Transmission Electron Microscopy of Semiconductors and Heterostructures

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Outline

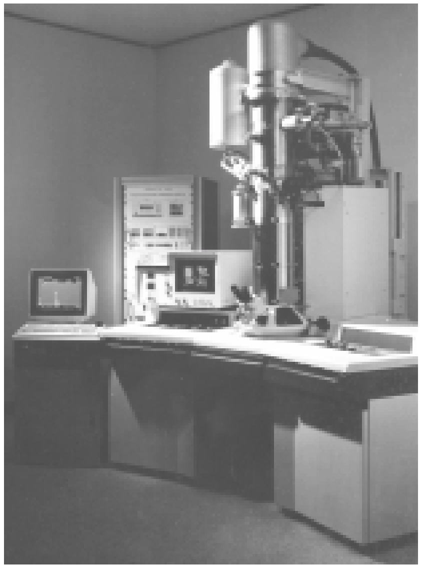
- Background
- Scattering theory
- Applications
  - Imaging (defects, interfaces, atomic structure …)
  - Diffraction (strain, polarity …)
  - Microanalysis (chemical composition etc)
- Recent developments

Transmission electron microscope (TEM)

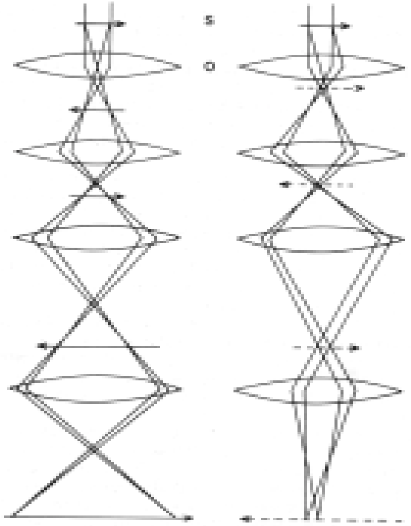
- Heated W, LaB6 or field emission source

- Electromagnetic lenses, giving direct imaging or diffraction using a parallel probe, or microanalysis using a focused probe

- Thin samples (10- 500 nm)



Hitachi HF2000 TEM

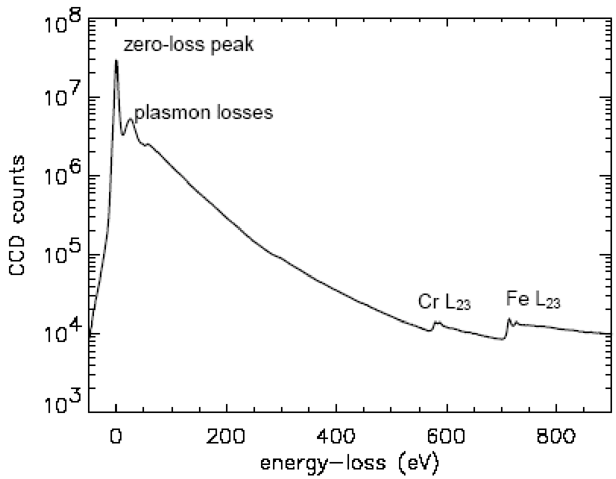


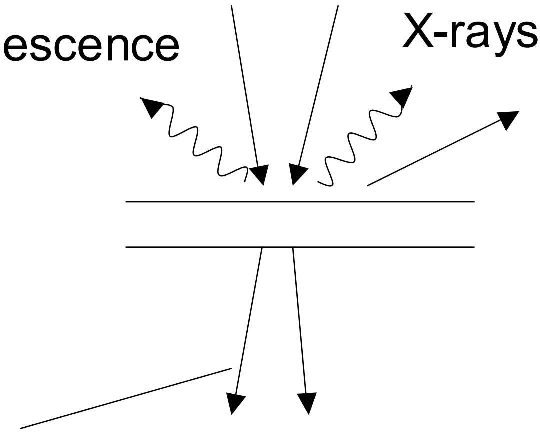
Imaging and diffraction!

Electrons as particles (200kV)

- Electrons travel at 0.7c (relativistic)
- Up to 10^{10} e/sec. Focused probe (field emission gun) can generate up to 1nA into 1nm probe, or greater than 10^{8} e/atom/sec
- An electron can transfer up to 44eV to a carbon atom in a head-on collision. This can generate point defects (bulk) and sputtering (surface)
- Less energetic collisions generate phonons, excitation of inner and outer shell electrons, plasmons and photons. This inelastic scattering gives microanalysis and imaging using a variety of signals
- Radiation damage can be a problem, with some organic materials damaging at down to 1 e/atom. Conversely there is potential for lithography and hole drilling

Inelastic scattering: some useful signals





Cathodoluminescence

X-rays (EDX)

Secondary electrons

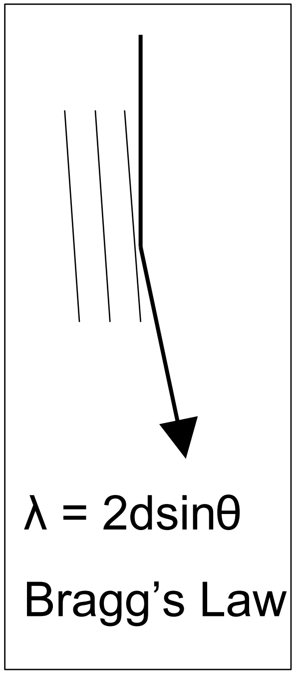
Electron energy loss spectroscopy (EELS)

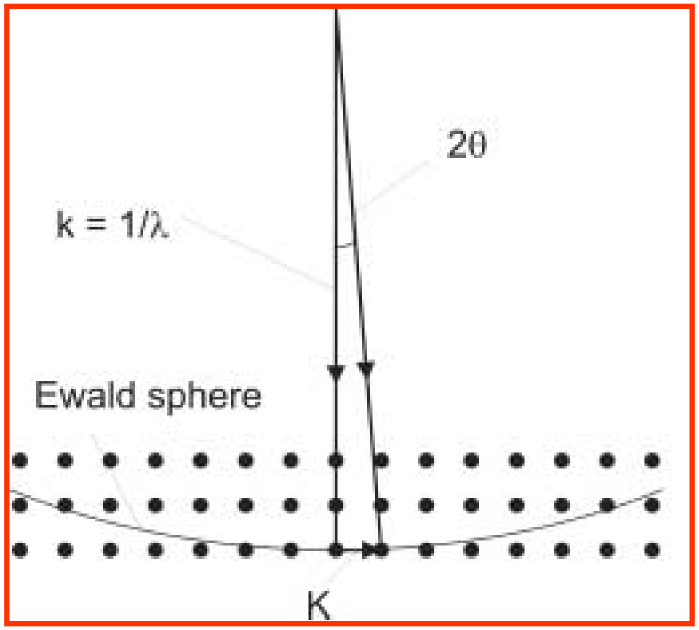
High angle annular dark field detection (HAADF)

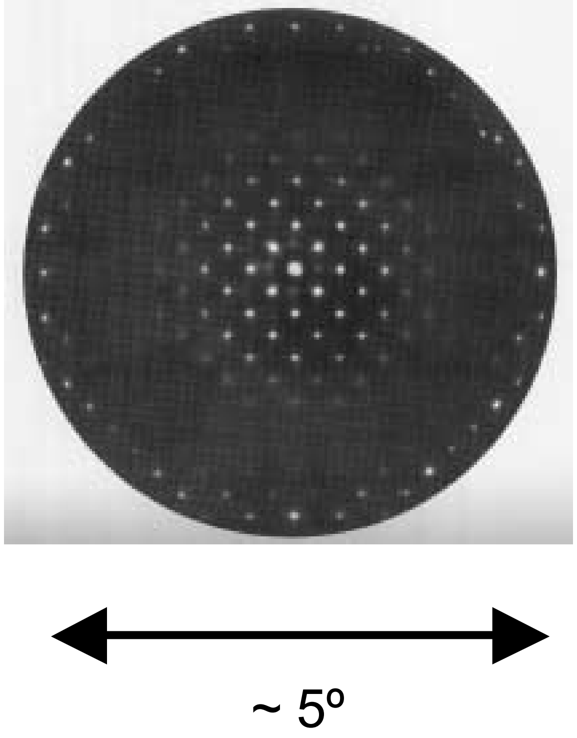
Electrons as waves: diffraction

λ = h/p (de Broglie) = 0.0025nm (200kV)

c.f. λ = 0.1 nm (X-rays), 500nm (light)



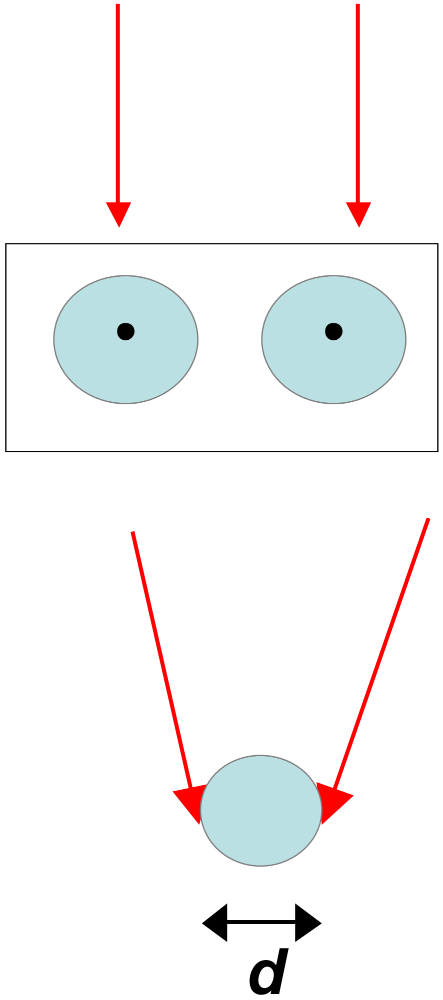




Spatial resolution

$$d = \lambda / \alpha$$

Abbe criterion (α = *convergence angle)*



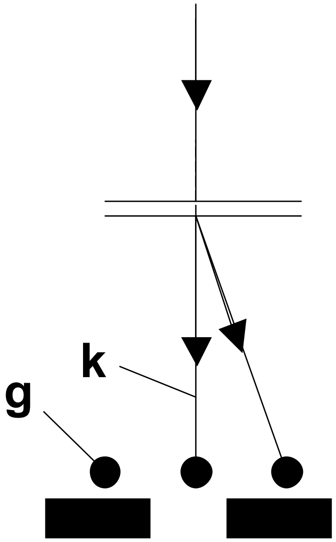
Light microscope: $\alpha \sim 1$ rad, d ~ λ

TEM: $\alpha \sim 10^{-2}$ rad, d ~ 100λ (0.2nm!)

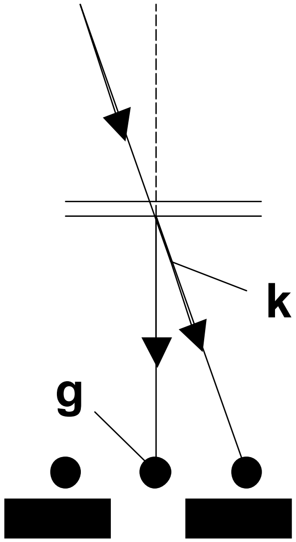
i.e. resolution is comparable to atom spacings, and α is comparable to the Bragg angle

Imaging modes

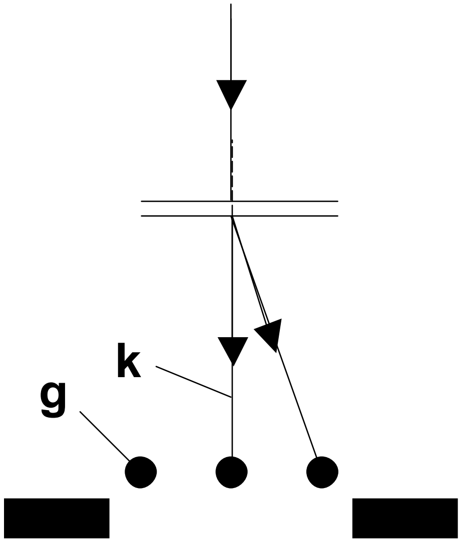
The presence of aberrations requires that imaged beams must be as close as possible to the optic axis. Selection is by means of an objective aperture



Bright field



Dark field

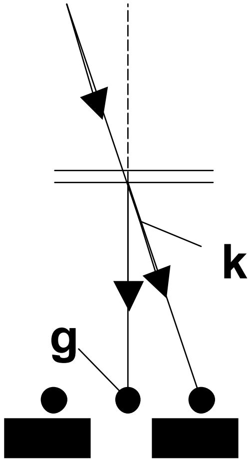


Lattice imaging

Scattering theory

Amplitude scattered into g (thin crystal limit):

$$\boxed{d\varphi_g / \varphi_0 = i\pi\Delta t / \xi_g}$$



Δt = specimen thickness

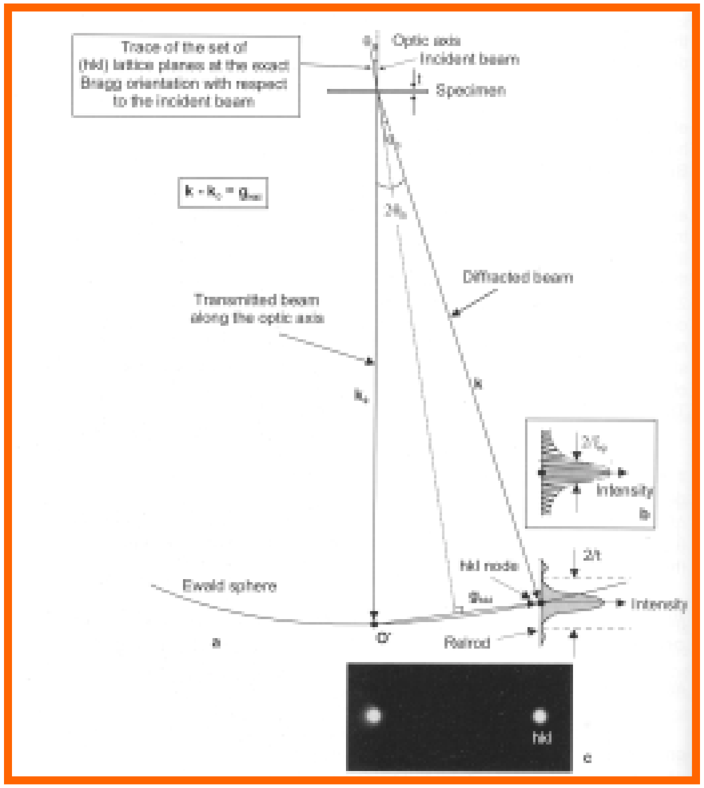
Φ = amplitude

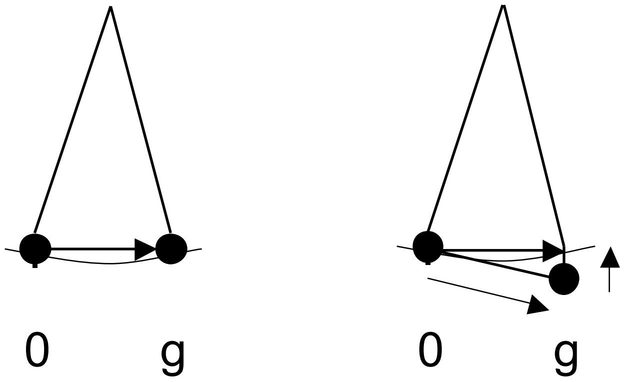
ξ_g = extinction distance

For electrons, ξ_g ~ 10 – 100 nm

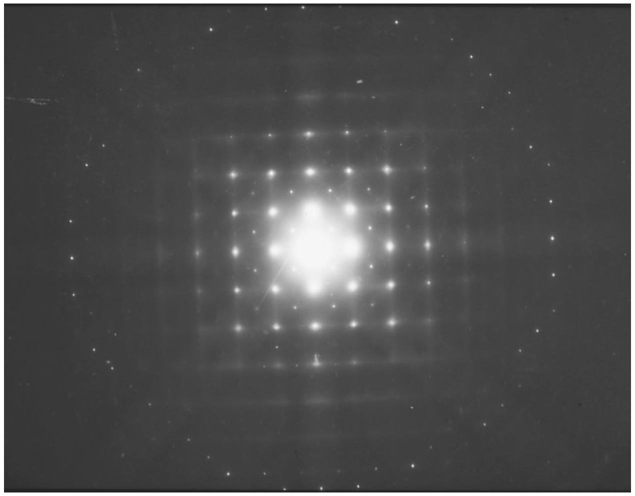
For X-rays, ξ_g ~ 2-3 orders of magnitude greater

TEM: why so many reflections?





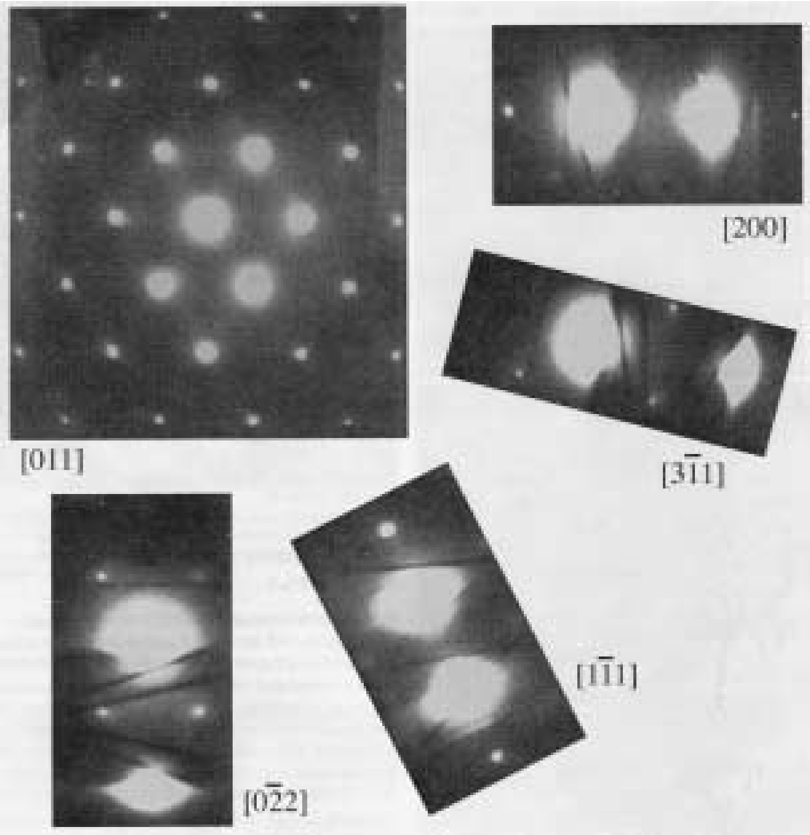
K = g **K = g + s**



Two-beam imaging

In general, electron diffraction is a **many beam problem**

Fortunately, it is possible to orient a single crystal sample until **only one diffracted beam is strong**. Understanding diffraction is then a relatively simple two-beam problem:



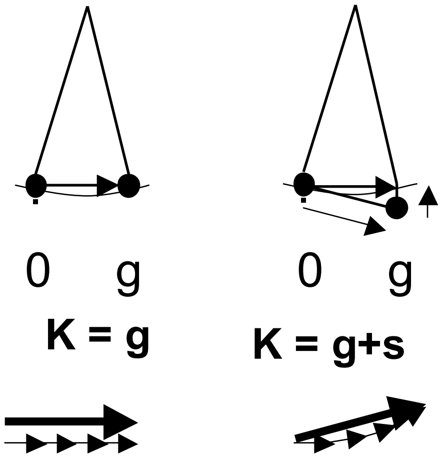
Two-beam imaging: significance of “deviation parameter” s

Large s is simple (kinematical):

$$\phi_g / \phi_0 = i\pi / \xi_g \int_t^0 \exp(-2\pi isz)dz$$

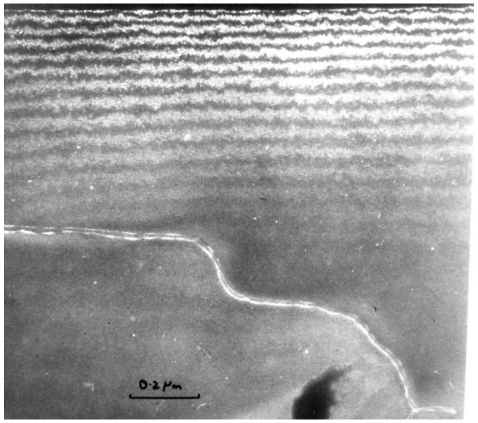
$$\phi_g^{\,2} / \phi_0^{\,2} = \pi^2 / \xi_g^{\,2} \; \sin^2(\pi ts) / (\pi s)^2$$

$$\Delta t = 1/s$$

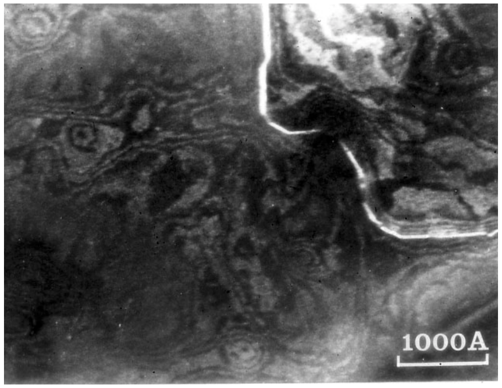


?

s = 0



s = 0.2nm^{-1}



s = 1.3nm^{-1}

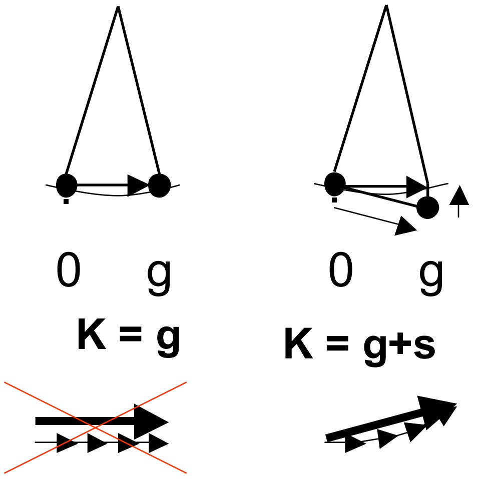
(surface steps)

Two-beam imaging: significance of "deviation parameter" s

s = 0: behaviour is dynamical:

$$\frac{\phi_g^{\,2}}{\phi_0^{\,2}} = \frac{\pi^2}{\xi_g^{\,2}} \, \frac{\sin^2 \pi t\sqrt{s^2+\xi_g^{\,-2}}}{\pi^2(s^2+\xi_g^{\,-2})}$$

$$\Delta t = 1\Big/\sqrt{s^2+\xi_g^{\,-2}}$$

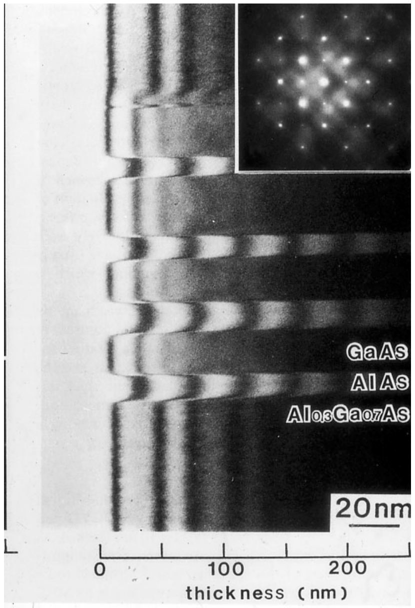


0 g

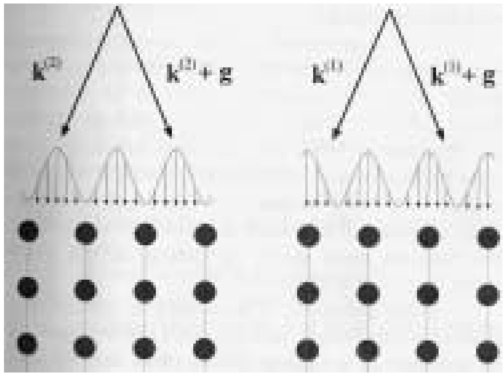
K = g

0 g

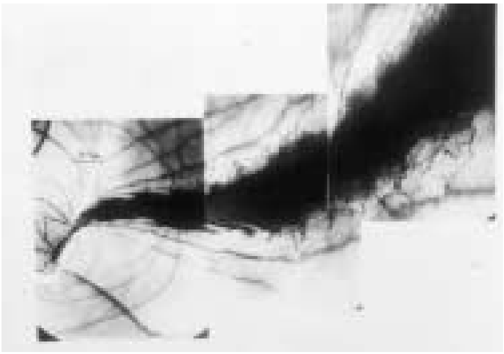
K = g+s



s = 0, Δt = 1/s = ξ_g



Bloch waves



Channelling

Two-beam imaging: defects

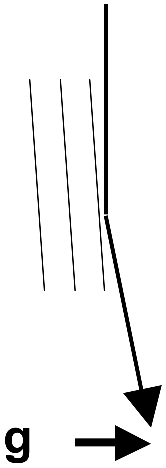
A good qualitative understanding of contrast can be obtained using the kinematical formula

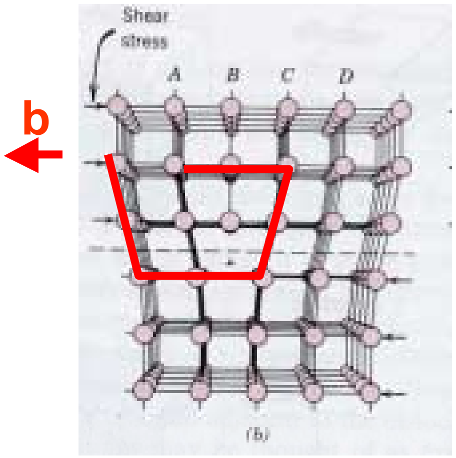
$$\phi_g / \phi_0 = i\pi / \xi_g \int_t^0 \exp(-2\pi i(sz + \underline{g}.\underline{R})dz$$

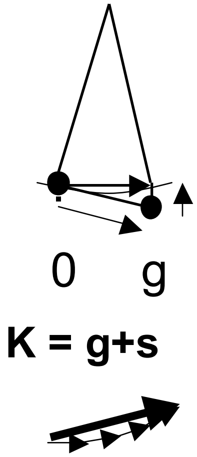
e.g. for dislocations **g.R** defines bending of diffracting planes

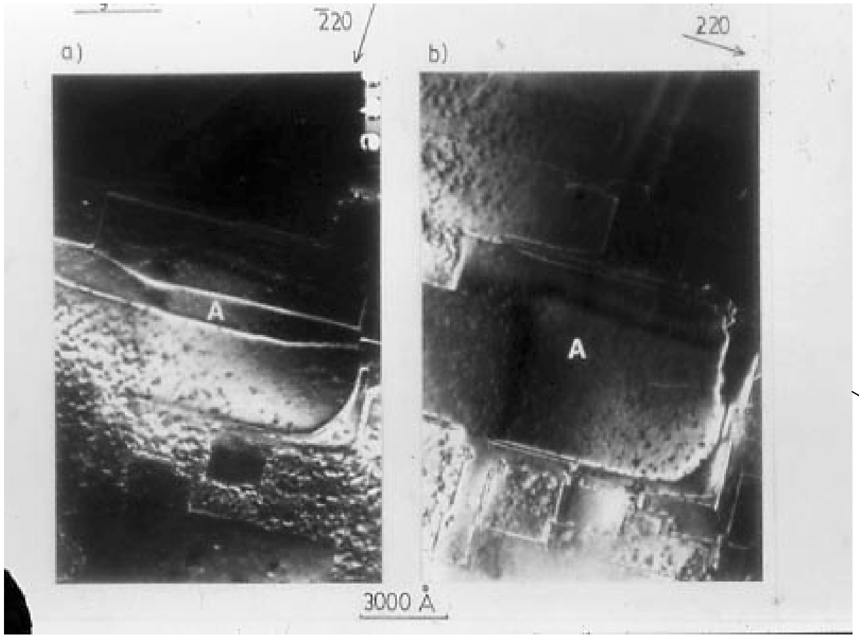
Two-beam imaging: defects

Dislocations are seen when the diffracting planes are distorted, i.e. when the dot product **g.b** is non-zero





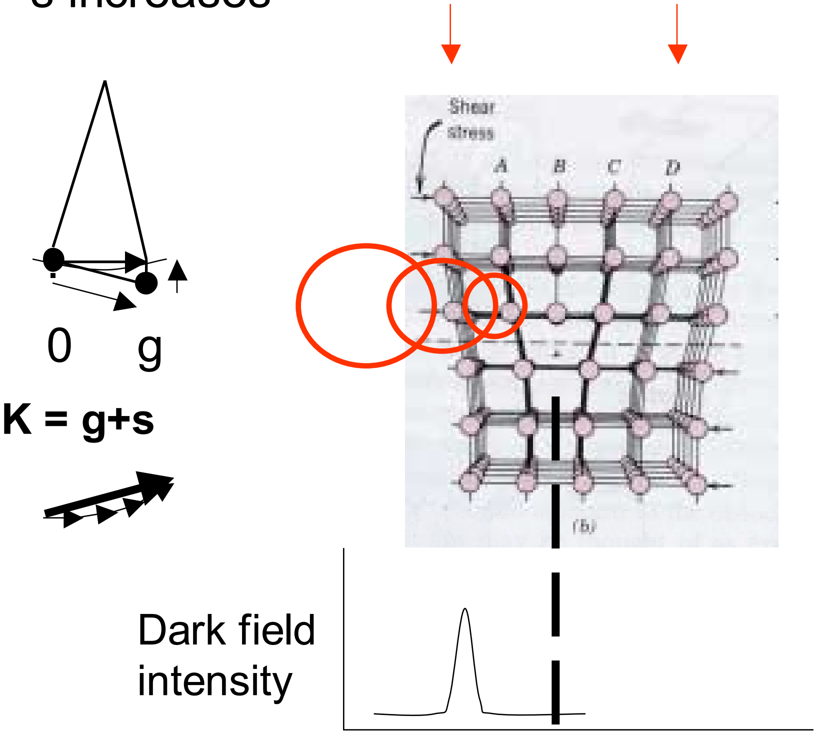


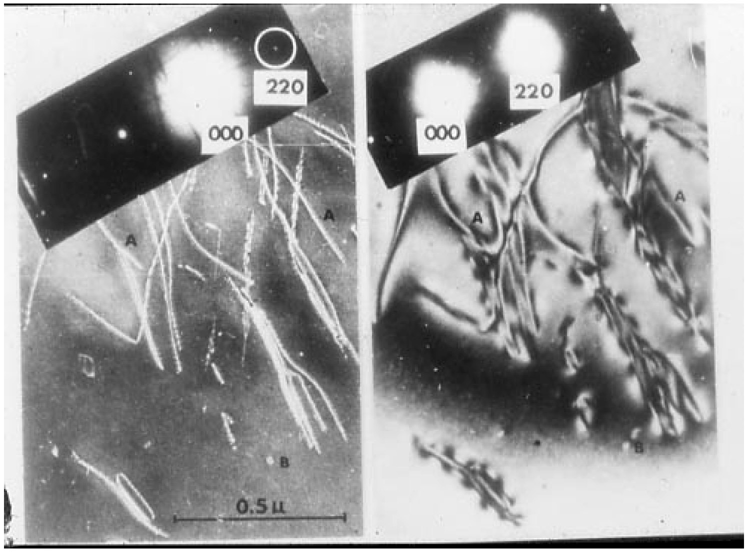


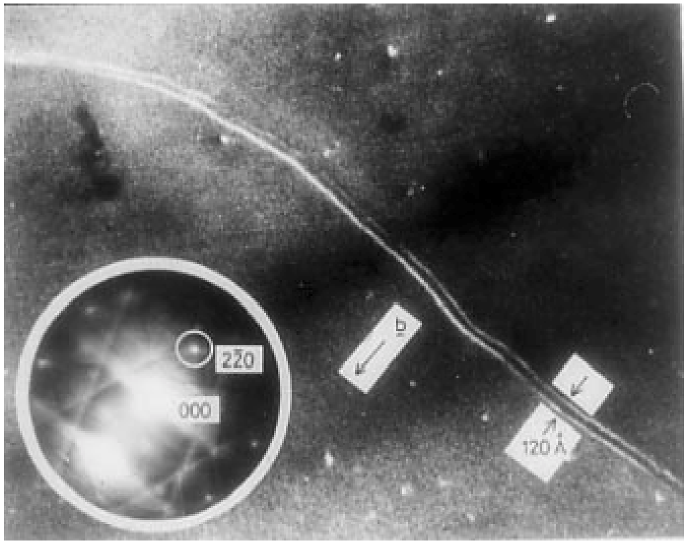
Analysis of misfit dislocations in $NiSi_2$/(001)Si interface

Core structure of dislocations: weak beam technique

Image is seen where planes are bent towards s = 0, i.e. progressively closer to the core as s increases



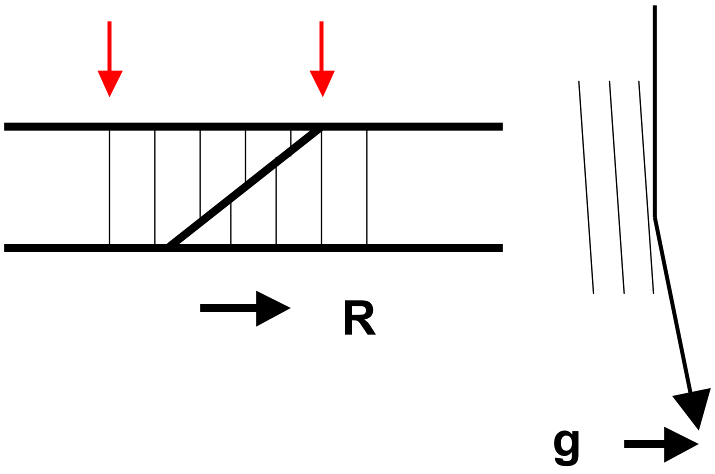


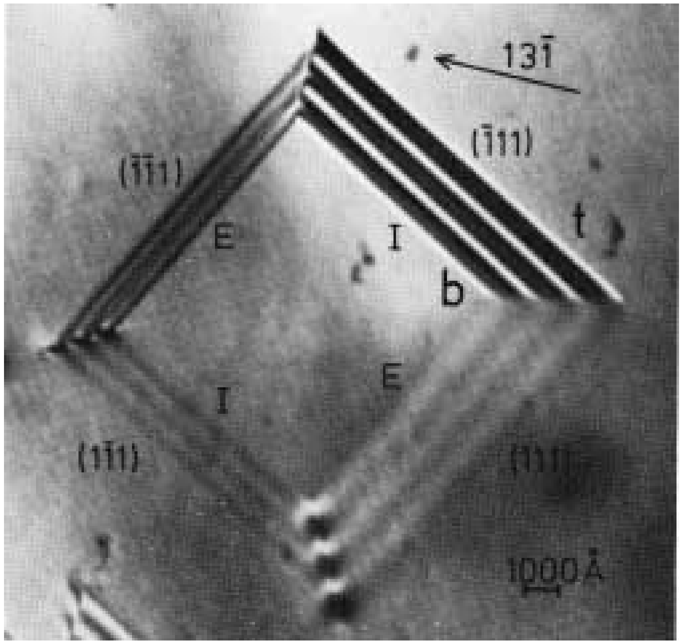


Dislocations in semiconductors are often dissociated

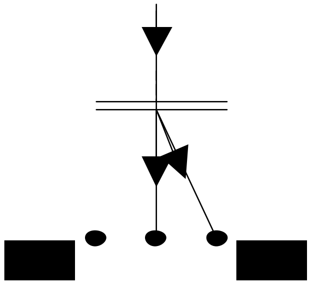
Two-beam imaging: defects

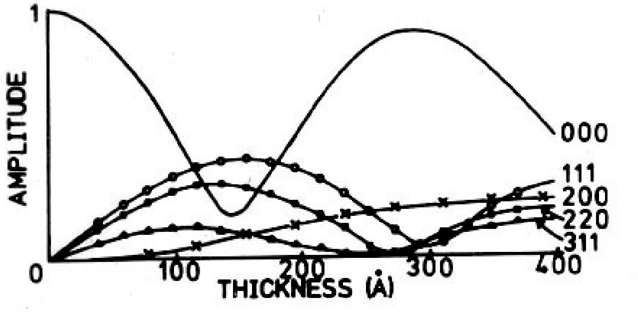
Stacking faults are visible when the diffracting planes are fractionally displaced, i.e. contrast depends on **g.R**





Lattice imaging: many (strong) beams

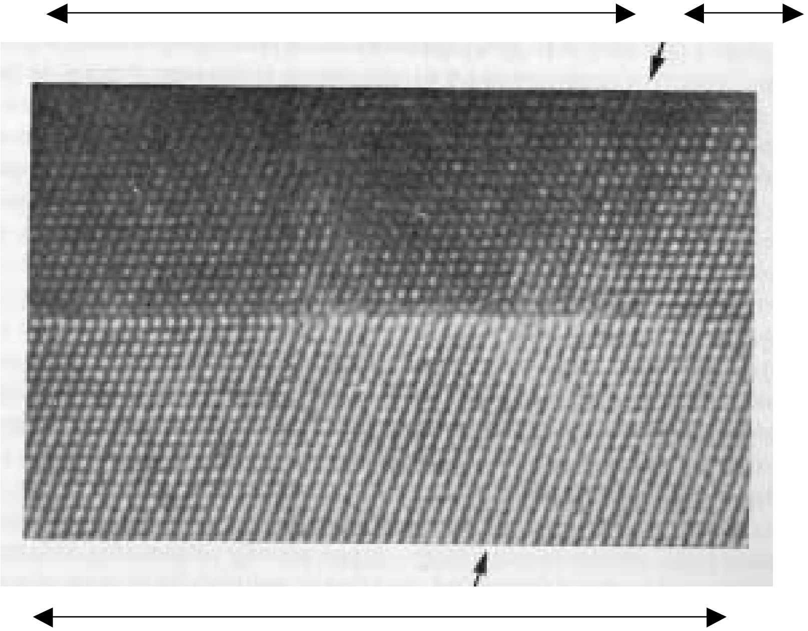




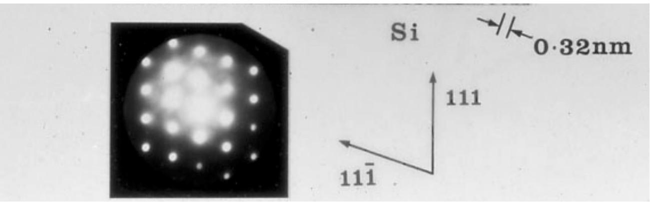
Scattered amplitudes from Si viewed along [110] as a function of film thickness. Phases vary also!

white atoms

black atoms



black atoms



NiSi2/(111)Si

Can we believe what we see?

Lattice imaging

Hence two problems:

- Seeing is not believing
- Limited resolution described by the contrast transfer function
- However, with computation many problems can be solved

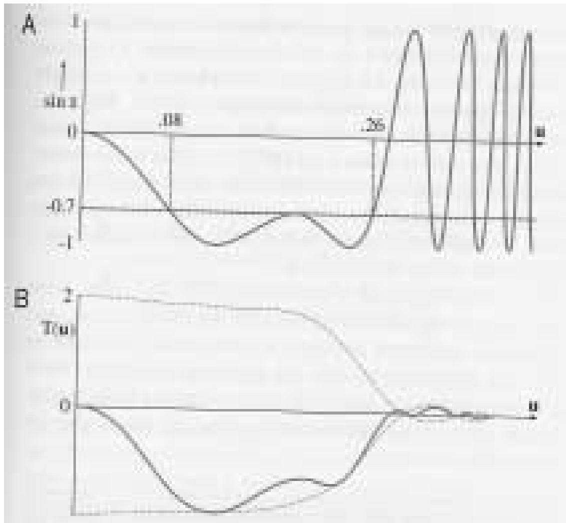
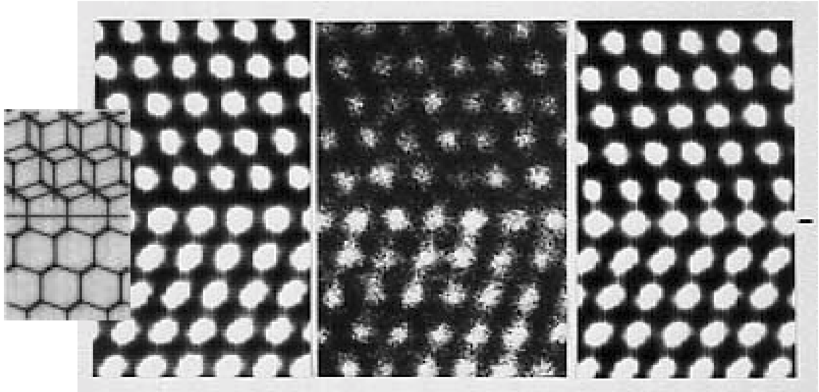
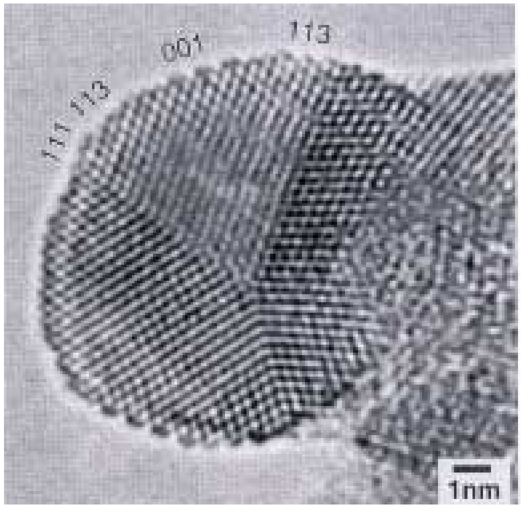


Figure 28.7. (A) sin χ(u) versus u without damping of the higher spatial frequencies. (B) T(u) versus u modified by the damping envelope (dashed line); $\Delta f = -100$ nm, $C_s = 2.2$ mm.



“B” NiSi2/(111)Si along [110]



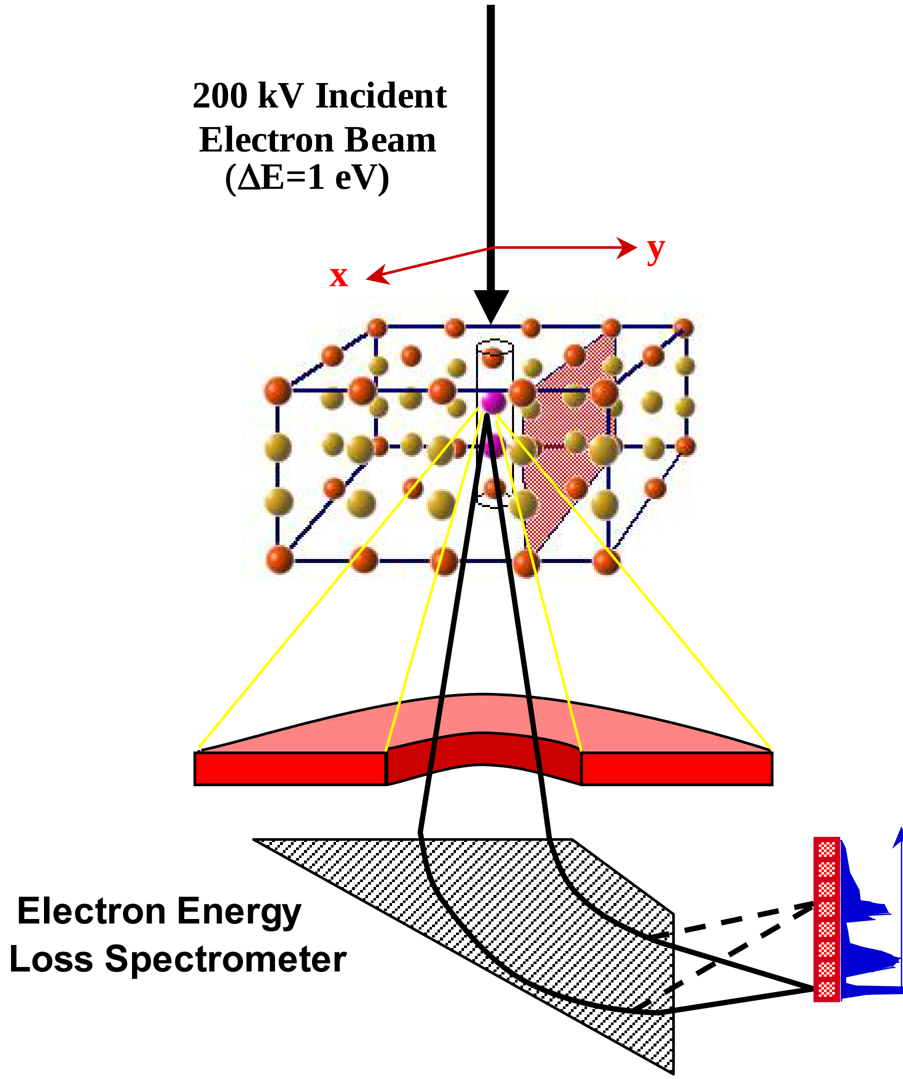
Si nanocrystal (Takeguchi JEM 48, 1087)

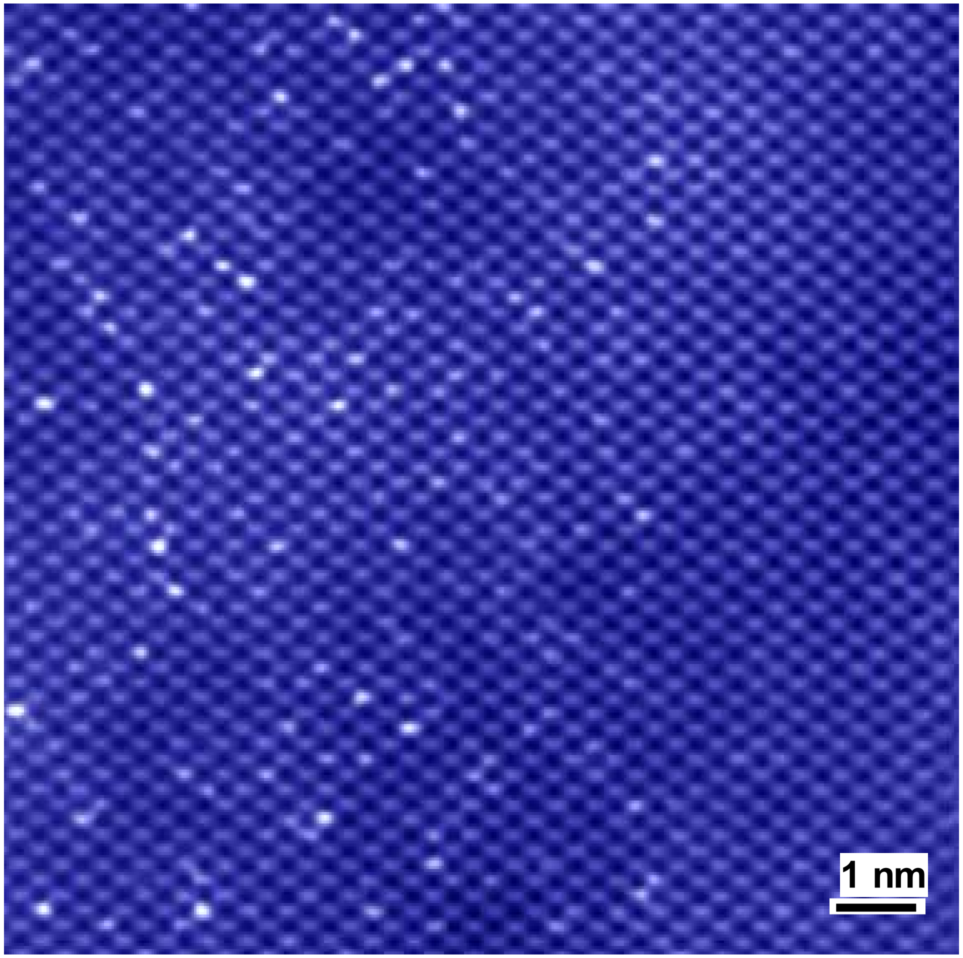
Lattice imaging

Current advances:

- Resolution improvements from 0.2nm to better than 0.1nm through aberration correction
- Smaller focused probes
- Improved resolution of structure (e.g closely spaced atoms in semiconductors), lattice imaging by scanning TEM (STEM) using chemically sensitive signals

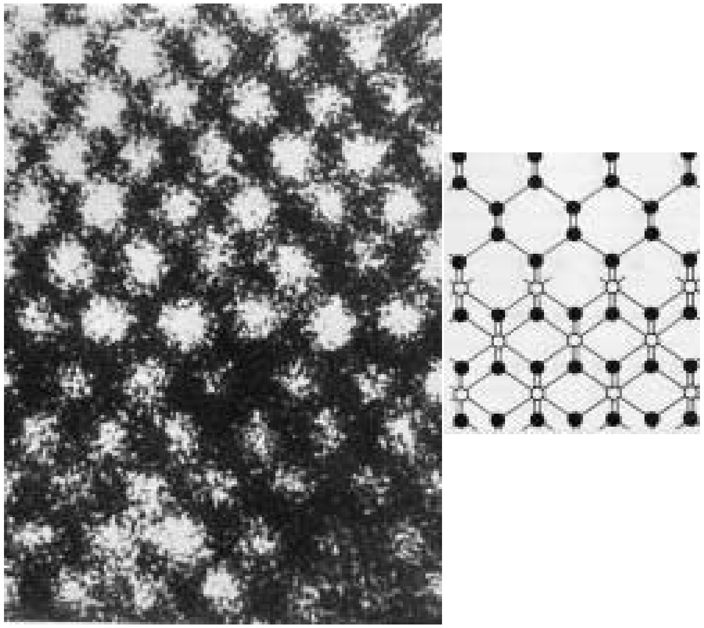
HAADF: Sb dopants in Si
(courtesy of D. Muller)



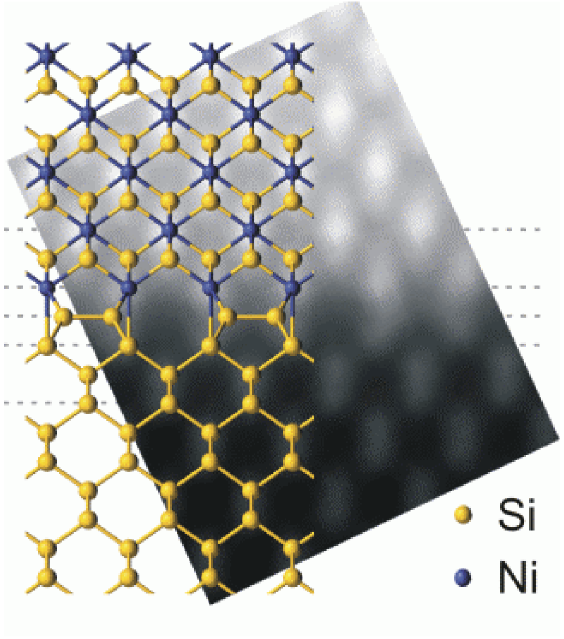


U. Kaiser, D. Muller, J. Grazul, M. Kawasaki, *Nature Materials*, **1** 102 (2002)

NiSi2/(001)Si 1984 - 2004

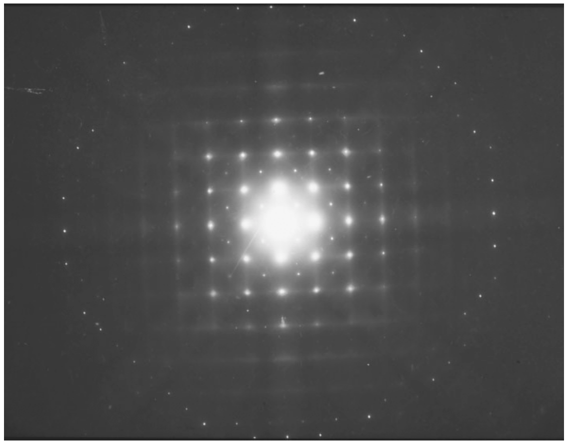


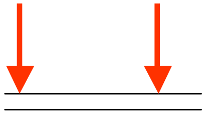
Direct image



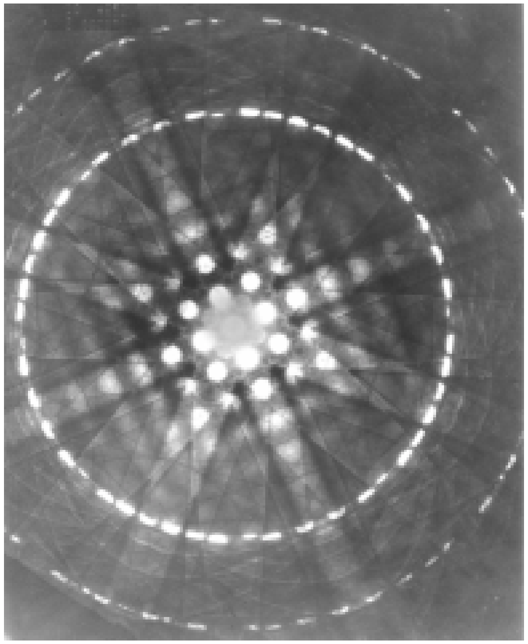
HAADF image, courtesy of A. Bleloch showing higher resolution and chemical sensitivity

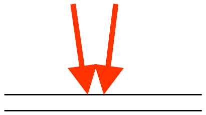
Convergent beam electron diffraction



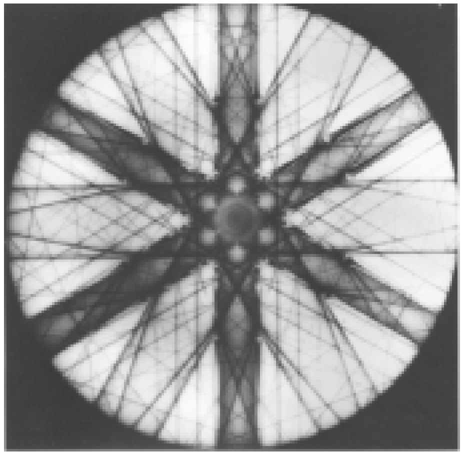


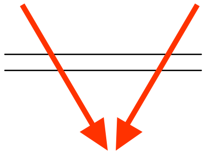
Selected area
diffraction





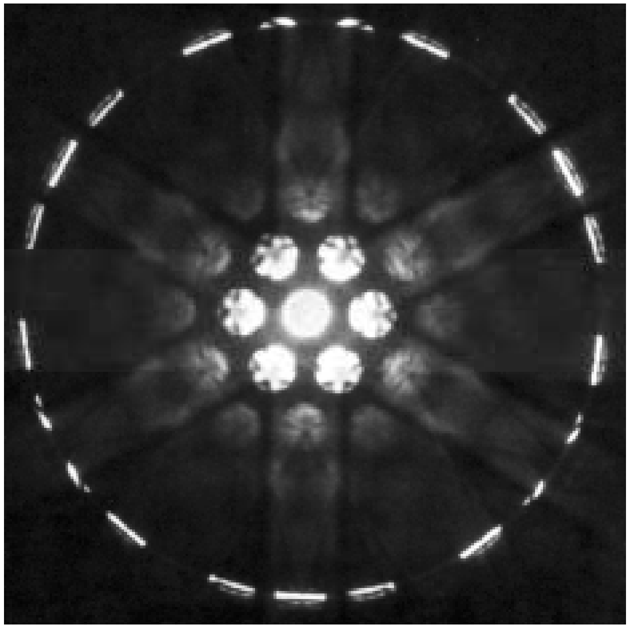
CBED

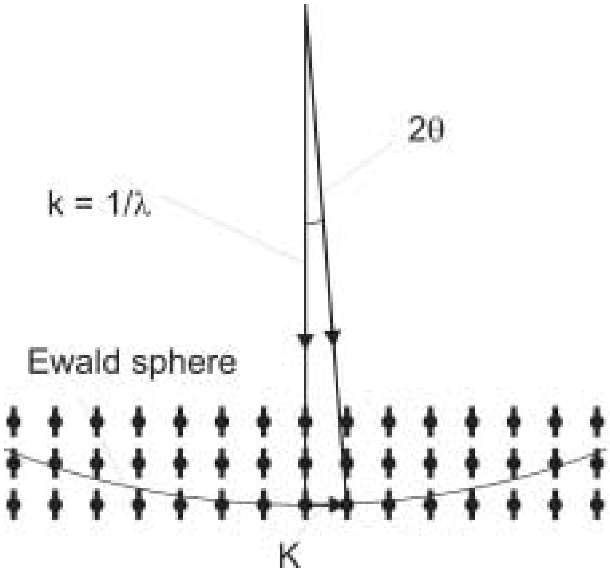


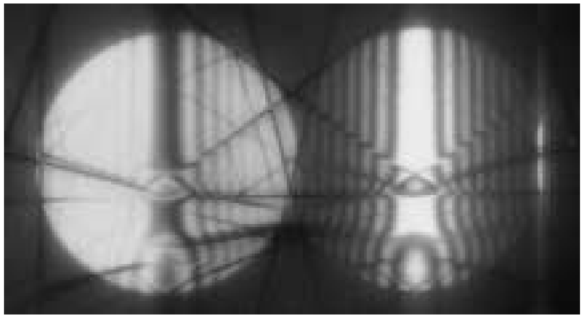


LACBED

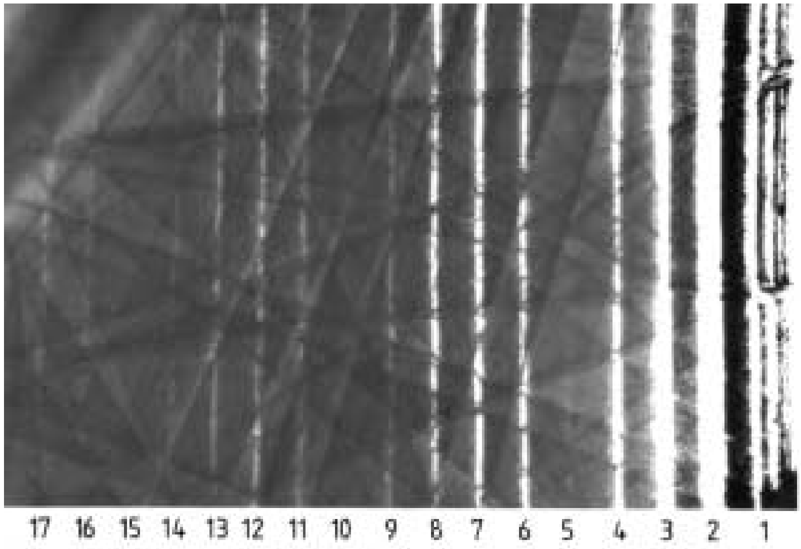
Electron rocking curves





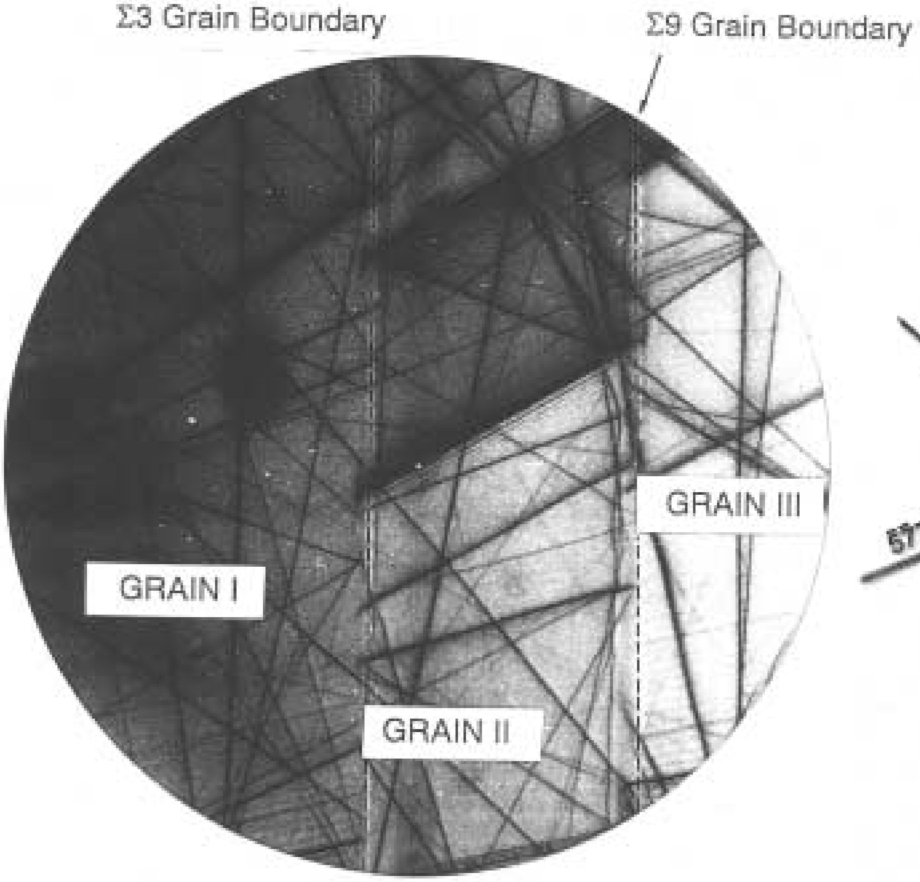


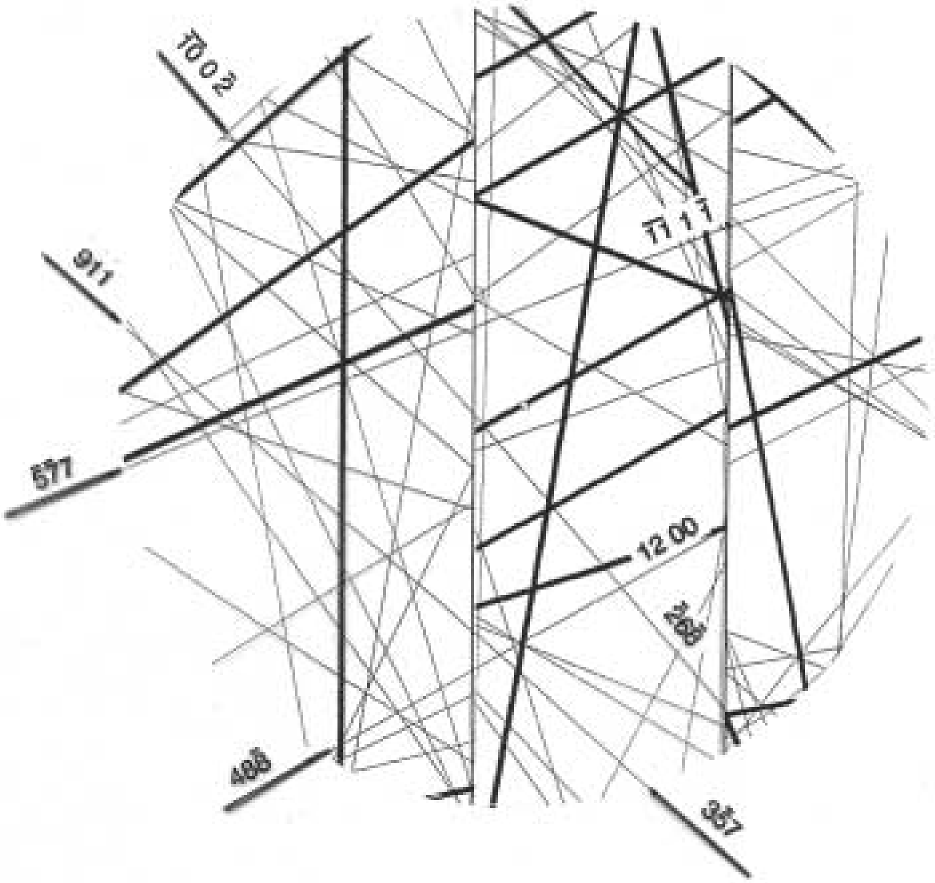
CBED Si 220



LACBED InP/InGaAs MQW 200 disc (Vincent et al Inst Phys Conf Ser 90, 233 (1987)

High order (weak) reflections: grain boundaries in Si



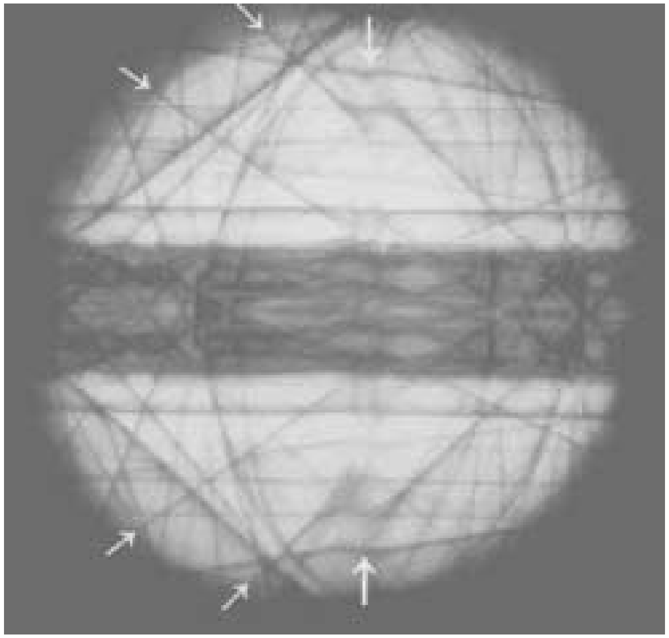


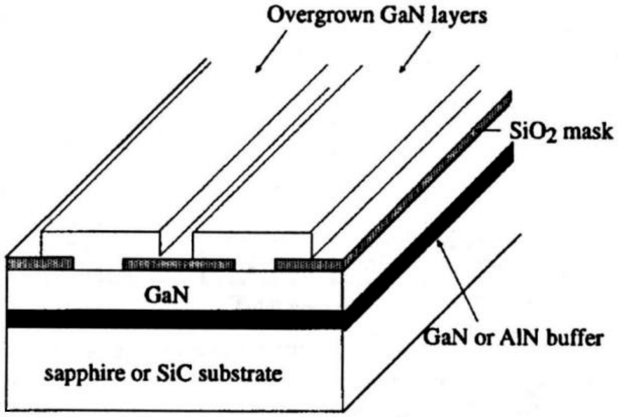
J-P Morniroli and D. Cherns, Ultramicroscopy **62**, 53 (1996)

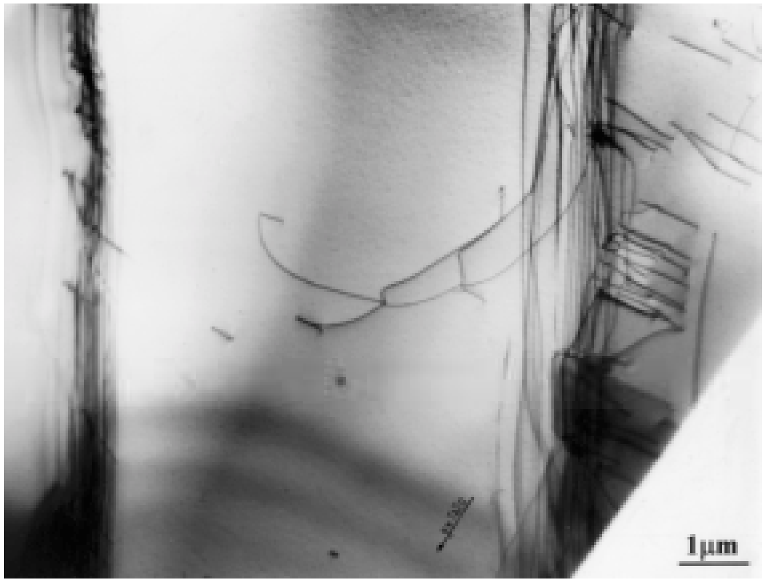
High order reflections:

Rotation of wings in GaN ELOG structures

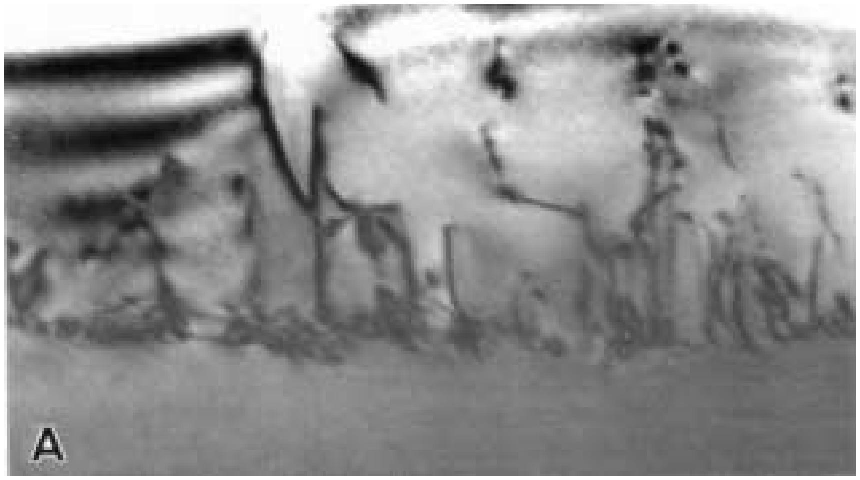
Z Liliental-Weber and D Cherns JAP **89** 7833 (2001)

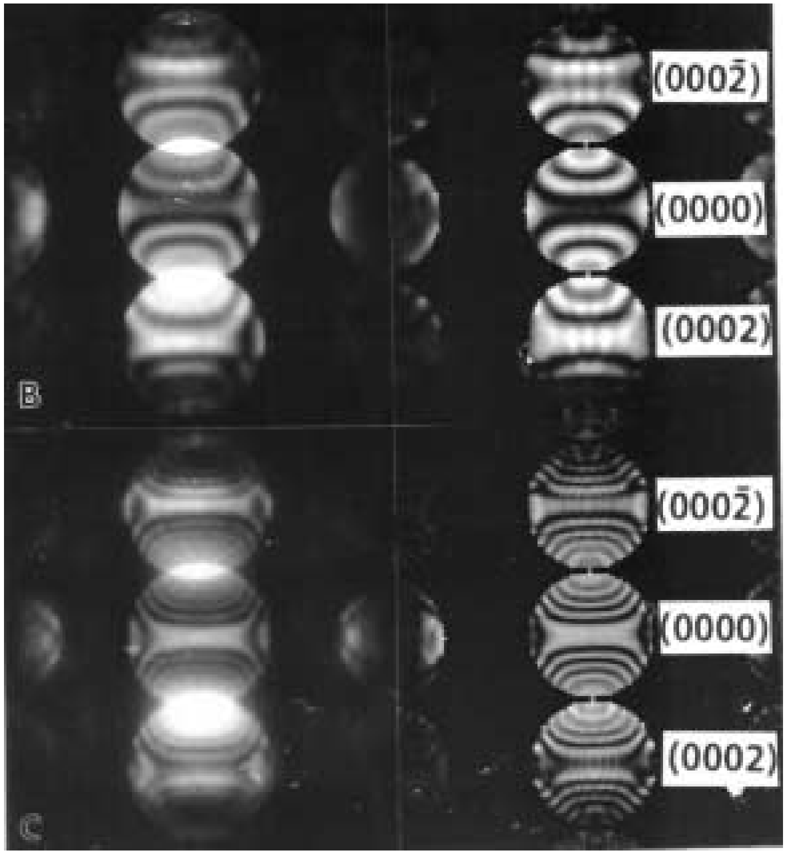






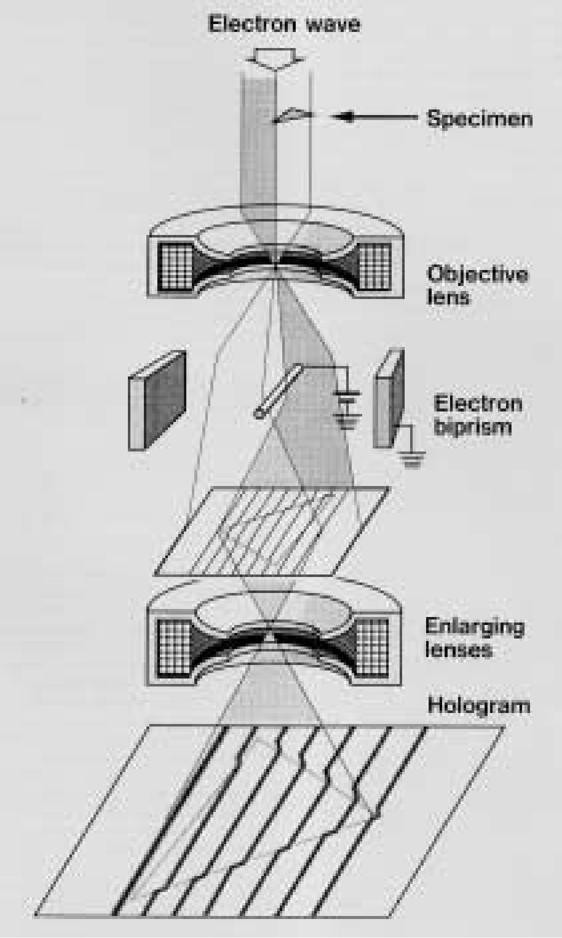
Low order (strong) reflections: polarity determination in GaN/GaN bicrystals

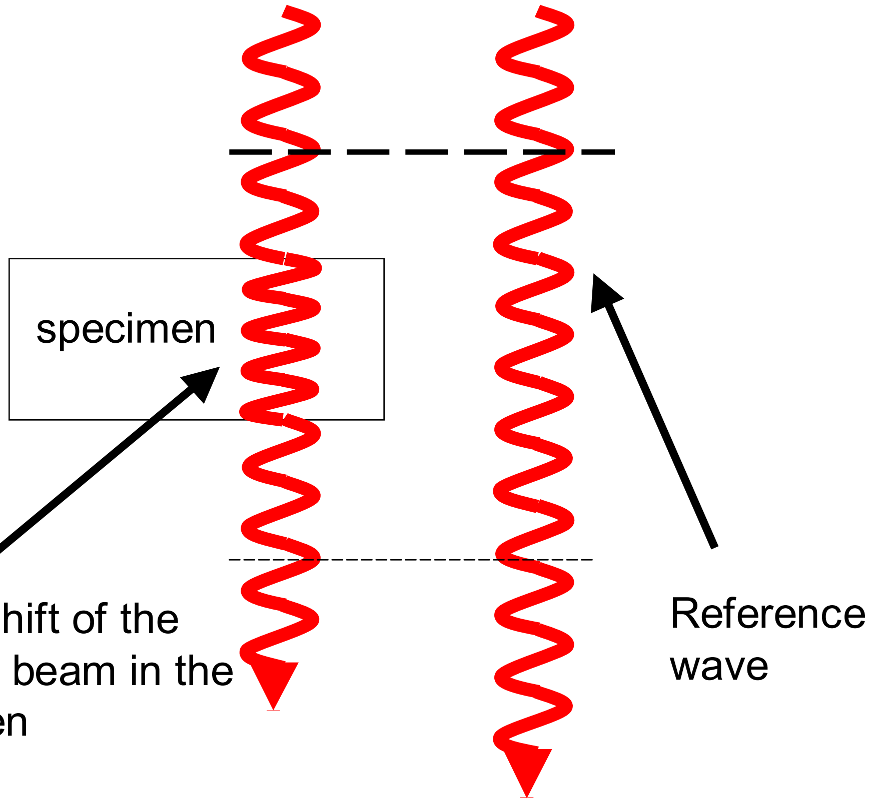




Asymmetry in the CBED patterns is a dynamical effect depending on double diffraction between 0002 and 000-2 reflections. It represents breaking of Friedel's Law…

Electron holography





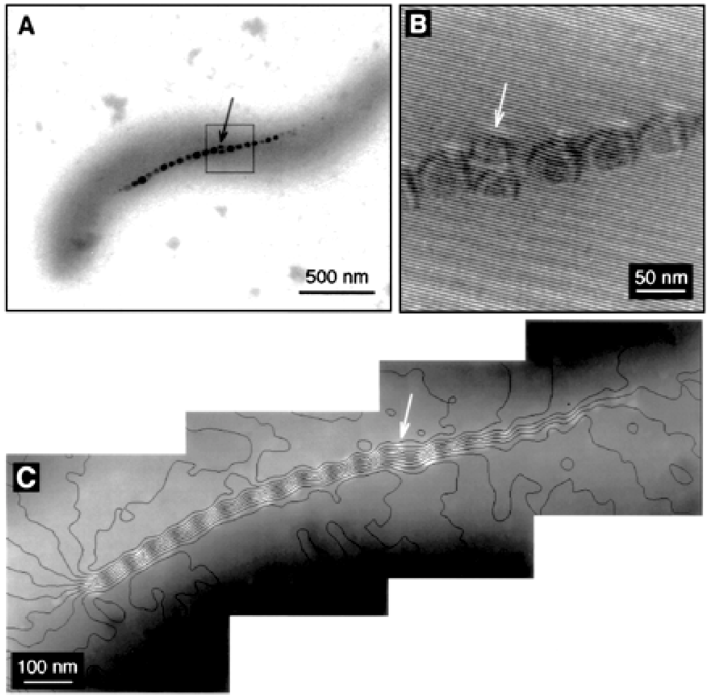
specimen

Phase shift of the electron beam in the specimen

Reference wave

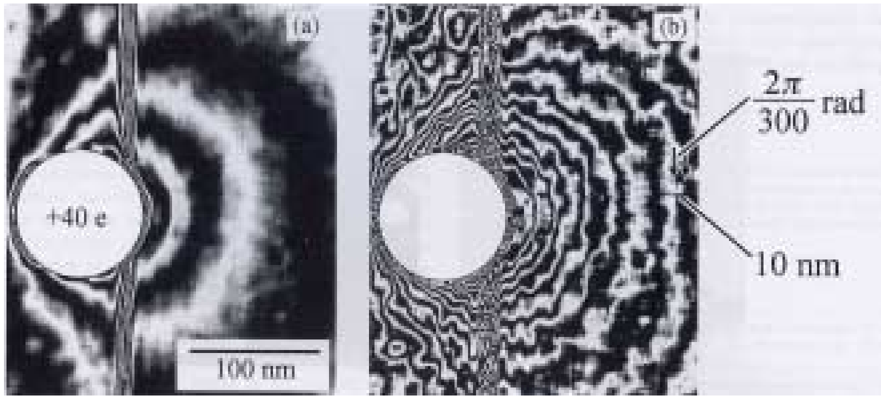
Phase shift depends on the “inner potential”, which can include contributions from internal (and external) magnetic or electric fields

Examples of holography

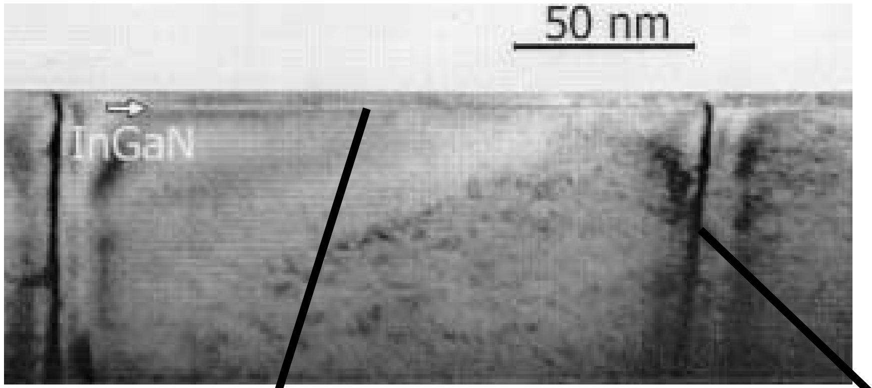


RE Dunin-Borkowski et al: electron holography of magnetotactic bacteria, Science 282 (1998) 1868

Phase map around a charged latex sphere (K Yamamoto et al, JEM 49 (2000) 31)

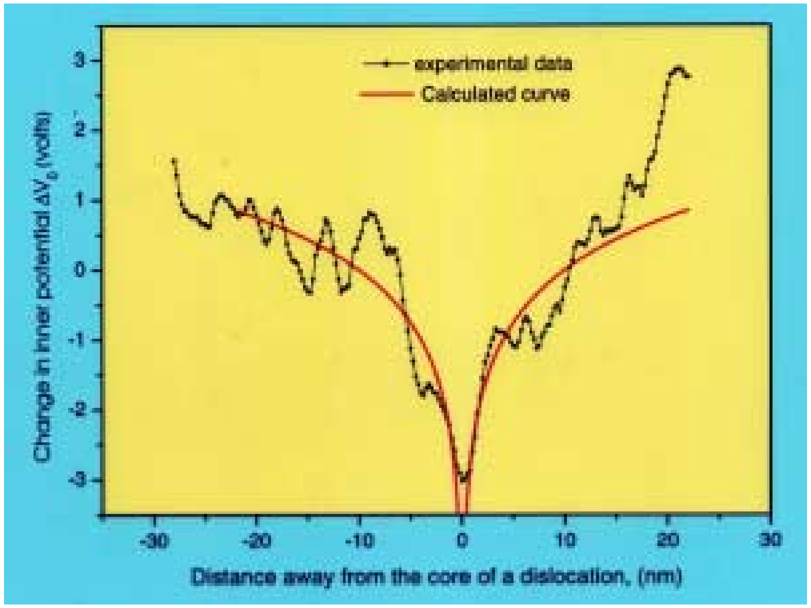


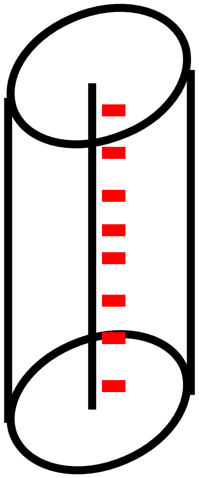
Electric fields in GaN/InGaN LEDs



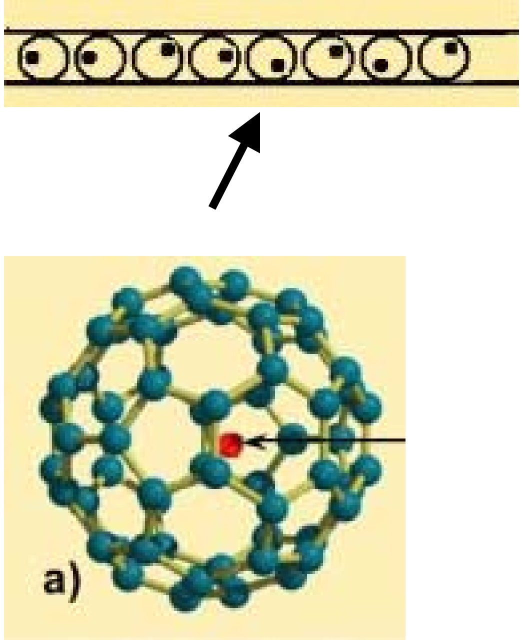
Cherns, Barnard and
Ponce: Solid State
Comm. **111**, 281 (1999)

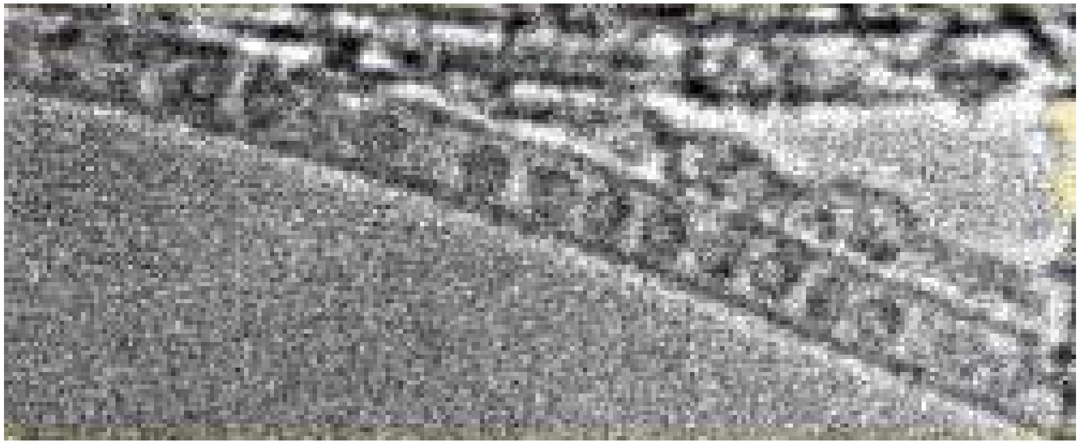
D. Cherns and C. Jiao
PRL **87**, 205504 (2001)

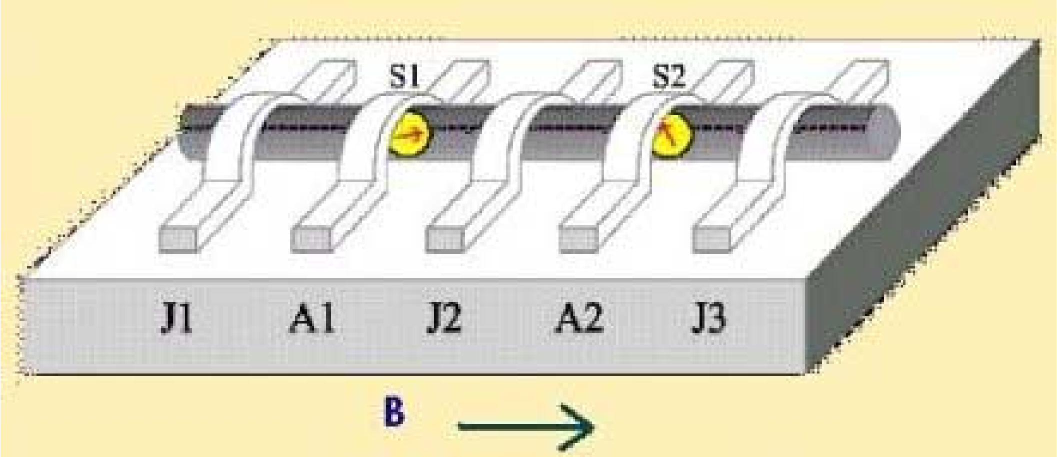




A problem requiring a combination of techniques!







Ref. A Briggs (www.nanotech.org)

Some references and acknowledgements

- P.B. Hirsch et al “Electron Microscopy of Thin Crystals”
- M.H. Loretto “Electron Beam Analysis of Materials”
- D.B. Williams and C.B. Carter “Transmission Electron Microscopy”
- J-P. Morniroli “Large Angle Convergent Beam Electron Diffraction”