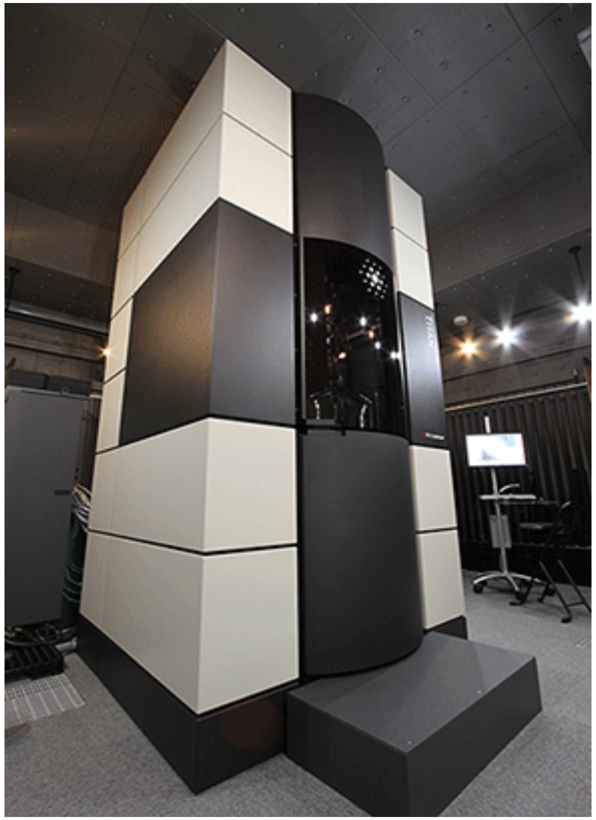
Introduction to Transmission Electron Microscopy: Electron Sources, Optics, Microscope Alignment, (Electron/Specimen Interactions (AS))

Jeremy Sloan

ARM200F



FEI TITAN³

Monochromated TEM (Zeiss)

SuperSTEM (Daresbury)

THE UNIVERSITY OF WARWICK

Introduction to Microscopy

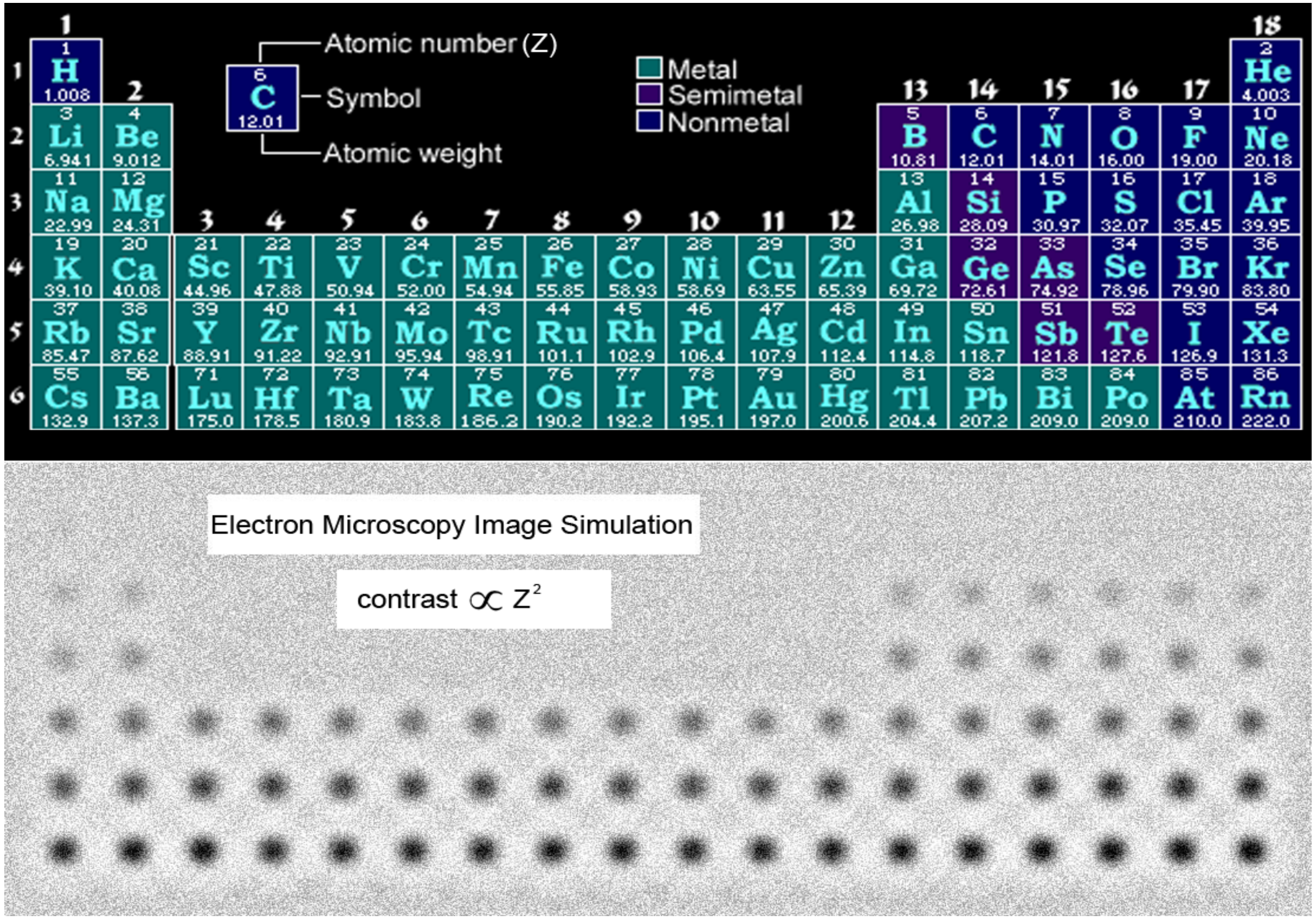
9th December 2014

Overview

HRTEM: High Resolution Transmission Electron Microscopy
or TEM:Transmission Electron Microscopy

- Overview
- How do Elements Image ?
- Design of a TEM – comparison with optical microscope
- What's the best electron source ?
- Formation of images and diffraction patterns
- Aligning the TEM
- Comparison of TEM and STEM (introduction)
- Aligning the STEM (introduction)
- => Electron Beam Interactions (AS)

Overview



A ‘periodic table’ of atom image contrast
in a HRTEM (contrast ∝ atomic No.)

What we see in a **Transmission Electron Microscope** is governed by a number of factors:

(I) The **resolving power** of the microscope

(II) The individual **scattering power** of the atoms comprising the specimen

(III) The **number of atoms** in the cross section of the sample (i.e. the specimen thickness)

(IV) The **crystallinity** of the sample.

(V) The **sensitivity** of the sample to the **electron beam**

So why TEM/HRTEM/STEM ?

•<u>H</u>igh <u>R</u>esolution <u>E</u>lectron <u>M</u>icroscopy can resolve object details smaller than 1 nm (10^{-9} m).

•It can be used to image the interior structure of the specimen (comparing to atomic resolution scanning tunneling microscopy, only at the surface).

•Comparing to atomic resolution provided by X-ray diffraction (average information), HREM can provide information on the <u>local</u> structure.

•Using the related technique of <u>electron diffraction</u>, we can also do <u>crystallography</u>

•Direct imaging of atom arrangements, in particular the structural defects, interface, dislocations.

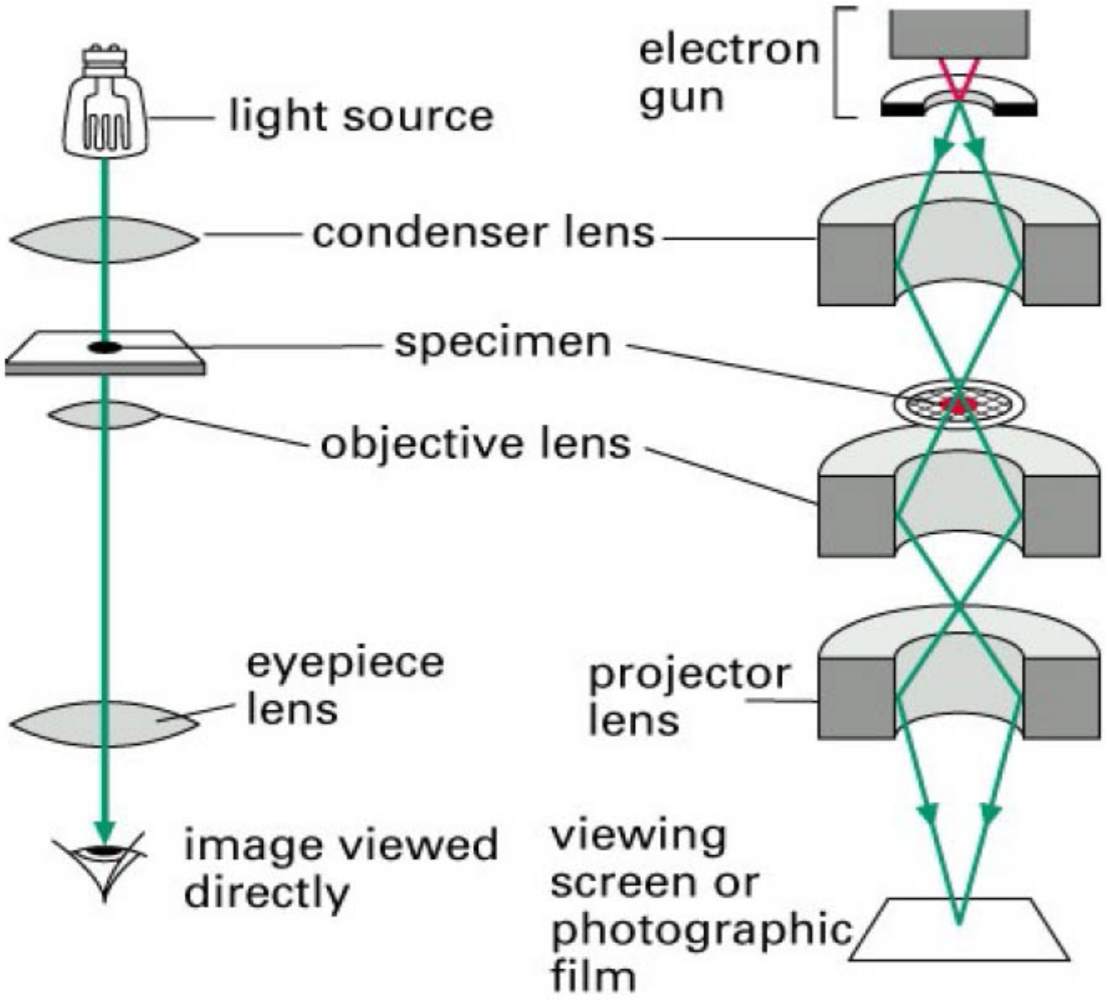
•Like SEM, HRTEM can be combined with spectroscopic techniques to give more chemical (and other) information about a specimen

TEM- comparison with optical microscope

Optical Microscope

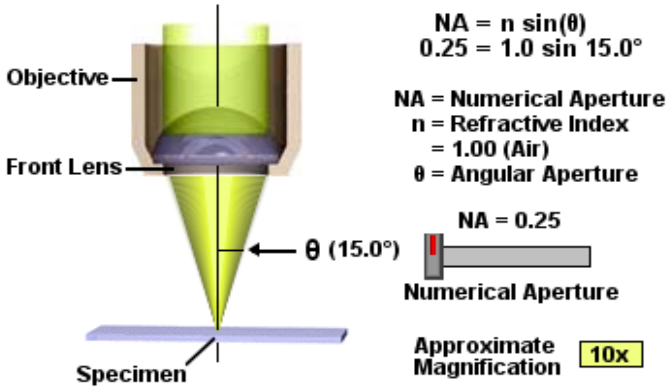
Transmission Electron Microscope

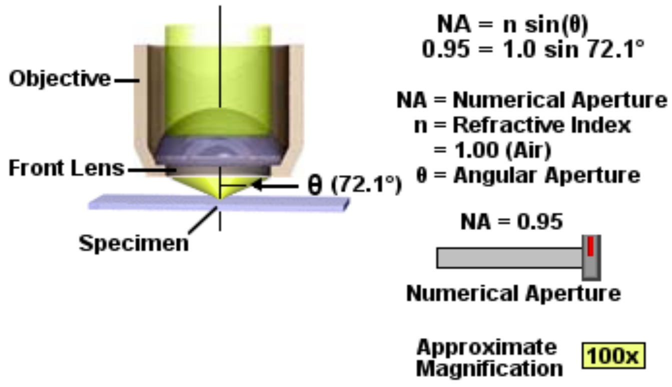
JEOL 3000F HRTEM, Oxford



Numerical aperture & Rayleigh Criterion for optical miroscope resolving power

The light-gathering ability of a microscope objective is quantitatively expressed in terms of the **numerical aperture**, which is a measure of the number of highly diffracted image-forming light rays captured by the objective. Higher values of numerical aperture allow increasingly oblique rays to enter the objective front lens, producing a more highly resolved image.





Resolution: smallest distance between two points on a specimen that can still be distinguished as two separate entities.

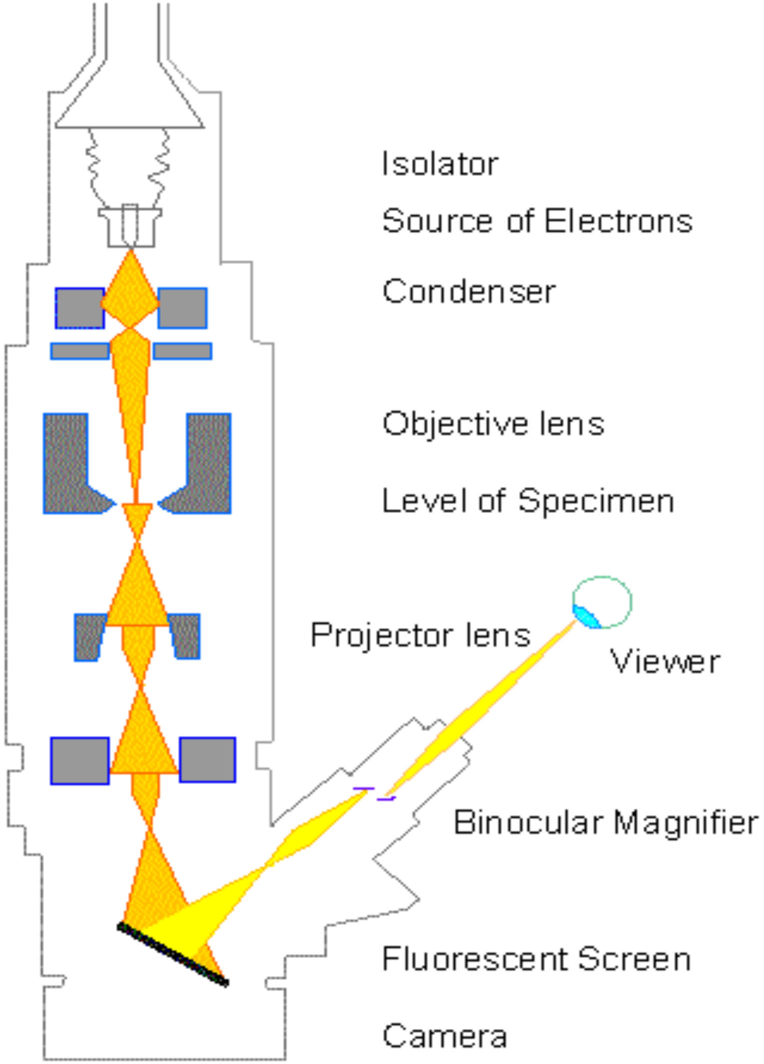
$R = 0.61\lambda/NA$ (1)

$R = 1.22\,\lambda/(NA(obj) + NA(cond))$ (2)

Raleigh criteria expressed for a **single lens (1)** and an optical microscope with **two lenses (2)**

Note resolving power $\propto \lambda$ $\therefore$ for a light microscope, (shortest wavelength = violet (380 nm) resolution is limited to ~<u>200nm (0.2 μm)</u>

TEM – electron wavelength



Ultimate resolution ~ <u>0.5(?)</u>-0.2 nm

- We know that electrons are small particles with wave-like characteristics, i.e.

  $\lambda = h/p$

  (h = Planck's constant; p = momentum (=mv); λ = electron wavelength)

  $\therefore$ $E=1/2\ m_0v^2$ or

  $\therefore$ $\lambda= h/(2m_0eV)^{0.5}$

  taking into account <u>relativistic effects</u>

  $\lambda= h/[2m_0eV(1+eV/(2m_0c^2))]^{0.5}$

  - c = speed of light; e and m_0 are the charge and rest mass of the electon respectively

Electron wavelengths

Equation for the electron wavelength (λ)

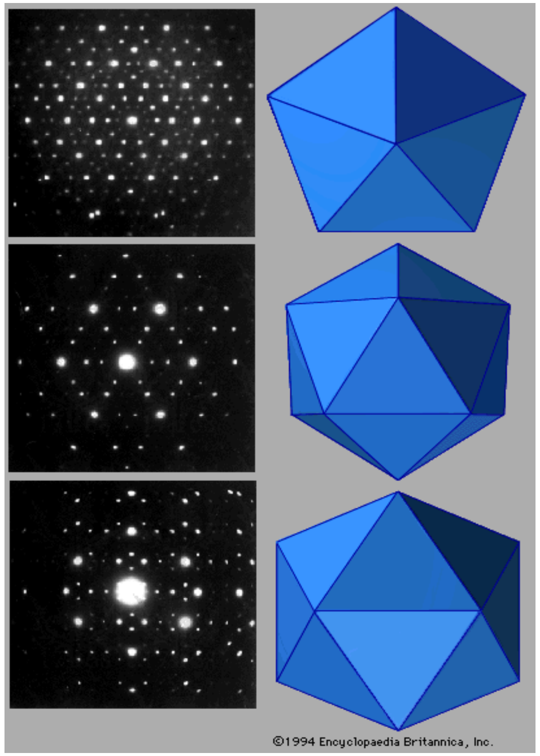
$$\lambda = h/[2m_0eV(1+eV/(2m_0c^2))]^{0.5}$$

m_0 = electron rest mass
V = voltage
e = electron charge
c = speed of light, h= Planck's constant
…apart from V, all are <u>constants</u> on RHS

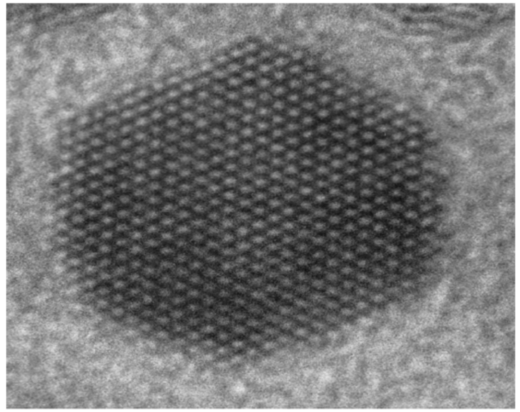
- V: accelerating voltage, non-relativistic
  - 100kV→ 0.0038nm,
  - 200kV→ 0.0035nm
  - 400kV→ 0.0023nm
- But electrons have speeds close to speed of light
- Relativistic wavelength
  - 200kV→ 0.0033nm, $2*10^8$m/s
  - 400kV→ 0.0016, $2.5*10^8$m/s
  - Increase in mass m/m_0= 1.78

  (Ana will recap !)

Electron diffraction



HRTEM

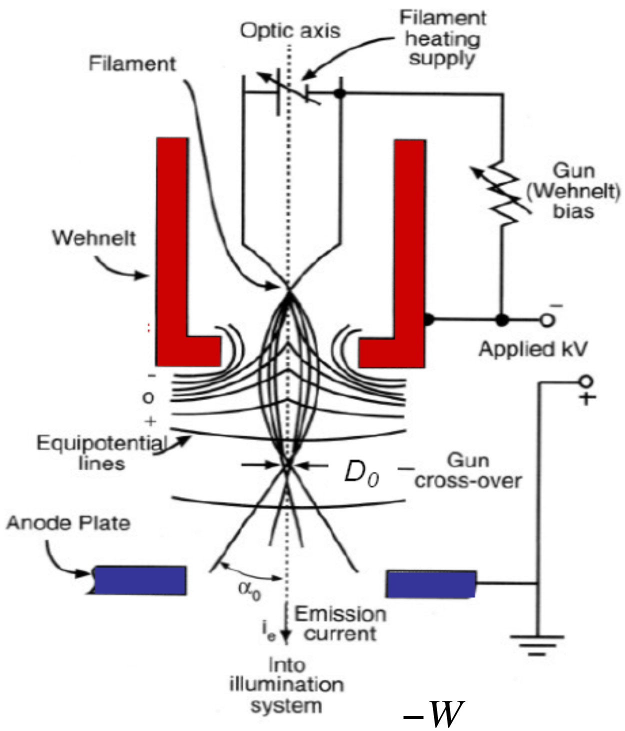


The Rosenthal Group at Vanderbilt

Phase contrast TEM image of a CdSe nanocrystal.

Electron sources –Thermionic Emitters

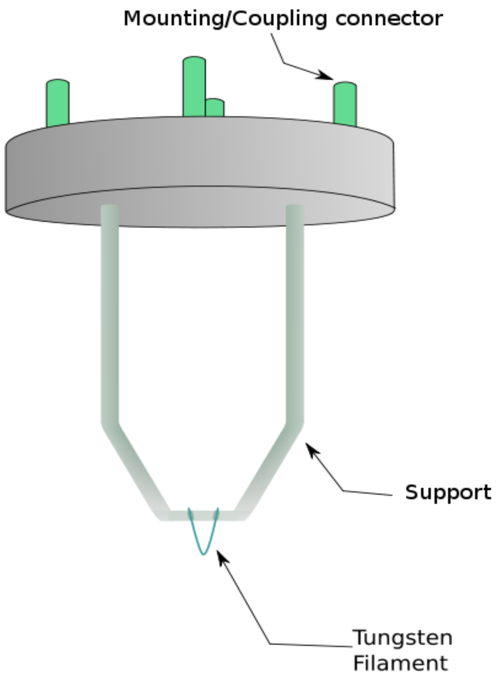
Thermionic emission is the heat-induced flow of charge carriers from a surface or over a potential-energy barrier

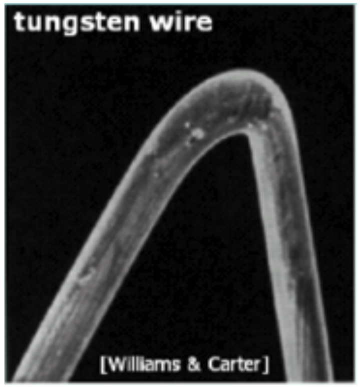


$$J = A_G T^2 e^{\frac{-W}{kT}}$$

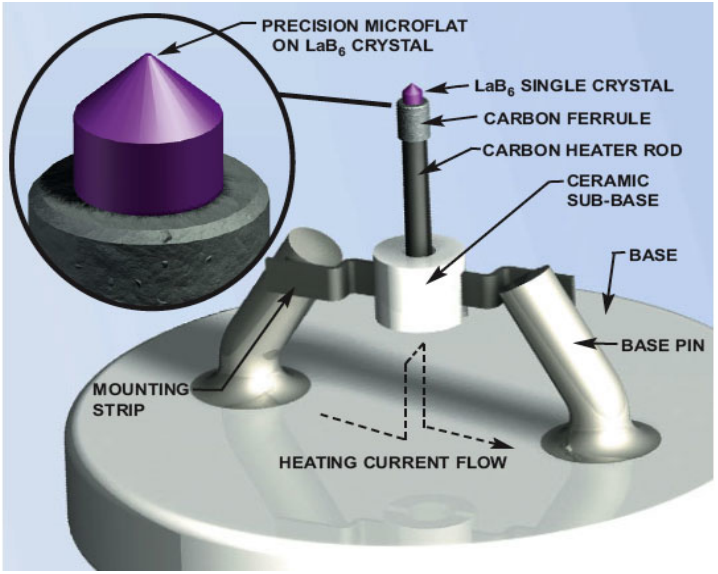
J = emission current density
A_G= 'Richardson's constant' (material dep.)
W = work function, T = temperature,
k = Boltzmann's Constant

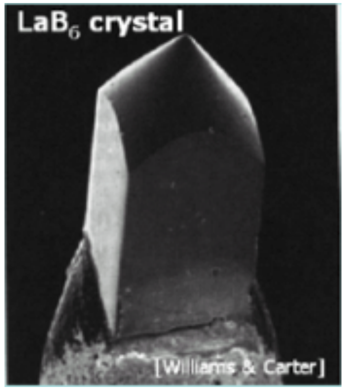
W hairpin





LaB_6 single crystal



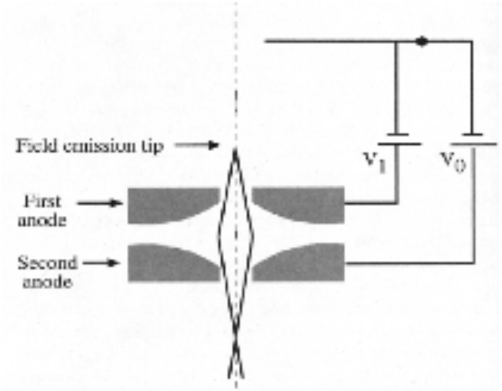


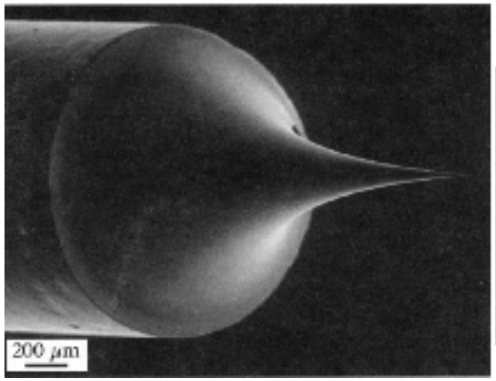
Electron Sources II - Field Emitters

Field emission (FE) is emission of electrons induced by an electrostatic field. Two kinds are common in electron microscopy (i) **Schottky Emission** and (ii) **Cold Field Emission (CFE)**

(i) **Schottky Emission** most electrons escape over the top of a field-reduced barrier, from states well above the Fermi level (**energy spread >0.7 eV**)

(ii) **CFE** most emitted electrons escape by **Fowler-Nordheim tunneling** from electron states close to the emitter Fermi level (**energy spread <0.3 eV**).

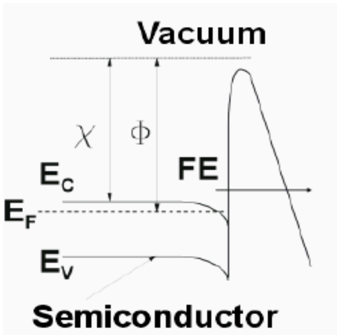


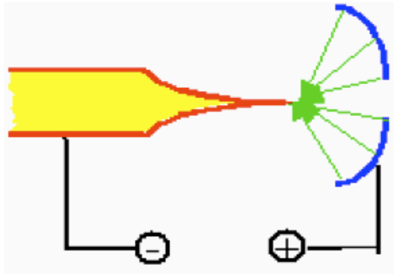


In general, FEG electron sources have much higher coherence and improved electron densities wrt to thermionic emitters

Emission

- Field emission

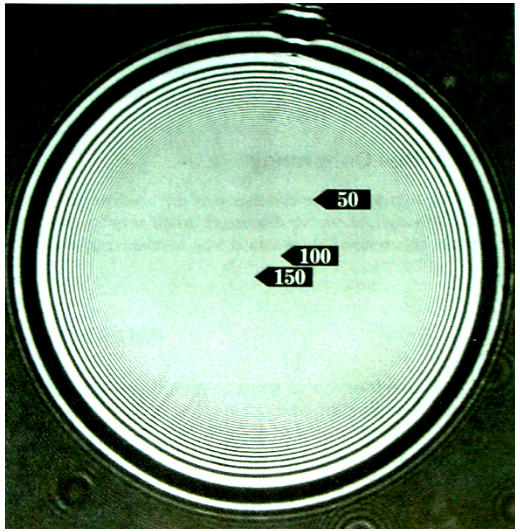




Field emission starts for E > 10^7 V/cm
High current density: $J(E) = A \cdot E^2\ \varphi \exp(-B\ \varphi^{1.5}/E)$

Strong nonlinear current-voltage characteristic
Very short switching time (t <ns)

Small spot size due to field enhancement at the tip apex



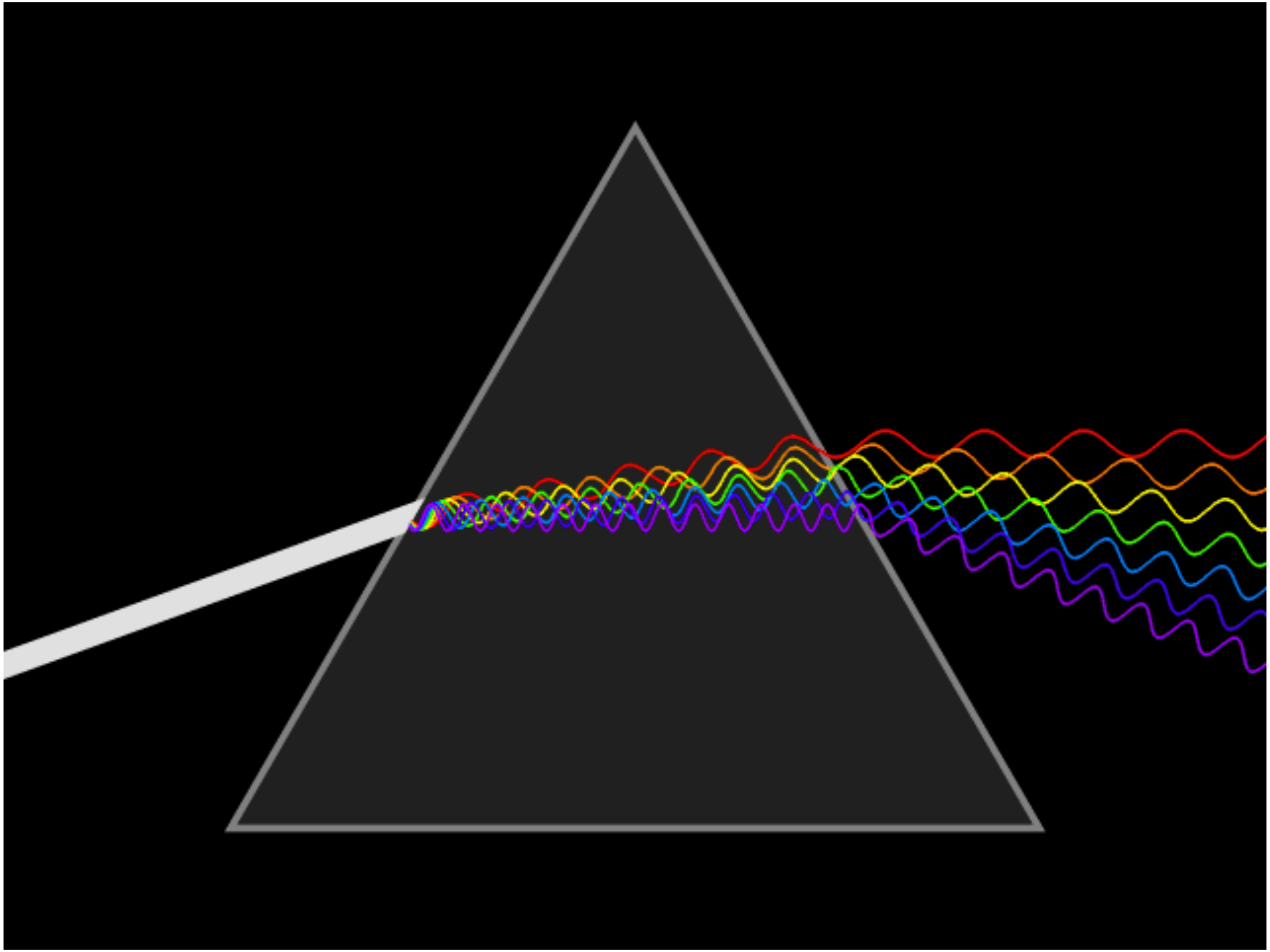
Fresnel Pattern produced with a W FEG source

…but can field emission be improved ?

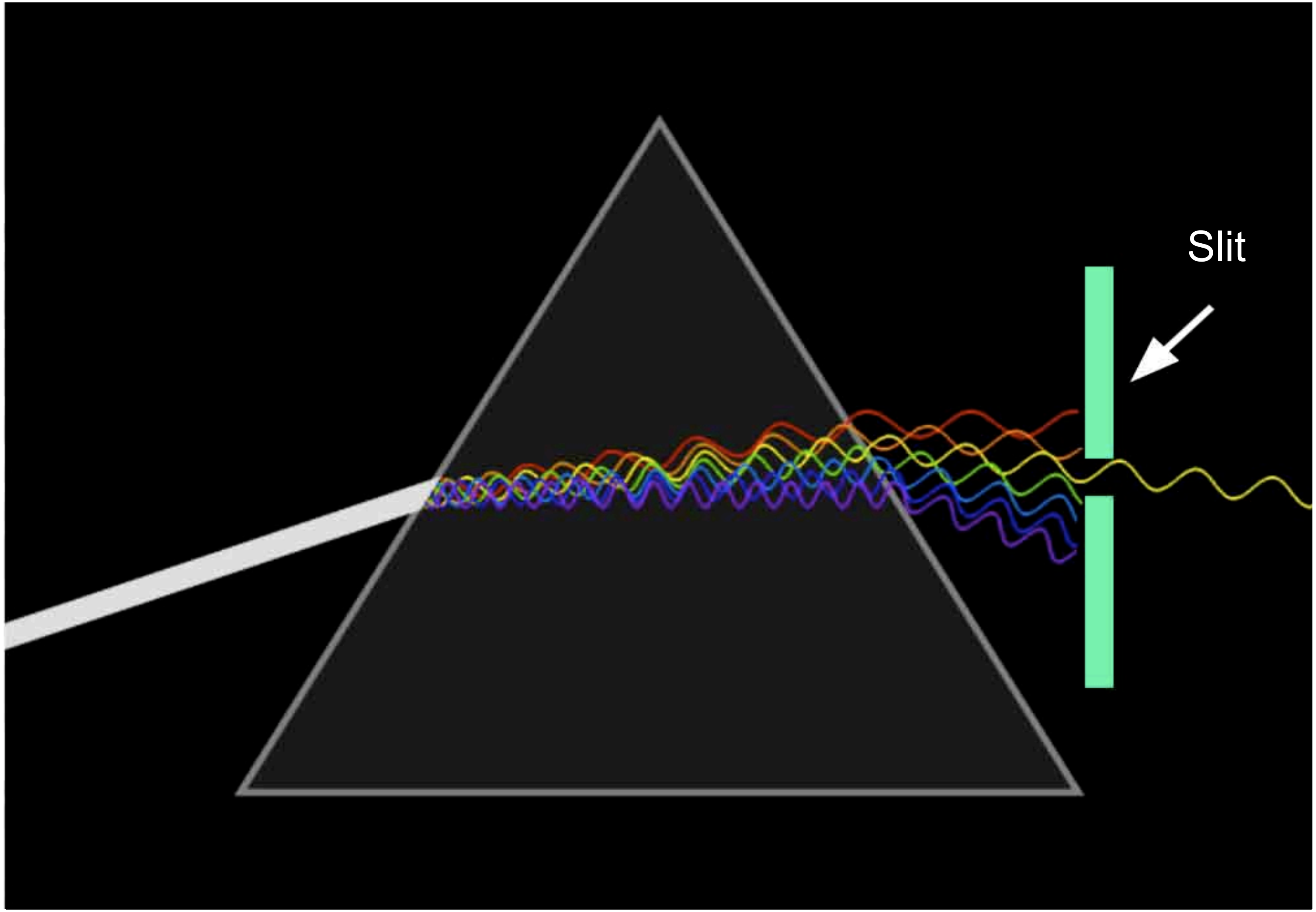
Characteristics of different electron sources

		Thermal emission		Field emission			
		W	LaB_6	Shottky ZrO/W	Thermal FE W (100)	Cold FE W (310)	
Brightness ($A/cm^2/sr$) at 200kV		~$5x10^5$	~$5x10^6$	~$5x10^8$	~$5x10^8$	~$5x10^8$	
Electron Source Size		$50\mu m$	$10\mu m$	$0.1\text{-}1\mu m$	10-100nm	10-100nm	
Energy Width (ev)		2.3	1.5	0.6-0.8	0.6-0.8	0.3-0.5	
Operating Conditions	Vacuum (Pa)	10^{-3}	10^{-5}	10^{-7}	10^{-7}	10^{-8}	10^{-9}
	Temperature (K)	2800	1800	1800	1600	300	
Emission	Current (μA)	~100	~20	~100	20-100	5-20	
	Short term stability	1%	1%	1%	7%	5%	2%
	Long term stability	1%/hr	3%/hr	1%/hr	6%/hr	20%	10%
Maintenance		Not necessary	Not necessary	Start-up takes time	Build up necessary after change	Flash every few hours	
Price & Operation		Low & simple	Low & simple	High & easy	High & easy	High & complicated ?	
Lifetime		3 months	1 year	>4 years (UIC 8 years +)	?	?	1 year
UIC instruments		JEM-100CX	JEM-3010	JEM-2010F	NA	NA	HB601

Principle of monochromation

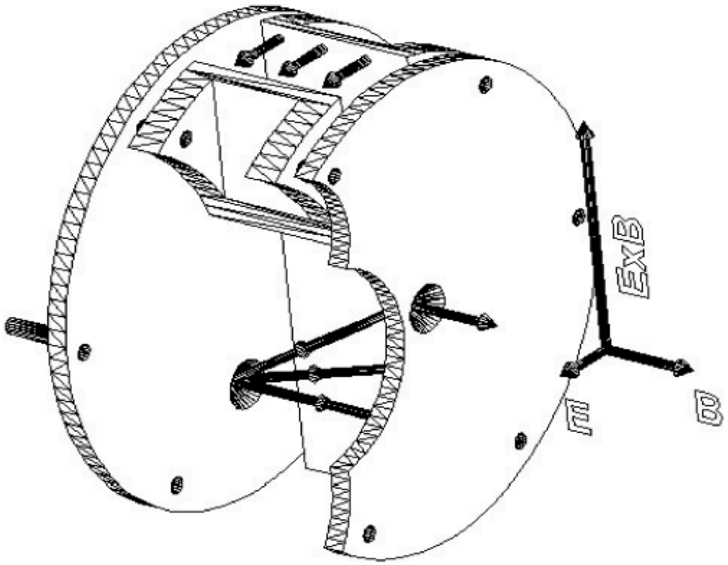


Principle of monochromation

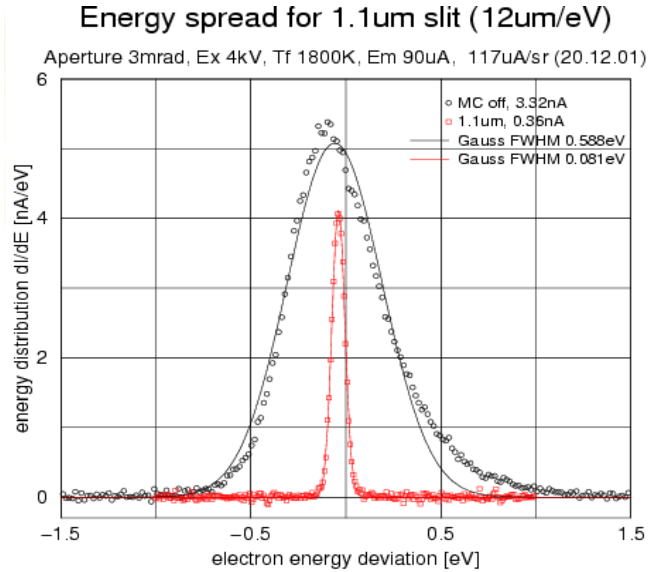


…doing this reduces the beam intensity, however, reducing S/N in spectroscopy

Monochromating the FEG improves the energy and imaging resolution further….



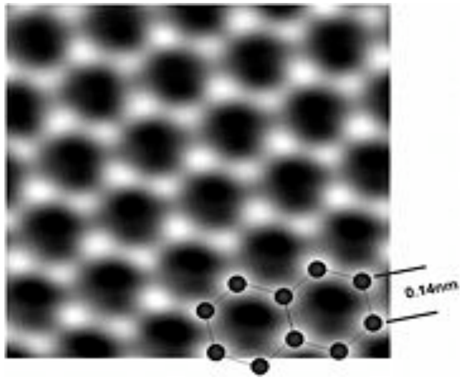
Good for spectroscopy…



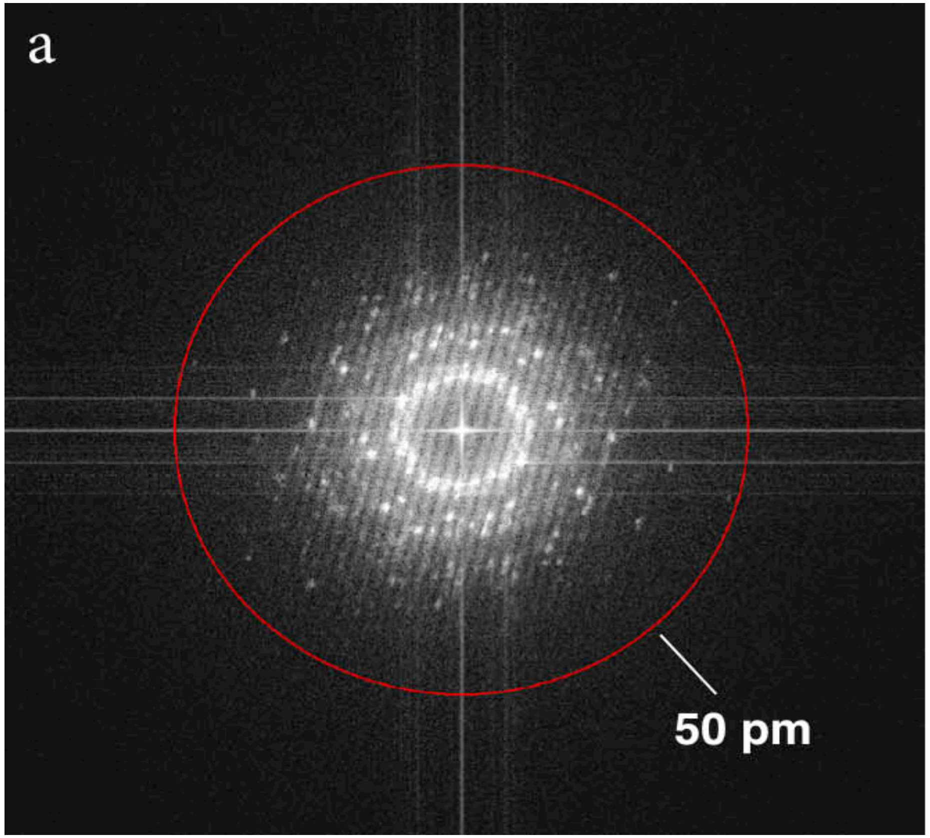
http://www.ceos-gmbh.de/mono.html

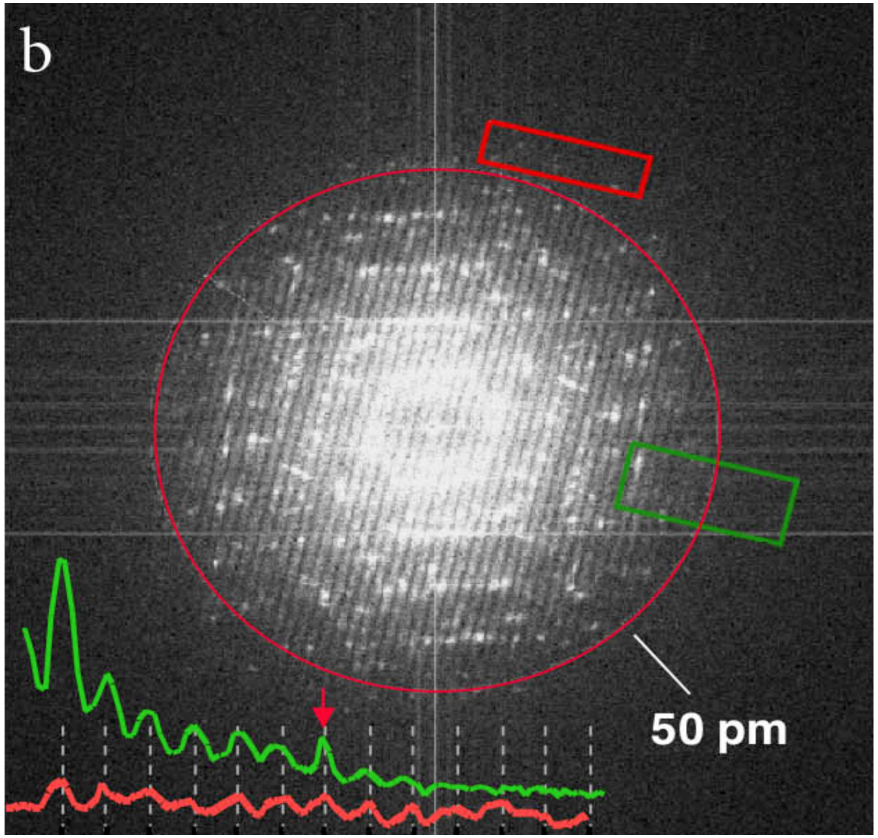
Schematic representation of a monochromator
The separation of the electrons occurs in the space with crossed perpendicular homogeneous electrostatic (E) and magnetic (B) fields.

…and for imaging



We can even see the effect of monochromation in electron diffraction

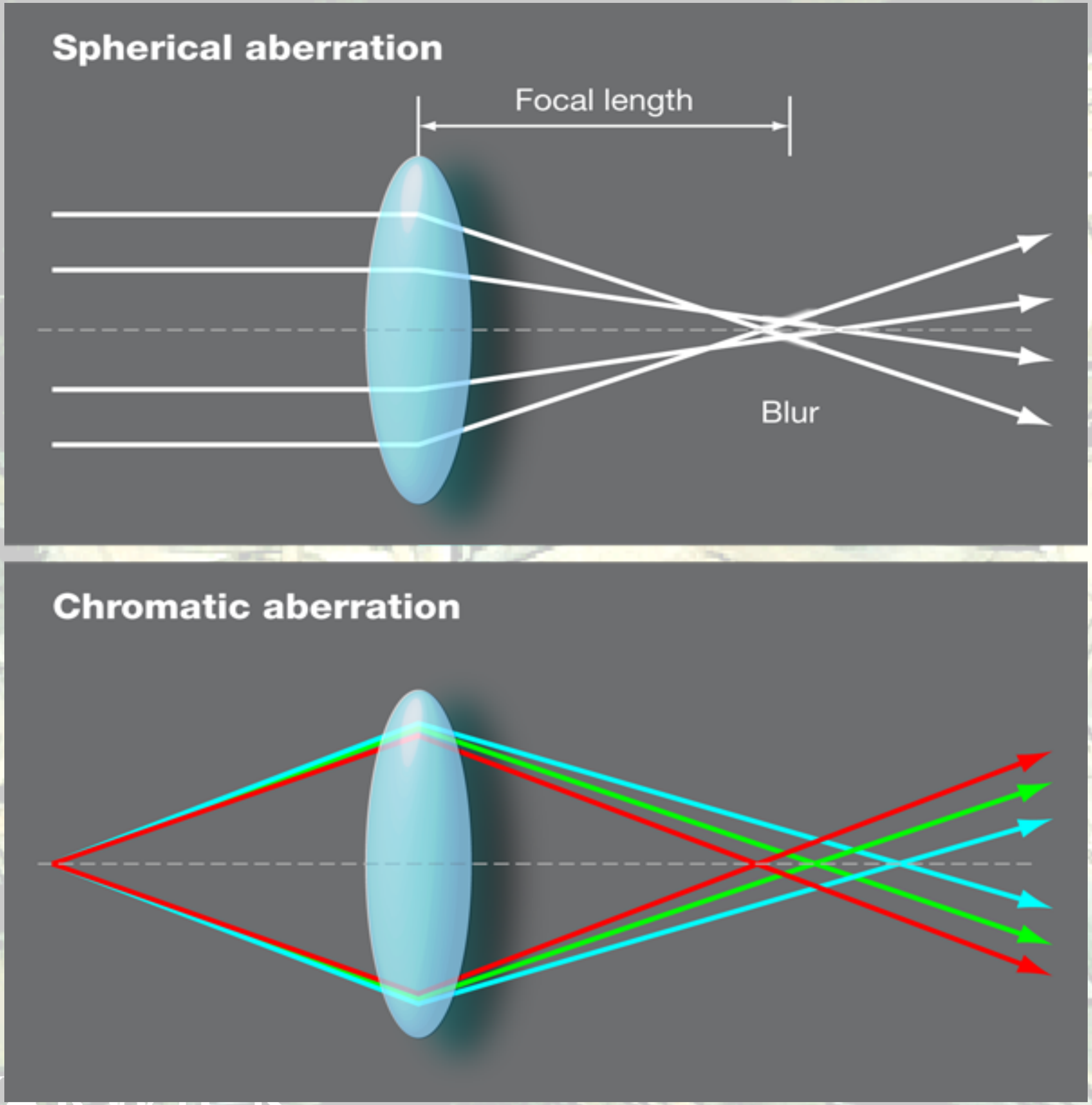




Young's fringe experiments with gold nanoparticles suspended on a carbon grid. With the monochromator **switched off**, **(a)**, the fringes extend to about 70 pm. With the monochromator **switched on**, **(b)**, the fringes extent to below 50 pm.

http://ncem.lbl.gov/frames/TEAM0.5.htm

But we still have to worry about lens aberrations…



These cause problems in **probe formation** – which adversely affects **spatial resolution** in spectroscopy.

And also **image formation – these** aberrations influence ultimate resolution (more on this later….).

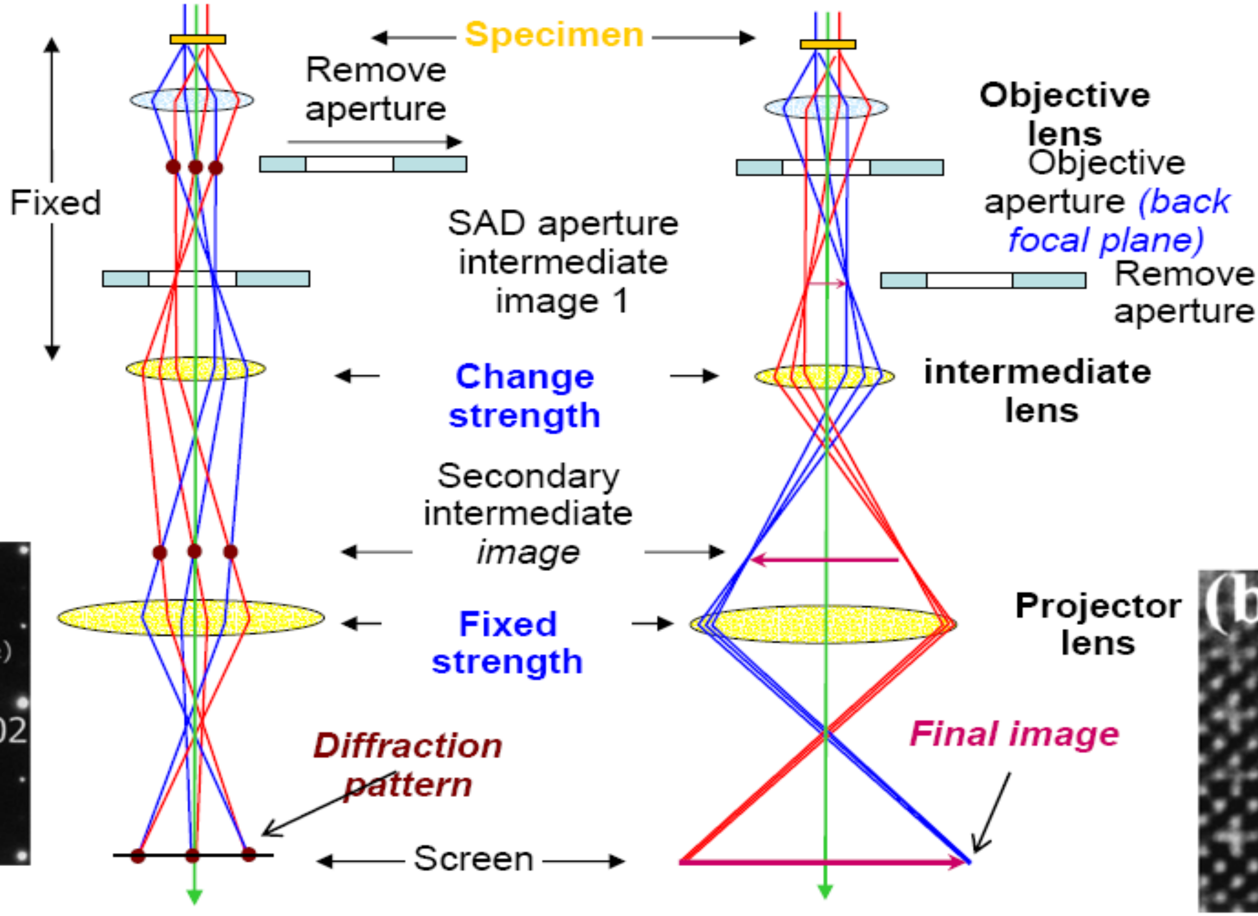
The best electron source is therefore a **high brightness** source that is **<u>monochromated</u>.**

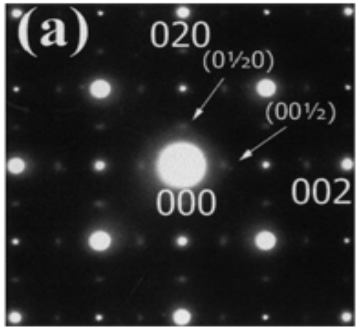
Current state of the art is ~0.08 eV
(still room for improvement !)

WARWICK

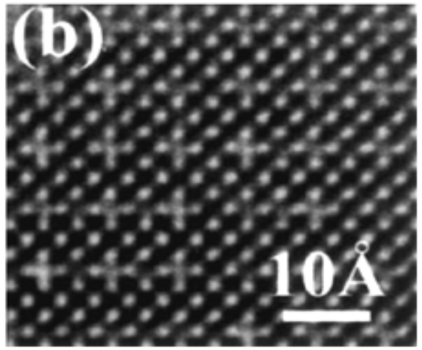
Image and diffraction pattern formation in a HRTEM

Diffraction mode Image mode





Electron diffraction pattern

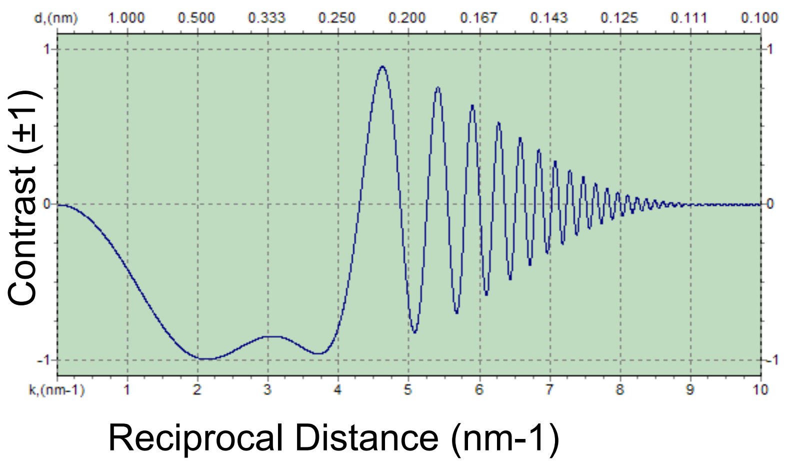


HRTEM Image (white crosses are <u>cation</u> vacancies in $La_xSr_yTiO_3$: an example of <u>defect</u> imaging)

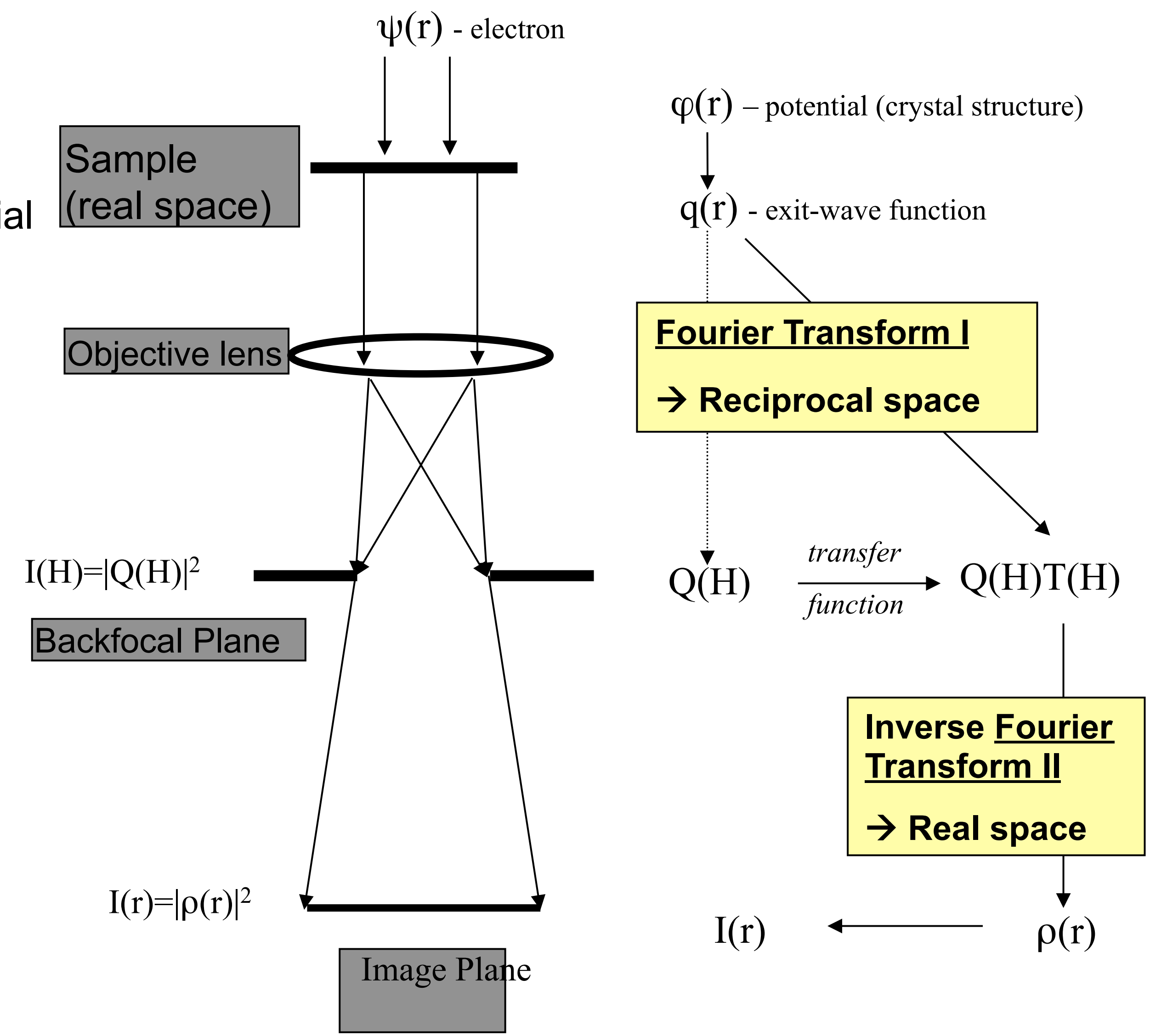
Contrast Transfer Function (T(H))

$T(H)=\sin(\pi C_s\lambda^3H^4/2+\pi\Delta f\lambda H^2)$.

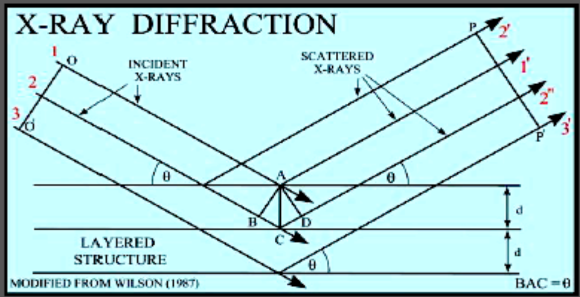
C_s: Spherical aberration constant; Δf: defocus value; λ = wavelength; H = spatial frequency (~atom periodicity)



Phase contrast transfer function calculated at Δf=-61 nm with Cs=1.0 mm.



Diffraction by crystals

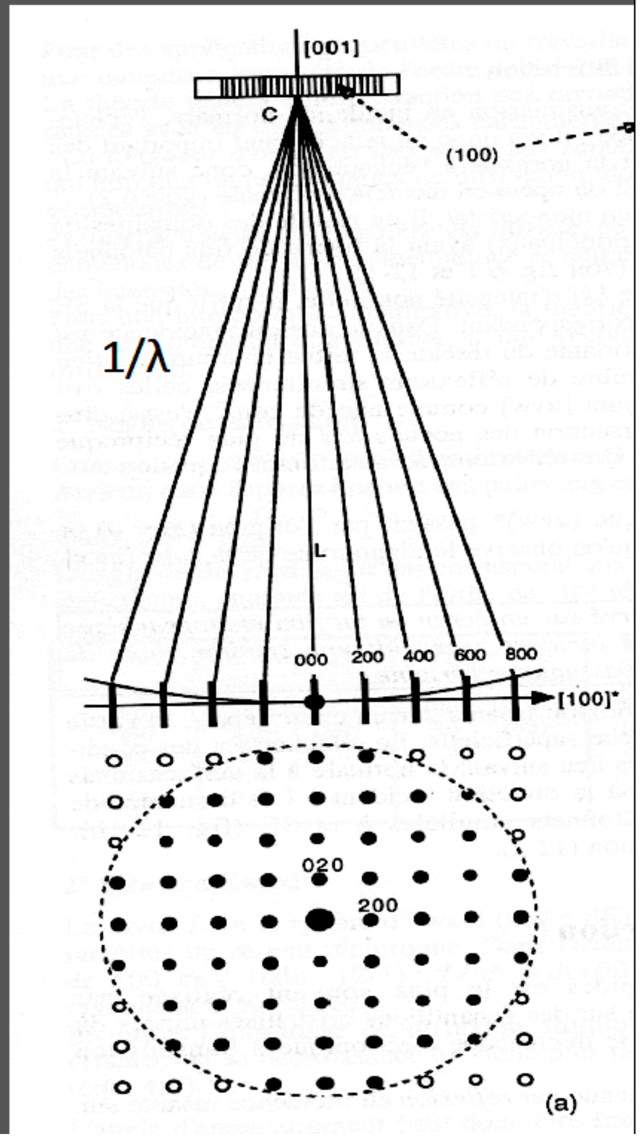


Bragg's law

$$2d_{(hkl)}\sin\theta = n\lambda$$

and for high-energy electrons:

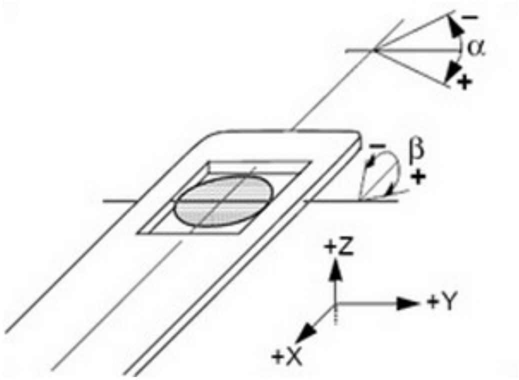
$$2d_{(hkl)}\theta \approx n\lambda$$



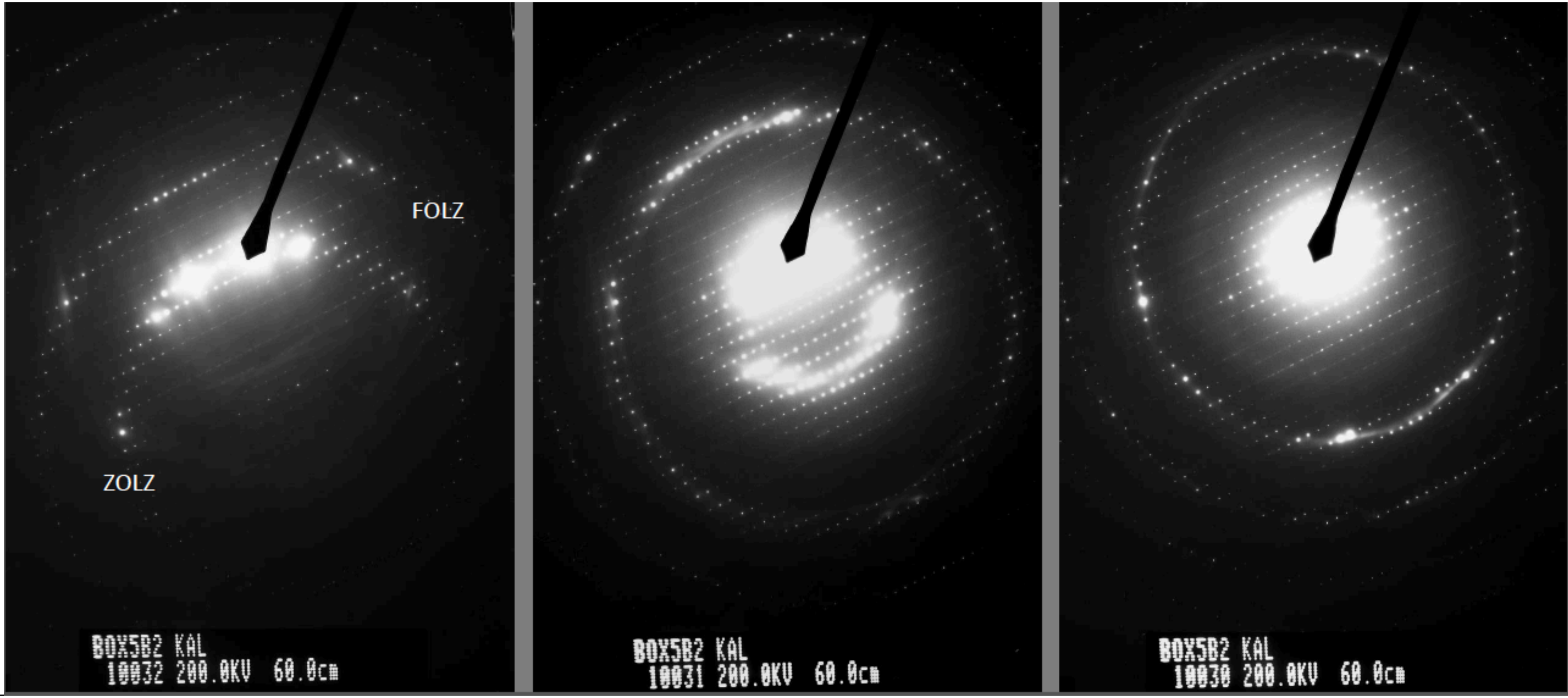
$\theta \approx < 1\text{-}2°$ ----->only lattice planes // to the incident beam are diffracting

very thin specimen ---> elongation of reciprocal lattice nodes --->relaxation of Bragg's conditions ------> many reflections at a time

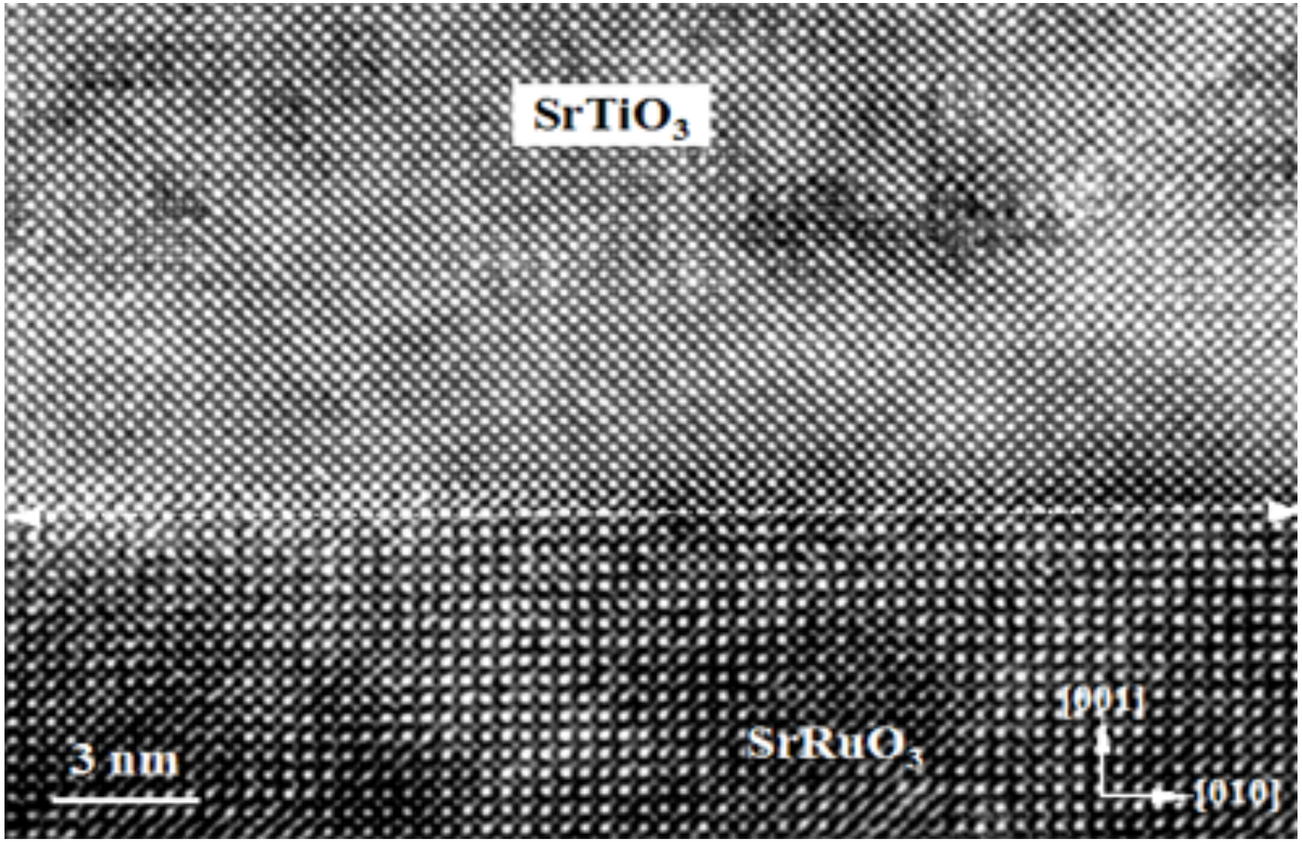
How to align a single crystal in a TEM (Diffraction Mode)



- incredibly important
- This is how we obtain HRTEM images in TEM/ HRTEM/STEM
- electron crystallography
- Need double tilt specimen holder



We need to be able to align crytsals in HRTEM to be able to study interfaces….



a=0.3905 nm

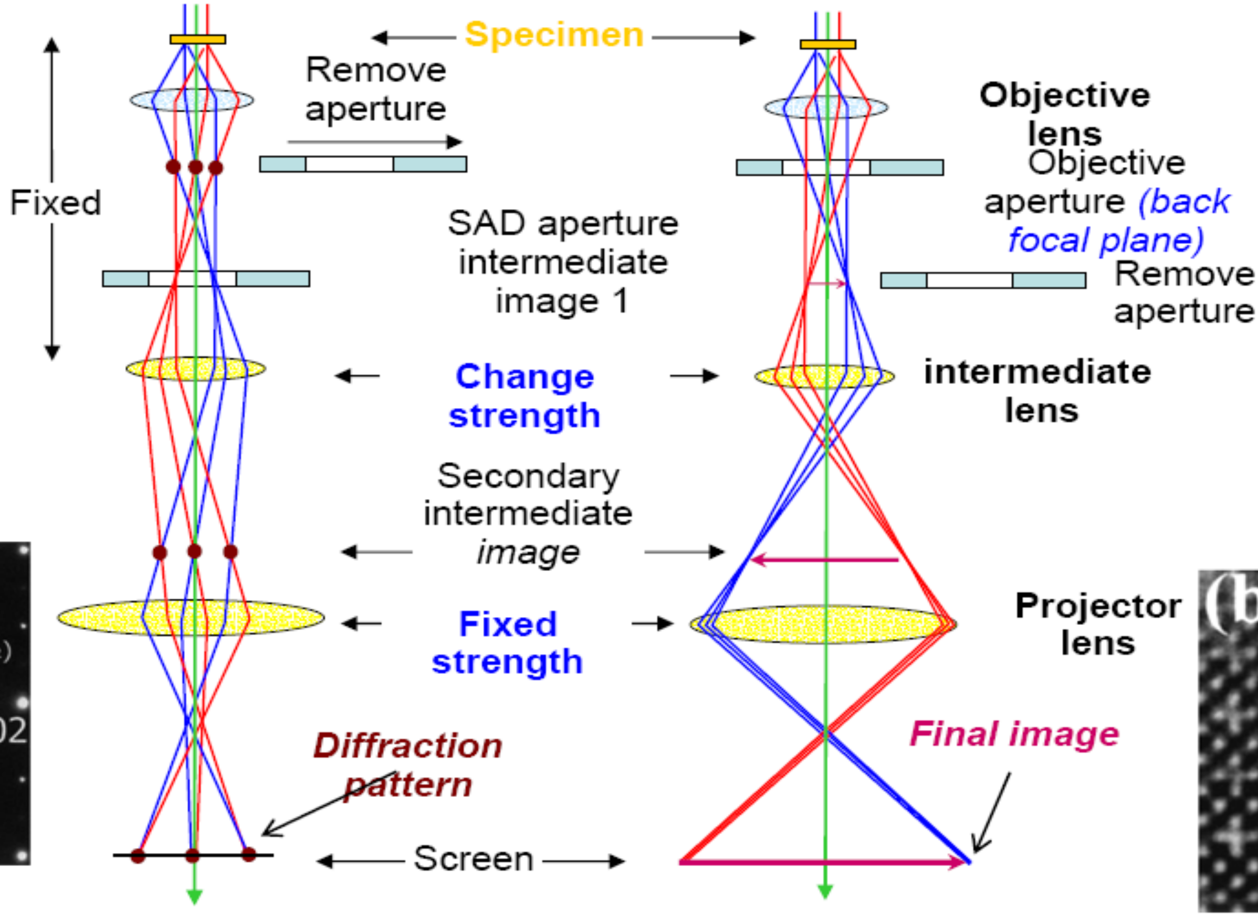
Misfit=0.64%

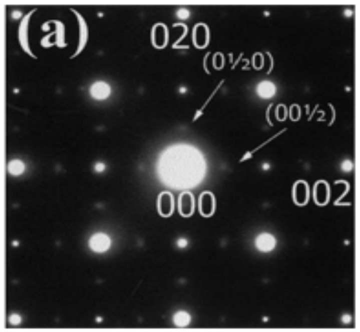
a=0.3982 nm

HREM image the coherent $SrTiO_3/SrRuO_3$ interface.

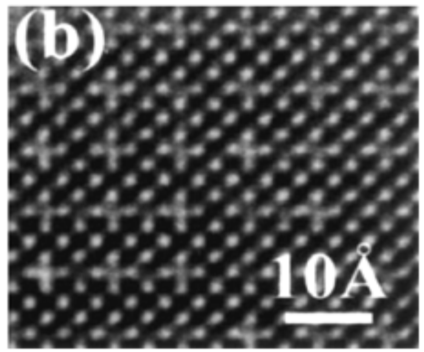
Image and diffraction pattern formation in a HRTEM

Diffraction mode / Image mode





Electron diffraction pattern



HRTEM Image (white crosses are cation vacancies in $La_xSr_yTiO_3$: an example of defect imaging)

Contrast Transfer Function (i.e. CTF or T(H)) & Scherzer defocus (optimum focusing conditions)

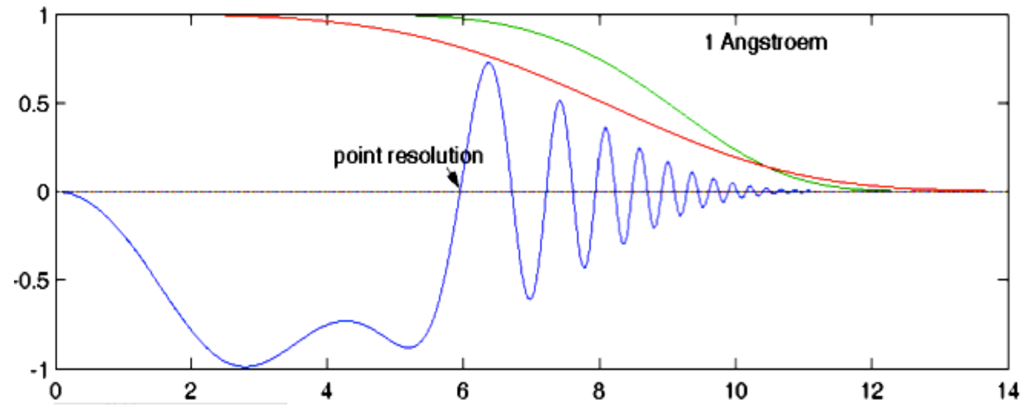
Mathematical Form of the CTF

$$T(H) = \sin\chi = \sin\left(\frac{1}{2}\pi C_s \lambda^3 H^4 + \pi \Delta f \lambda H^2\right)$$

C_s: Spherical aberration constant; Δf: defocus value; λ = wavelength; H = spatial frequency (~atom periodicity).

"Sin χ" is the "Contrast"

Graphical Form of the CTF



http://www.maxsidorov.com/ctfexplorer/webhelp/background.htm

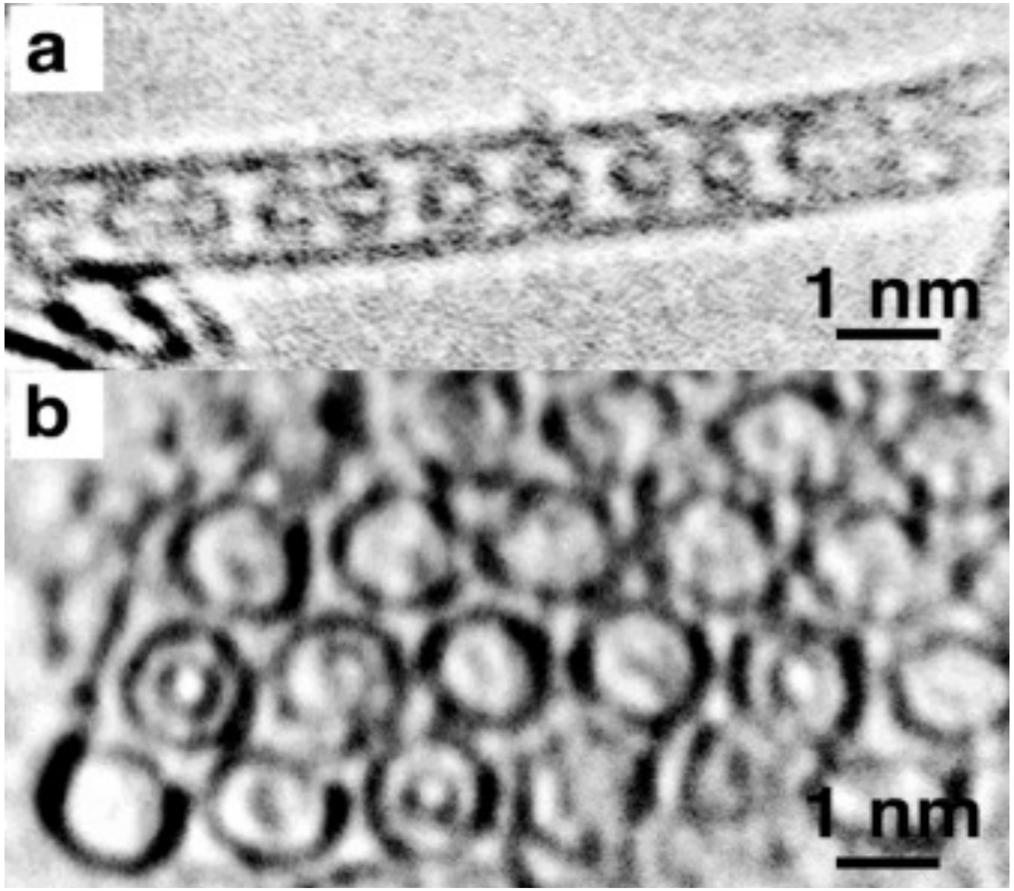
In **Scherzer defocus**, one aims to choose the right defocus value *Δf* whereby low spatial frequencies *u* are transferred into image intensity with a similar phase. In 1949, Scherzer found that the optimum defocus depends on microscope properties like the spherical aberration C_s and the accelerating voltage (through *λ*) in the following way:

$$\Delta f_{Scherzer} = -1.2\sqrt{C_s \lambda}$$

where the factor 1.2 defines the extended Scherzer defocus. For example, when C_s=*0.6* and an accelerating voltage is 300keV result in $\Delta f_{Scherzer}$ = *-41,25 nm*.

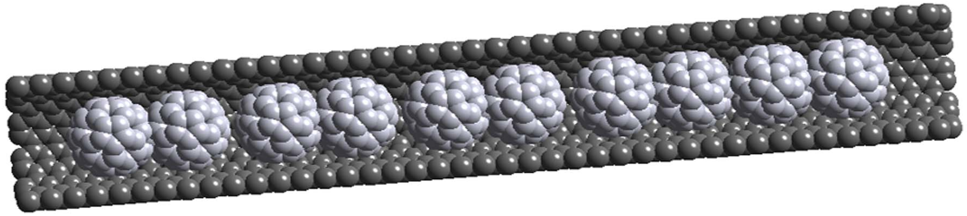
In the contrast transfer function (CTF), the **point resolution of a microscope** is defined as the spatial frequency u_{res} where the CTF crosses the X-axis for the first time. At Scherzer defocus this **value is maximized**.

Imaging of molecular structures within SWNTs (C_{60})



SWNT = Single Walled Carbon Nanotube

c

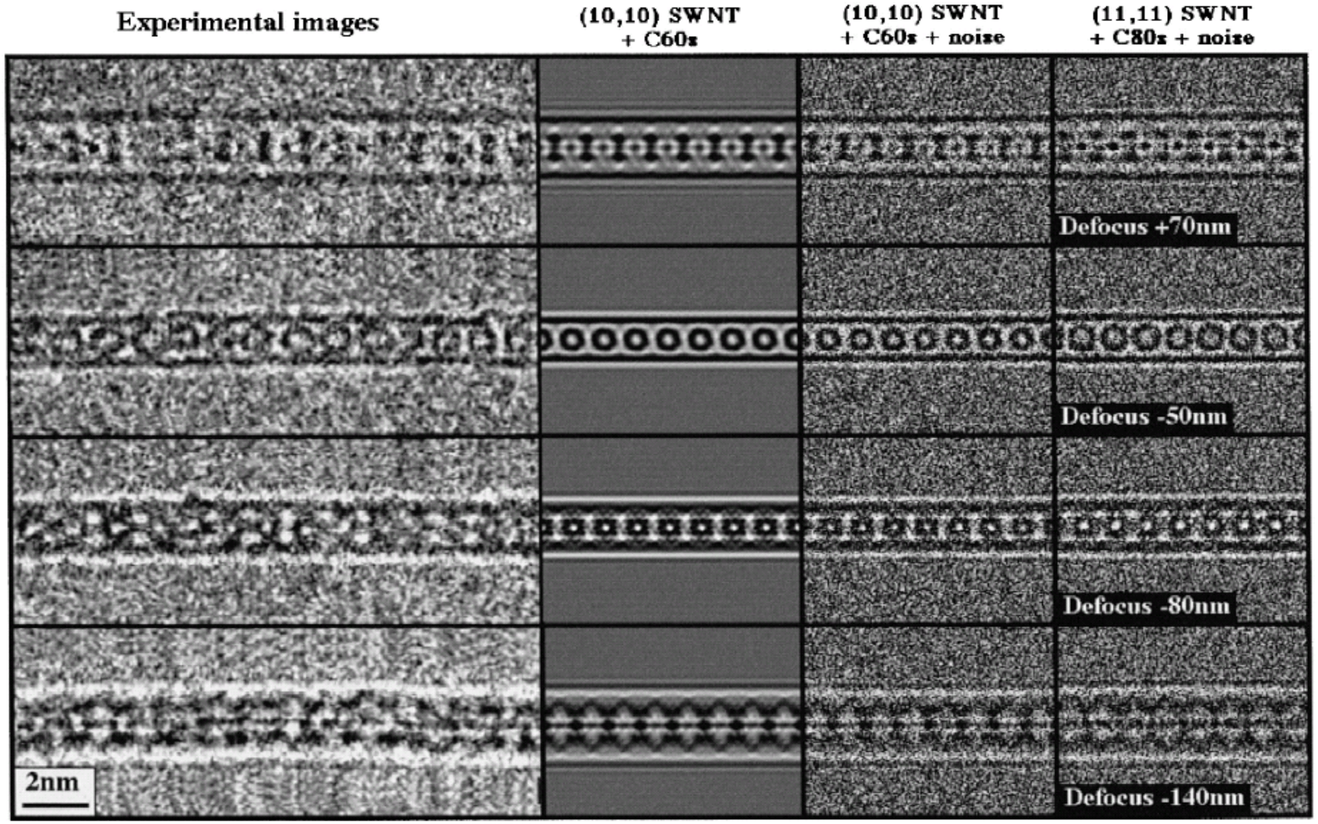


B.W. Smith, M. Monthioux, and D.E. Luzzi, Nature 396 (1998) p. 323.

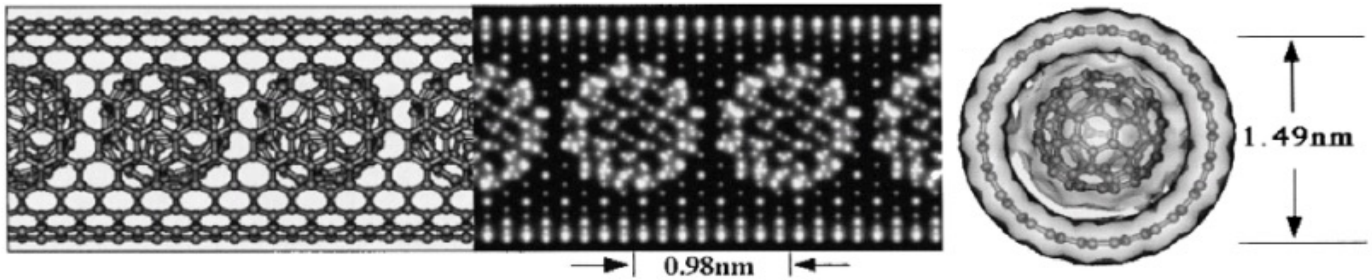
A SWNT is one example of an ultrathin specimen support

Imaging and simulation of C_{60} and C_{82} within SWNTs

Focal Series



Simulations

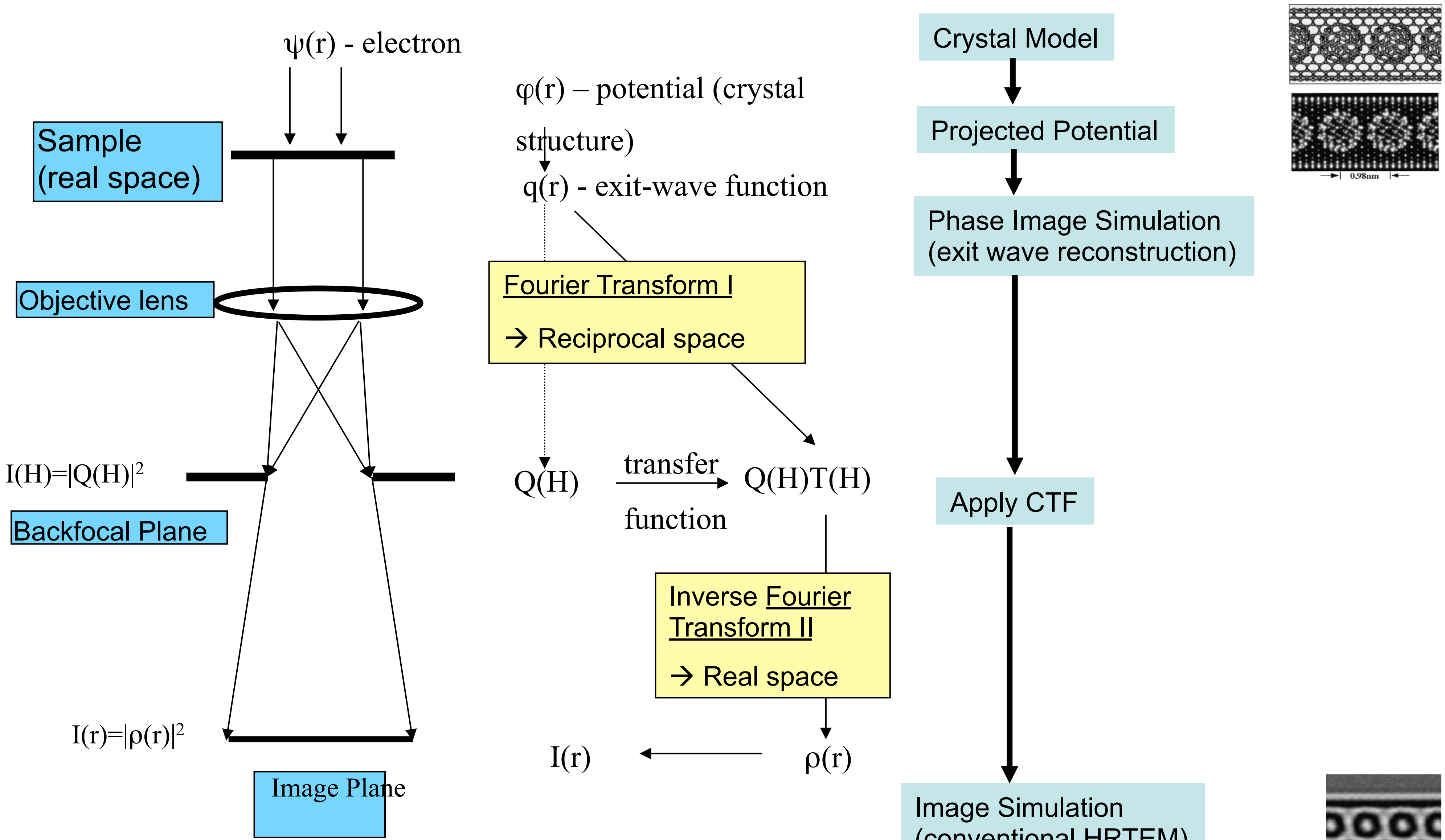


Structure model

Projected potential

Chem. Phys. Lett. 316(2000)191–198

Schematic Procedure for Image Simulation



Cowley, J. M.; Moodie, A. F., Acta Cryst., 1957, 10, 609-619; Goodman, P.; Moodie, A. F. Acta Cryst., 1974, A30, 280290.

So How to Align a TEM for the Best Performance ?

Short Answer – Best Learned Hands-on
However there is an online course (http://www.rodenburg.org/guide/t1300.html)

Overview

Objective: to utilize the atomic resolution capabilities of the HREM to characterize the nanostructure of the samples.

One should be concentrated on the phase contrast imaging with (partially) coherent illumination, large (or possible no) objective aperture, and optimum Objective Lens defocus (i.e. to obtain ideal Scherzer Defocus Conditions for maximum transfer of information).

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"Preliminaries"

• Basic alignment/adjustment of illumination/imaging system:

1) Gun shift
2) Gun tilt
3) Condenser lens alignment (pre-aligned)
4) Condenser aperture
5) Condenser astigmatism
6) Projecter lens system (pre-aligned)

So How to Align a TEM for the Best Performance ?

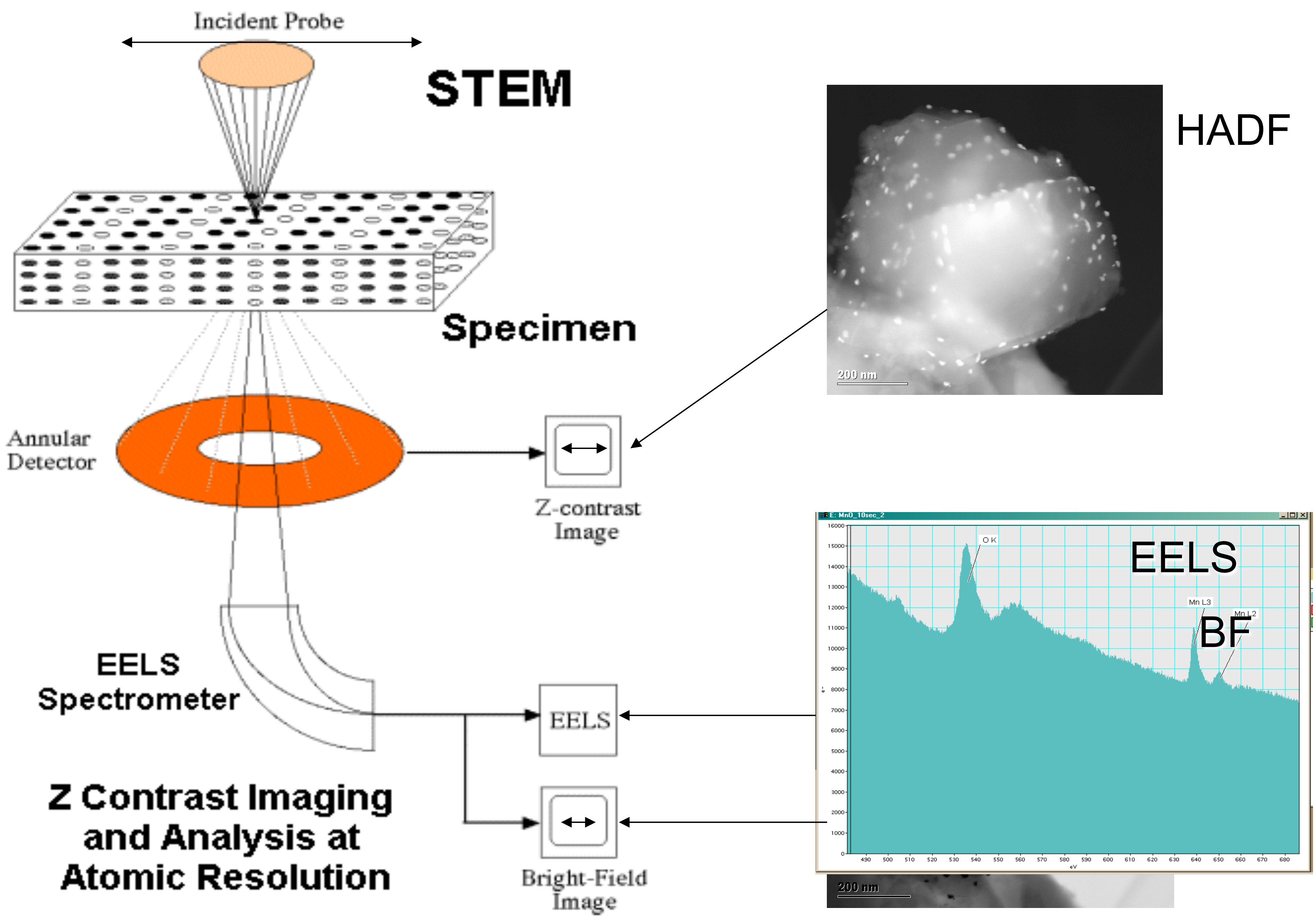
Important parameters (calibration)

1) Camera length
2) Electron-optical magnification
3) Defocus/defocus steps
4) Spherical aberration coefficient
5) Objective lens astigmatism
6) Illumination angle
7) Spread of focus
8) Resolution

So How to Align a TEM for the Best Performance ?

Improving the Resolution (see next lecture)

- This improvement can be done by changing some parameters in the microscope:
  - Δf (Objective lens defocus)
  - λ (wave length of the beam, i.e. beam energy), but the more energy the more radiation damage we produce.
- Or by improving:
  - C_S (Spherical Aberration of the objective lens)
  - C_c (Chromatic Aberration of the objective lens)
  - Coherence (monochromation)



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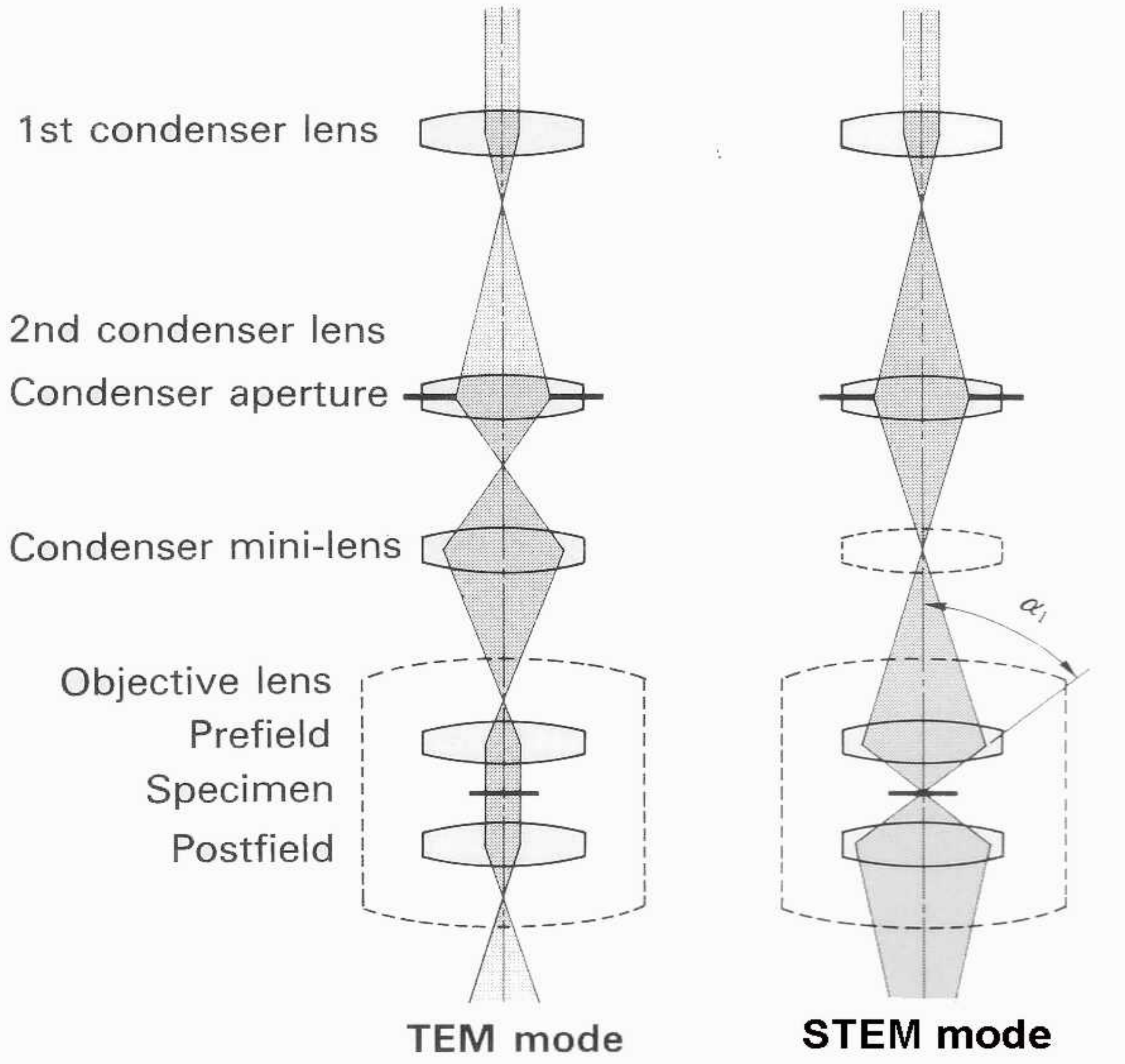


TEM>STEM

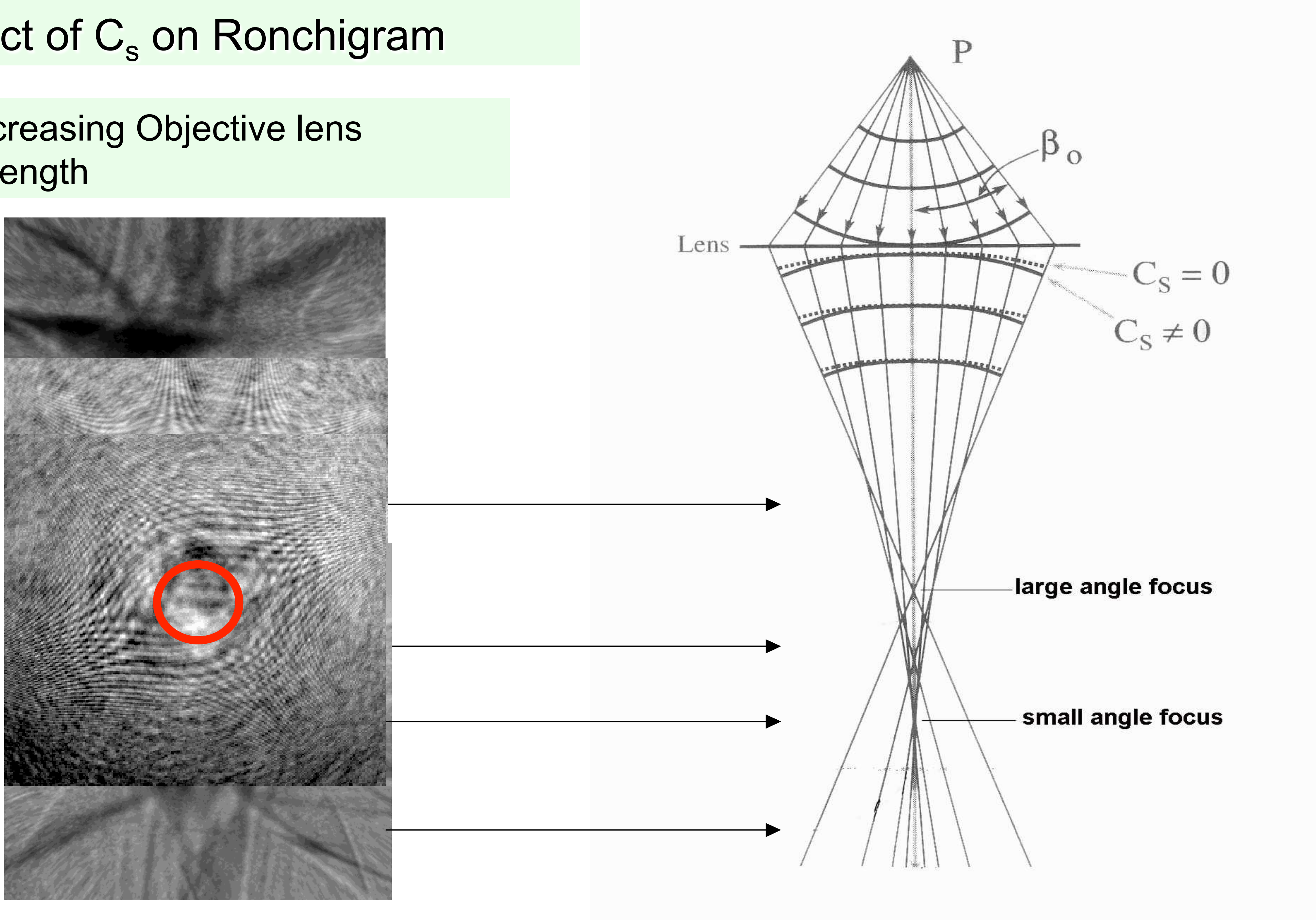
Microscope should be aligned in TEM mode before entering STEM mode. If EELS spectra to be collected this should include GIF alignment.

If you need to adjust A2 to optimize the probe this should be done before TEM alignment.

A2 adjustments are only necessary for ultimate imaging resolution and should be done slowly!

Effect of C_s on Ronchigram

Increasing Objective lens strength



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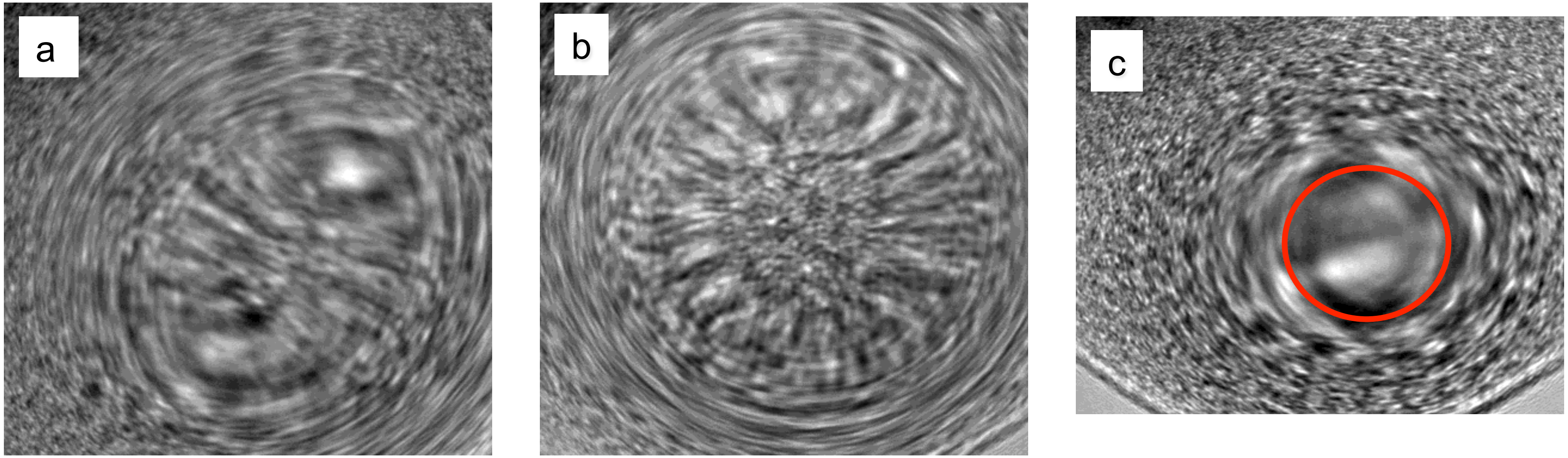
Stigmating using the Ronchigram



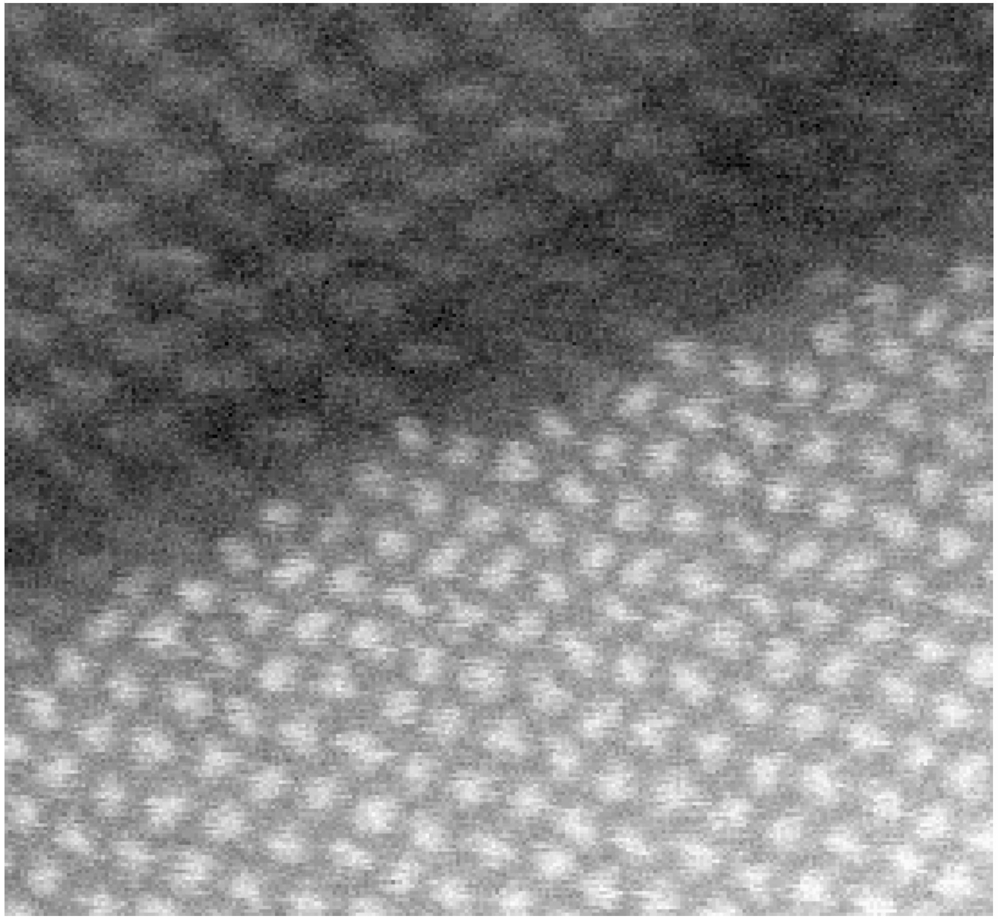
Ronchigram from amorphous area. Select Scan mode Spot 1 and a magnification above 100Kx.

a) underfocus astigmatic

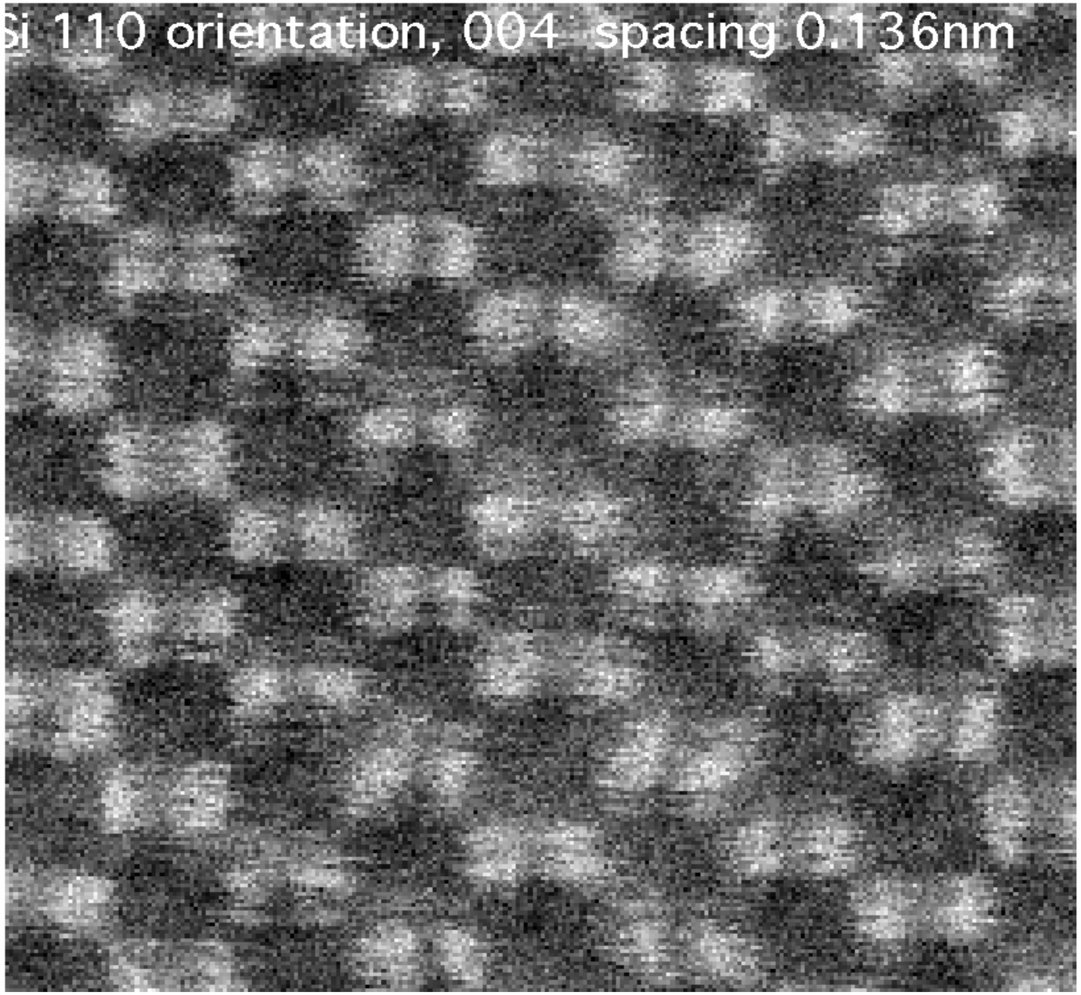
b) underfocus stigmated

c) Gausian focus stigmated (almost!)

Red circle marks unaberrated part of Ronchigram that should be selected by Objective aperture



< Au contact layer on GaAs - 0.2nm probe, (C1 6.06; C2 4.65; A2 7.3)



Si dumbells resolving 0.136nm 004 spacing with 0.13nm probe > (C1 6.06; C2 4.65, A2 6.8)

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Suggested Reading

‘Transmission Electron Microscopy’, Volumes 1-4 (or single volume) By David B. Williams and C. Barry Carter, Plenum Press, 1996.

‘Electron Microscopy and Analysis’ By Peter J. Goodhew, F. J. Humphreys, R. Beanland, Taylor and Francis, 2001.

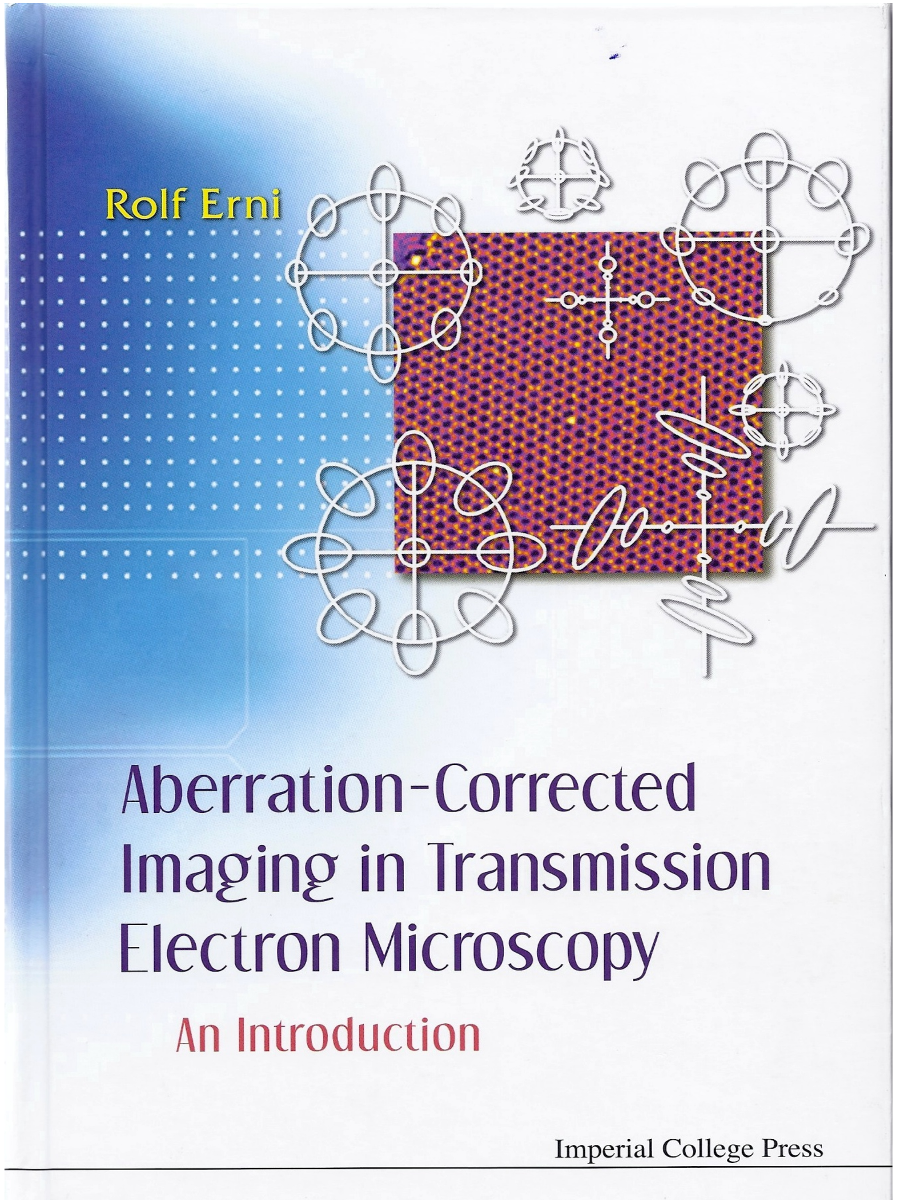
‘Electron Energy Loss Spectroscopy’, Rik Brydson, Royal Microscopical Society Handbooks, 2001.

‘Electron Energy-Loss Spectroscopy in the Electron Microscope’ 2nd Ed. R. F. Egerton, Plenum, 1996.

‘Transmission Electron Microscopy; Physics of Image Formation.’ L. Reimer and H. Kohl, 5th Ed., Springer, 2008.

+ any recent scientific literature on electron microscopy – the technology is improving all the time !

The next generation….



To the list of essential texts for any student of high-end electron microscopy, which will include Williams and Carter, Ray Egerton, any or all of a number of volumes by John Cowley and colleagues, Reimer and Kohl, (insert your favourites here), make a space on your bookshelf for Rolf Erni's "Aberration-Corrected Imaging in Transmission Electron Microscopy – An Introduction"- do it now!

Dr. Jeremy Sloan
Department of Physics, University of Warwick, UK

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