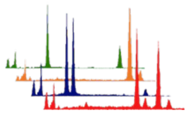
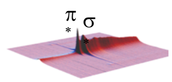
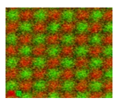
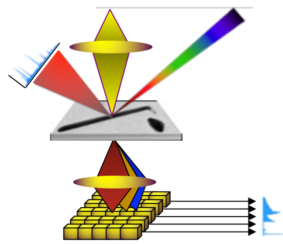
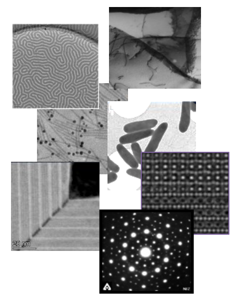
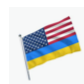
Introduction to
Transmission, Scanning Transmission & Analytical Electron Microscopy

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1

Workshop Outline

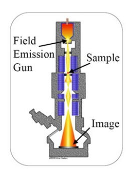
* EM & Challenges in Todays Materials
* Basic Instrumentation & Optics
* Basic Diffraction : SAED and some CBED
* Advanced/Applied CBED
* Basic Imaging Elastic Scattering TEM/STEM/HREM
* Dynamical Theory and Diffraction Contrast
* Basic Inelastic Scattering/AEM – XEDS/EELS
* Advanced Inelastic Scattering
* Other topics

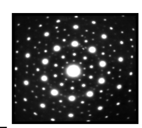
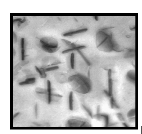
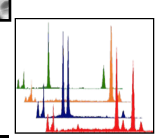
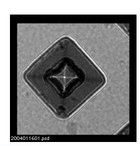
In-situ EM, Radiation Damage,

Tomography, Lorentz, Fluctuation Microscopy…

Computational Microscopy….

https://www.dropbox.com/s/c8edb7ri9czajkg/AMAS-2024-ZaluzecWorkshopLectures.zip?dl=0



2

2

Acknowledgements

R&D Support U.S. DoE / ANL / UofChicago

Colleagues contributing to these lectures include:

ANL EMC Group, Fraser, Mansfield, Eades, Calderon,
Jiao, Newbury, O 'Keefe, Weyland, Muller, ….
numerous text books, and others.

Apologies to all others from whom I can't remember
collecting images/figures over the years.

Permission granted to take photo's of anything presented

These Lecture Notes are Downloadable as PDFs

3

Materials Research Today

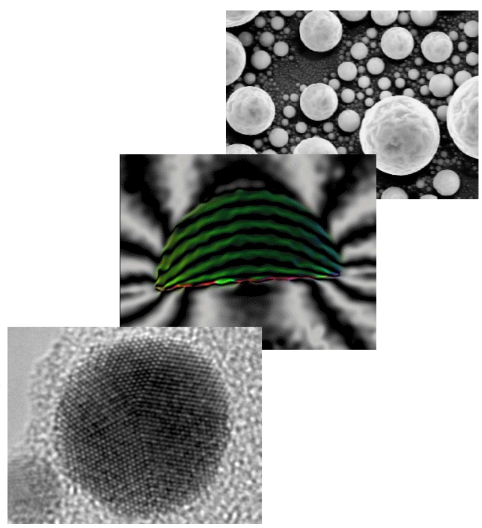
Understanding the Synergistic Relationships Between Structure and Properties

New Materials and Phenomena

- *Create / Discover*
- *Explore/Understand*
- *Control / Apply*

Motivation:

- *Serve National Strategic Mission*
  - *Energy/ Economy*
  - *Health*
  - *Defense*
- *Enable other Science Breakthroughs*

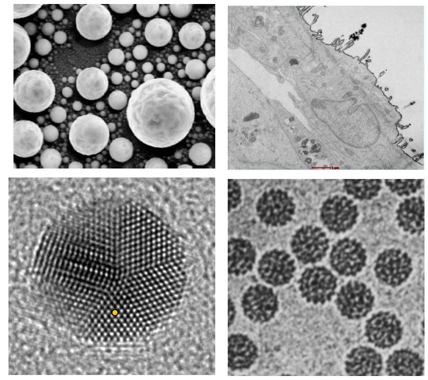


4

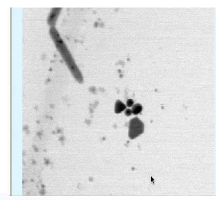
What are the basic questions we all ask ?

- **State**
  - Morphology
  - Crystallography
  - Elemental/Chemical Constituents
  - Bonding/Electronic Structure

- **Static vs Dynamic**
  - Temporal
  - Temperature
  - Stress/Strain/Mechanical Deformation
  - Vacuum/Gaseous/Liquid Environment
  - EM Fields
  - Irradiation Environment
    - Charged Particles
    - Photons….



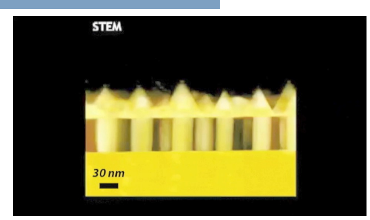
- **Key Challenges:**
  - In-situ observation of real time processes
  - In-situ high-spatial resolution elemental analysis
  - Simultaneous imaging of hard/soft components
  - Dynamics - Fast detection schemes, detectors, and sources



5

What are the basic questions most PI's Ask?

- *What does it look like?*
- *How big is it?*
- *What is it made of?*
- *Where are the important bits?*
- *This isn't what I expected why?*



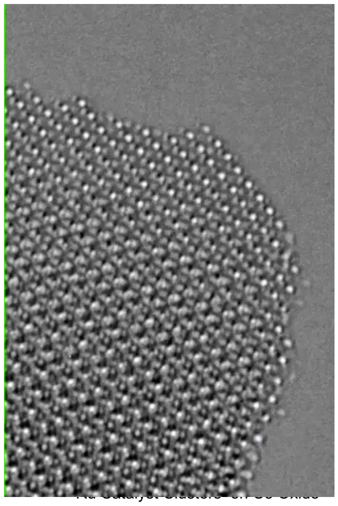
What are the limiting factors?

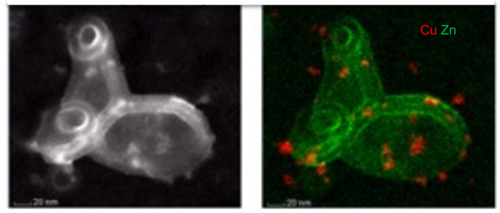
- *Barriers to entry*
  - *Bureaucracy*
  - *Funding*
  - *Staffing*
  - *Access*
- *Barriers to getting results*
  - *Specimens*
  - *Expertise*
  - *Infrastructure/Instrumentation*

6

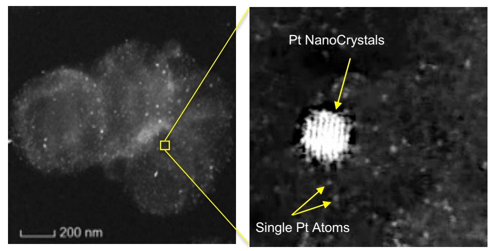
Today's Challenge : Multi-Modal / Analytical Sensitivity at the Sub-nm to Near-Atomic Level in Hard/Soft Matter

In Real Time





Heavy Metal Segregation - Polyamide Nanomembranes



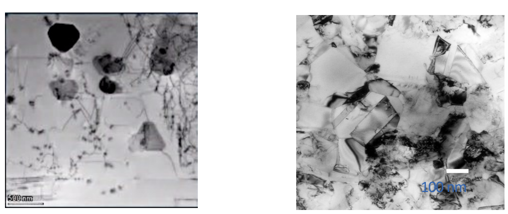
Sub-Angstrom Imaging in Photocatalysts in Thylakoids

Count every electron and make every electron count

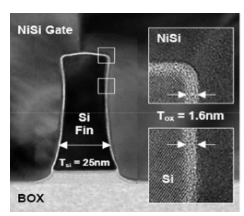
High Performance Materials: defects and interfaces

In order to understand materials behavior, we need to understand the defects that control materials behavior, we must characterize them.

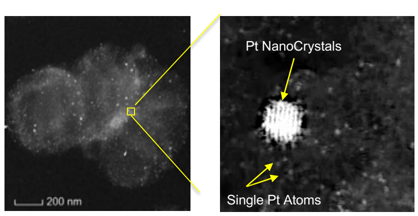
Dislocation motion often governs mechanical behavior in engineered alloys



… interfaces provide unique properties



A few atoms can dominate material behavior …



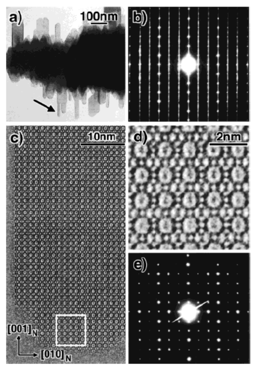
Sub-Angstrom Imaging in Photocatalysts in Thylakoids

Challenges:

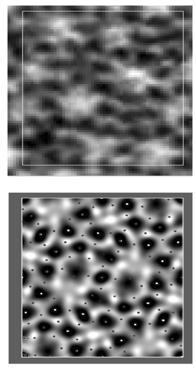
- 3D atomic-scale structure and chemistry
- "real" systems, not "special" interfaces
- real time dynamics
- in situ probes for properties

Small Particles: nanomaterials and catalysis

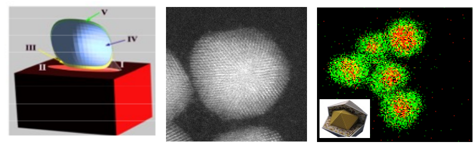
Nanomaterials and processes challenge our ability to understand them:



Zeolite structure solved by imaging and diffraction (Terasaki)



$Mo_1V_{0.32}Nb_{0.1}Te_{0.25}O_x$[1]
HREM image (top) simulation (bottom)
(Zajac - Inovene)



Multimetallic Electrocatalyst Nanoparticles

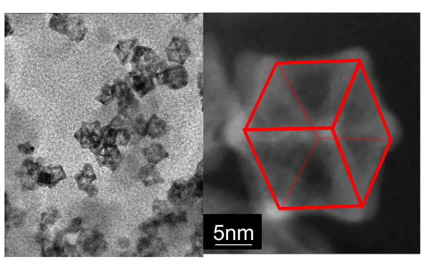
Challenges:

- ⧫ Quantification - nanocrystallography at atomic resolution (automated w/ real time modeling and simulation)
- ⧫ atomic scale 3D imaging and diffraction
- ⧫ In situ: controlled environments for simultaneous imaging and spectroscopy during reactions
- ⧫ Dynamics: real-time observation

9

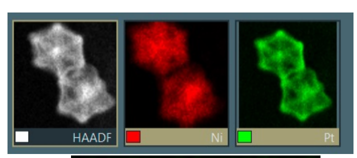
Engineered Nanomaterials and Processes

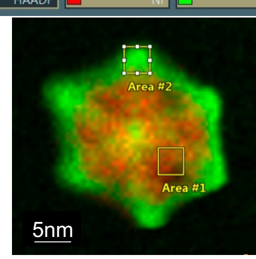
Can challenge our ability to understand them and characterize them

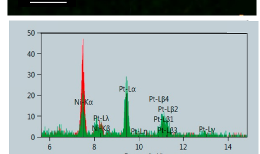


TEM - BF STEM - ADF

Metal Oxide Framework structure solved by imaging and spectroscopy







Key Challenges:

- ⧫ Quantification : Imaging Diffraction Spectroscopy vs Radiation Effects
- ⧫In situ: controlled environments for simultaneous imaging and spectroscopy during reactions
- ⧫ Dynamics: real-time observations

10

Environments: in-situ, growth processes and hard/soft materials

Materials behave differently in growth environments - understanding growth processes, especially self-assembly via organic or bio, presents a challenge

Nanowires grown in situ, characterized post-growth (F. Ross, IBM)

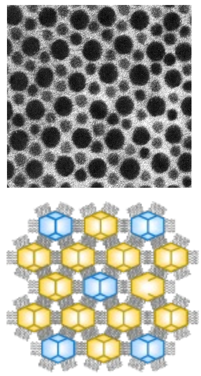
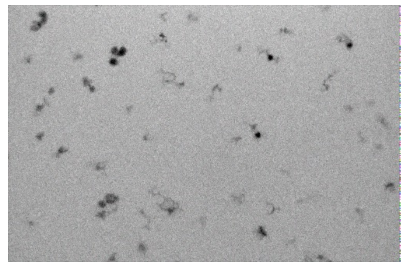


Image (top) and model (bottom) of self-assembled metamaterial (C. Kiely, Lehigh)



Key Challenges:

- In-situ observation of growth processes - at atomic resolution in growth environment
- In-situ high-spatial resolution chemical analysis
- Simultaneous imaging of hard/soft components
- Dynamics - Fast detection schemes, detectors, and sources

11

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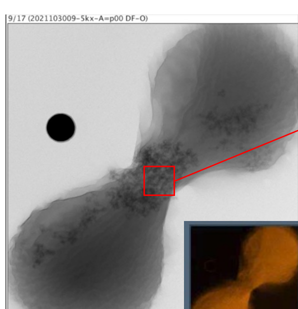
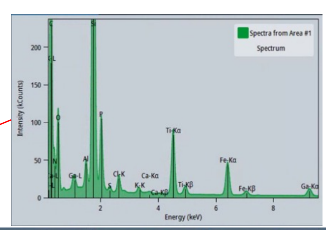
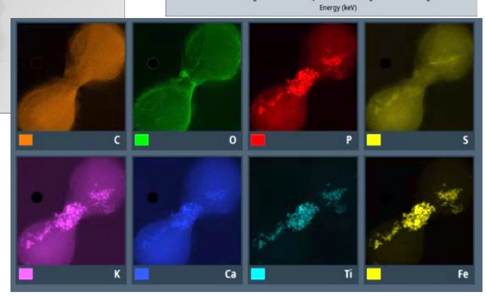
Biological Systems

Dramatically challenge our ability to understand them and characterize them

Nanotechnology, particularly with the usage of nanoparticles, has led to promising results in cancer diagnosis and treatment. Gaining insights into the interactions between nanomaterials and subcellular structures is crucial prior to their wide application *in-vivo*



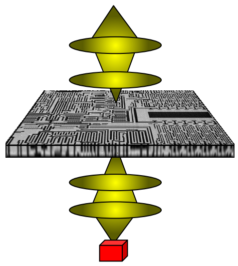




Challenge: the detection of biologically relevant transitional metals such as Fe, Zn, Cu, Ni and/or other heavy metals such as Hg and Se to further reveal their pathological significance and impacts in complex biological systems.

12

Types of Microscopes

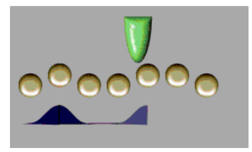


Microscopes with Lenses

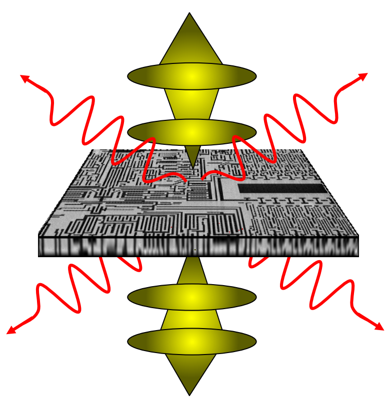
Optical Microscopes
Electron Microscopes
X-ray Microscopes

Microscopes without Lenses

Field Ion Microscopes
Point Projection Microscopes
Scanning Probe Microscopes



Reflection / Scanning
Deals Mainly with Near Surface Region

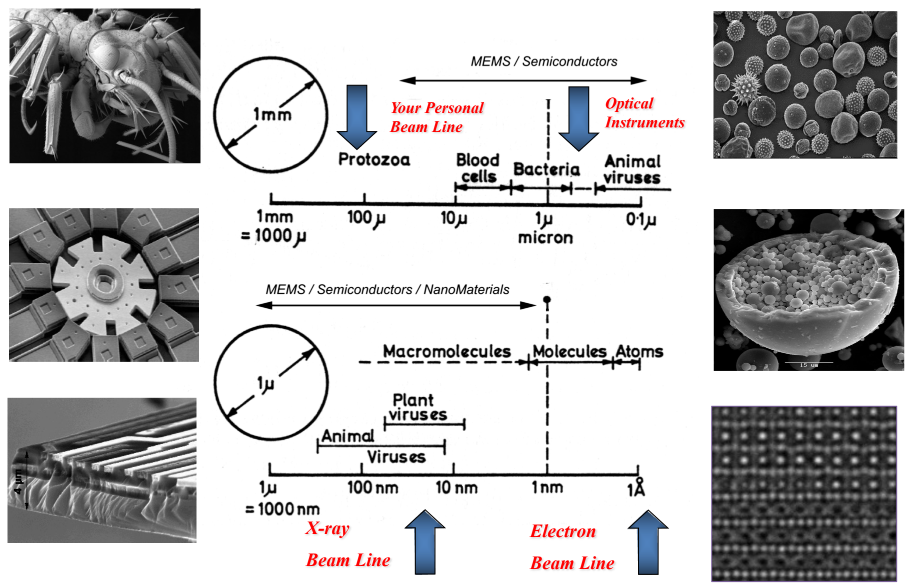


Specimen

Transmission
Deals Mainly with Internal Structure

Modern Instruments can depending upon the specimen operate in both modes

Various "Beam Lines" have resolution limits and capabilities

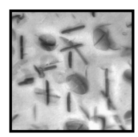


15

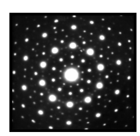
Electron Microscopy & Microanalysis

Experimental methodologies which employs (electron-optical) instrumentation to spatially characterize matter on scales which range from tenths of a millimeter to tenths of a nanometer. The principle modalities employed are:

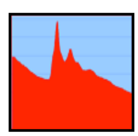
Imaging
- Scanning Electron Microscopy
- Transmission Electron Microscopy
- Scanning Transmission Electron Microscopy



Diffraction
- Electron Backscattered Difrraction
- Selected Area Electron Diffraction
- Convergent Beam Electron Diffraction
- Reflection High Energy Electron Diffraction

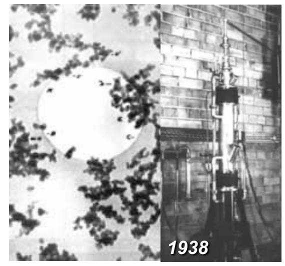


Spectroscopy
- X-ray Energy Dispersive
- Electron Energy Loss
- Auger Electron
- Cathodoluminescence

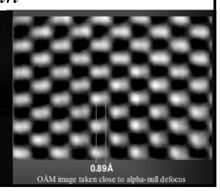


16

A Historical Time Line in Electron Optical Instrumentation



1897	JJ Thompson - Discovery of the Electron
1926	H. Bush Magnetic/Electric Fields as Lenses
1929	E. Ruska PhD Thesis Magnetic lenses
1931	Knoll and Ruska 1st EM built
1932	Davisson and Calbrick - Electrostatic Lenses
1934	Driest & Muller - EM surpases LM
1939	von Borries & Ruska - 1st Commerical EM ~ 10 nm resolution
1945	~ 1.0 nm resolution (Multiple Organizations)
1965	~ 0.2 nm resolution (Multiple Organizations)
1968	A. Crewe - U.of Chicago - Scanning Transmission Electron Microscope ~ 0.3 nm resolution probe - practical Field Emission Gun
1986	Ruska etal - Nobel Prize
1999	< 0.1 nm resolution achieved (OÅM)
2009	0.05 nm (US DoE TEAM project)
2020's	High Resolution/Sensitivity Spectroscopy



Comparison Beam Line Source Characteristics

Source	Brightness (particles/cm^2/sR/eV)	Elastic Mean Free Path (nm)	Absorption Pathlength (nm)	Attainable Probe Size (nm)
Neutrons	10^{14}	10^{7}	10^{8}	10^{6}
X-rays	10^{26}	10^{3}	10^{5}	~ 30
Electrons	10^{29}	10^{1}	10^{2}	< 0.1

Information about the specimen in ALL Instruments is derived as a result of Scattering Events

Elastic Scattering

Little to no Energy change but Momentum/Direction changes

Inelastic Scattering

Energy and/or Momentum/Direction changes

- *How can we use these scattering processes to characterize modern materials?*
- *What are the limitation and future prospects?*

Elastic/Pseudo-Elastic Scattering

Electron Microscopy (EM),

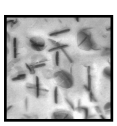
Transmission Electron Microscopy (TEM)

- *Amplitude/Diffraction/Phase Contrast Imaging*
- *Selected Area Electron Diffraction (SAED)*
- *Convergent Beam Electron Diffraction (CBED)*
- *High Resolution Electron Microscopy (HREM)*
- *Fluctuation Electron Microscopy (FEM)*
- *Lorentz Microscopy*
- *Electron Holography*

Scanning Transmission Electron Microscopy (STEM)

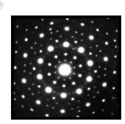
Scanning Electron Microscopy (SEM),

- *SEM-based Electron Channeling Patterns (ECP),*



Reflection High Energy Electron Diffraction (RHEED)

Low Energy Electron Diffraction (LEED)



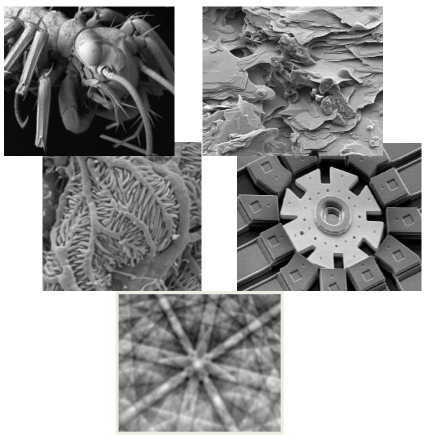
Traditional Electron Microscopy

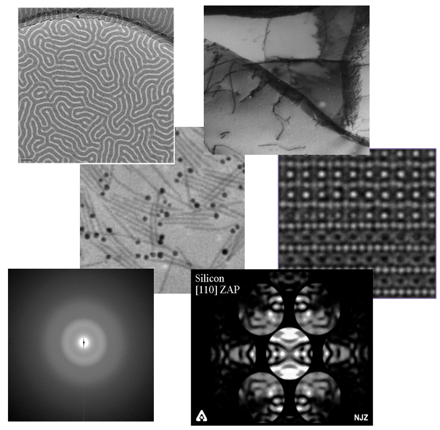
SEM

Scanning Electron Microscopy

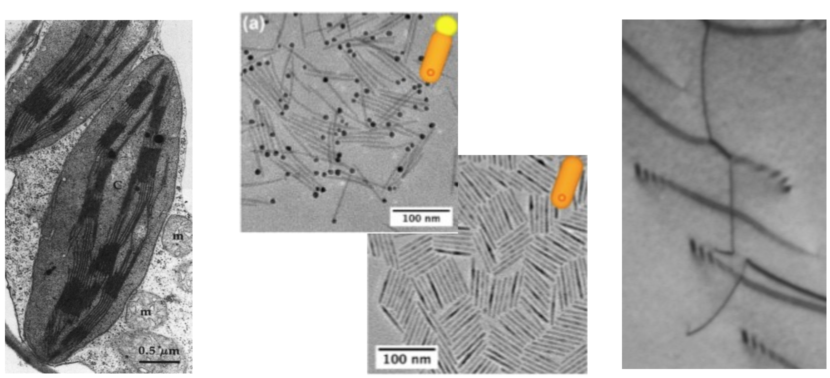
TEM / STEM / HREM

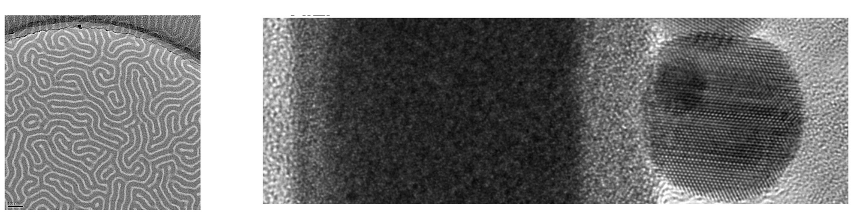
Transmission - Scanning Transmission - High Resolution Electron Microscopy



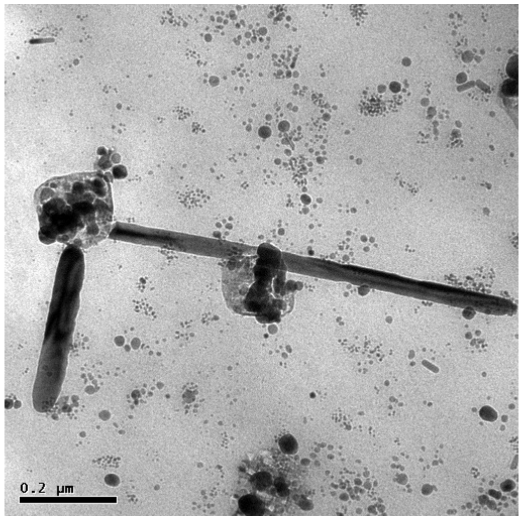


Microstructural Characterization using Imaging



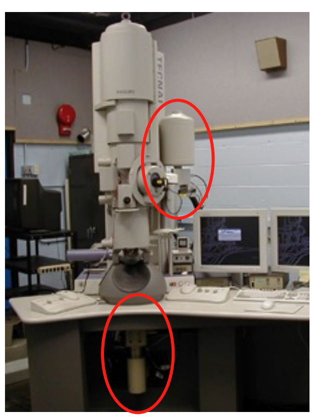


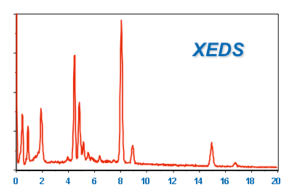
Nanoparticle Soup
What is it made up of ??

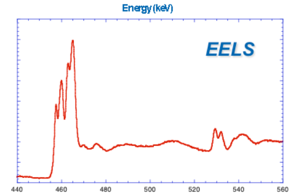


23

Analytical Electron Microscopy
Information from Inelastic Scattering Events







24

Inelastic Scattering Spectroscopies

	Type
Electron Energy Loss Spectroscopy (EELS), *EXtended Energy Loss Fine Structure (EXELFS),* *Energy Loss Near Edge Fine Structure (ELNES),* *Auger Electron Spectroscopy (AES),*	$e^- \rightarrow e^-$
X-ray Emission Spectroscopy (XES), *X-ray Energy Dispersive Spectroscopy (XEDS),* *Wavelength Dispersive Spectroscopy (WDS),* *Cathodoluminescence (CL)*	$e^- \rightarrow \lambda$
X-ray Photoelectron Spectroscopy (XPS), *X-ray Photoelectron Microscopy (XPM),* *Ultraviolet Photoelectron Spectroscopy (UPS),*	$\lambda \rightarrow e^-$
X-ray Absorption Spectroscopy (XAS), *EXtended X-ray Absorption Fine Structure (EXAFS),* *X-ray Absorption Near Edge Fine Structure (XANES)* *X-Ray Fluorescence (XRF).*	$\lambda \rightarrow \lambda$

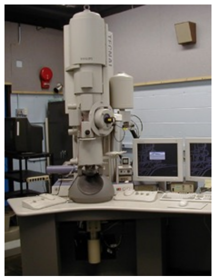
25

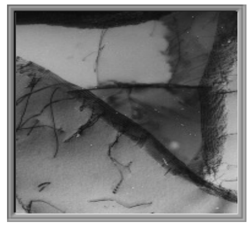
25

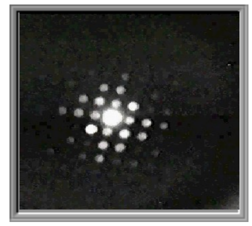
5E61 aluminum alloy: Al-6Mg-0.9Mn-0.07Zr-0.2Er (wt.%)

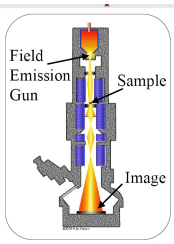
26

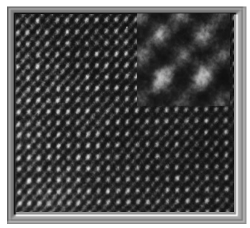
Basic Instrumentation & Optics

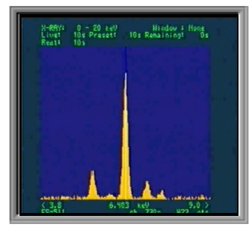












27

Some Fundamental Properties of Electrons

- Electron wavelength: based on de Broglie's ideas of wave-particle duality we know $\lambda = h/p$, where p is the electron momentum , h is Planck's constant, and λ is corresponding wavelength of the electron.
- In the TEM we impart momentum to the electron by accelerating it through a potential drop, V, giving it a kinetic energy *eV* This potential energy must equal the kinetic energy:

$$eV = \frac{m_0 v^2}{2}$$

$$p = mv = \sqrt{2m_0 eV}$$

For nonrelativistic electron wavelength

$$\lambda(\AA) = \frac{h}{p} = \frac{h}{\sqrt{2m_0 eV}} \approx \frac{12.27}{\sqrt{V(volts)}}$$

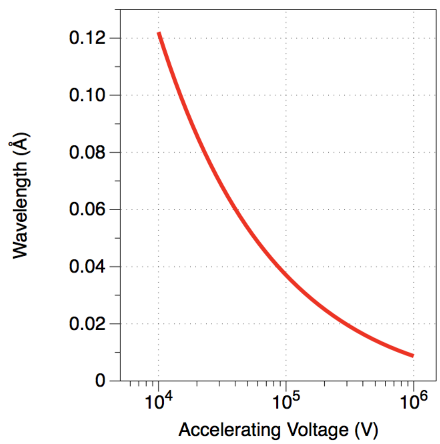
- However, for electron microscopy, relativistic effect cannot be ignored at 100-keV energies and above because the velocity of the electron become greater than half the speed of light. So the corrected (relativistic effect is considered) electron wavelength is:

$$\lambda = \frac{h}{p} = \frac{h}{\sqrt{2m_0 eV\left(1 + \frac{eV}{2m_0 c^2}\right)}} \approx \frac{12.27}{\sqrt{V(1 + 0.978x10^{-6}V)}}$$

28

Electron Properties as a Function of Accelerating Voltage

Eo(keV)	Beta (β= v/c)	To(keV)	Lambda(Å)
10	0.194985588	9.713933639	0.122006809
20	0.271865885	18.88424091	0.085858423
30	0.328376144	27.5507422	0.069770360
40	0.374059778	35.74967755	0.060138800
50	0.412686137	43.51408608	0.053539219
60	0.446224575	50.87413939	0.048648835
70	0.475865846	57.85743509	0.044834105
80	0.502398843	64.4892559	0.041748422
90	0.526380132	70.7927988	0.039184029
100	0.548220817	76.78937814	0.037007783
120	0.586670979	87.93855317	0.033487213
140	0.619564277	98.0760223	0.030736068
160	0.648106642	107.3205897	0.028507488
180	0.673148202	115.7741193	0.026653306
200	0.695314426	123.5243525	0.025078437
250	0.741018616	140.2969866	0.021986933
300	0.776525452	154.0641159	0.019689150
400	0.827868919	175.1109328	0.016442523
600	0.887947128	201.4486306	0.012572465
800	0.920908699	216.6822100	0.010274471
1000	0.941079211	226.2780694	0.008724368



Basic Components of All Microscopes That Use Lenses

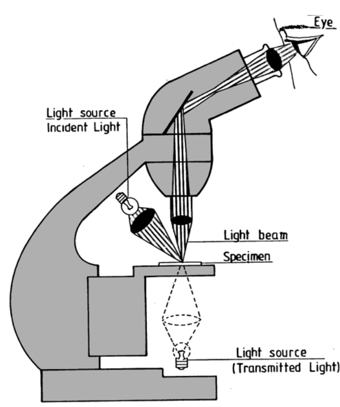
Illumination Source

Illumination Lens

Specimen

Magnifying Lens

Detector/Viewer



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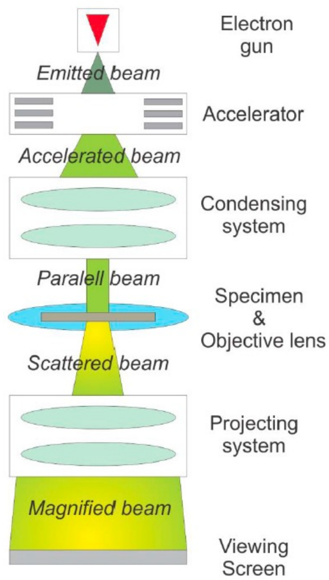
Illumination Source

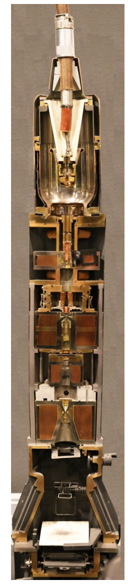
Illumination Lens

Specimen

Magnifying Lens

Detector/Viewer





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Chicago Museum of Science and Industry

31

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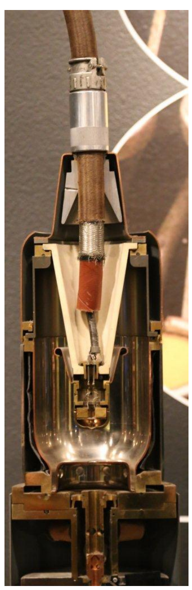
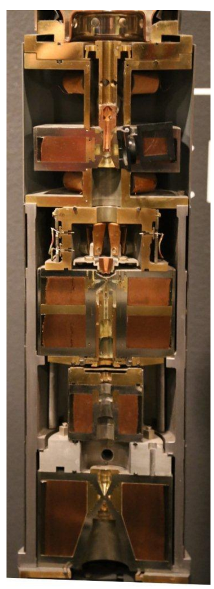
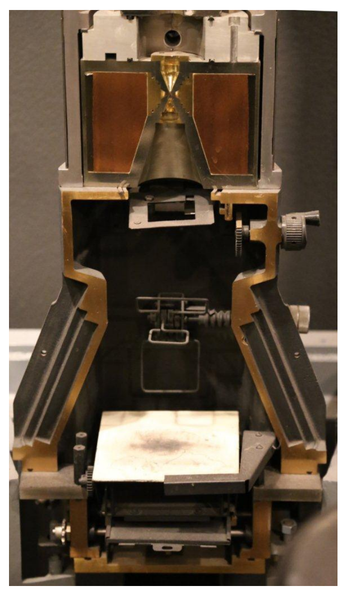


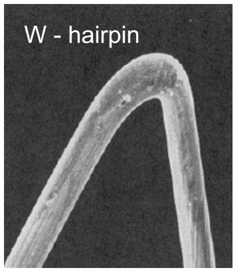


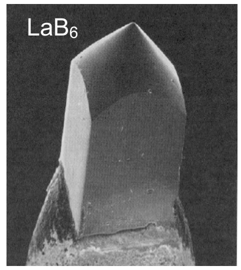


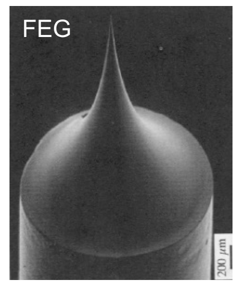
32

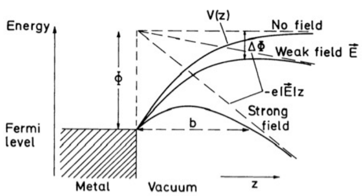
32

Sources for Electron Microscopy
Thermionic, Thermally Assisted,
And Field Emission







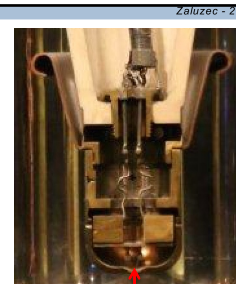


Conduction electrons must overcome the work function ϕ if they are to be emitted from the cathode into the vacuum.

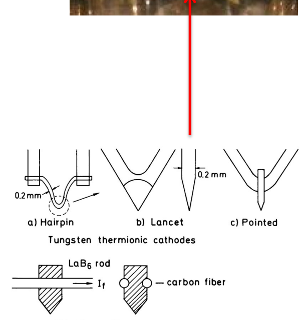
Thermionic sources

Richardson law gives the current density :

$$j_c = AT_C^2 \exp(-\phi / kT_C)$$



- k is Boltzmann's constant
- T_C is the cathode temperature
  - W has T_C of 2500-3000 K (T_M3650 K)
  - LaB_6 has a T_C of 1400-2000 K
- A and ϕ are material constants
- Note that $j_c \propto$ T.



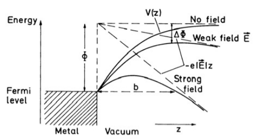
Heating usually produced by running a current through the material!

Field emission and Schottky sources

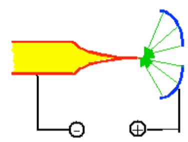
The width b of the potential barrier at the metal-vacuum boundary decreases with increasing electric field **E**.

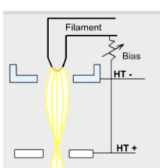
For |**E**|>10^7 V/cm the width b < 10 nm and electrons can penetrate the potential barrier by the wave mechanical tunneling effect.

The current density of field emission can be estimated from the Fowler-Nordheim formula:

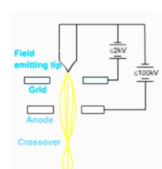


$$j = \frac{k_1 |\mathbf{E}|}{\phi} \exp\left(\frac{k_2 \phi^{3/2}}{|\mathbf{E}|} \right)$$





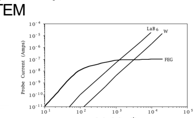
Sources for Electron Microscopy: Thermionic, Thermally Assisted, And Field Emission



Comparison of Electron Sources

Type		Brightness β/Vo A/cm²/sr/eV	Source Size (mm)	Energy Spread (eV)	Temporal Coherence (μm)	Shot Noise	Current Stability	Spatial Coherence	Vacuum (Torr)
Thermonic	Hairpin	1	50	2-3	0.4	Low	Good	Low	$<10^{-4}$
	Pointed	5	10				Fair	Moderate	$<10^{-5}$
LaB_6	Poly Crystal	10-30	10	~1		Low	Good	Moderate	$<10^{-6}$
	Single Crystal	20-50	5						
Field Emission	Thermal Assist	100-500	5	~.3	4	Fair	Moderate	High	$<10^{-8}$
	Cold	~1000	0.001	<.1			Fair		$<10^{-10}$

Total emission current is not usually the most important consideration
Brightness – defined as the current density per solid angle is more important
Spatial Coherence *for* parallel beam TEM this is important;
Spatial coherence is controlled by the effective source size..
A small source size leads to a narrow spread in electron phases leading to increased interpretability
Field emission guns (FEGS) produce the highest coherence – best for atomic resolution TEM
A **small source size** is also critical for scanning TEM (STEM) systems for high resolution work
Temporal Coherence is important in electron spectroscopy



Spatial Coherence = $\lambda / 2\alpha$ *Temporal Coherence* = $2 E \lambda / \Delta E$

Roles of the Lenses

Gun Lens

Helps form probe

Condenser Lens

Mainly controls:
Spot Size
hence total beam current

Objective Lens

Mainly controls
Focus, 1st Magnification

Diffraction/Intermediate Lens

Controls Mode

Projector Lens

Magnification

Most TEM/STEM have 7-9 Lenses

1 Gun Lens
2-3 Condensers
1 Objective
1-2 Intermediate
1-2 Projectors

Most instruments Have Electromagnetic Round Lenses

Exception:
Aberration Corrected Systems

Note the locations of the various Apertures.

Optimum aperture sizes are needed for various imaging functions.

37

37

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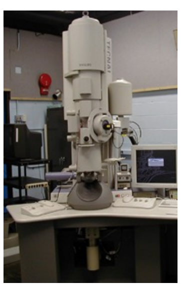
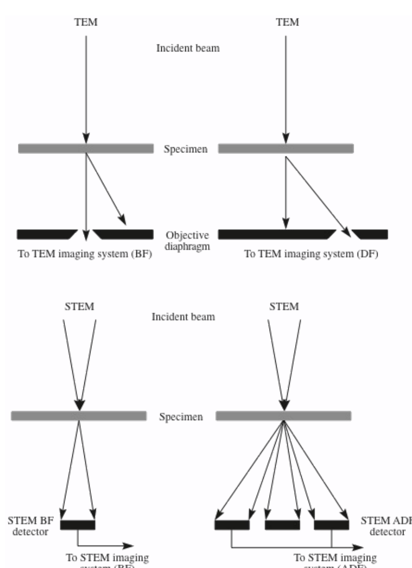
TEM vs STEM





38

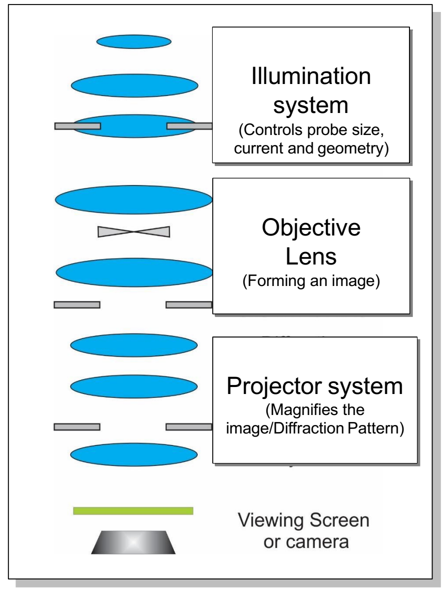
The TEM column

Can split into:
illumination system,
objective system
projector system;

i) The illumination controls the formation of the electron probe on the specimen

ii) The objective lens forms a parallel probe AND collects the electrons that have passed through the specimen and forms an image

iii) The projection system magnify or demagnify this image or diffraction pattern onto the viewing screen /camera

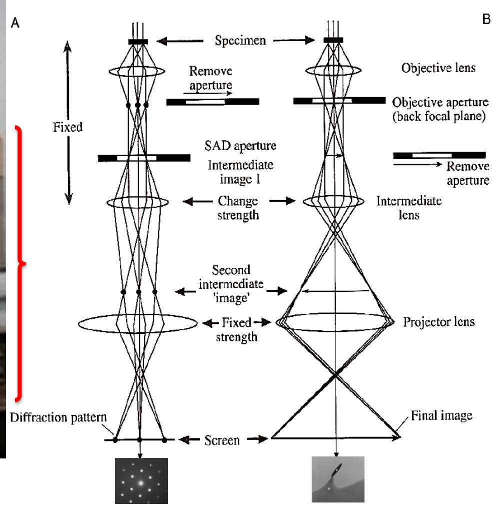


39

Projection/Imaging System

Projects/Magnifies the Diffraction Pattern (Back Focal Plane)

Projects/Magnifies the Image (Image Plane)



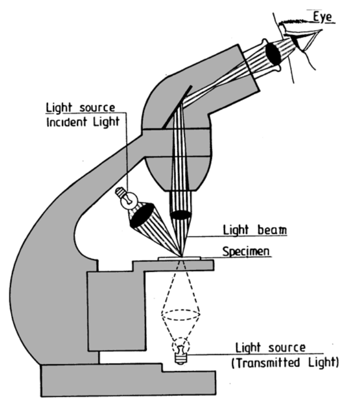
- The TEM in unique in that we can carry easily carry out **imaging** and **diffraction** in the *same instrument*

This is carried out by adjusting the strength of the Intermediate lenses in the **projection system**

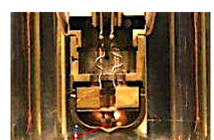
Other instruments can do this but it is much more difficult!

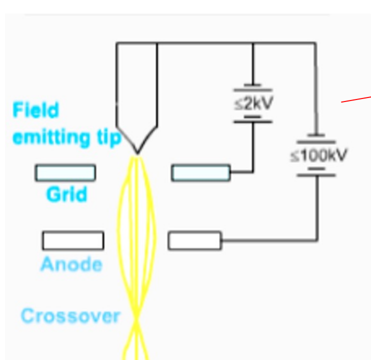
40

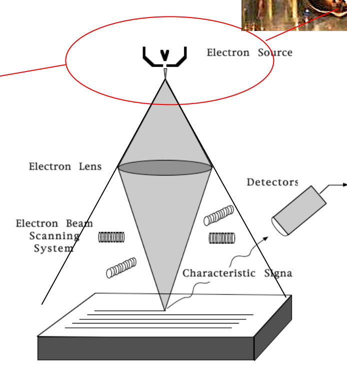
Why do we need a lens?



Why do we need a lens?







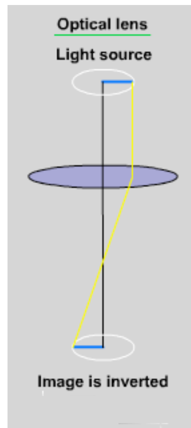
Because all electron sources generally produce a diverging beam of electrons. This beam must be "focussed" onto the specimen, to increase the intensity and thus to making the probe "smaller".

Lenses
and Magnification

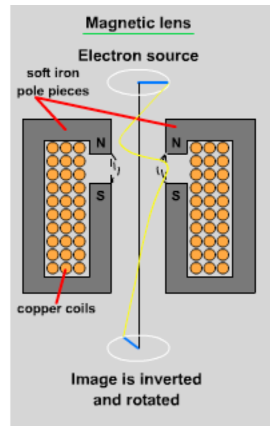
$$\frac{1}{f} = \frac{1}{d_o} + \frac{1}{d_i}$$

$$M = -\frac{d_i}{d_o}$$

Thin Lens
Formulae



Focus achieved
using Refraction



Focus achieved
using Lorentz Force

Electron Lenses

Electrons are charged particles and are influenced by Electromagnetic Fields.
Lenses in an TEM/STEM utilize either or combinations of Magnetic and
Electrostatic Fields to direct the beams as desired.

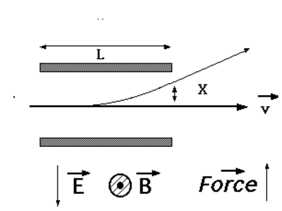
Force (F) and displacements (X) on electrons by different types of fields yields a deflection in their trajectory. In a uniform field region the electrons drift at a characteristic radius (R).

Electrostatic

$\vec{F}_E = q\,\vec{E}$ $\qquad X_E = \frac{1}{2}\frac{qEL^2}{m_o v^2}$ $\qquad R_E = \frac{m_o v^2}{qE}$

Electromagnetic

$\vec{F}_B = q\left[\vec{v} \times \vec{B}\right]$ $\qquad X_B = \frac{1}{2}\frac{qBL^2}{m_o v}$ $\qquad R_B = \frac{m_o v}{qB}$



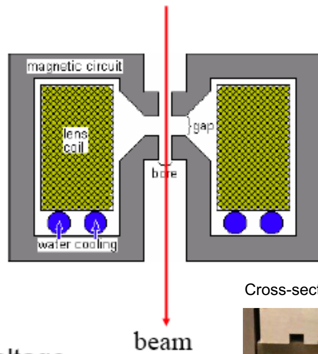
The Round Electromagnetic Lens

Lenses

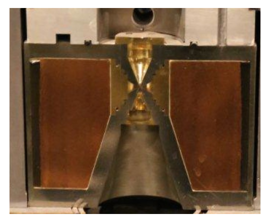
- the focal length is given by:

$$f = \frac{K \cdot U}{(N \cdot I)^2}$$

K : constant
U : accelerating voltage
N : windings
I : lens current



Cross-section of a Projector Lens



Types of Electron Lenses

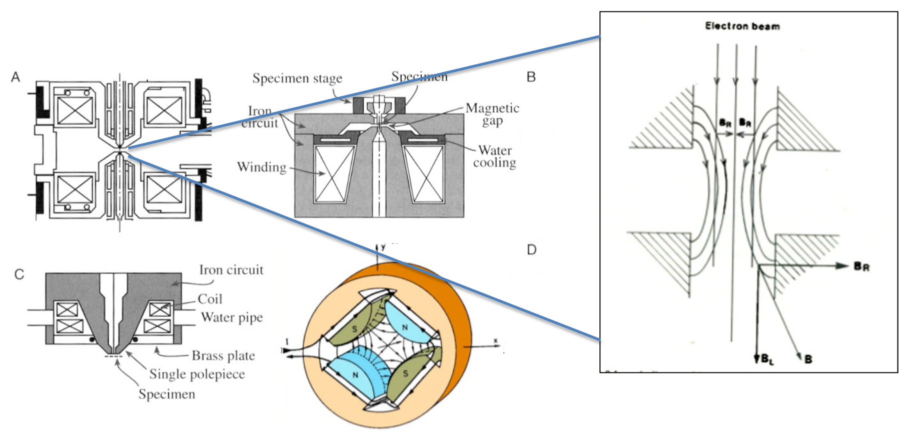
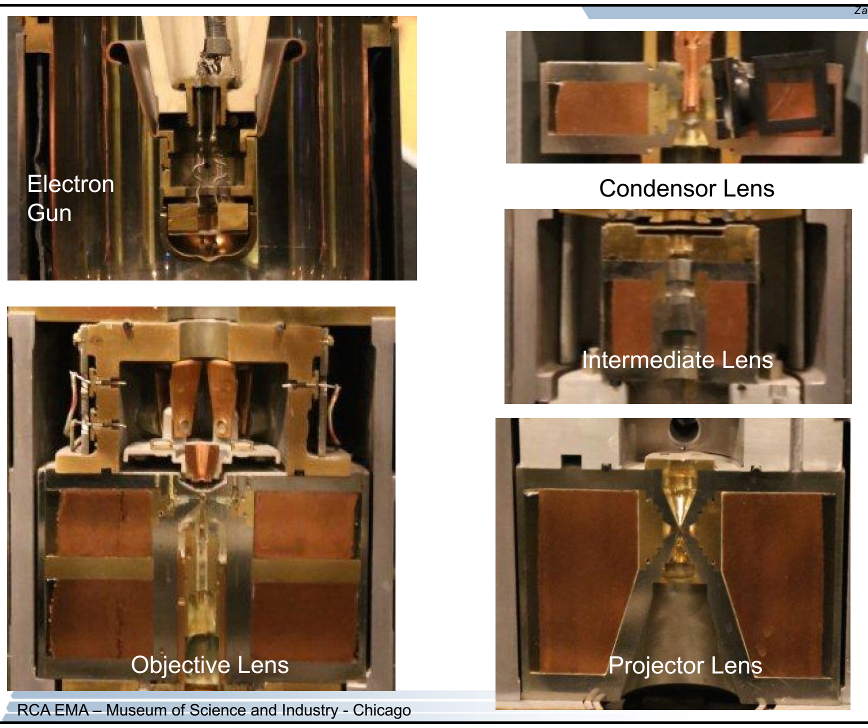


Figure 6.8. A selection of different lenses: (A) a split polepiece objective lens, (B) a top-entry immersion lens, (C) a snorkel lens, and (D) a quadrupole lens.

Condenser Lenses ~ Type A, Objective Lenses ~ Type A B or C, Stigmators Type D



RCA EMA – Museum of Science and Industry - Chicago

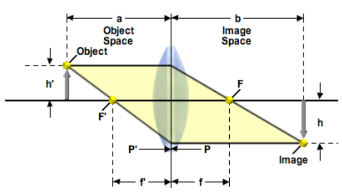
Magnification is achieved by

Stacking Lenses

$M = M_1 * M_2 * M_3$

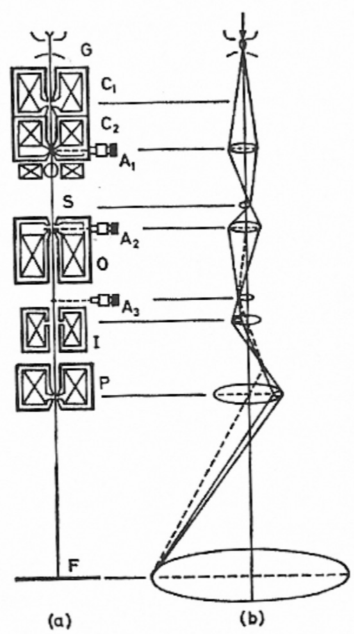
How Accurate is M ?

What are the limiting Factors?



$$\frac{1}{f} = \frac{1}{a} + \frac{1}{b}$$

$$M = \frac{h}{h'} = -\frac{b}{a}$$



At “low/medium magnifications” use gratings to calibrate magnification

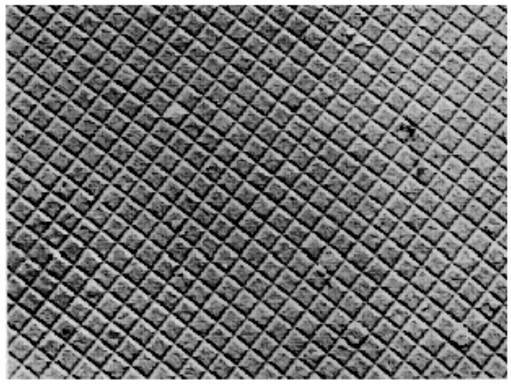
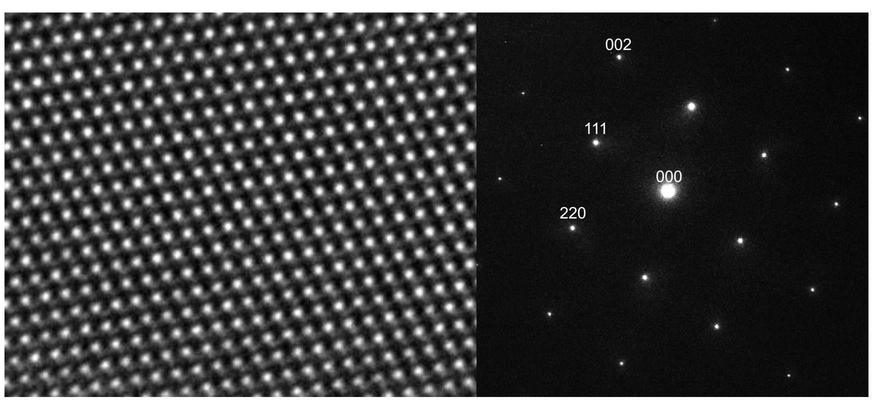


Fig. I.130. Replica of cross-ruleddiffraction grating with 2160 lines/mm used as a calibration specimen.(From Agar, p.162)

At “high magnifications” use lattice images to calibrate distances

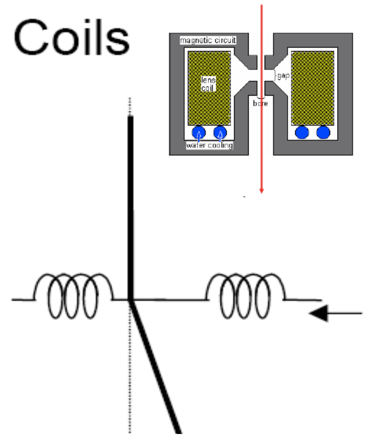


Silicon {110}
$d_{<111>} = 0.3134$
$d_{<220>} = 0.1919$
$d_{<002>} = 0.1635$

Alignment of the Beam : Mechanical vs Electronic

Deflection Coils

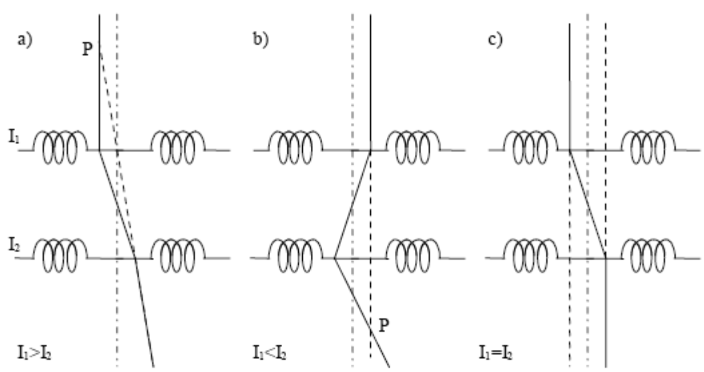
- Basic Principle
  - Gun coils
  - Beam coils
  - Image coils
  - Scanning coil
  - ....
  - ......



$\vec{F_B} = q\left[\vec{v} \times \vec{B}\right]$ $\quad X_B = \frac{1}{2}\frac{qBL^2}{m_o v}$

Modern Instrument use Double Deflection Coils

Deflection Coils

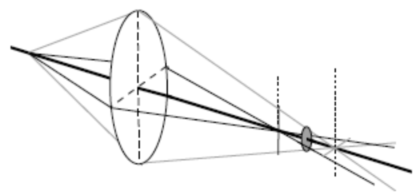


Tilting the Beam

Translating the Beam

Astigmatism

- Lens defect caused by magnetic field asymmetry

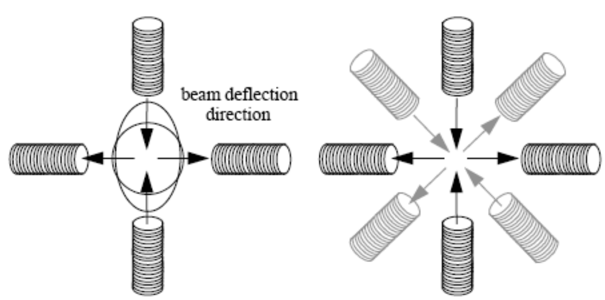


- can be corrected using stigmators!

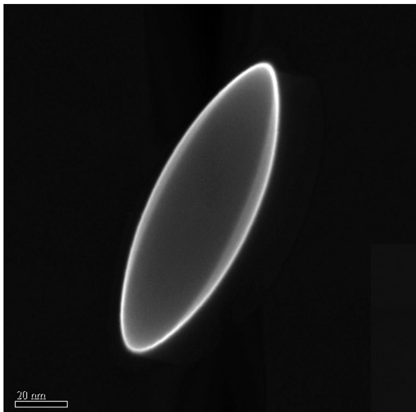
53

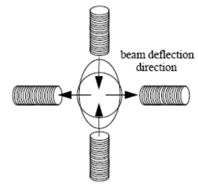
Stigmators

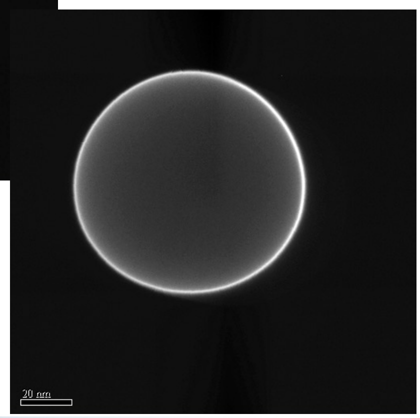
- Working Principle



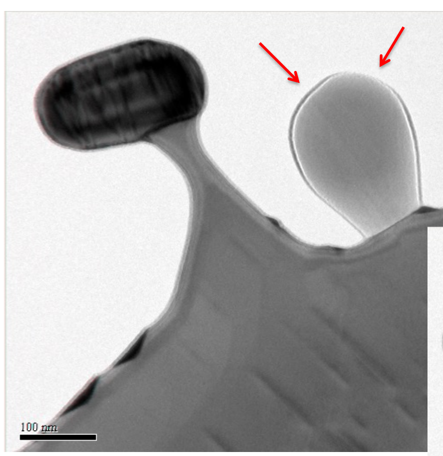
54

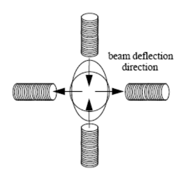






Probe Astigmatism





Slightly overfocussed image

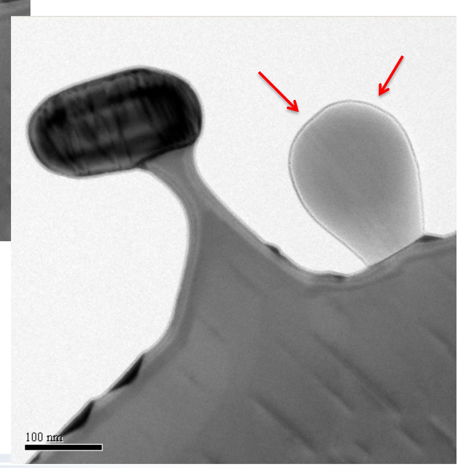
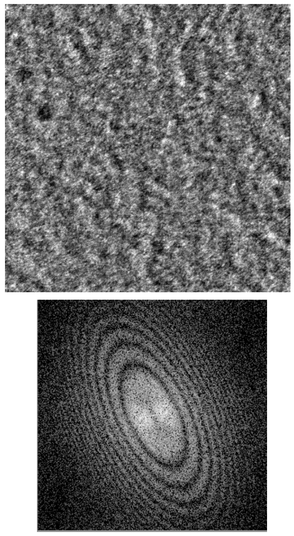
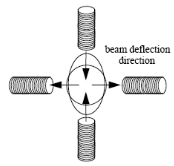
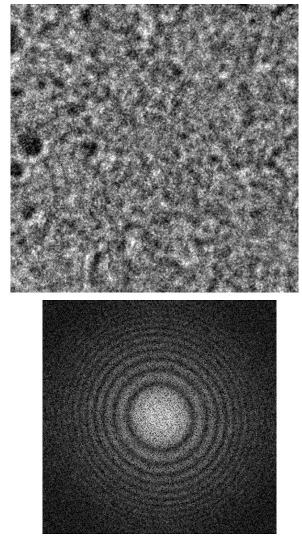


Image Astigmatism

Astigmatism in Amorphous Carbon Films
M ≥ 100 kx





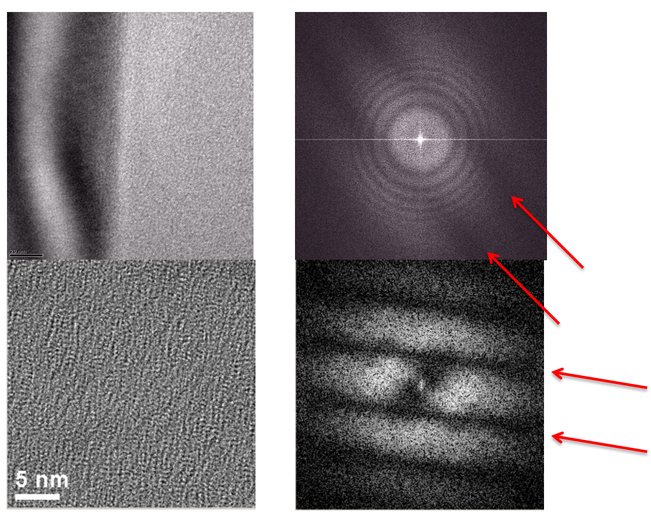


FFT of Amorphous Carbon for Astigmatism Correction

57

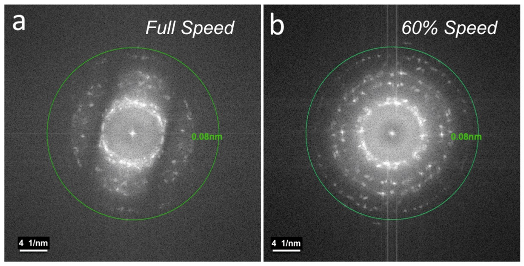
57

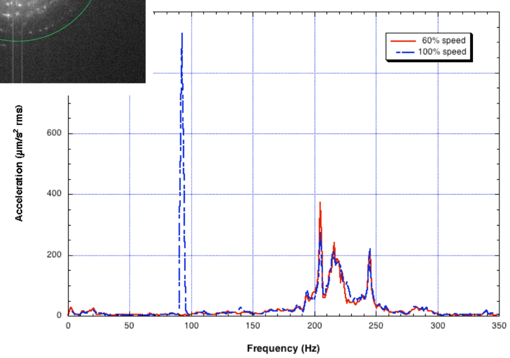
FFT of also “reveals” other problems…



Sample drifting during exposure

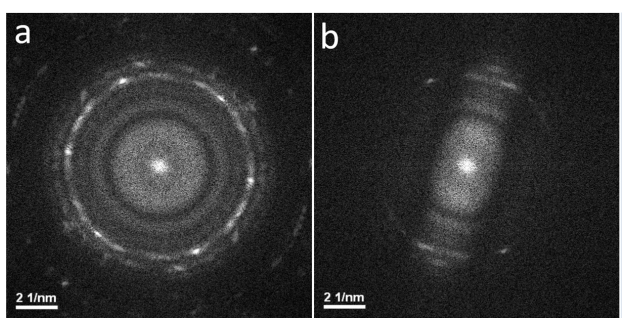
58





Bearing starting to
fail on Turbopump

Electrical interference
High stability Power Supplies
In an Aberrration Correction system



Cell Phone OFF

Cell Phone ON
No one is speaking
the phone is just ON!

Roles of the Lenses

Gun Lens

Helps form probe

Condenser Lens

Mainly controls:
Spot Size
hence total beam current

Objective Lens

Mainly controls
Focus, 1st Magnification

Diffraction/Intermediate Lens

Controls Mode

Projector Lens

Magnification

Most TEM/STEM have 7-9 Lenses

1 Gun Lens
2-3 Condensers
1 Objective
1-2 Intermediate
1-2 Projectors

Most instruments Have Electromagnetic Round Lenses

Exception:
Aberration Corrected Systems

Note the locations of the various Apertures.

Optimum aperture sizes are needed for various imaging functions.

Apertures vs Lenses

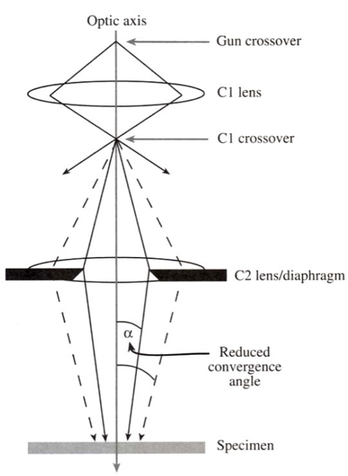


Figure 9.2. Effect of the C2 aperture on the beam coherence: a smaller aperture creates a more parallel, more coherent beam.

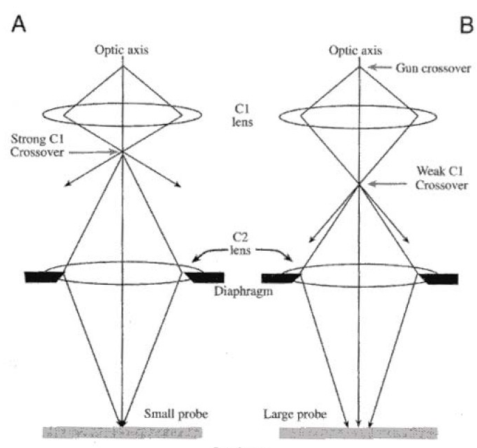
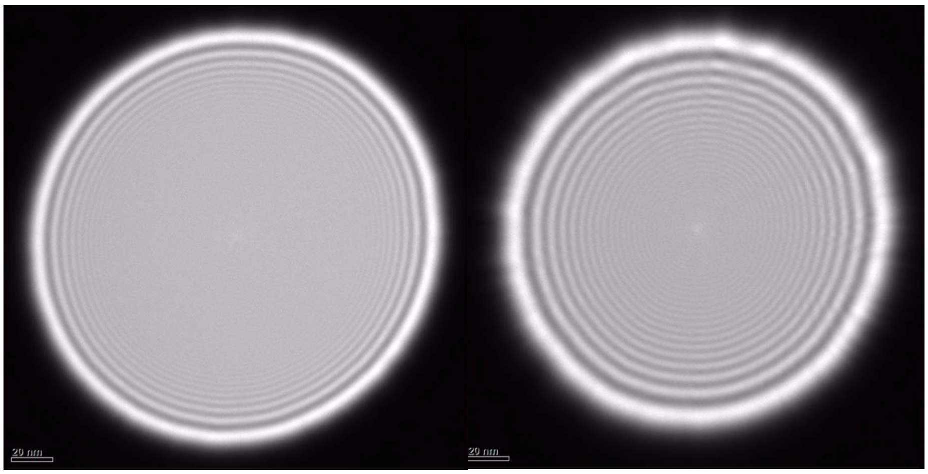


Figure 9.5. Effect of the C1 lens strength on probe size: a stronger C1 lens (A) results in greater demagnification by any subsequent lens (C2 or C3), giving a smaller electron beam at the specimen. A weaker lens (B) gives a broader probe.

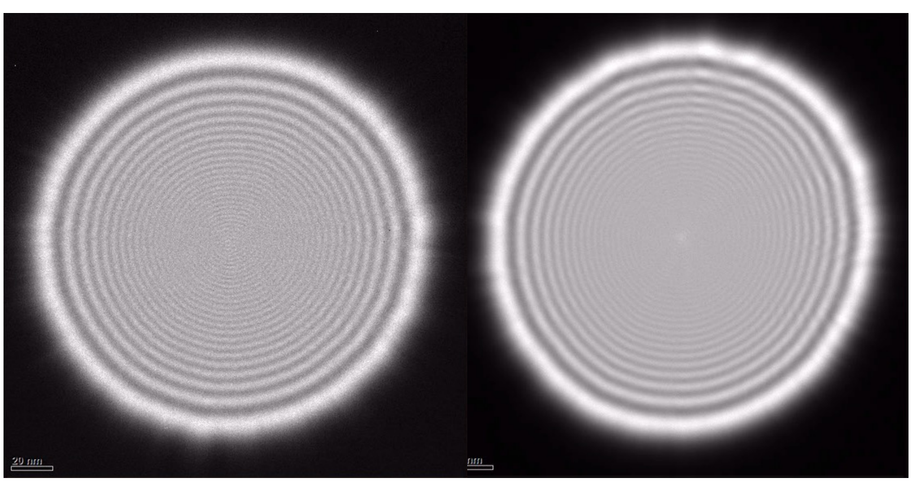
70 um SS 3 | 30 um SS 3



Coherence of the Source Improves your Images at High Resolution f (aperture)

63

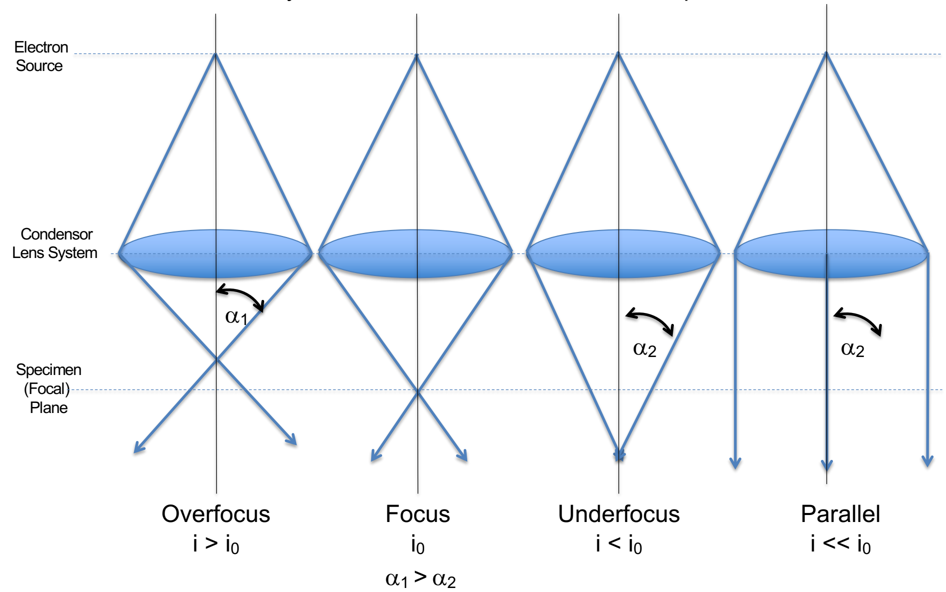
30 um SS 9 | 30 um SS 3



Coherence of the Source Improves your Images at High Resolution f (C1/Spot Size)

64

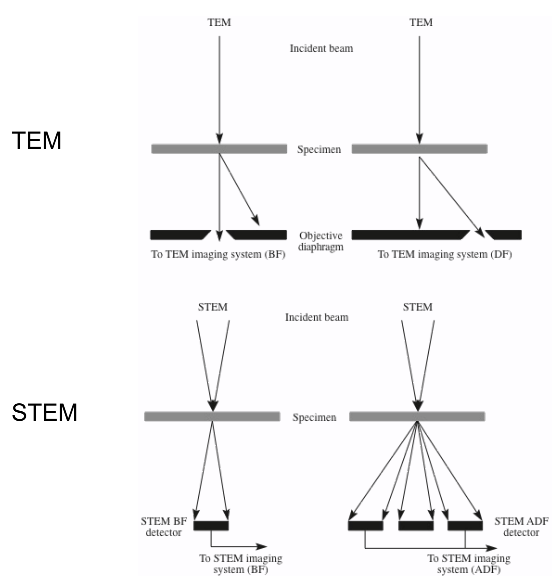
Illumination system controls the "Probe" Size, Shape, Coherence



Underfocus is more "parallel" than Overfocus

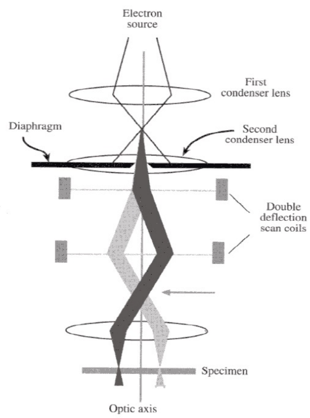
65

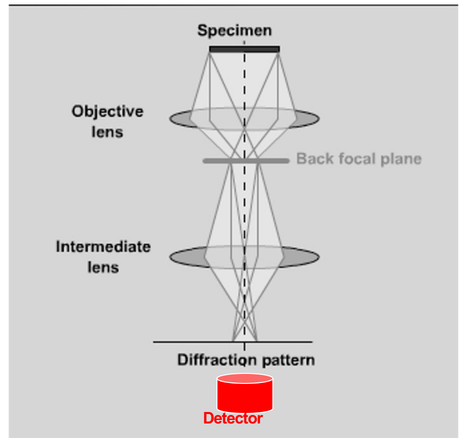
TEM vs STEM instruments



66

STEM Pre/Post Specimen Optics





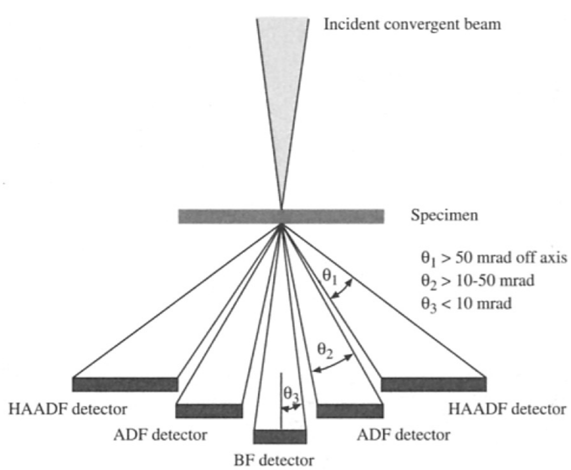
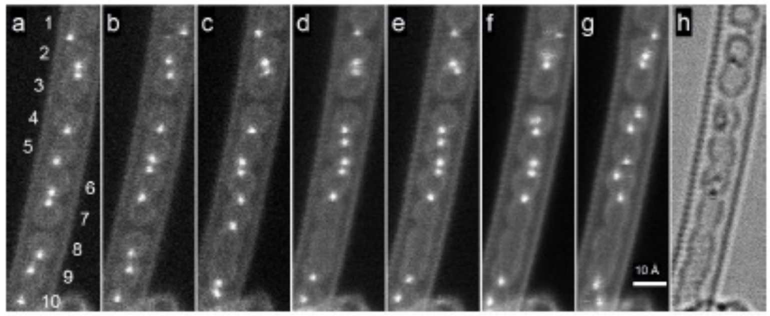
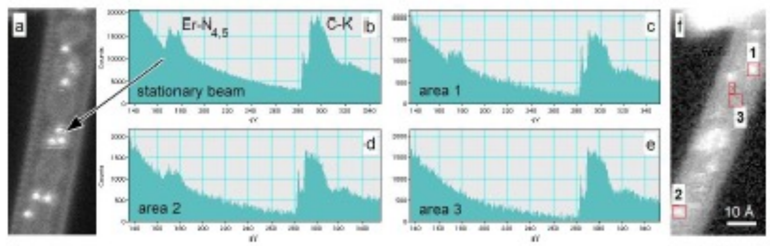


Figure 22.13. Schematic of the HAADF detector set-up for *Z*-contrast imaging in a STEM. The conventional ADF and BF detectors are also shown along with the range of electron scattering angles gathered by each detector.





Krivanek etal 2010

Roles of the Lenses

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Helps form probe

Condenser Lens

Mainly controls:
Spot Size
hence total beam current

Objective Lens

Mainly controls
Focus, 1st Magnification

Diffraction/Intermediate Lens

Controls Mode

Projector Lens

Magnification

Most TEM/STEM have 7-9 Lenses

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- 1-2 Intermediate
- 1-2 Projectors

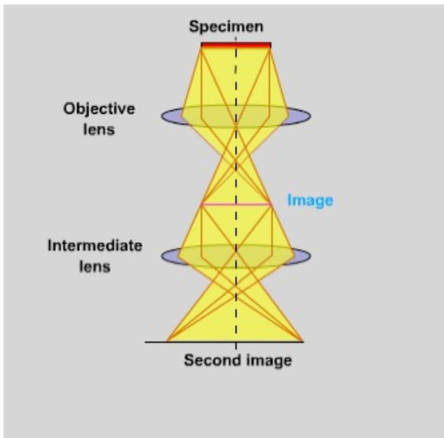
Most instruments Have Electromagnetic Round Lenses

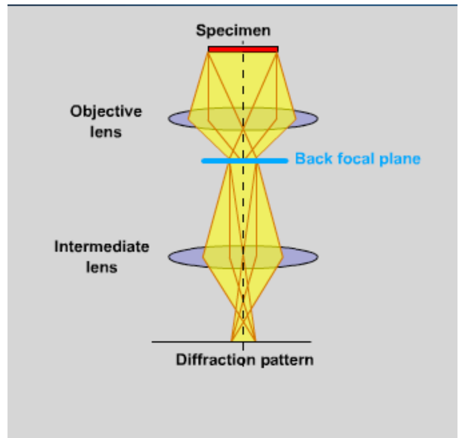
Exception:
Aberration Corrected Systems

Note the locations of the various Apertures.

Optimum aperture sizes are needed for various imaging functions.

TEM Mode Imaging vs Diffraction:
Post Specimen Intermediate Lenses determine the mode



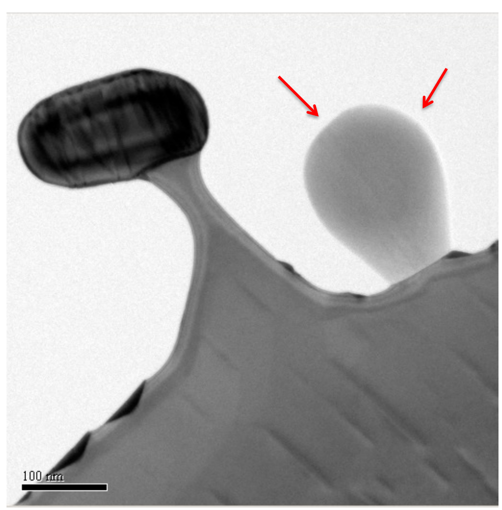


When is the Image in Focus?

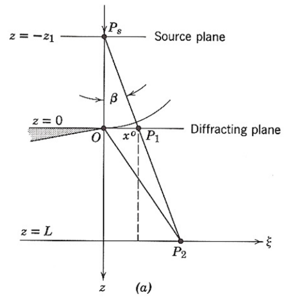
Look for Absence of Fresnel Fringes

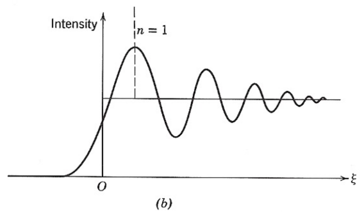
Or

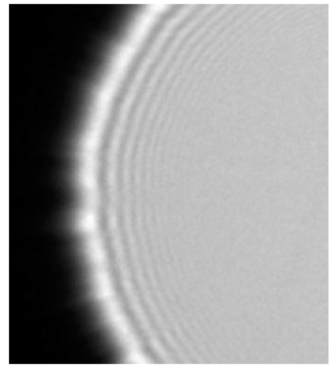
Minimum Contrast



Fresnel Fringes - Diffraction (Interference) from an Edge





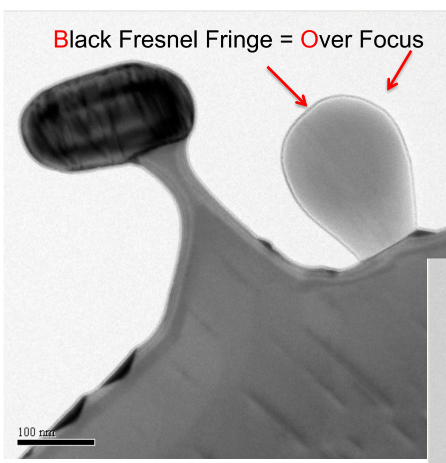


$$\nabla^2\Psi(r) + \frac{8\pi^2 me}{h^2}[E + V(r)]\Psi(r) = 0$$

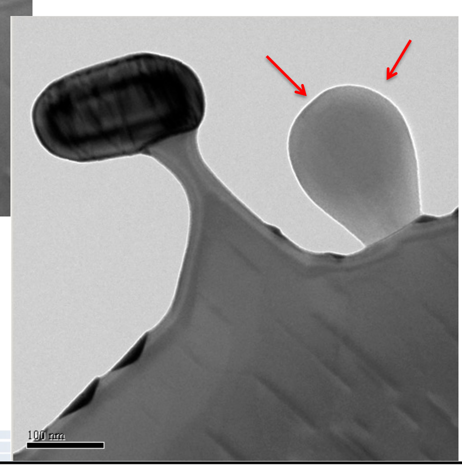
$$\Psi_1(r,z) = \frac{\exp(2\pi ikr)}{r} \qquad \Psi(r,z) = \sum \Psi_i(r,z)$$

$$I = \Psi(r,z)\Psi^*(r,z)$$

$$\xi_{\max} \simeq [\Delta f(2n-1)\lambda]^{1/2}$$



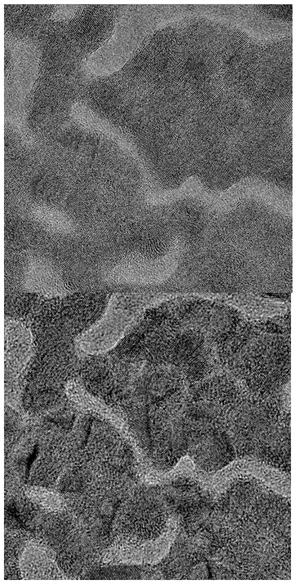
White Fresnel Fringe = Under Focus



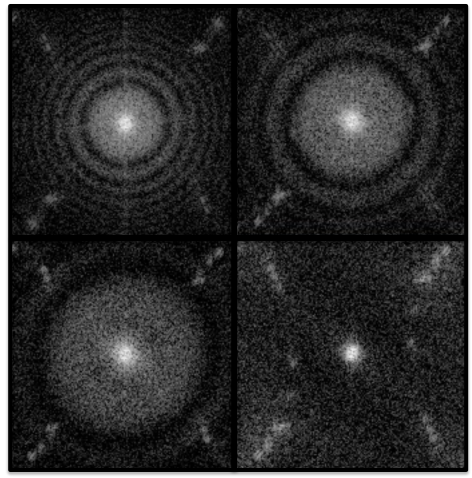
B O ⟷ W U

Minimum Contrast/ No Fringe = Focus

Which Image is in Focus



Scherzer Focus using FFT



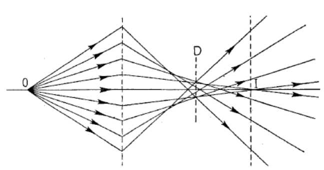
M $\geq$ 100 kX

75

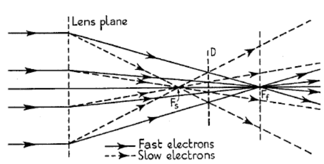
What limits our ability to perfectly focus?

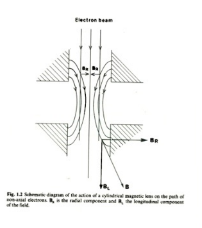
Aberrations

- Spherical



- Chromatic





$$r_{sph} = C_s \beta^3$$

$$r_{chr} = C_c \frac{\Delta E}{E} \beta$$

Lens Aberrations

Divide into 2 Regions

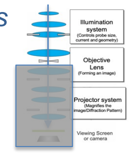
Pre-Specimen

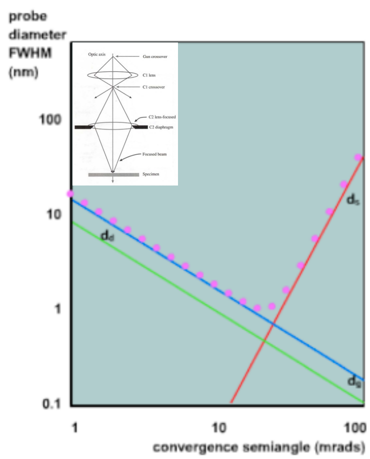
Post-Specimen

76

Aberrations and Probe (Pre-Specimen) Related Parameters

Aberrations Mainly Controlled by C_s and α





$$d_g = \frac{2}{\pi}\frac{1}{\alpha}\sqrt{\frac{i}{B}}$$

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

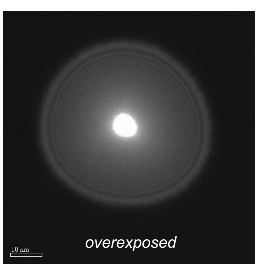
$$d_d = 1.22\frac{\lambda}{\alpha}$$

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

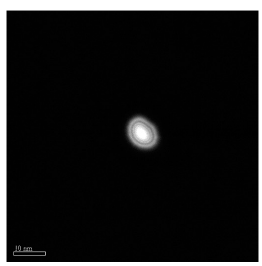
Gaussian/Geometric Limits
Spherical Aberration
Diffraction Limits

77

Can we see probe aberrations ? Yes if you look for them.
Condensor Apertures control α

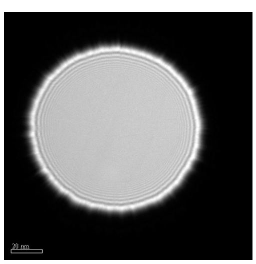


150 µm

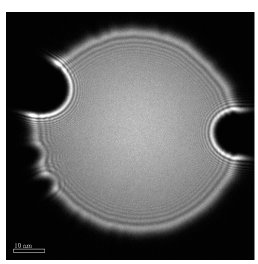


70 µm

30 µm



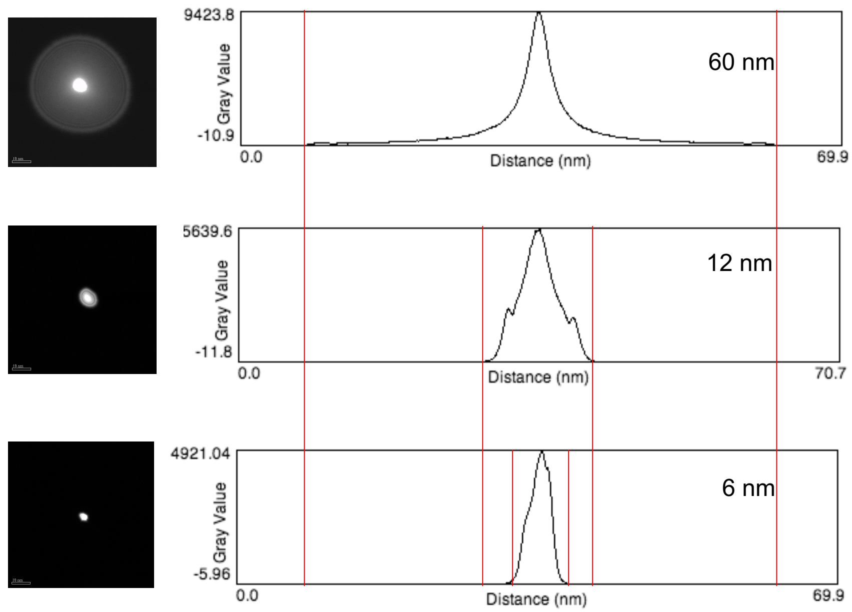
70 µm Clean



100 µm Dirty

100 µm Dirty

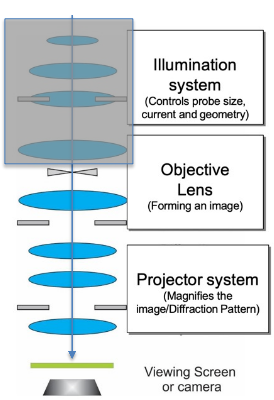
78

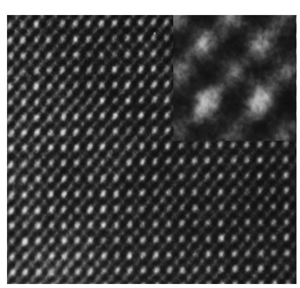


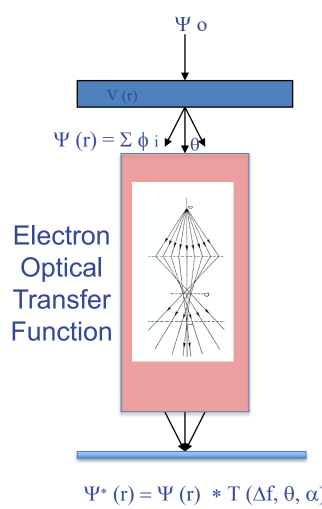
79

What Limits TEM Image (Post-Specimen) Resolution?

Lens Aberrations cause: Losses in Resolution , Contrast , & Image Delocalization
These effects are described by the Contrast Transfer Function $T\ (\Delta f, \theta, \alpha)$ of the "System"



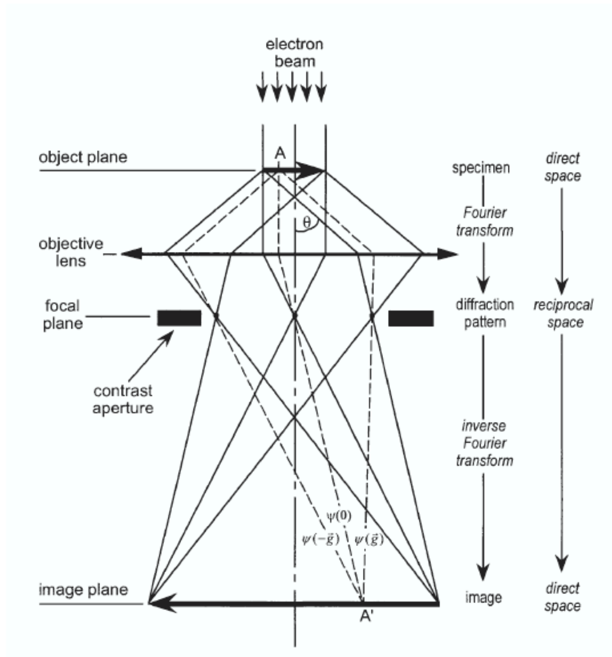




80

Post
Specimen
Aberrations
are incorporated
into the CTF

T (Δf, θ, α)

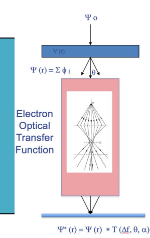


Contrast & Information Transfer: TEM

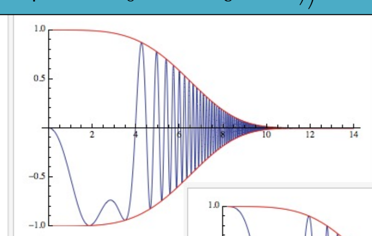
Contrast Transfer function: *T (Δf, θ, α)*

$$T_{phase}(q) = \sin\left(\frac{(2\pi)}{\lambda}\left(\frac{1}{2}C_1\lambda^2q^2 + \frac{1}{4}C_3\lambda^4q^4 + \frac{1}{6}C_5\lambda^6q^6 + \frac{1}{8}C_7\lambda^8q^8\right)\right) * E_t * E_s;$$

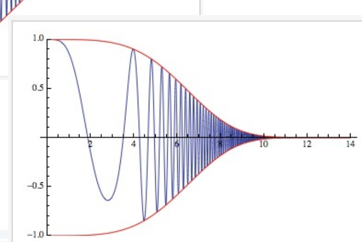
$$T_{amplitude}(q) = \cos\left(\frac{(2\pi)}{\lambda}\left(\frac{1}{2}C_1\lambda^2q^2 + \frac{1}{4}C_3\lambda^4q^4 + \frac{1}{6}C_5\lambda^6q^6 + \frac{1}{8}C_7\lambda^8q^8\right)\right) * E_t * E_s;$$



E_t: damping envelope of
temporal coherence
E_s: damping envelope of
spatial coherence



C_1: defocus = Δf
C_3: Spherical Aberration Coefficient (=C_s)
C_5, C_7: Higher Order Aberration Coefficients
Information limit:
temporal coherence → C_c, ΔE



Lens Systems & Microscope Resolution

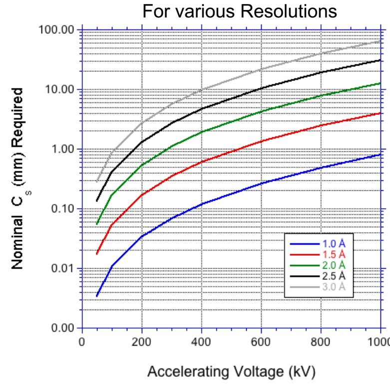
Generally TEM Resolution is limited by the Objective Lens:

- C_s of the Objective Lens
  - Scattering Angles required for HR Phase Contrast Image Formation
    $\alpha \sim \lambda/d \Rightarrow$ 200 kV & 1 Å $\Rightarrow \alpha \sim$ 25 mR
- C_c of the Objective Lens
  - Effective Energy Spread of the Source
- Electron Wavelength
- Other Lenses are less critical ~ f(α, M)

$$\delta = 0.66 \times C_s^{1/4} \lambda^{3/4}$$

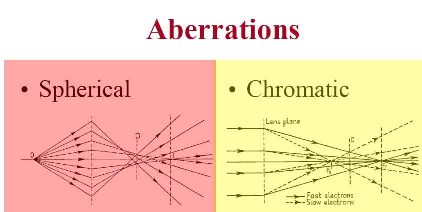
200 kV & 3 Å $\Rightarrow C_s \sim$ 2.7 mm
200 kV & 2 Å $\Rightarrow C_s \sim$ 0.5 mm
200 kV & 1 Å $\Rightarrow C_s \sim$ 30 μm

C_s vs kV
For various Resolutions

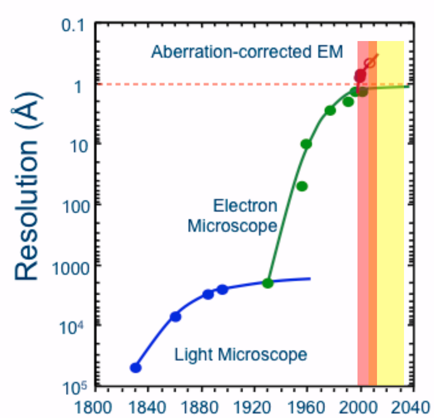


83

What are the limitations in Elastic/Inelastic Imaging (& Spectroscopy) ?



$r_{sph} = C_s \beta^3$ $\qquad r_{chr} = C_c \frac{\Delta E}{E} \beta$

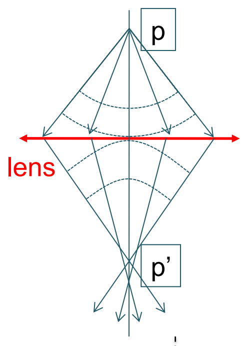


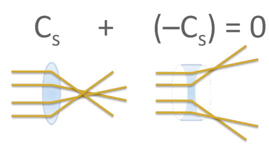
The source and solution to "resolution limitations" has been known for nearly 50 years

84

Spherical (C_s) Aberration Corrector

Spherical aberration

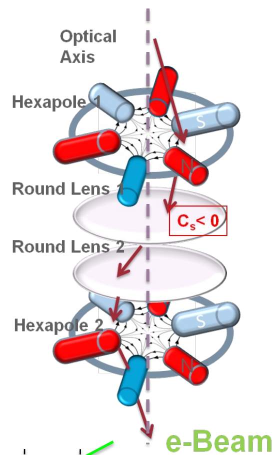


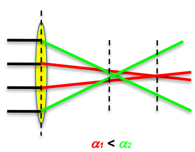


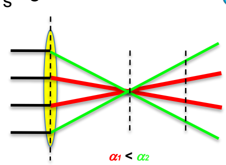
Electromagnetic lens

Round lens → $C_s>0$

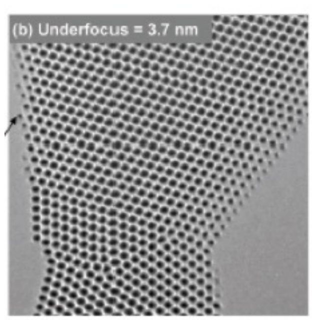
Quadrupole
Hexapole
Octupole
Dodecapole } $C_s<0$



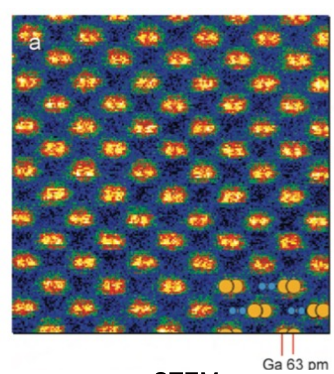




TEAM Project



TEM



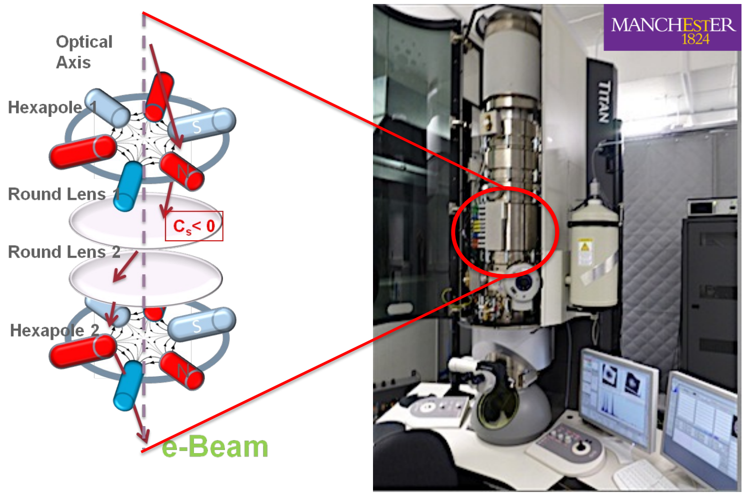
STEM

TEAM I
Commissioned Fall 2009
≤ 0.5 Å in TEM & STEM

LBNL, ANL, ORNL, UIUC

Microsc. Microanal. 14, 454–462, 2008

C_s Probe Corrector
(pre specimen)



University of Manchester
Probe Corrected TEM/STEM

Capabilities of C_s-Correction: TEM

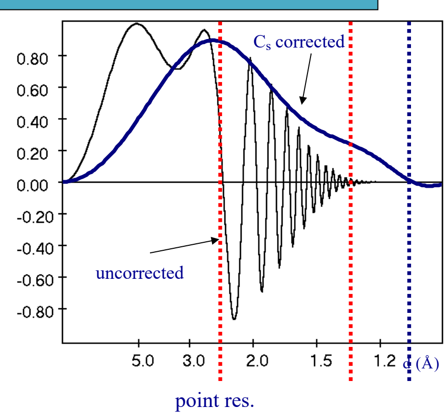
System Contrast Transfer function

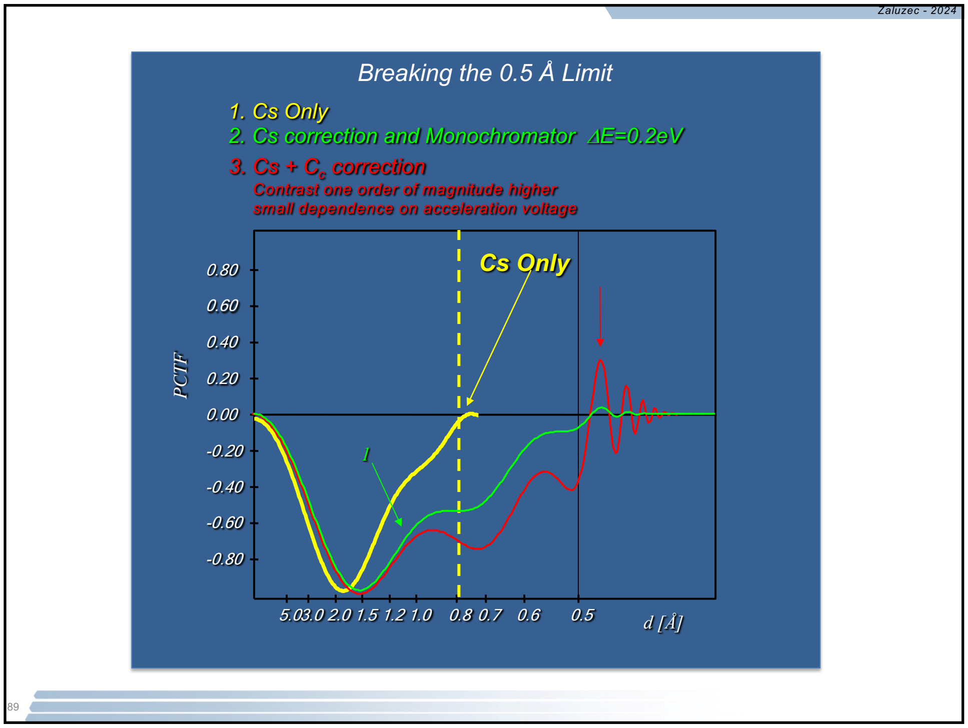
$$T(q) = \sin\left(\frac{(2\pi)}{\lambda}\left(\frac{1}{2}C_1\lambda^2 q^2 + \frac{1}{4}C_3\lambda^4 q^4 + \frac{1}{6}C_5\lambda^6 q^6 + \frac{1}{8}C_7\lambda^8 q^8\right)\right) * E_t * E_s;$$

E_t: damping envelope of temporal coherence
E_s: damping envelope of spatial coherence

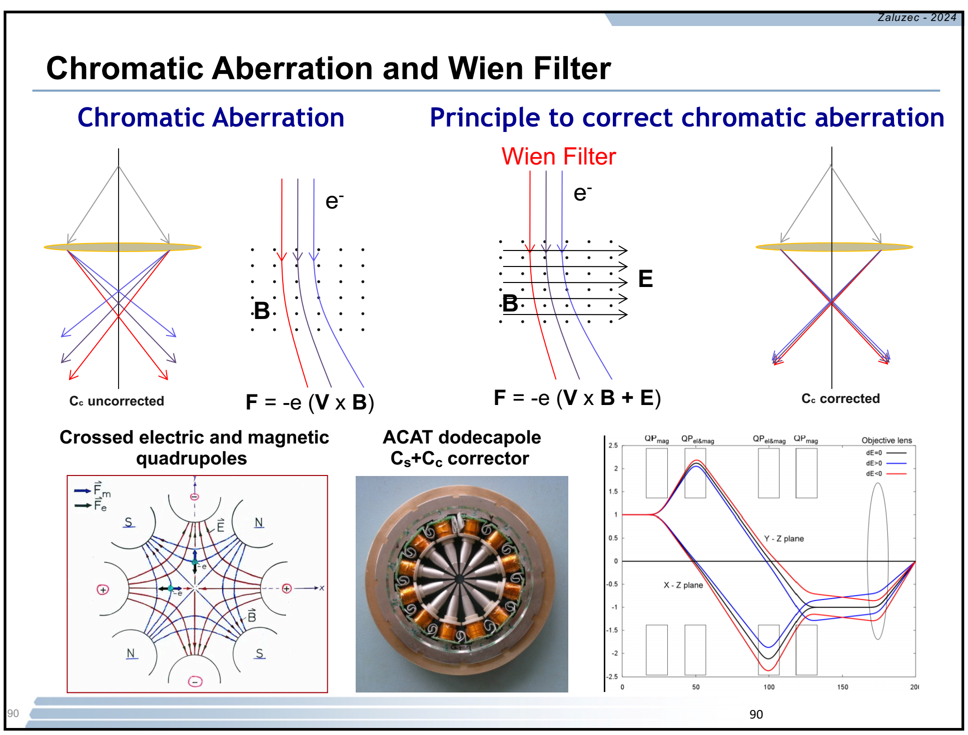
C_1: defocus - Δf
C_3: Spherical Aberration Coefficient C_s

Information limit:
temporal coherence → C_c, ΔE




Zaluzec - 2024
Breaking the 0.5 Å Limit
1. Cs Only
2. Cs correction and Monochromator ΔE=0.2eV
3. Cs + Cc correction
Contrast one order of magnitude higher
small dependence on acceleration voltage
Cs Only
PCTF
0.80
0.60
0.40
0.20
0.00
-0.20
-0.40
-0.60
-0.80
5.0 3.0 2.0 1.5 1.2 1.0 0.8 0.7 0.6 0.5
d [Å]
89

89


Zaluzec - 2024
Chromatic Aberration and Wien Filter
Chromatic Aberration
Principle to correct chromatic aberration
Wien Filter
e-
B
E
Cc uncorrected
F = -e (V x B)
F = -e (V x B + E)
Cc corrected
Crossed electric and magnetic quadrupoles
ACAT dodecapole Cs+Cc corrector
Y - Z plane
X - Z plane
Objective lens
90

90

ANL EMC Prototype C_s + C_c Corrector (DoE TEAM Project)

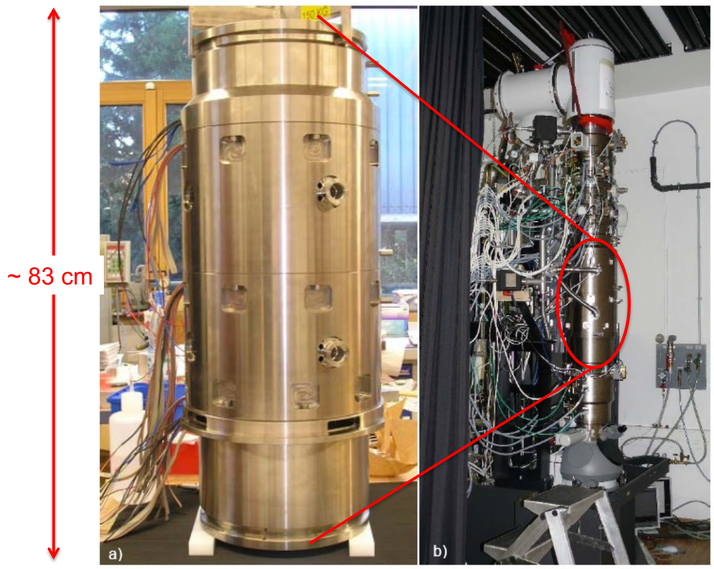
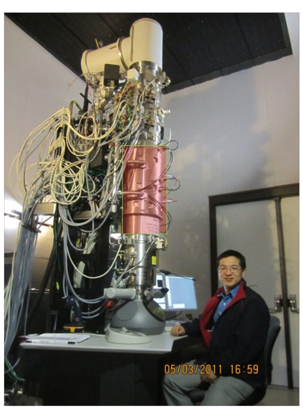
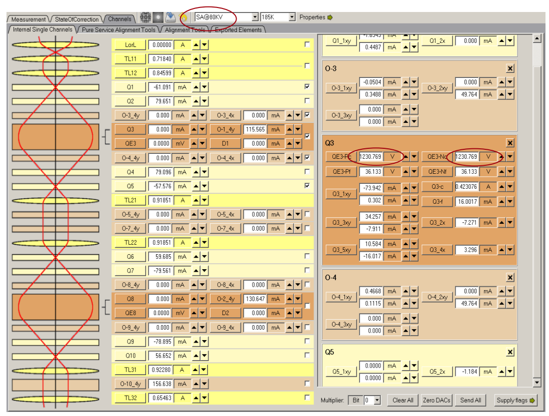


Fig. 1: *a) The Cc/Cs-corrector (C-COR) after construction showing the total length (83 cm) without filter boxes and vacuum lines and b) the Titan with the C-COR incorporated.*

Argonne Chromatic Aberration-corrected TEM (ACAT)





- FEI Titan 80-300 ST
- Pole-piece gap: 5.4 mm
- C_s/C_c image corrector

Power supply only for C_s/C_c corrector about 140 channels

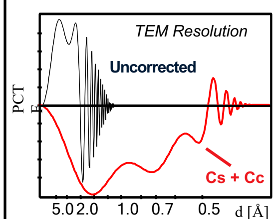
ANL TEAM Prototype

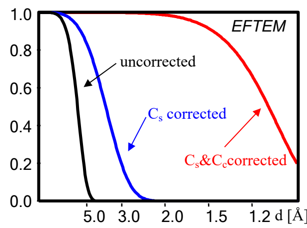
The Argonne Chromatic Aberration-corrected TEM

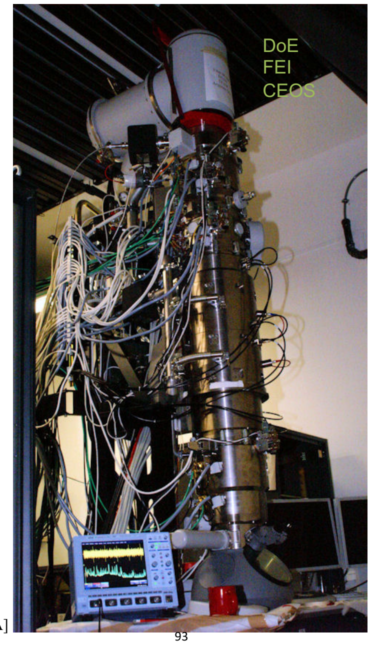
- developed as part of TEAM project, now on-line as a *user* facility instrument

Key capabilities:

- Improved resolution and signal-to-noise at *low voltage* operation
- EFTEM Imaging with a large field of view
- Can be operated in C_s and $C_s + C_c$ modes

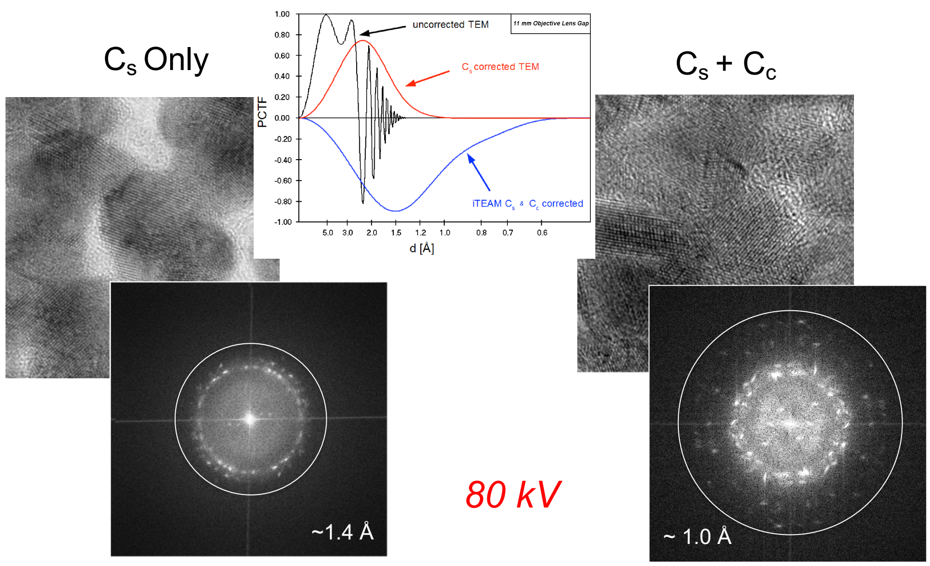






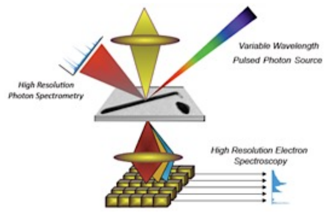
93

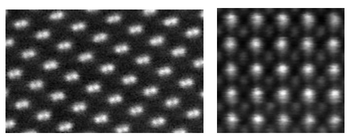
Low Voltage Benefits of Chromatic Aberration Correction to HREM/CTEM

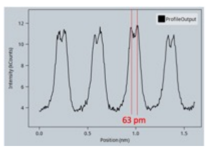


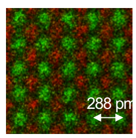
J.G. Wen etal – ANL

The PSC Analytical PicoProbe Electron Optical Beam Line









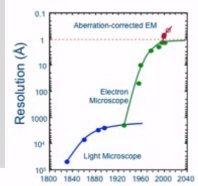
<211> GaN sub-atomic image
Ga-Ga = 63 pm

β – NiAl [001]
HyperSpectral Imaging

Key Features/Attributes at Commissioning

- E_o: 30-300 kV Electron Source
- High Brightness Coherent Field Emission Gun
- Probe Corrector (δx < 55 pm)
- Monochromator (δE ≤ 30 meV)
- XPAD 4.5 sR X-ray Spectrometer
- ZTwin Lens
- Next Generation Multi-Port Octagon
- HR/LowDrift Piezo Goniometer
- 4Kx4Kx16 bit CMOS Imaging Detector
- 128x128x30bit Diffraction Camera
- CTEM/STEM/iDPC /4DSTEM Modes
- Analytical LBHV DTilt Holder
- Ambient Tomography Holder
- Pytchography/Tomography Holder
- Ambient Liquid Cell Holder
- Cryo Tomography/Spectroscopy Holder
- Dynamic Imaging ~ 0-300 fps
- Multi-Modal Enabled
- Telepresence Enabled
- Electron Energy Filter / UltraMono - Spring 2023

Image Resolution
CTEM < 100 pm / STEM ~ 55 pm
Temporal Resolution
0- 300+ fps (CTEM) ≤ 1 usec/pixel (STEM)
Spectroscopy
- Worlds most sensitive AEM XEDS detector (>4sR)
- HR Energy Filter-Spectrometer - (< 30 meV)
Environmental
Commercial and Custom (Ambient) in-Situ Holders
Direct Electron Detectors
Cryo EM Imaging for Soft/Hard, Energy & Quantum Materials
Cryo EM Prep / Inert transfer
Future
In-situ Gaseous Media @ Temperature



95

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96

96