Physics of Materials

High Resolution Scanning Electron Microscopy (STEM)

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Masters Course PHYS-307

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Why do we use *electrons* as a probe?
High energy = short wavelengths= high spatial resolution

Electromagnetic radiation: $E = hc/\lambda$ so if $\lambda < 5$ nm, $E > 1$ keV

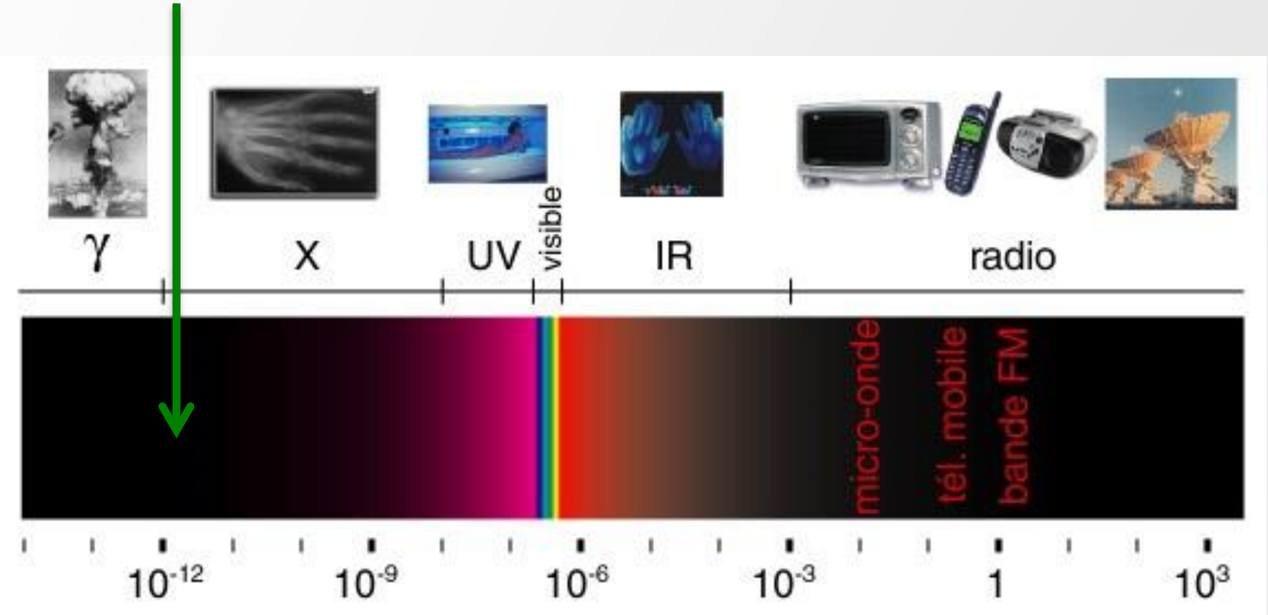
Electron wavelength according to de Broglie equation: $\lambda = \hbar/p$

with $p = m_o v = (2m_o eV)^{1/2}$

non relativistic (<50keV):

$$\lambda = \frac{h}{\left(2m_0 eV\right)^{1/2}}$$

electrons



Relativistic correction (>50keV or >1% speed) :

$$\lambda = \frac{h}{\left[2m_0 eV\left(1 + \frac{eV}{2m_0 c^2}\right)\right]^{1/2}}$$

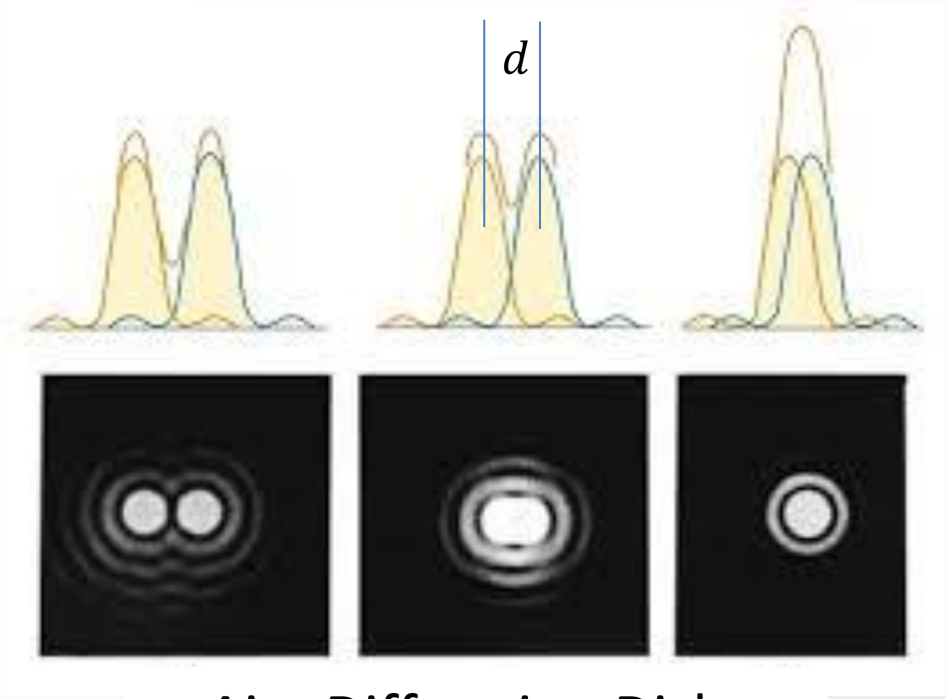
Why do we use *electrons* as probe?

Electron charge (e)	-1.602 x 10^{-19} C
1 eV	1.602 x 10^{-19} J
Electron rest mass (m_o)	9.109 x 10^{-31} kg
Electron rest energy ($m_o c^2$)	511 keV
Kinetic energy (charge x tension)	1.602 x 10^{-19} Nm (per 1 volt)
Plank's constant (h)	6.626 x 10^{-34} N-m-s
1 amp	1 C/sec
Light speed in vacuum (c)	2.998 x 10^{8} m/sec

Accelerating voltage [KV]	**Nonrelativistic λ [nm]**	**Relativistic λ [nm]**	**Mass [x m_o]**	**Velocity [x 10^8 m/s]**
1	0.03879	0.03878	1.002	0.13
10	0.01227	0.01221	1.02	0.42
80	0.00434	0.00418	1.157	1.1
200	0.00274	0.00251	1.391	1.59
300	0.00224	0.00197	1.587	1.82
1000	0.00123	0.00087	2.957	2.44

Why do we use electron as a probe?

Raleigh's Criterion



Airy Diffraction Disks

$$d = 0.61\frac{\lambda}{n \sin\theta} = 0.61\frac{\lambda}{\beta}$$

Green light : $\lambda \approx 532$ nm,
β (objective collection angle)~1 rad

n= 1.7 for oil immersion lens

*** d=200nm

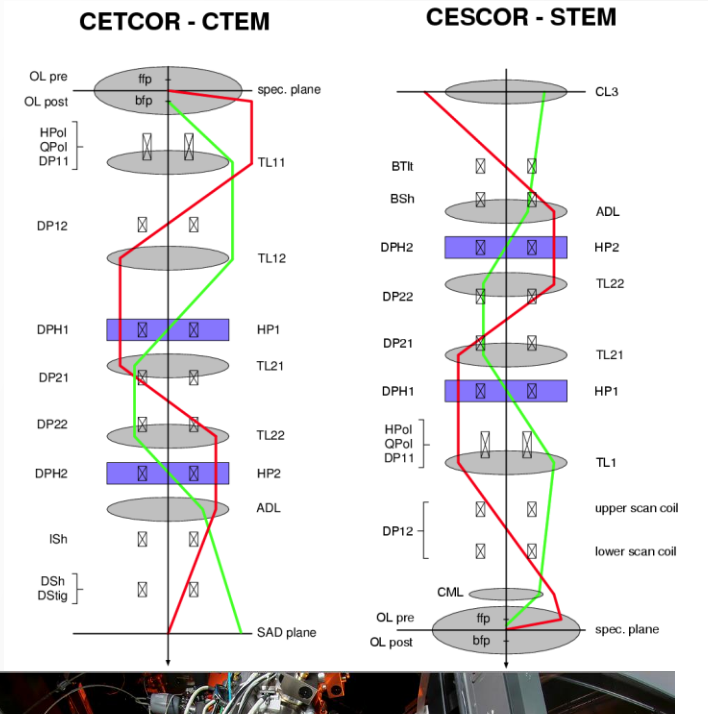
Electrons 200 keV : $\lambda \approx 0.00251$nm

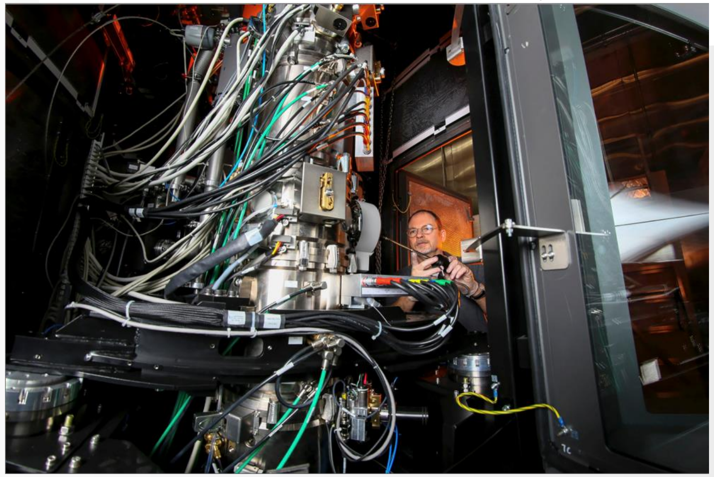
n=1 for vacuum
max β=0.1 rad given TEM geometry

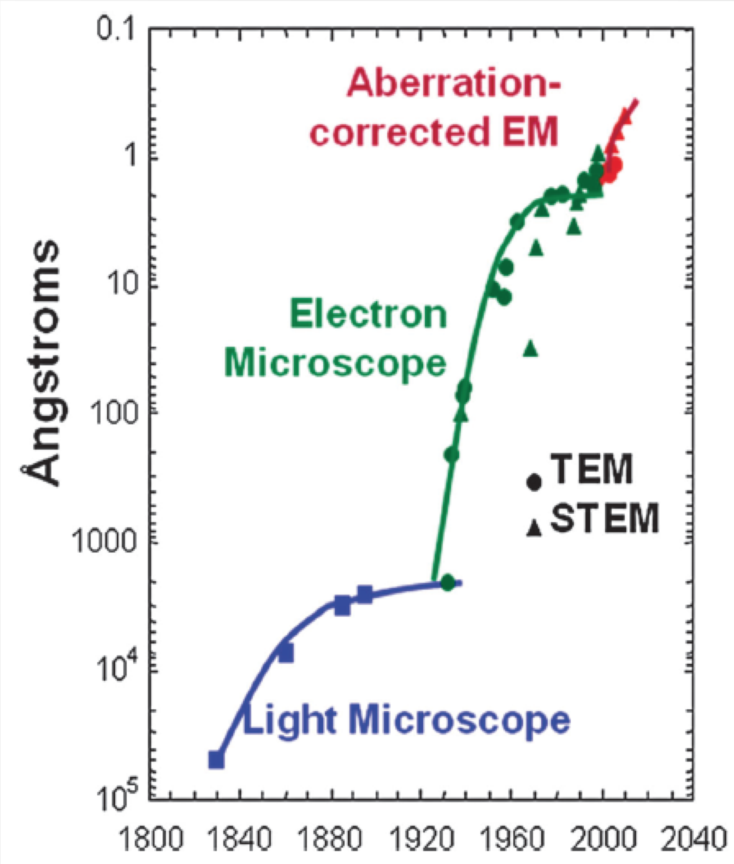
***d=0.015 nm

- ***Lens aberrations limit spatial resolution***
- Spherical and Chromatic Aberration corrections allow for 0.05 nm resolution

Aberration corrected (AC) HRTEM: Cs correctors







Uncorrected resolution is ~150 X diffraction limit

Cs-Corrected resolution is ~20 x diffraction limit

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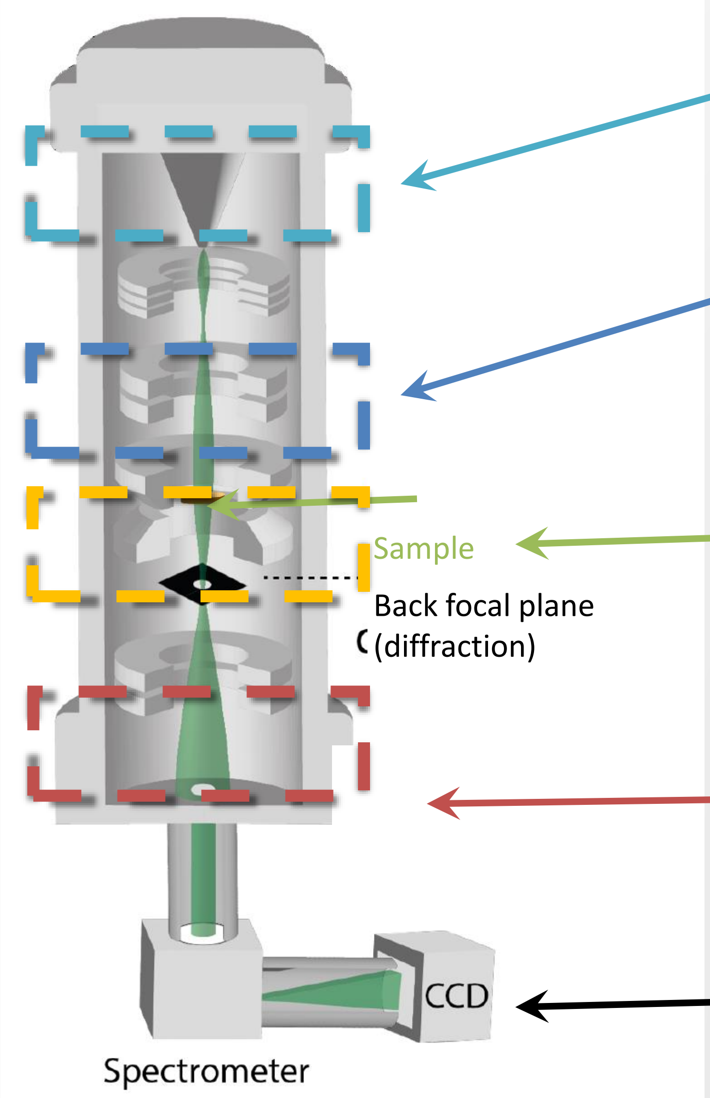
Transmission electron microscope



1. Gun – Thermionic or Field emission. Electrons are accelerated to different voltages (wavelengths)
2. Condenser lenses- changes spot size, illumination area, and convergence angle on the sample (forms STEM probes)
3. Objective lens – images the sample. The electron diffraction patterns are formed in the back focal plane, where the objective aperture is also located.
4. Intermediate lenses- changes modes from imaging to diffraction, magnification, and camera lengths
5. Detectors- cameras, STEM detectors, electron spectrometer

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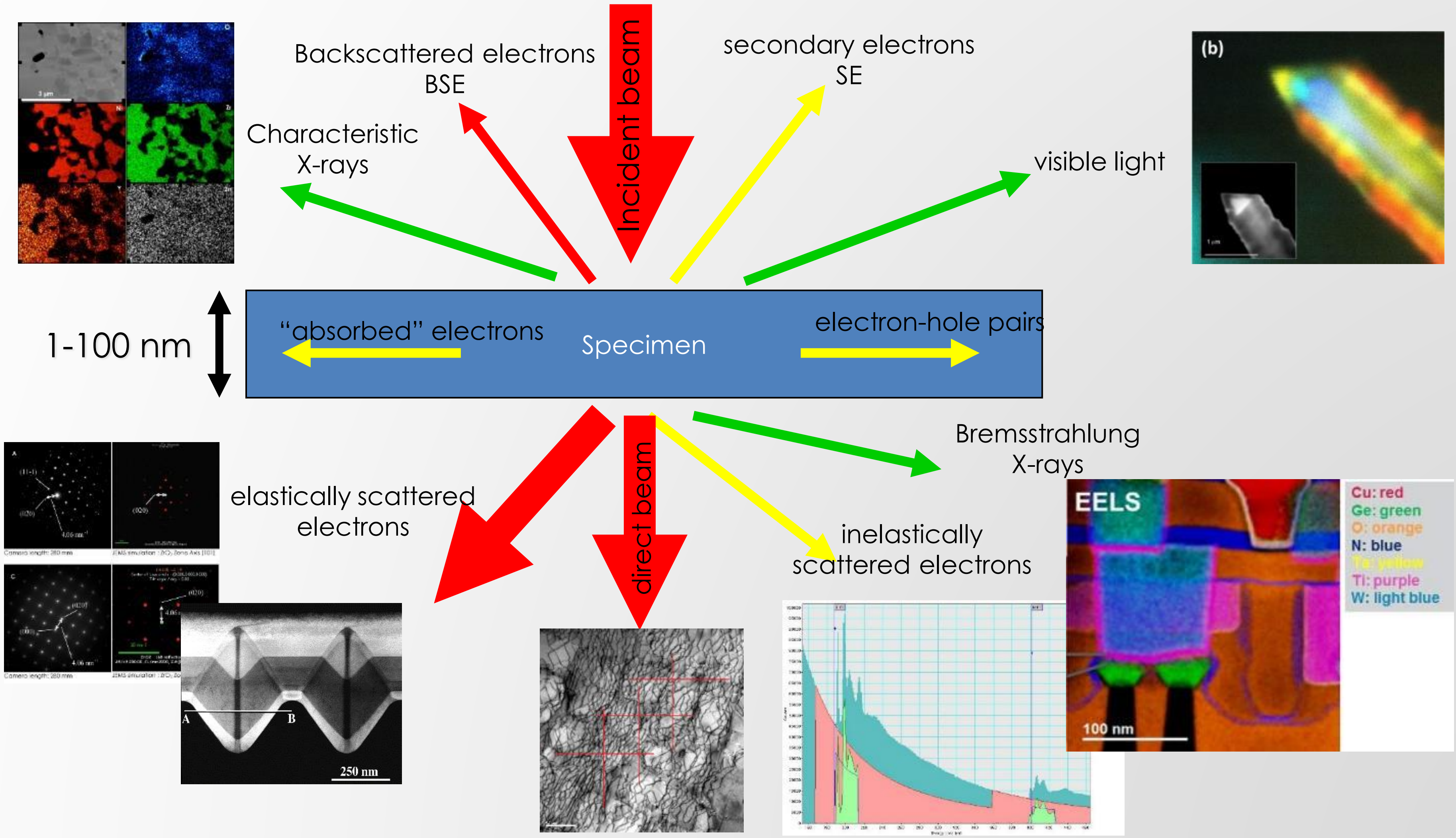
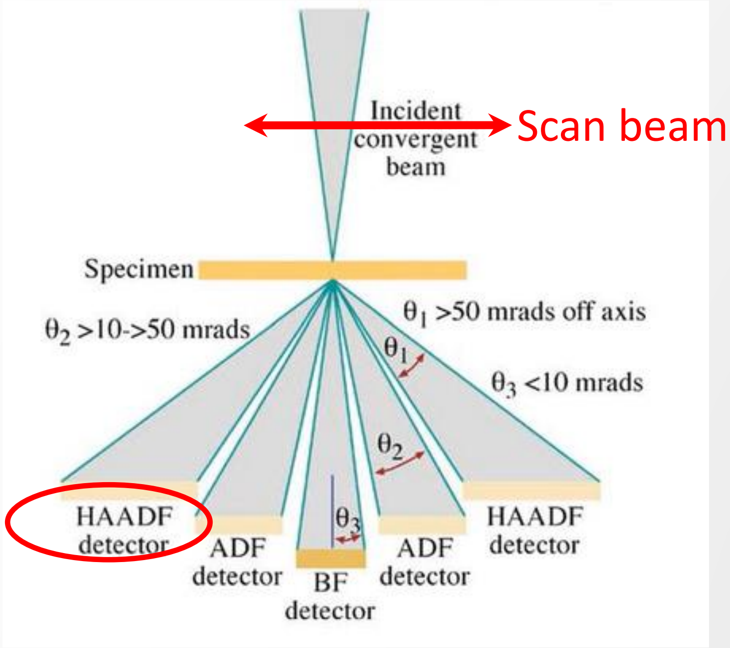
Electron-matter interactions in a thin sample



Electron-matter interactions in a thin sample: Incoherent elastic scattering - STEM

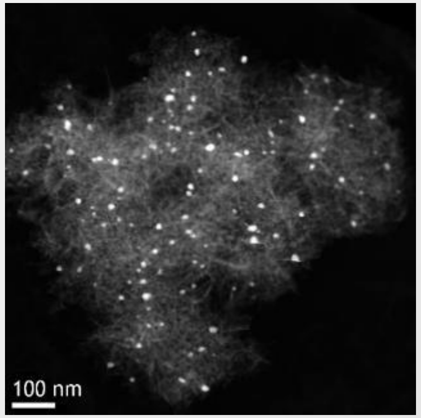
Focused e$^-$ probe scanned on sample; disc and annular detectors in back focal (diffraction) plane



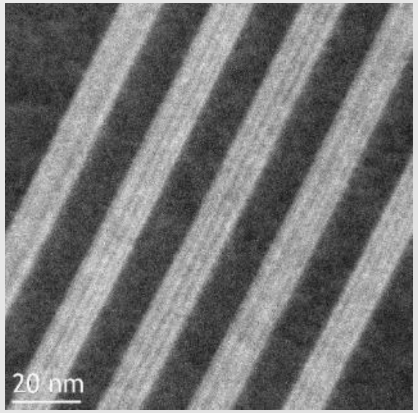
EELS spectrometer

High-angle annular dark-field => compositional contrast:
intensity ∝ Z^2t
(thickness *t*, atomic number *Z*)

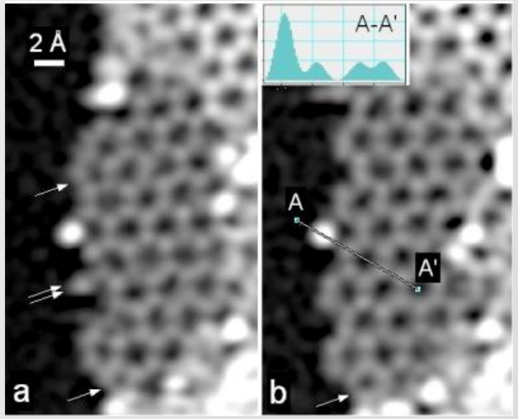
Z-contrast examples:



Pt catalyst on Al_2O_3

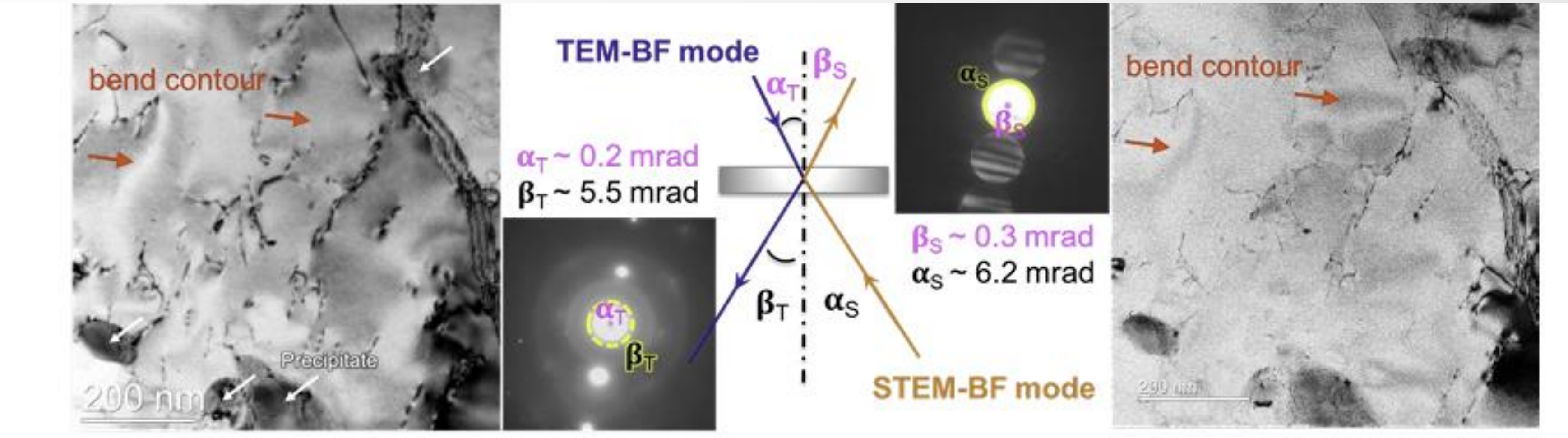


Si-Ge/Si multilayer

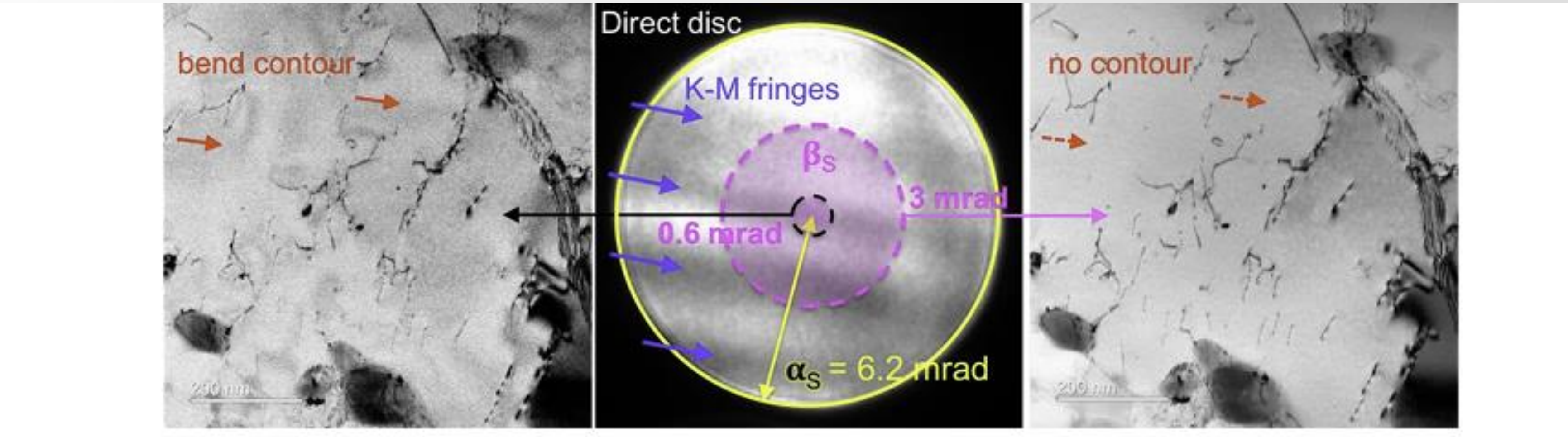


Cs-orcrected - graphene with dopant atoms (Krivanek et al., Nion)

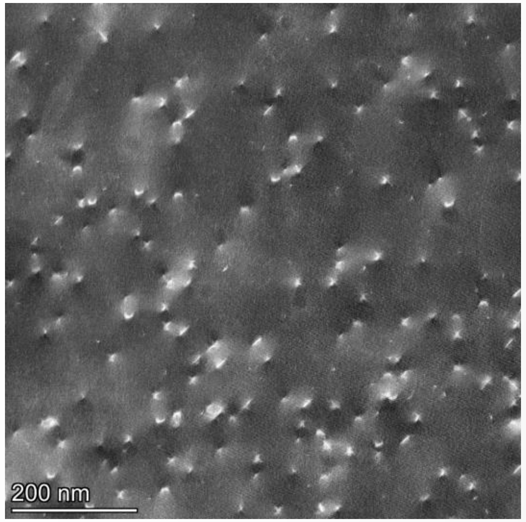
STEM imaging mode suppresses bend contours

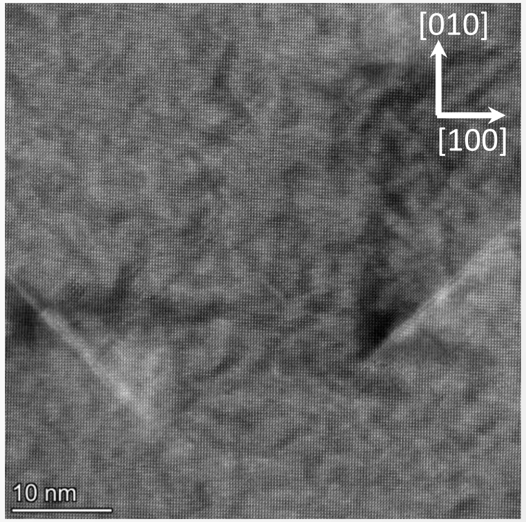


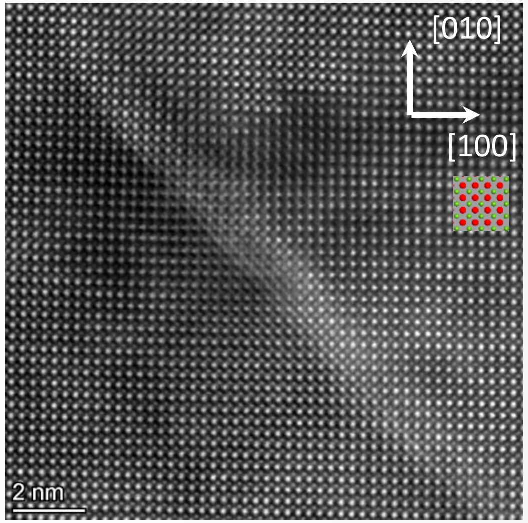
Bend Contour contrast is suppressed when $\beta_S \approx \alpha_S$

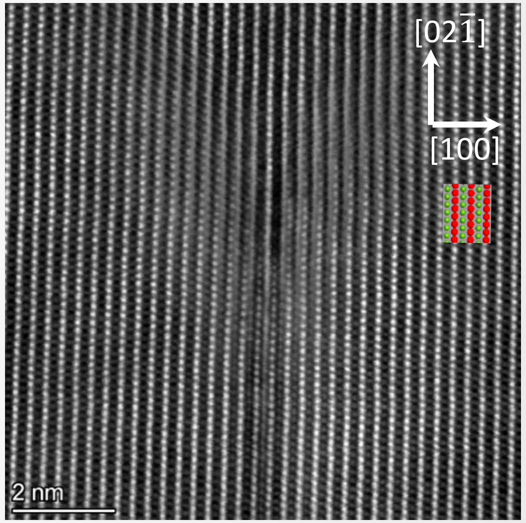


HR-STEM images of the Dislocations

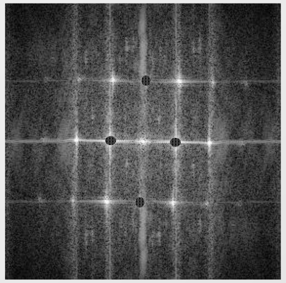


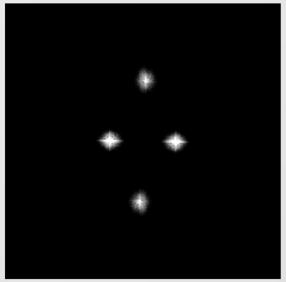




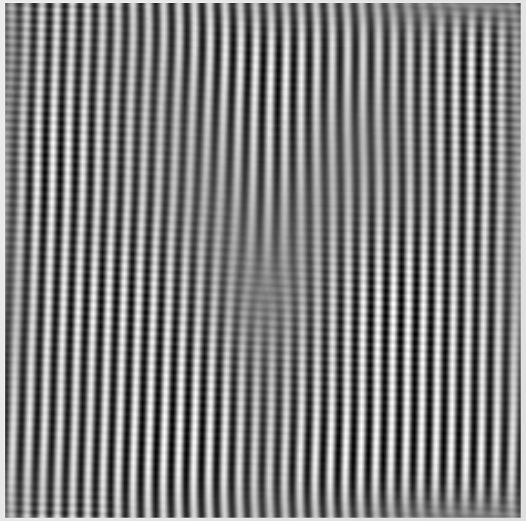


Fourier Filtering

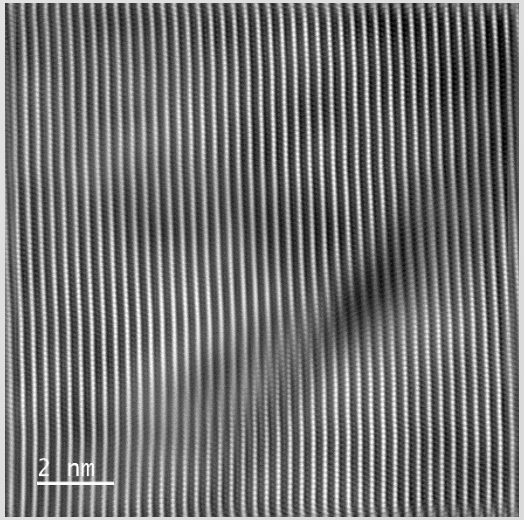




$\vec{b} = a[100]$



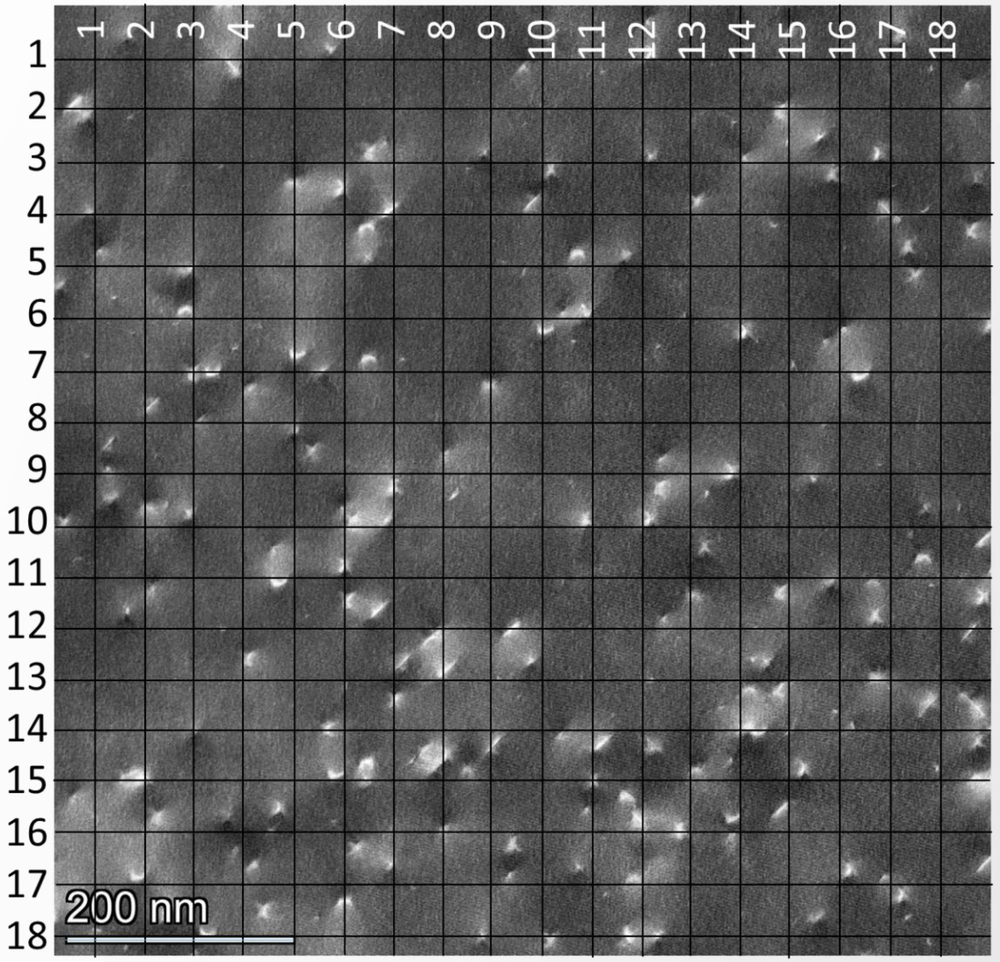
$\vec{b} = a[111]$



Dislocation density measurements (Orowan equation)

HAADF $\beta_S(24mrad) \approx \alpha_S(20\text{mrad})$

$\Lambda \approx 5X10^9$ cm^{-2}



Line Intercept Method

- Make a grid having a total line length L
- Count the number of intersections between a given grid and the dislocations, N
- Measure TEM sample thickness, t (EELS, CBED, X-ray reflection, etc.)
- The dislocation density, Λ, is given by the following equation.

$$\Lambda = 2N/tL$$

- This method is valid for dislocation density in the metal is less than 10^{14} cm^{-2}.

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