline Pd

HRTEM image of a Kr nanocluster on a Mg substrate showing Moiré fringes. The lattice parameter of Kr can be calculated from the Moiré fringe spacing:

\[
\frac{1}{d_{\text{fringes}}} = |(1/d_{\text{MgO}}) - (1/d_{\text{Kr}})|
\]

HAADF-STEM images of the interface between the IGF and the prismatic surface of an β-Si₃N₄ grain.

THANK YOU