II

High Resolution

Transmission Electron Microscopy HR TEM/ HREM

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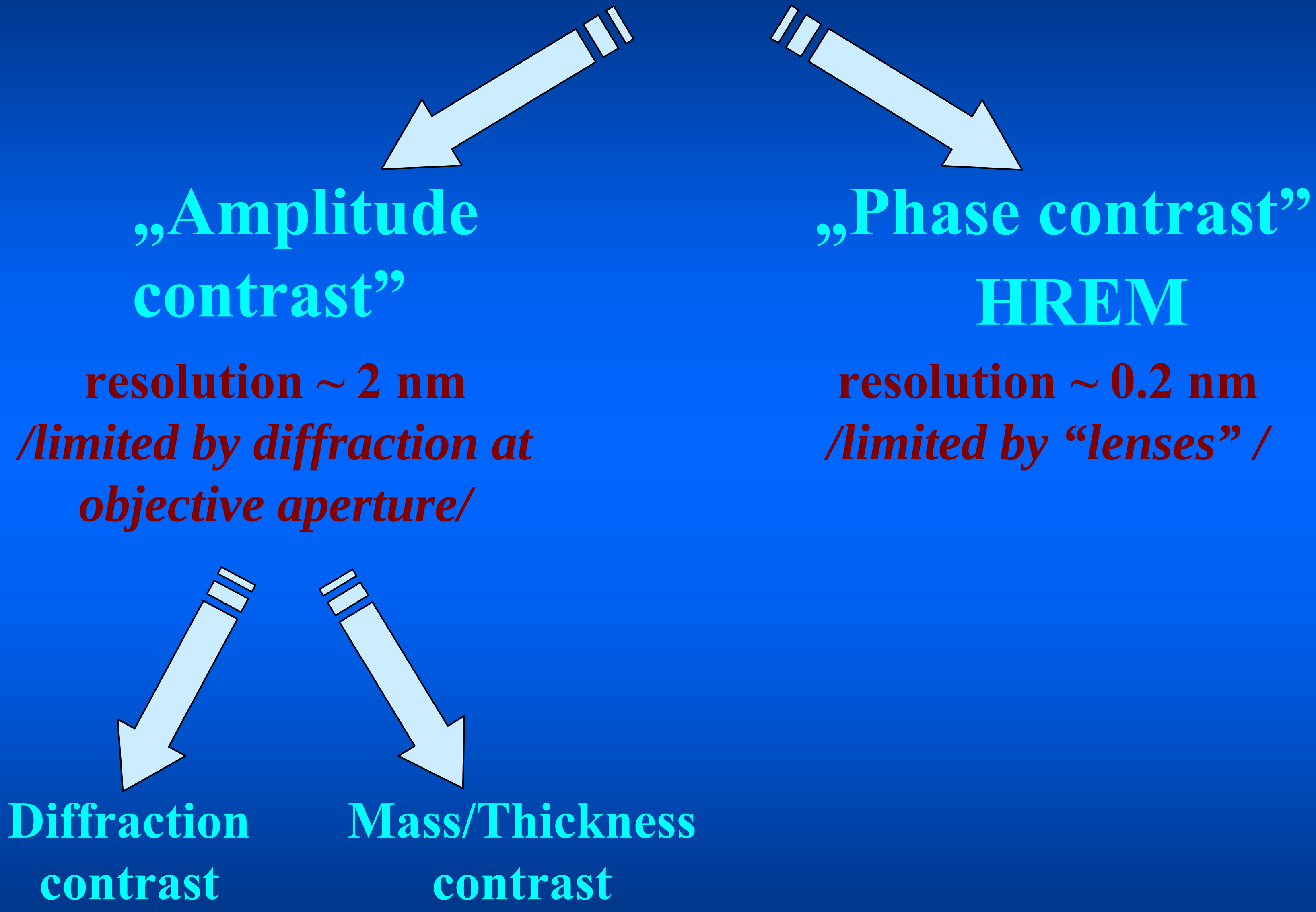
TEM
LAB

IMIM PAN – KRAKÓW – 2019

Contrast in TEM



„Amplitude contrast”

resolution ~ 2 nm

/limited by diffraction at objective aperture/

„Phase contrast” HREM

resolution ~ 0.2 nm

/limited by “lenses” /

Diffraction contrast

Mass/Thickness contrast

“idea” of PHASE CONTRAST

- electron plane wave interact with crystal in a way:
  „**some electrons** passing regions of positive potential, i.e. atomic nuclei are accelerated, λ is reduced and the **phase is advanced** by an amount proportional to the potential at the scattering site
  /in reference to “passing” i.e. nondiffracted beam/

- for a **thin crystal** i.e. <10 nm (weak phase object - WPO) :
  = amplitude changes caused by inelastic scattering are small
  = phase changes caused by elastic /dyf/ scattering are small
  (electrons are diffracted only once!)
  one can regard crystal as a **weak phase object**
  and apply **kinematical theory of electron diffraction**
  (otherwise multiple scattering =>**dynamical theory**)

J.L.Hutchinson, School on EM, Berlin 2000

Phase contrast is generated when primary and scattered beams recombine

for a WPO with ~$\pi/2$ phase shift nearly no amplitude contrast

wave function in image plane: $\Phi_T(R) = \mathrm{F.T.}^{-1}\{\mathrm{F.T.}\,[\Psi(k)]\,\exp(-i\,\chi)\}$

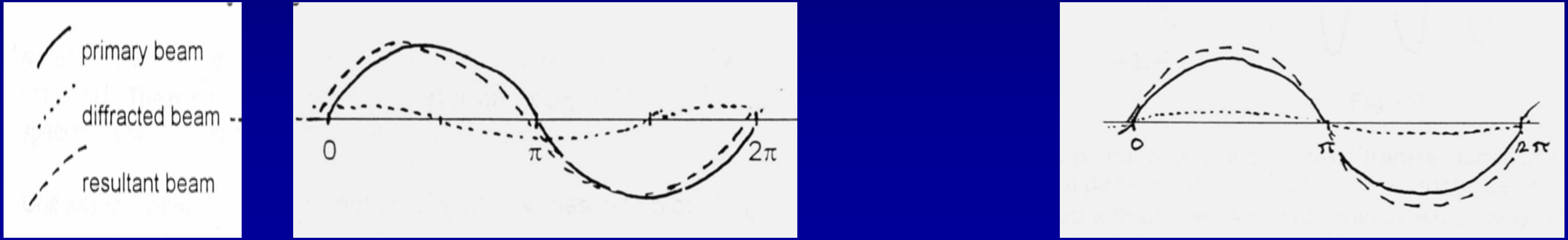


image contrast depends on: $I(R) = |\Phi_T(R)|^2$

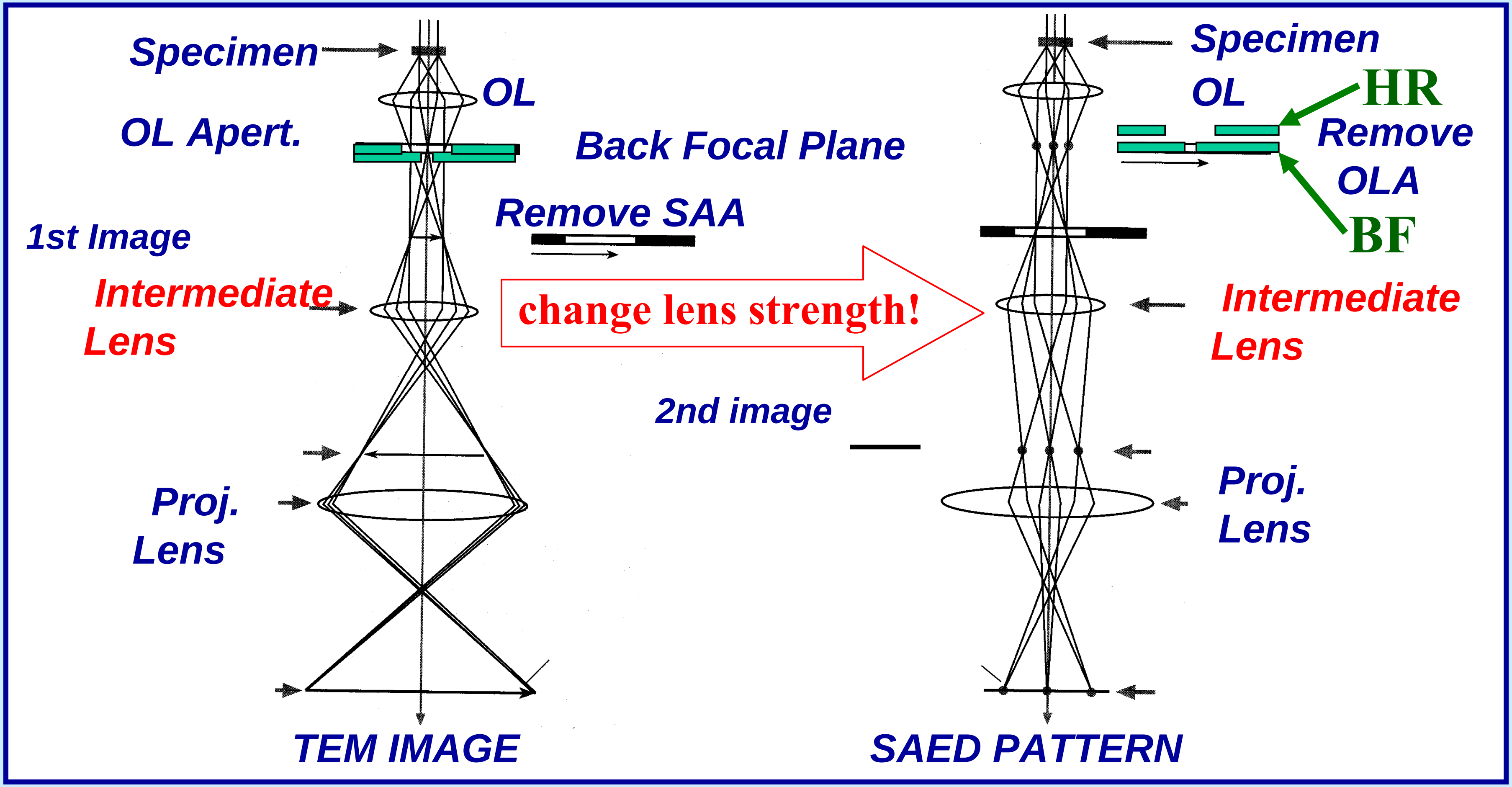
1949 - Scherzer: relation between
phase shift χ and {Δf/defocus + C_s/obj. ast.. + $\theta(1/d)$/diff. angle }

$$\chi = \frac{\pi}{2\lambda}\left(C_S\theta^4 - 2\Delta f\theta^2\right)$$

dla jednego θ (1/d)!

sin χ is close to unity over large range of $1/d_{hkl}$ at
„Scherzer focus” $\Delta f_{Scherzer} = - C_S^{1/2} S\lambda$

Conditions for HREM imaging and Selected Area Diffraction



Specimen

OL

OL Apert.

Back Focal Plane

Remove SAA

1st Image

Intermediate
Lens

change lens strength!

2nd image

Proj.
Lens

TEM IMAGE

Specimen

OL

HR

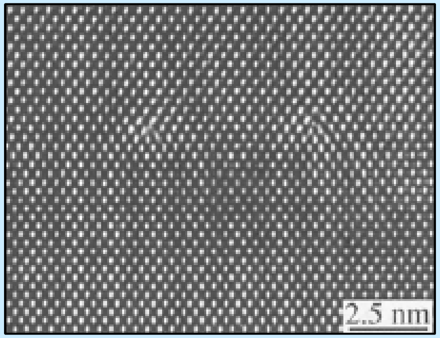
Remove
OLA

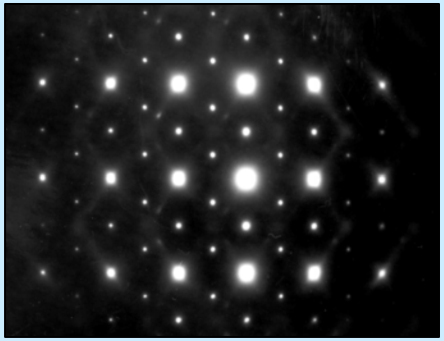
BF

Intermediate
Lens

Proj.
Lens

SAED PATTERN

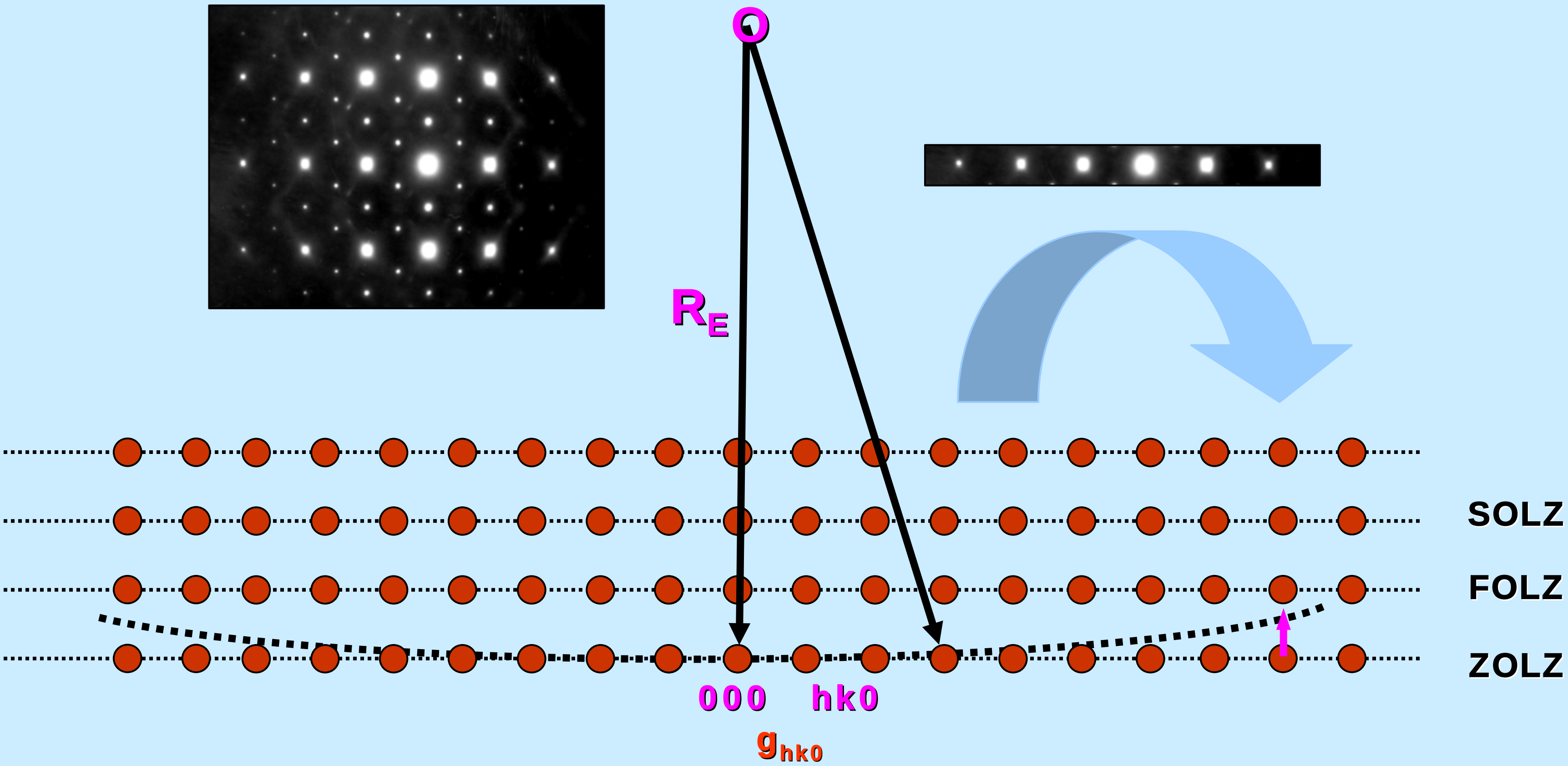




Conditions for obtaininf electron diffraction/ Ewaldasphere

Usual d- spacings (10 Å -1 Å) >>> λ

Radius of Ewald sphere (R_E= 1/ λ)>>> g spacings

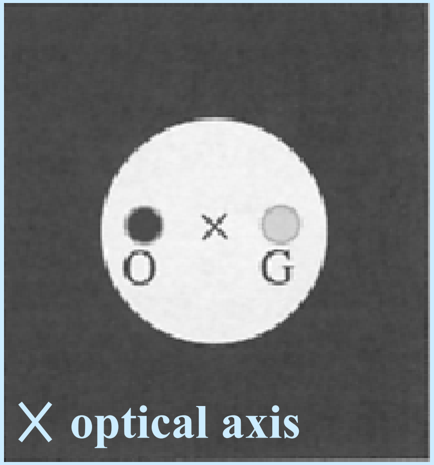


g_{hkl} - diffraction vector in reciprocal space

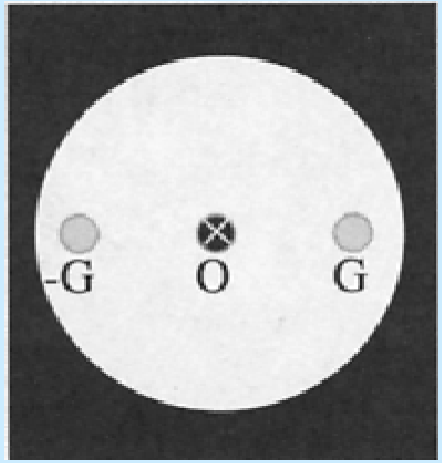
↑ s - deviation from exact Bragg condition

Lattice imaging/ lattice fringes

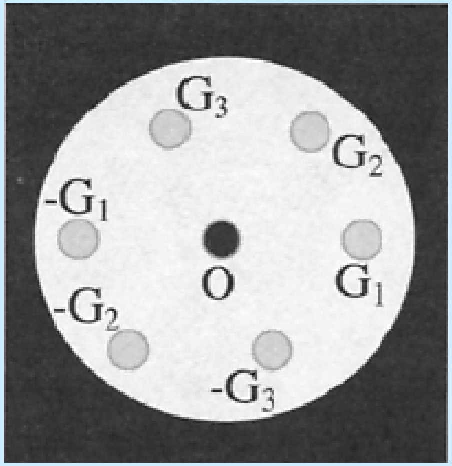
„tilted beam" illumination

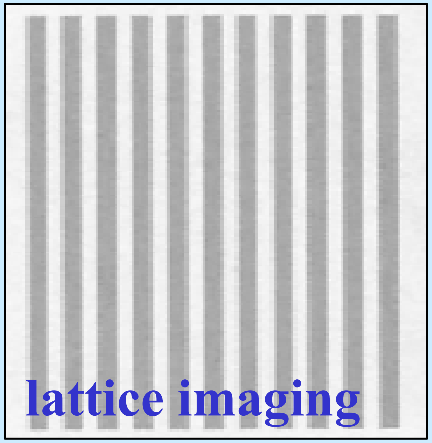


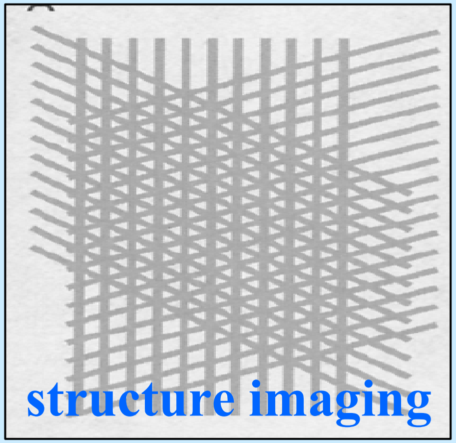
„on axis" two beam illumination



„on axis" two beam illumination



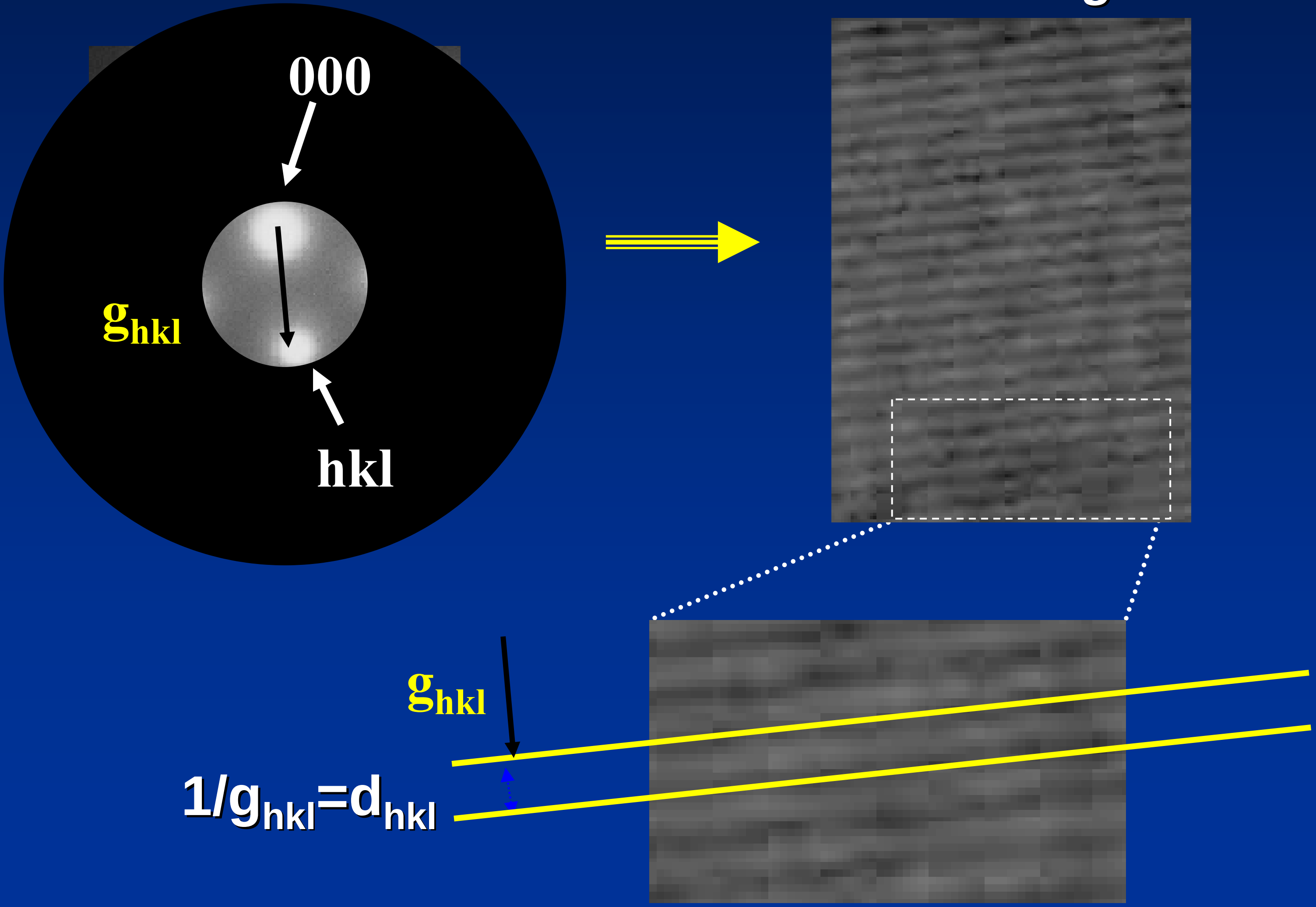

lattice imaging


structure imaging

- spacing of fringes/ spots equals to spacing of diffracting planes
- fringes/ spots may show no relation with position of planes/ atomic columns !!!

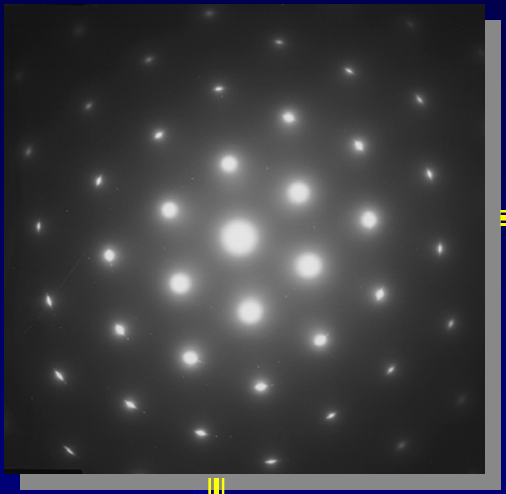
HREM : Lattice imaging

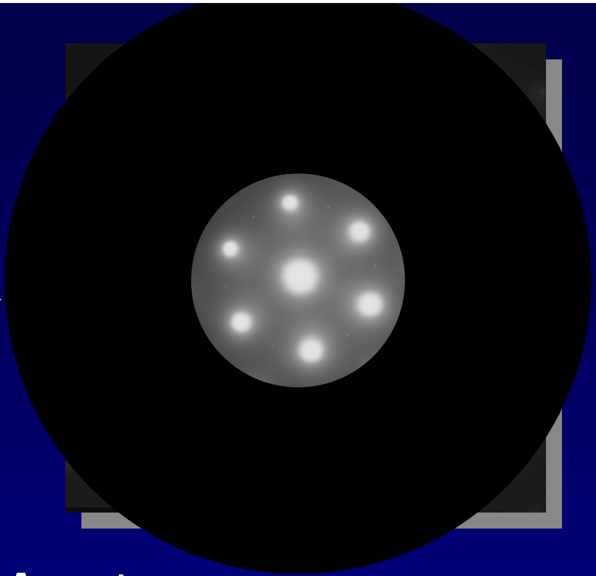
Lattice Fringes



$1/g_{hkl}=d_{hkl}$

HREM : Structure imaging

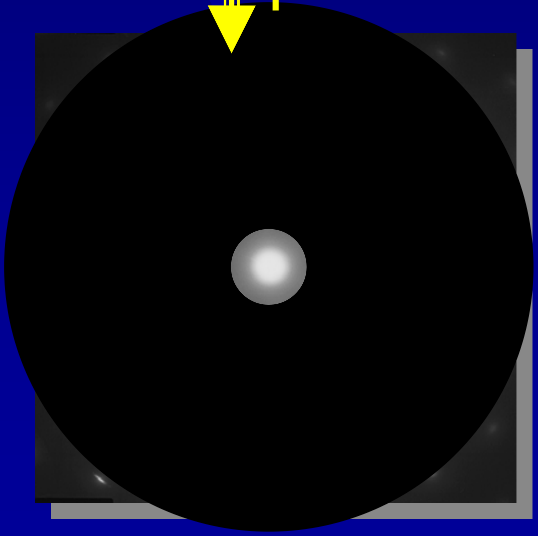




Imaging with Multiple Beams (HREM)

Increasing number of beams increases resolution!

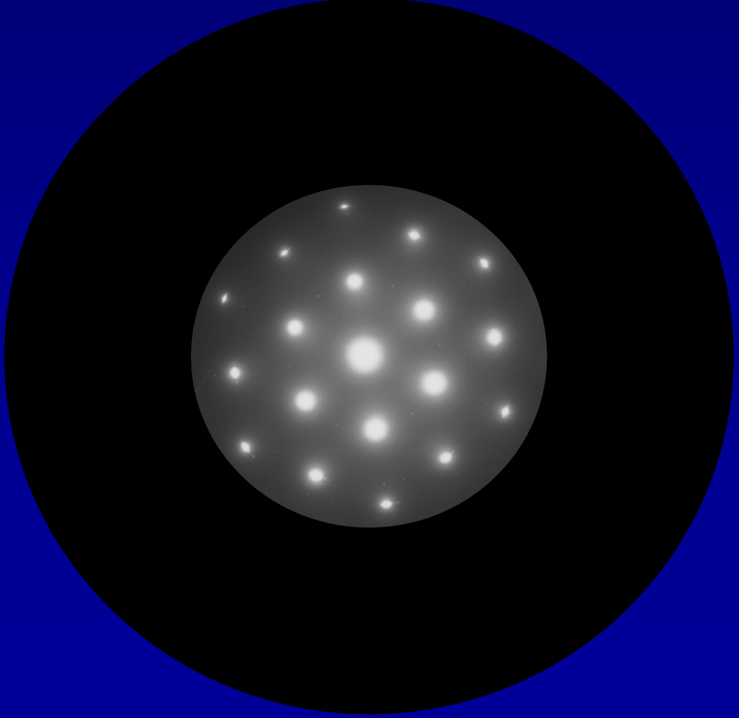
OL Aperture



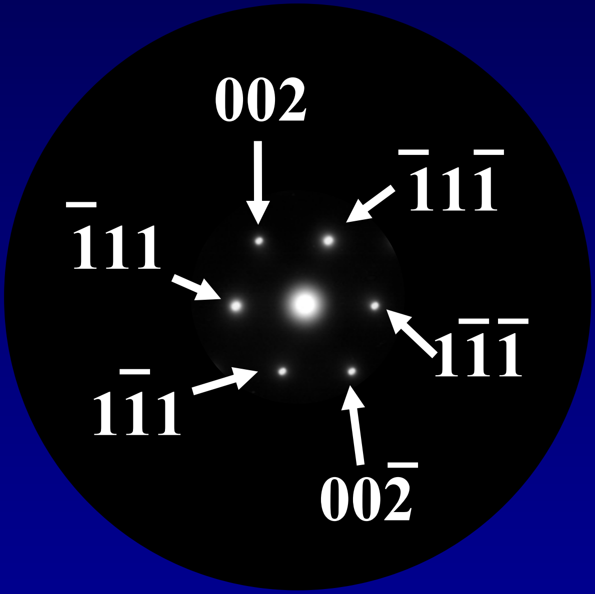
Imaging with only 1 beam

Diffraction contrast (BF,DF)

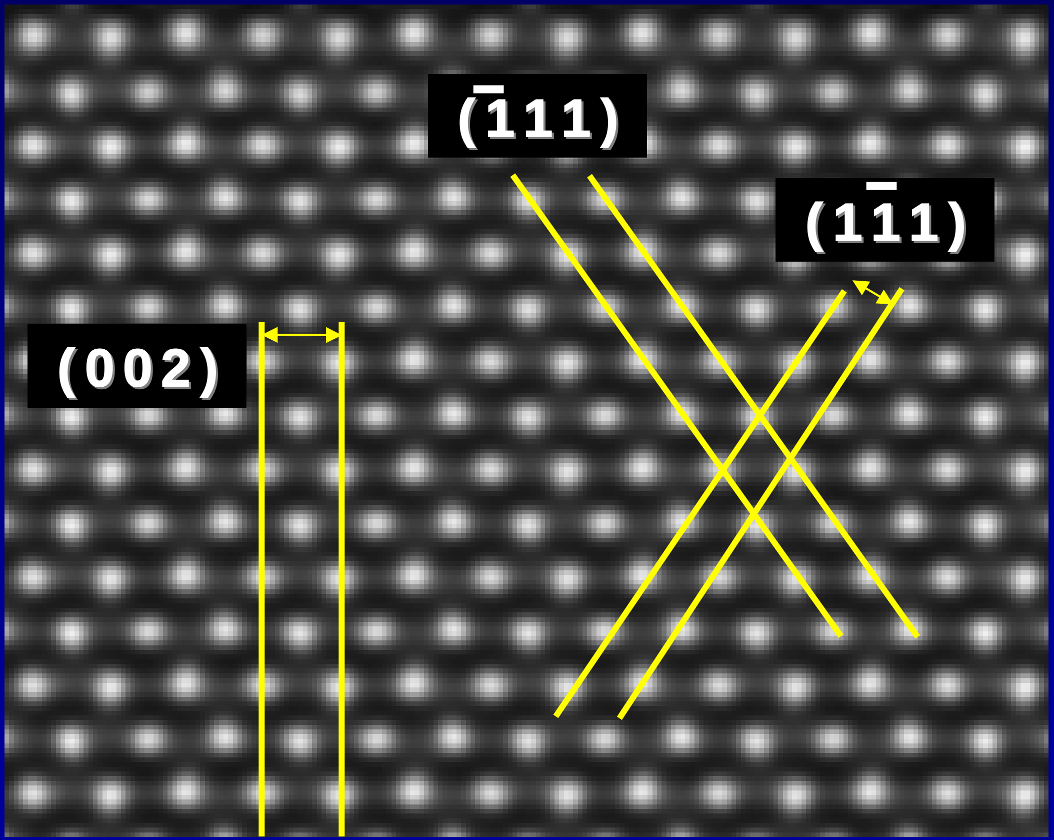
Defect Analysis



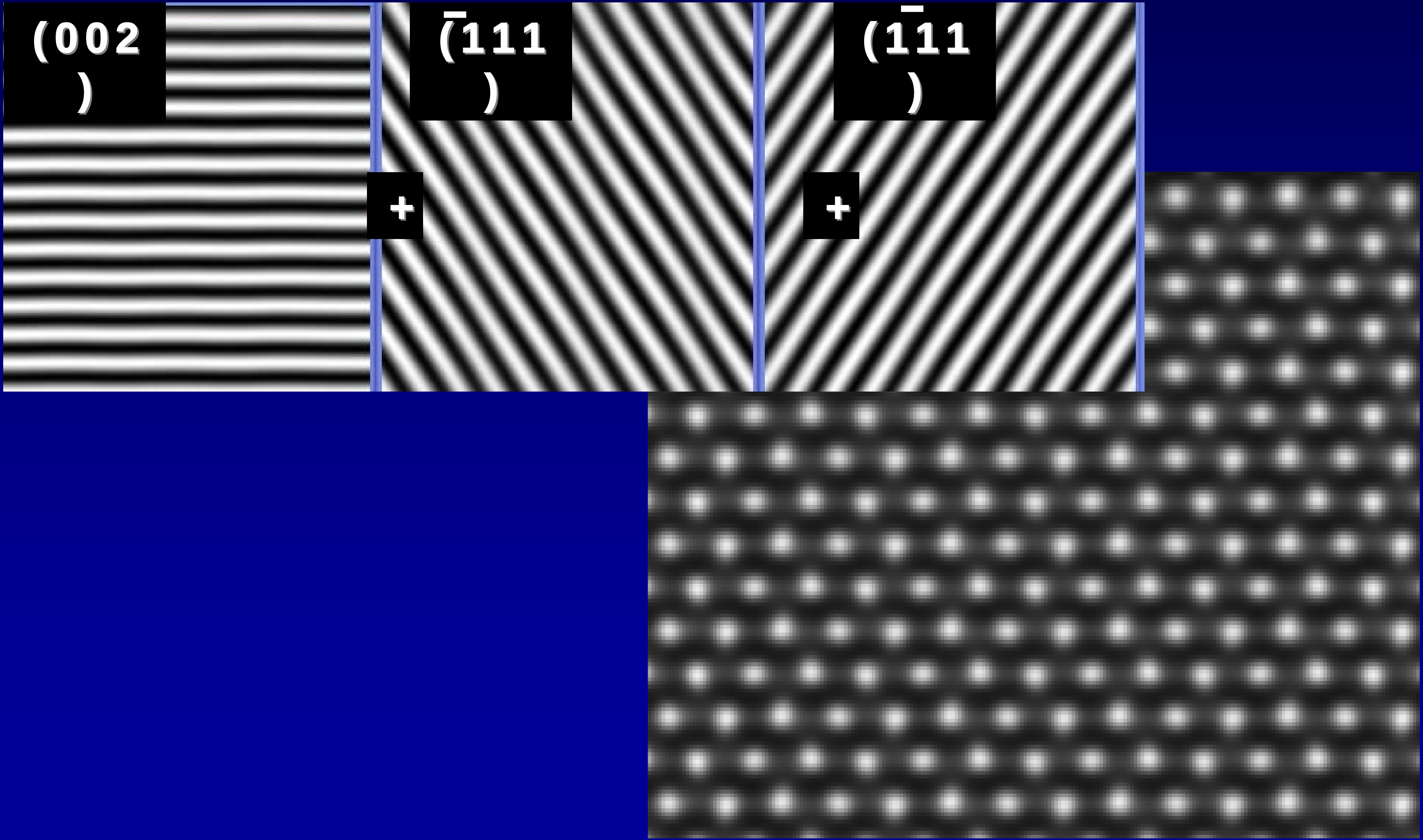
HREM : Structure imaging



7 beams HREM (lattice) image



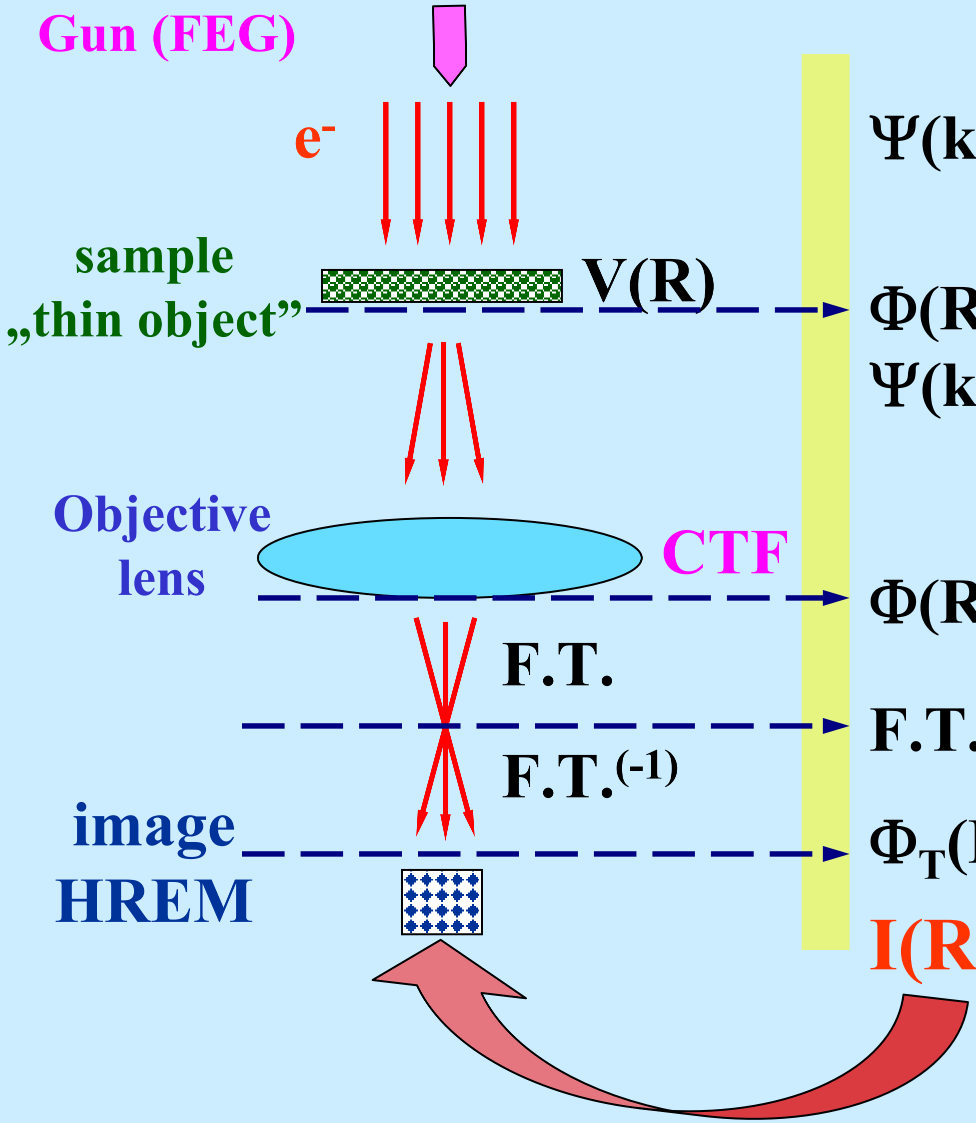
HREM : The imaging step



HREM - Part I.

„Classical" approach = „thin object" + „Scherzer defokus"

(⇒ „direct (?) corelation of image with the structure")



$\Psi(k)$

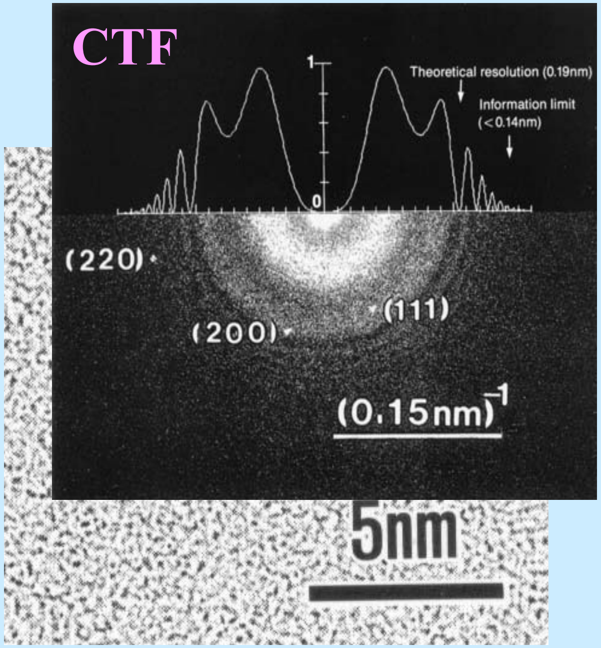
$\Phi(R) = \Psi(k) * V(R)$

$\Phi(R) * \mathrm{CTF}$

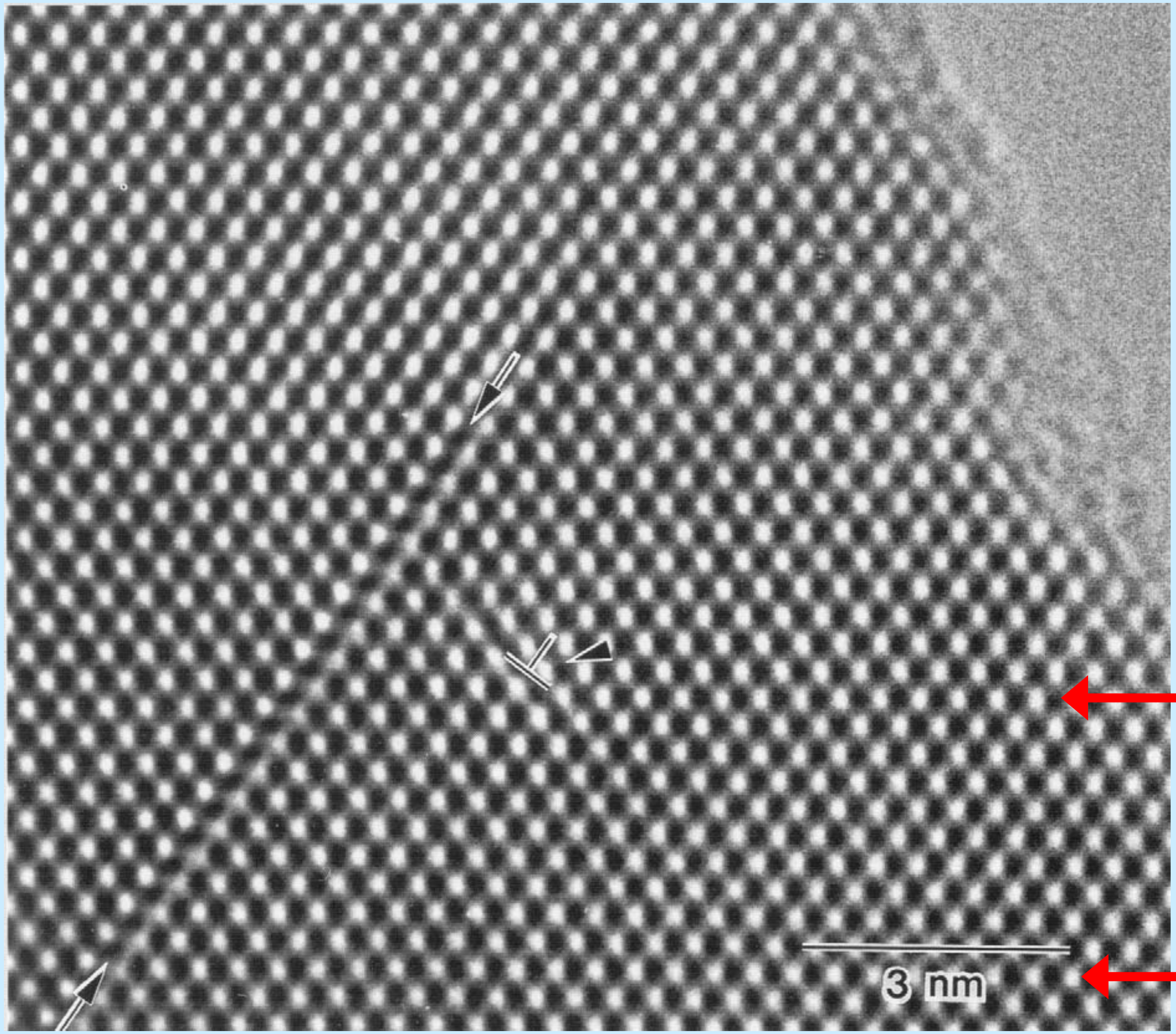
$\mathrm{F.T.}\{\Phi(R) * \mathrm{CTF}\}$

$\Phi_T(R) = \mathrm{F.T.}^{(-1)}\{\mathrm{F.T.}[\Phi(R) * \mathrm{CTF}]\}$

$I(R) = |\Phi_T(R)|^2$



HREM - „achievements”



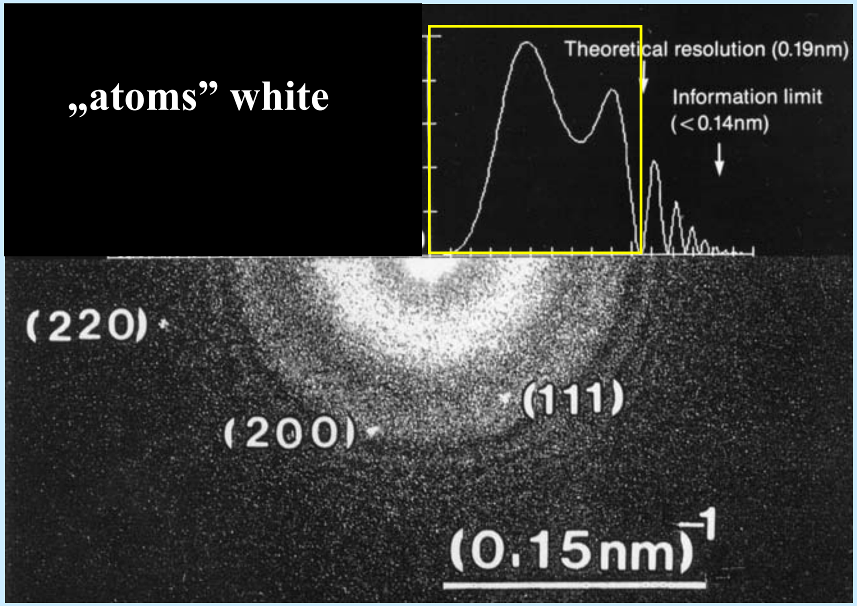
CdTe: [110] zone axis
Scherzer defocus

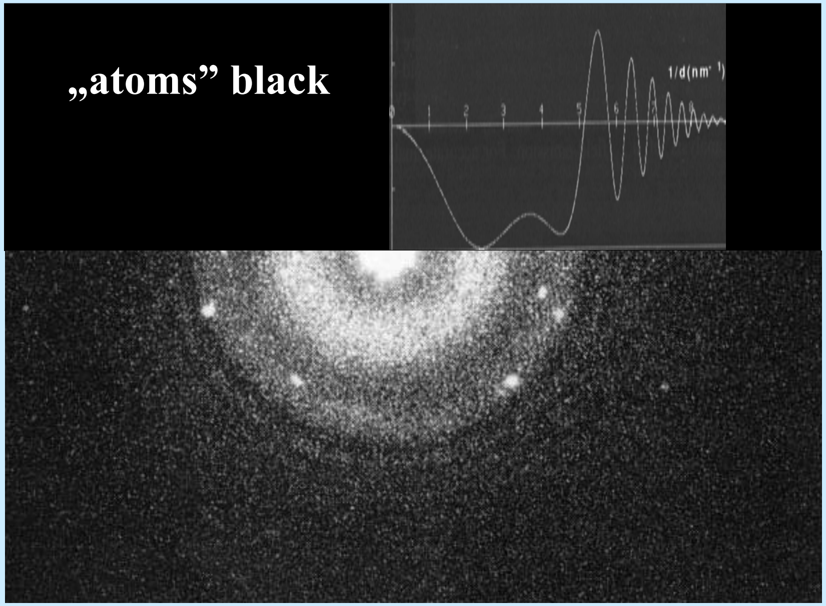
/Stacking fault + edge dislocation; note bending of SF caused dislocation strain field/

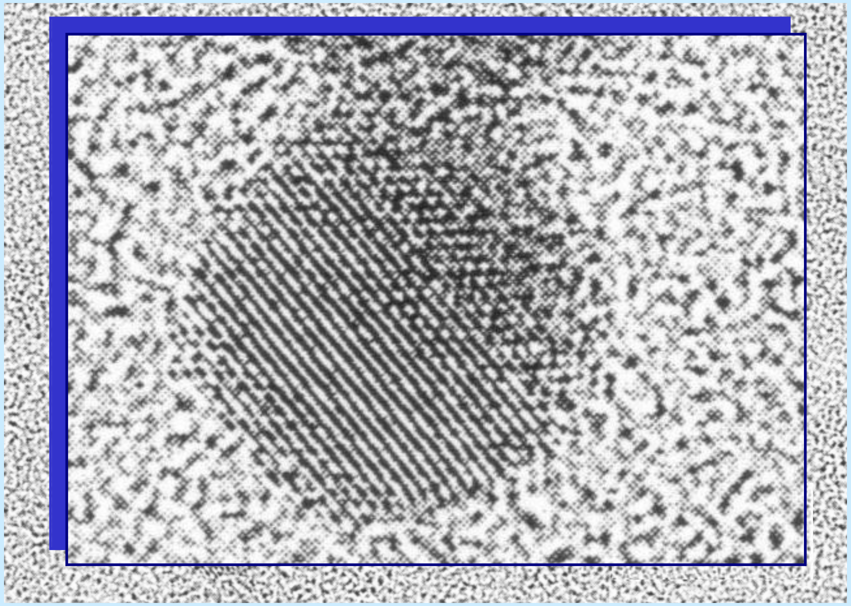
Scherzer underfocus /obj. lens weakened from Gasian „focus”; atoms „black” • • •

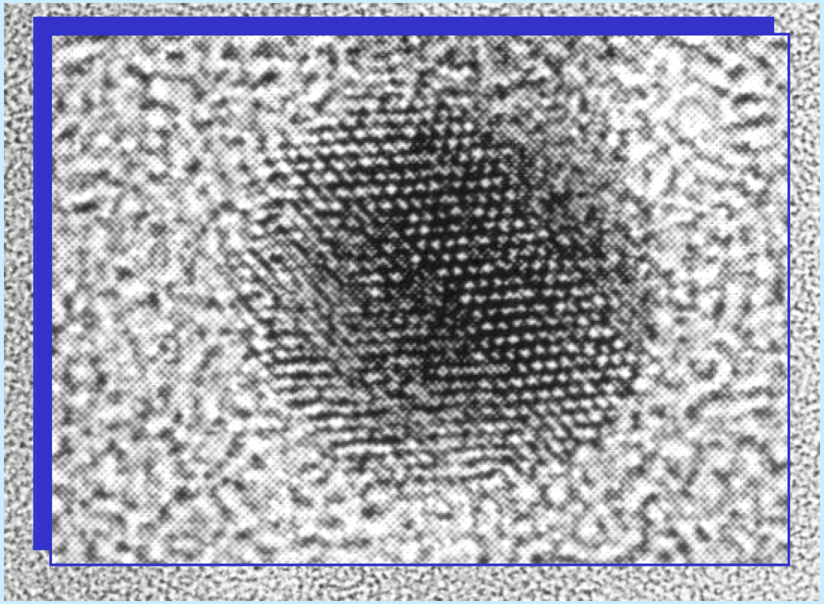
Scherzer overfocus /obj. lens excited over Gasian „focus”; atoms „white” • • •

Au/ amorphous Ge (CTF + Optical Diffraction Pattern, + HREM Image


„atoms” white

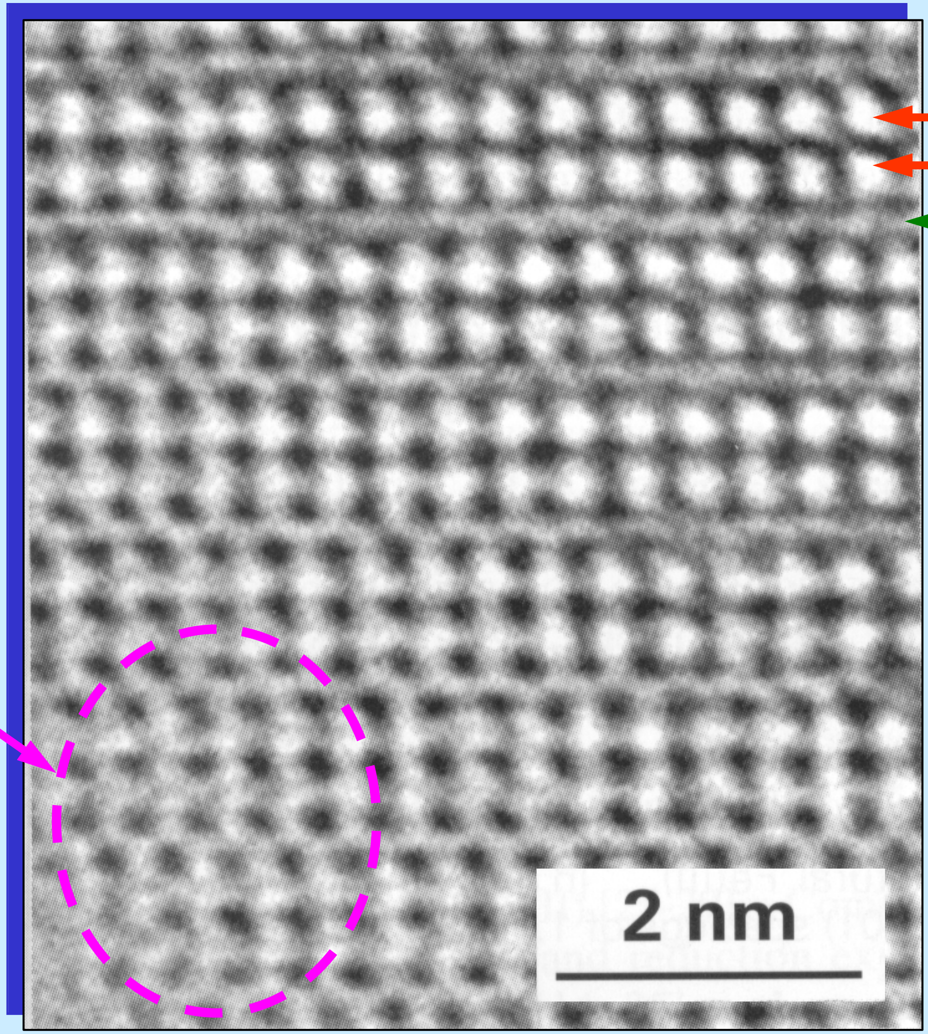

„atoms” black





HREM - „light” & „heavy” atoms

NbO_x



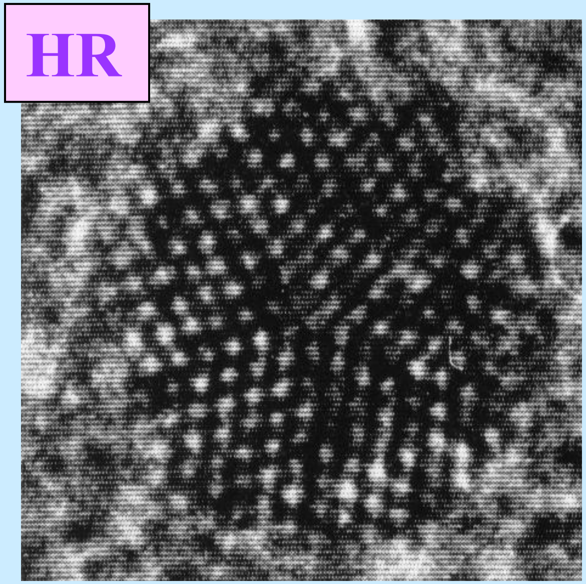
Nb

O

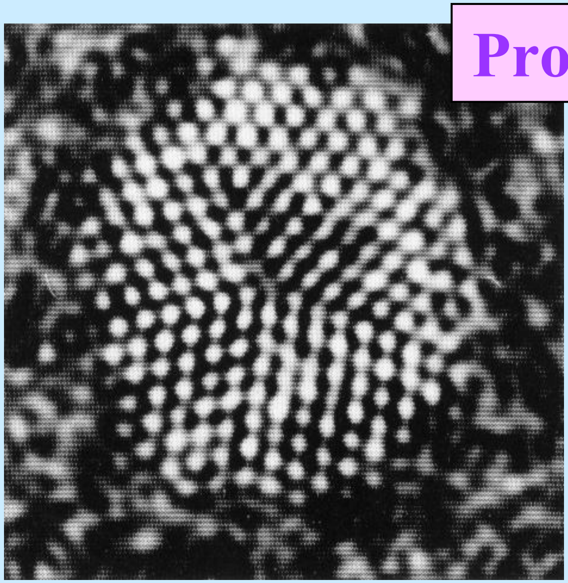
radiation damage

Hutchinson at. al., JEOL News, 37E(2002)2

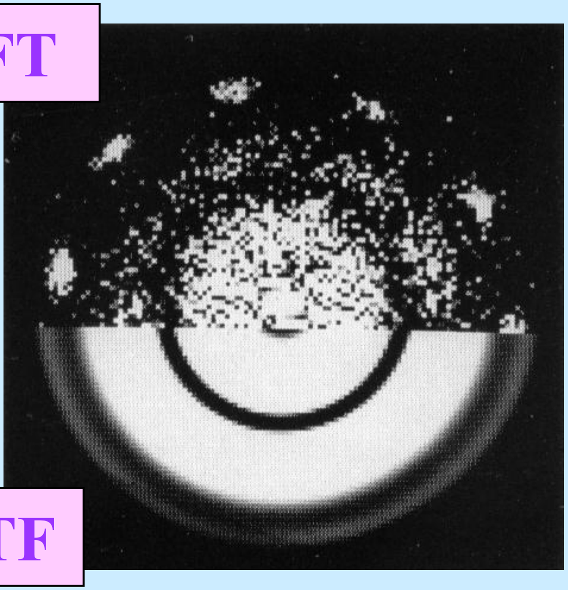
Removal of noise



HR

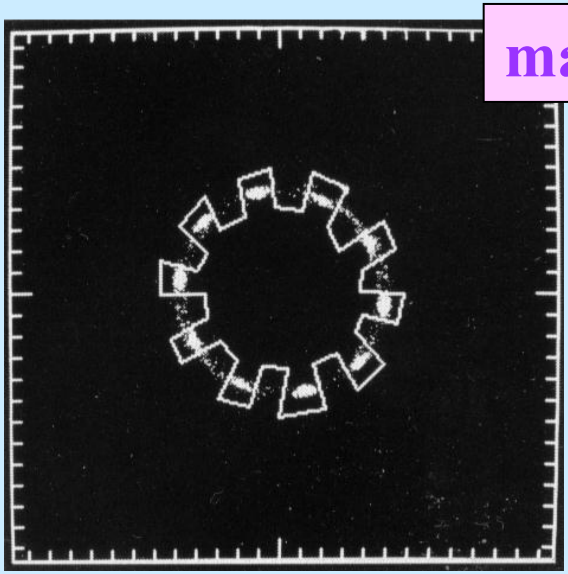


Procesed HR



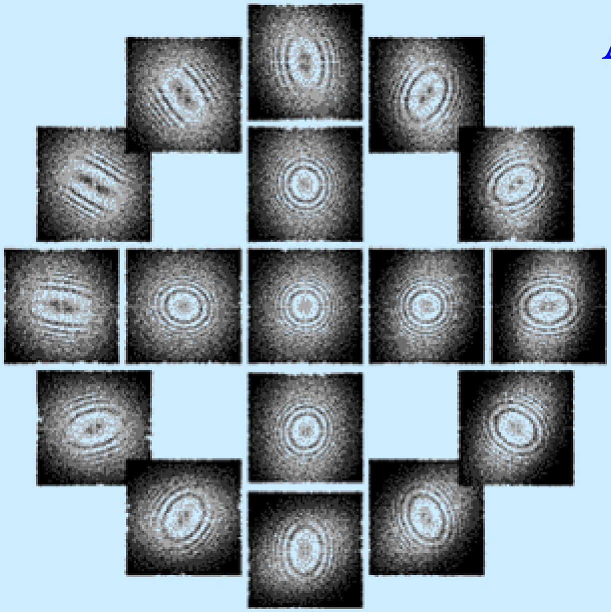
FT

CTF

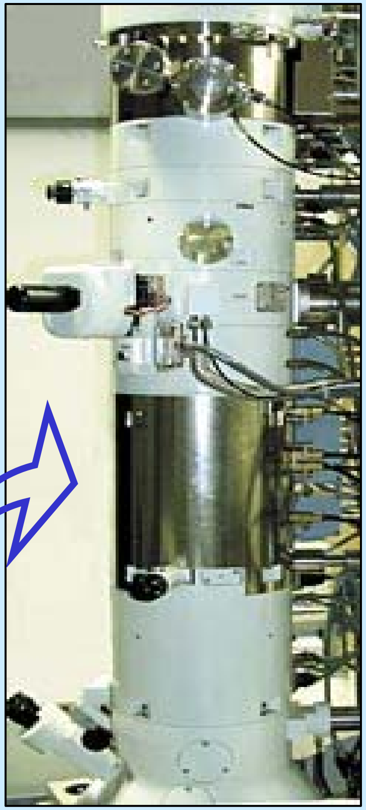


mask

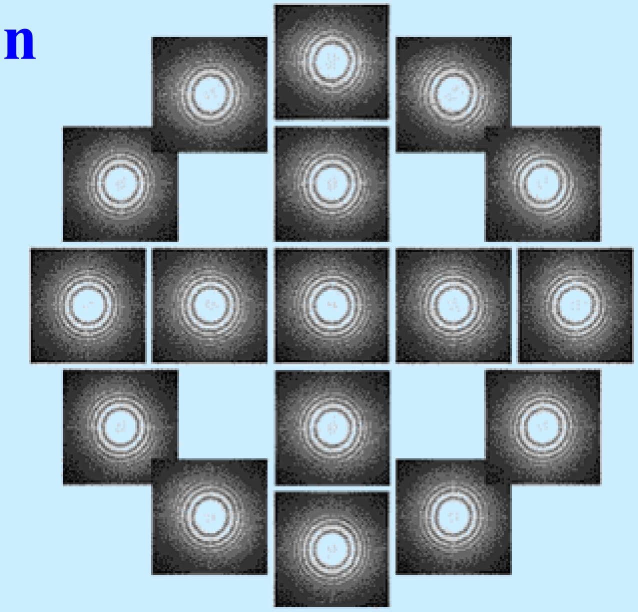
Astigmatism correction



2-fold astigmatism corrected

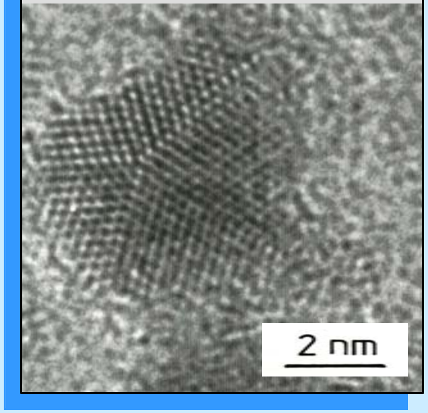


von Rose corrector:
series of two hexapole
and two transfer lenses



3-fold astigmatism corrected

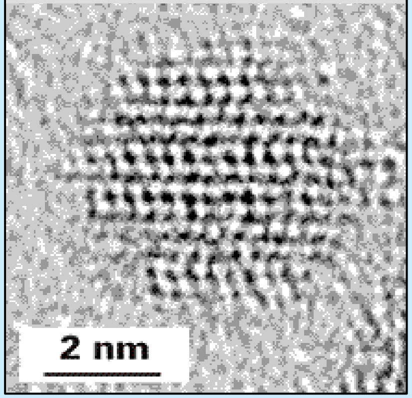
Au/C, Hewitt 89



silny „phase kontrast”

Hutchinson, JEOL News 37E(2002)2

CdSe/C Hutchinson 02



obniżony „phase kontrast”

cont.

„3- fold astimatism increases diameter of diffraction discs producing spurious contrast up to several nm”

Hutchinson 2002

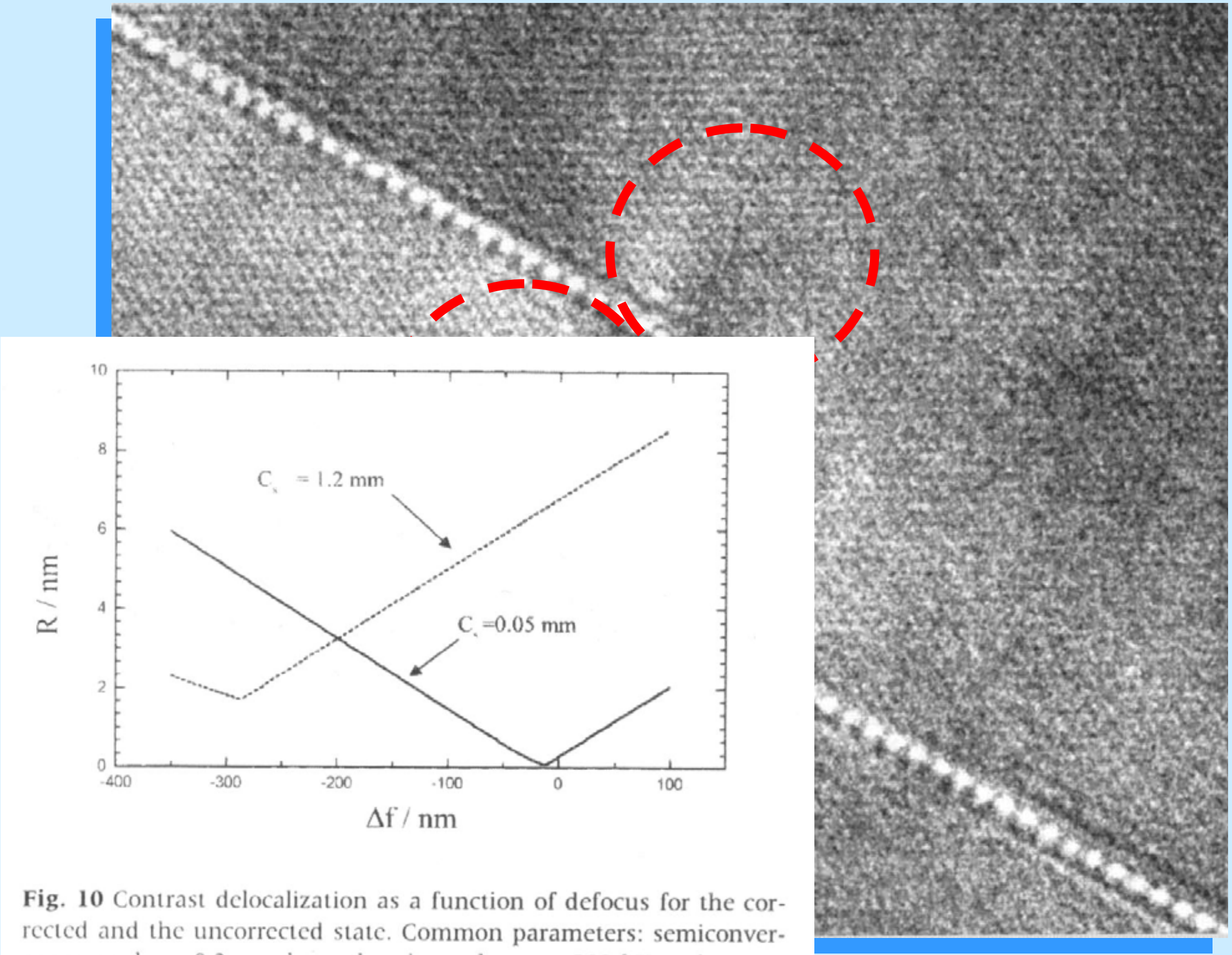


Fig. 10 Contrast delocalization as a function of defocus for the corrected and the uncorrected state. Common parameters: semiconvergence angle = 0.2 mrad, acceleration voltage = 200 kV, and q_{max} = 0.14 nm.

Granica Σ3
folia Au
„zlokalizowany”
kontrast na uskoku
(po usunięciu
astygmatyzmu
trójosiowego)

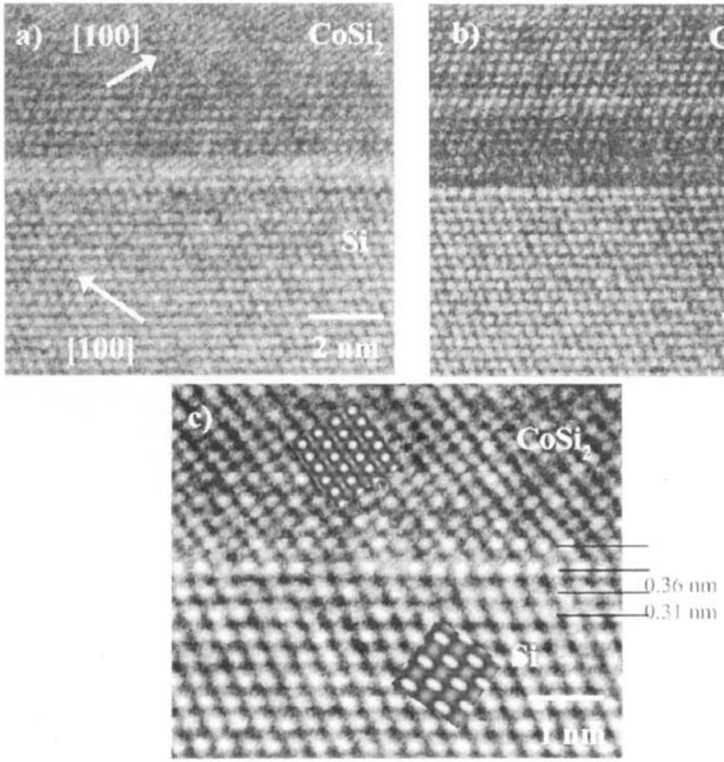
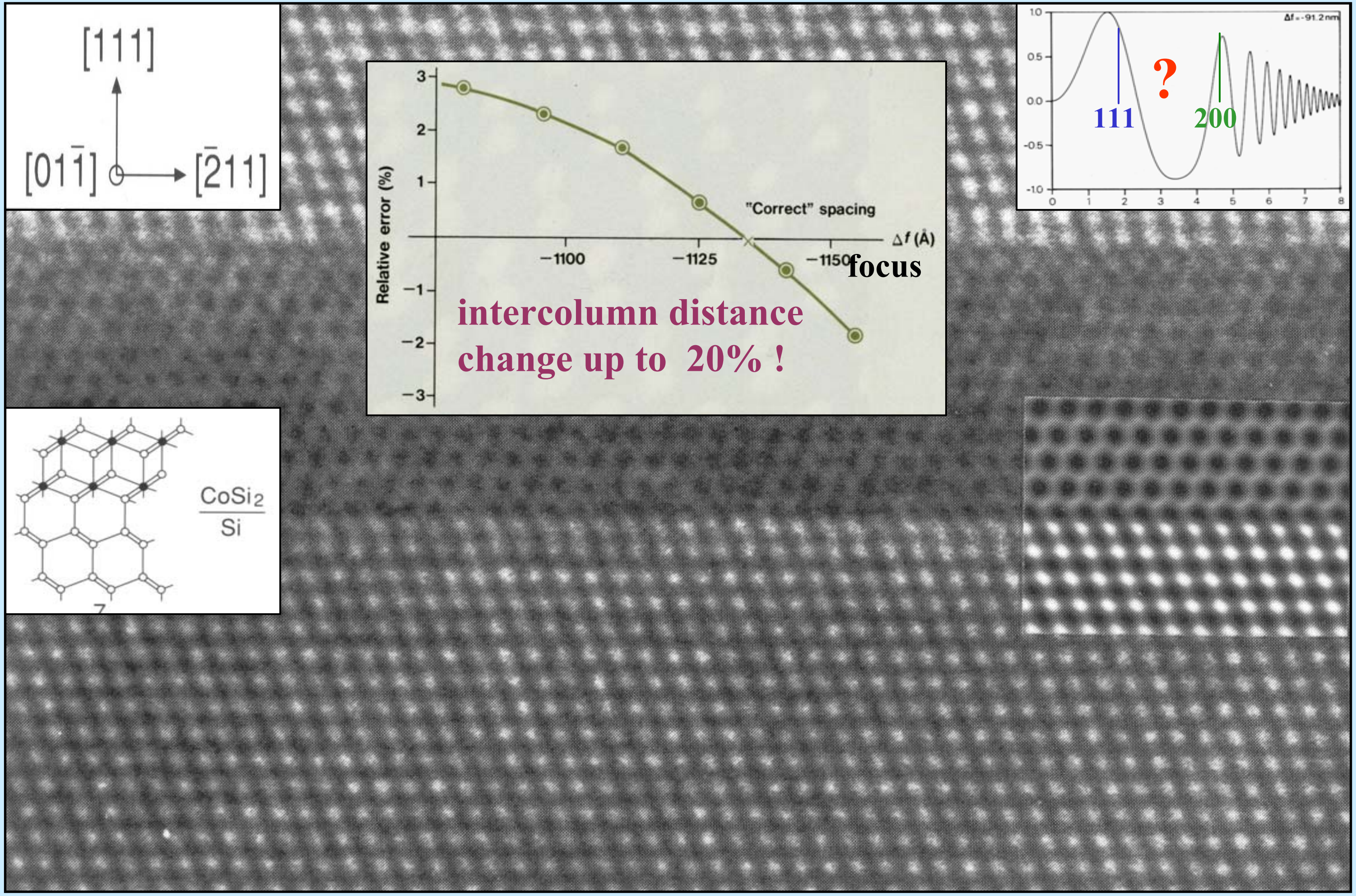


Fig. 4 High-resolution images of an epitaxial Si(111)/CoSi$_2$ interface demonstrating the influence of the spherical aberration on contrast delocalization. Images (a) and (b) were taken with a C_s of 1.2 mm at –67 nm and at –257 nm, respectively. Image (c) was recorded in the aberration-corrected state at a defocus of –12 nm and a C_s value of 50 µm.

HREM: limitation of „classical” approach; boundaries

Si / CoSi2: type CaF (difference between $d_{111}$and d_{200} ~1.2%)



intercolumn distance change up to 20% !

200 kV, defocus f = -90 nm, thickness = 6 nm

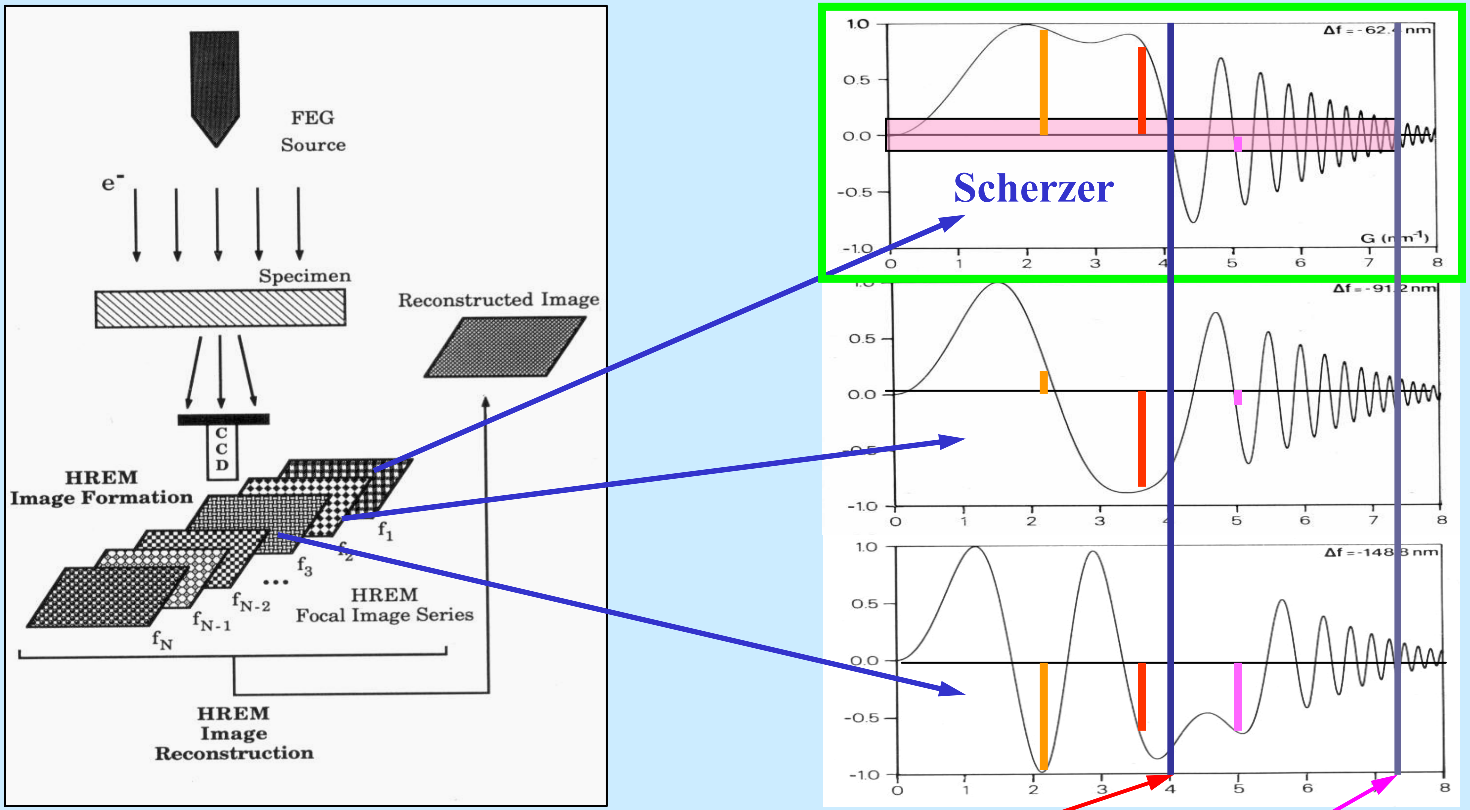
approximation „thin object” O.K. for Si – not O.K. for $CoSi_2$

Coene at. al. Phillips Electron Optics Bulletin, 132(1992)15

HREM (Ultra HREM) - part II

Image reconstruction - „through focus image series"
„on axis-" or „on - line holography"
way beyond „Scherzer defocus" up to „information limit"

$(h_1k_1l_1)$, $(h_2k_2l_2)$, (h_3,k_3,l_3)



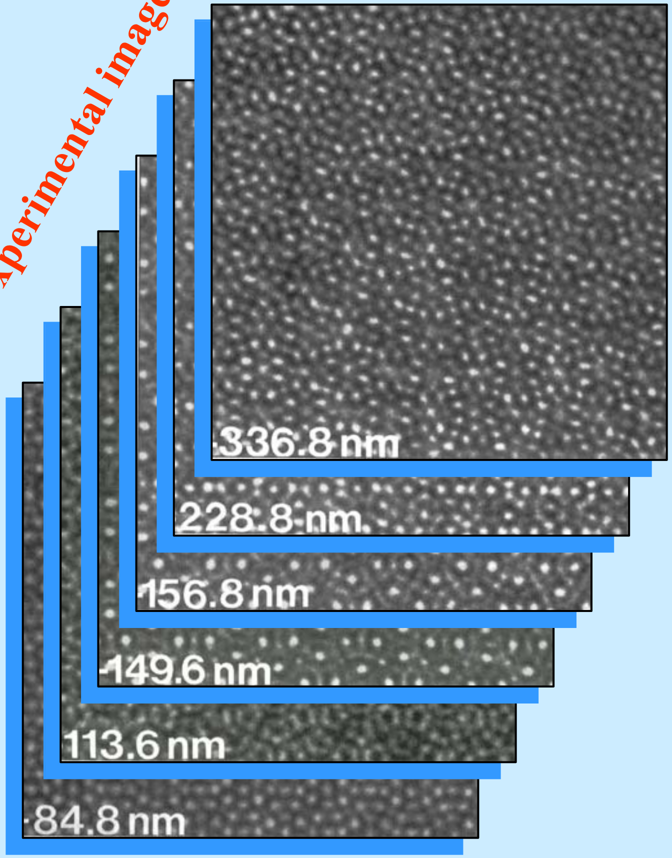
theoretical resolution, information limit

HREM - „on-axis” holography - application

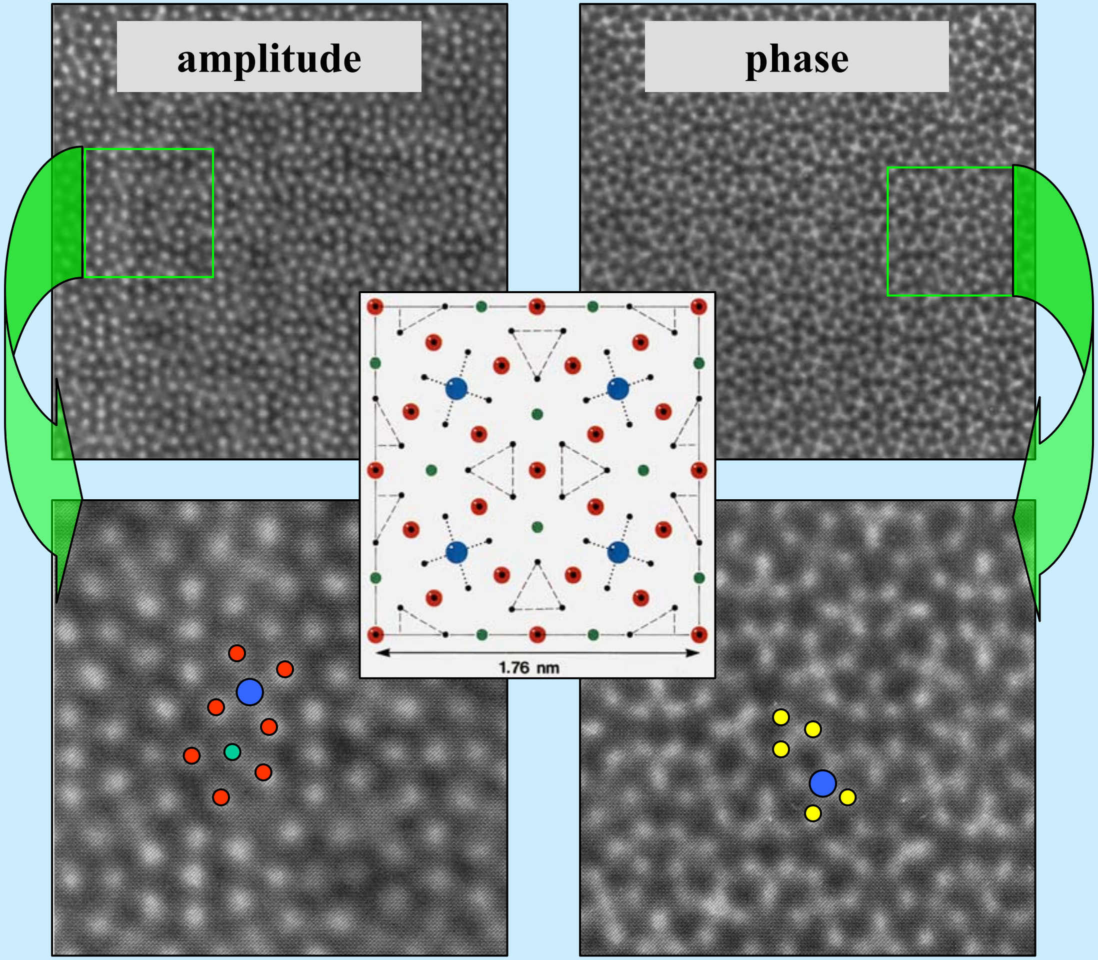
+ info. o grubości folii

experimental images (!)

$Ba_2NaNb_5O_{15}$



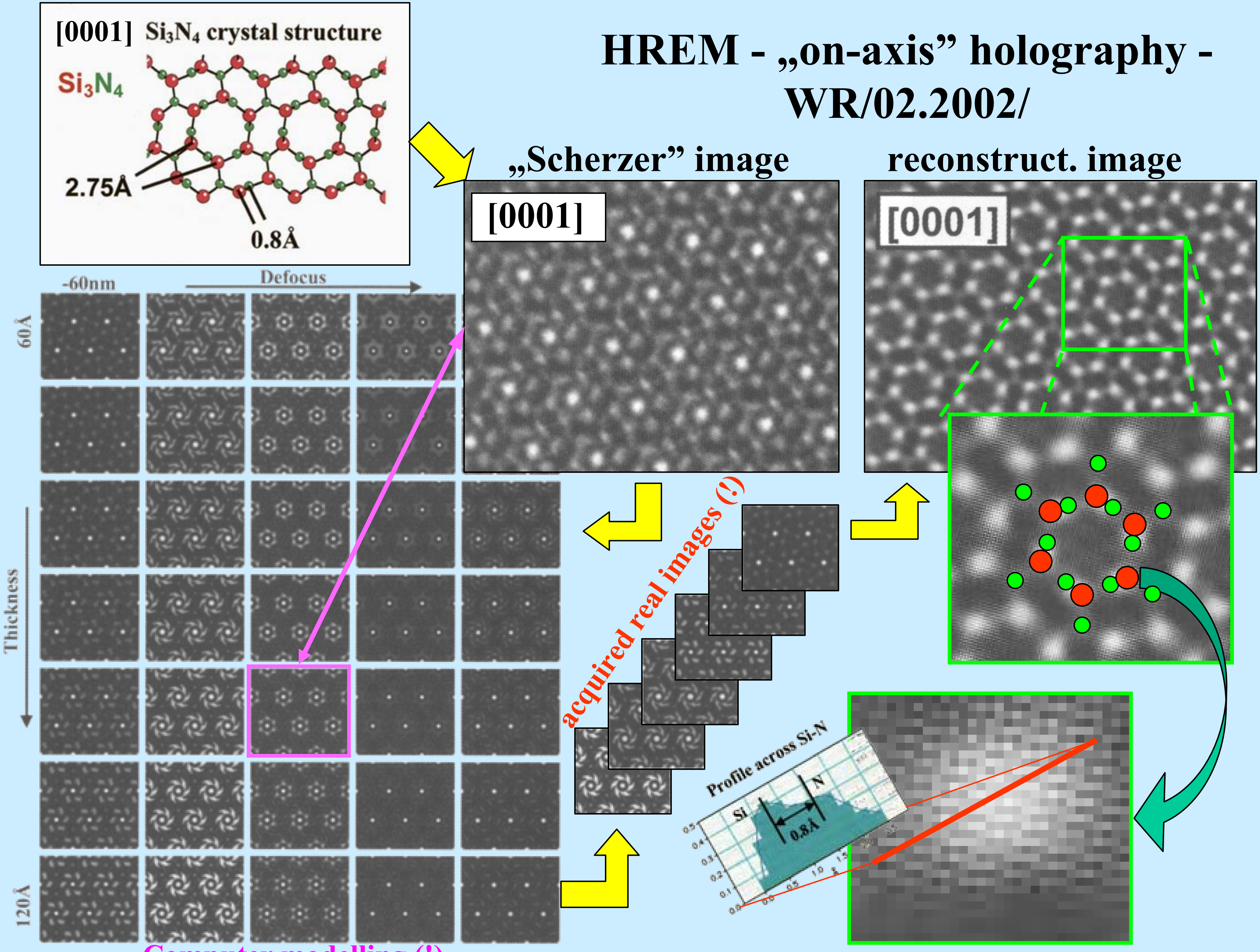
images reconstructed (!) from focal series



Coene at al. Electron Optics Bulletin, 132 (1992) 15

$Ba_2NaNb_5O_{15}$

HREM - „on-axis" holography - WR/02.2002/



„Scherzer" image

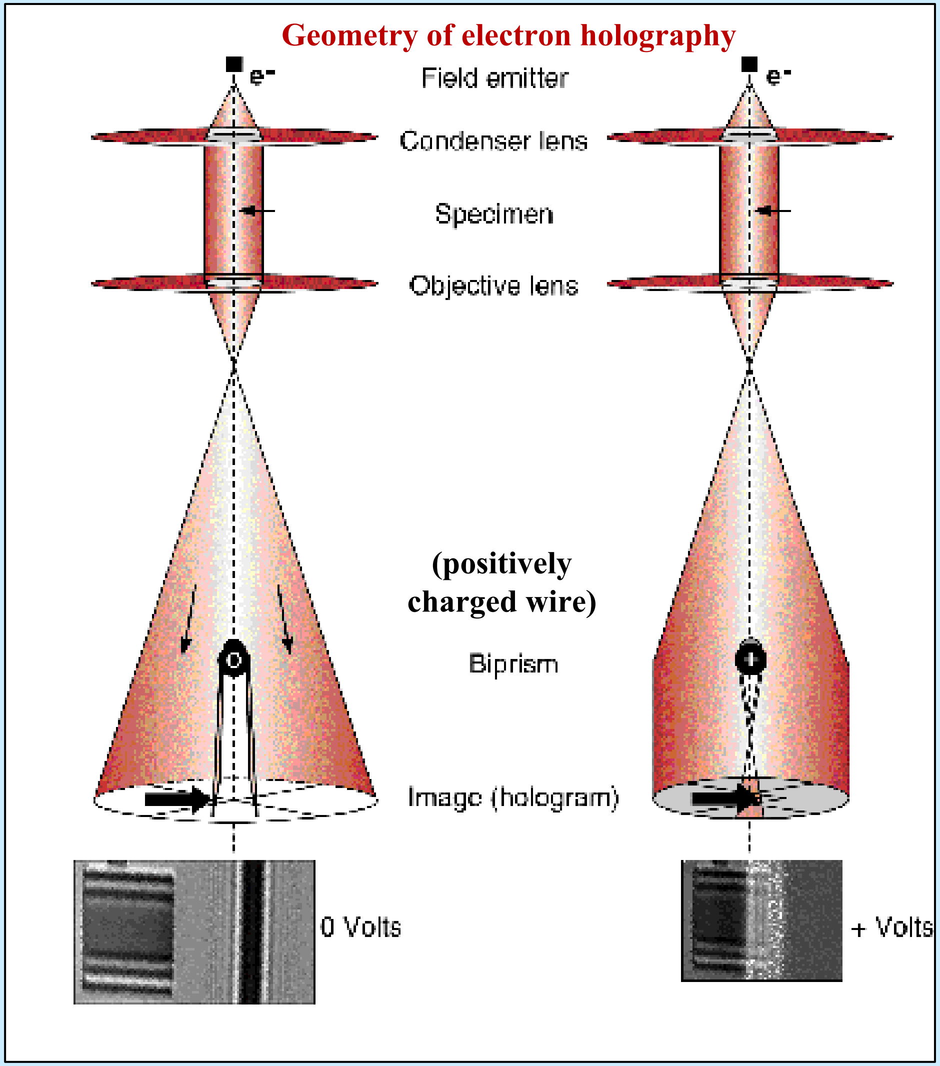
reconstruct. image

acquired real images (!)

Computer modelling (!)

HREM - „of-axis” holography

Gabor ⇒ Möllenstedt and Düker in 1955

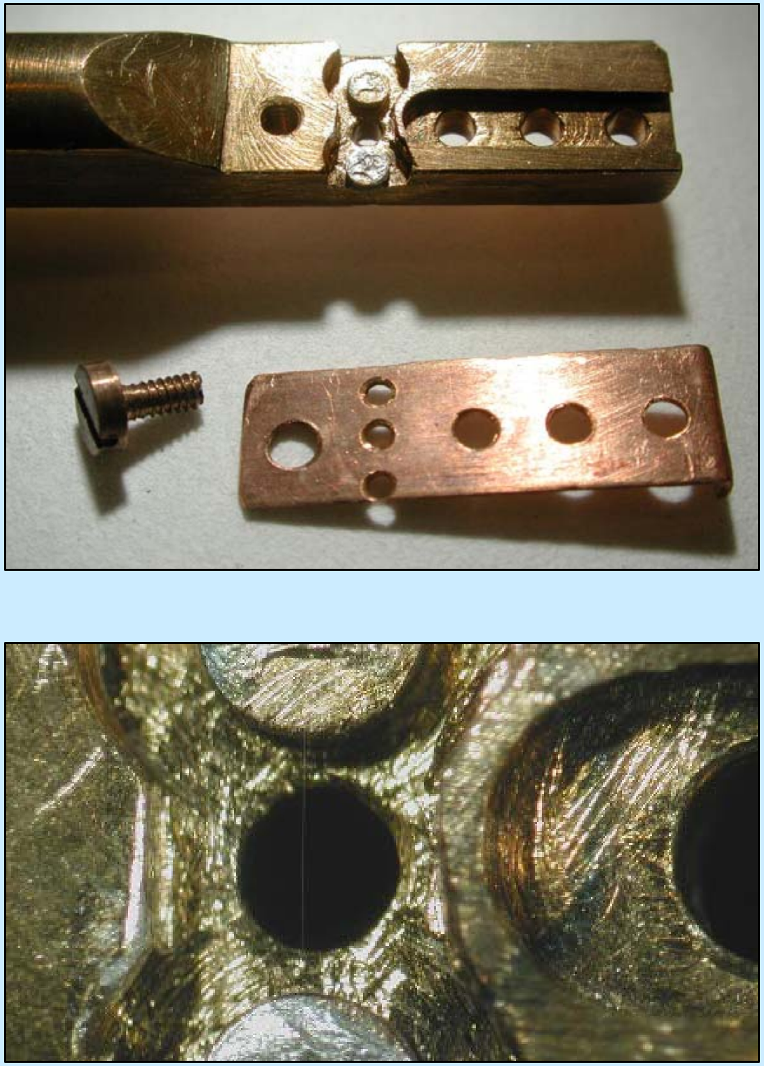


Geometry of electron holography

no voltage on biprism — positive voltage on biprism

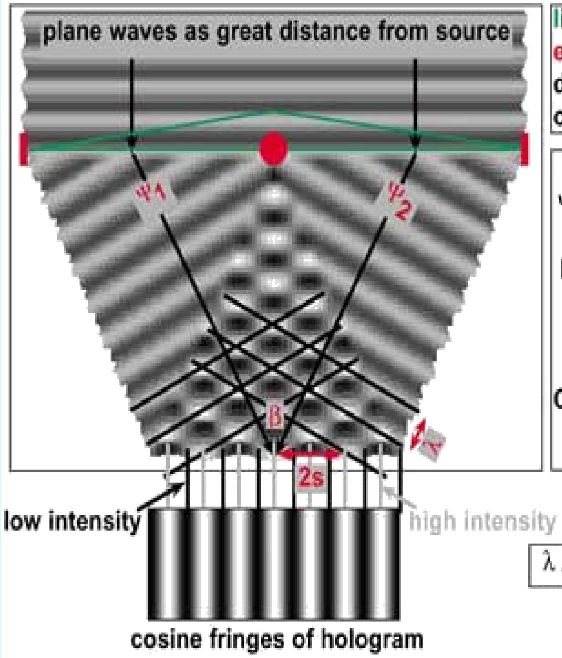
waves from object and reference

. not overerlap — overlap (forming hologram)



Möllenstedt Biprism

HREM - „of-axis” holography (c.d.)



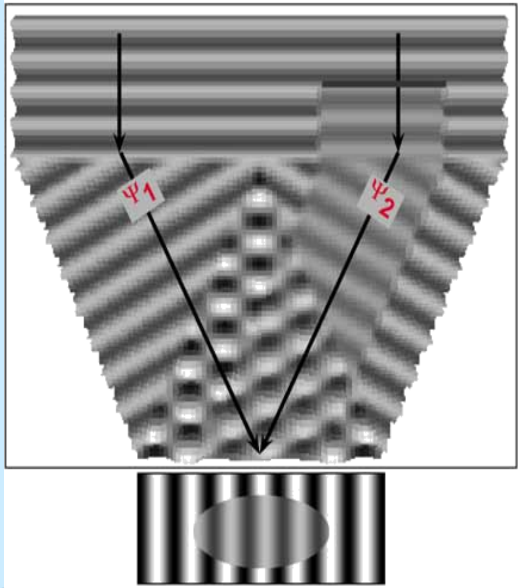
light optical biprism
electron optical biprism
deflection angle independent of distance from biprism

$\Psi_1 = a \exp[i\varphi_1]$ $\Psi_2 = a \exp[i\varphi_2]$

$I = (\Psi_1 + \Psi_2)^2$
$= 2a^2 (1 + \cos[\Delta\varphi])$
$\Delta\varphi = \varphi_1 - \varphi_2$

$C = (I_{max} - I_{min}) / (I_{max} + I_{min}) = 1$

$\lambda / 2s = \sin[\beta / 2]$ -> $s = \lambda / \beta$



amplitude causes decrease in fringe contrast
phase causes displacement of fringes

no imaging lens displayed for reasons of simplicity

$\Psi_1 = a_r \exp[i\varphi_1]$
(plane reference wave)

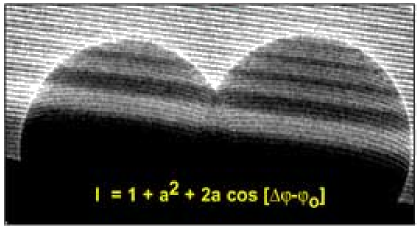
$\Psi_2 = a_r a_o \exp[i(\varphi_2 + \varphi_o)]$

$I_{holo} = (\Psi_1 + \Psi_2)^2 =$
$a_r^2 (1 + a_o^2 + 2a_o \cos[\Delta\varphi - \varphi_o])$

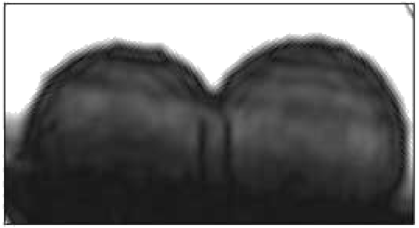
$a_o = \exp[-t / (2\lambda_i)]$
$\varphi_o = CE \int V dz$

$C = (I_{max} - I_{min}) / (I_{max} + I_{min})$
$= 2a_o / (1 + a_o^2)$

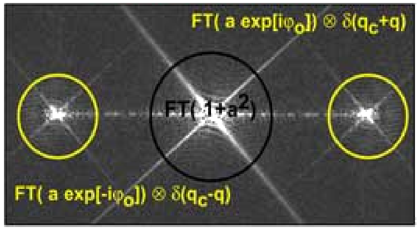
Hologram



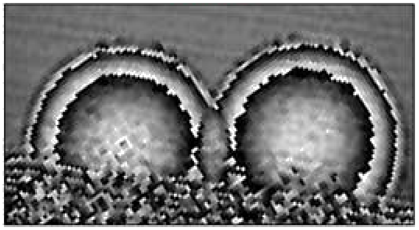
Amplitude



Fourier Transform

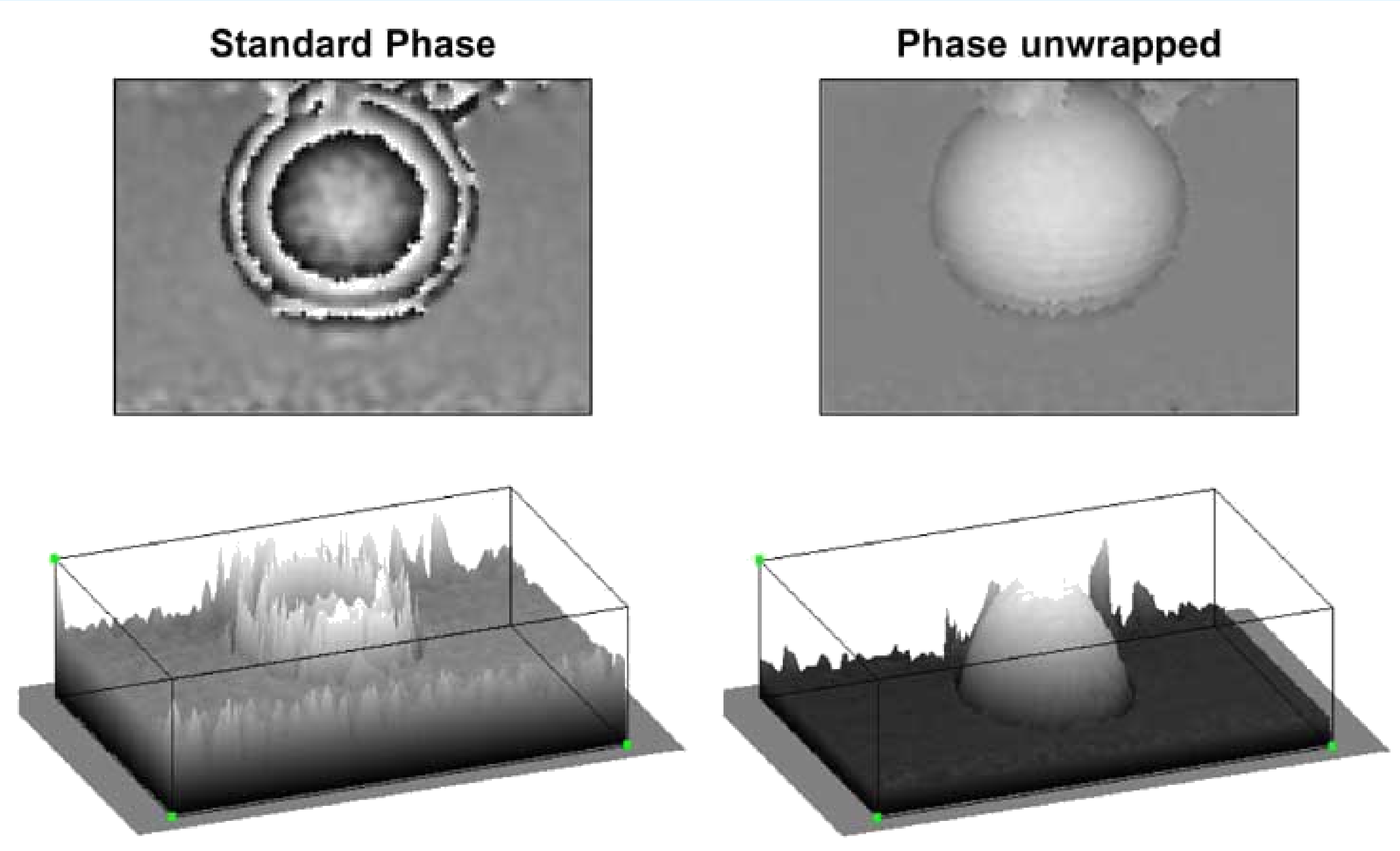


Phase



Hollography allows to obrain:
- 3D thin foil thickness maps
- 2D electric & magnetic potential maps

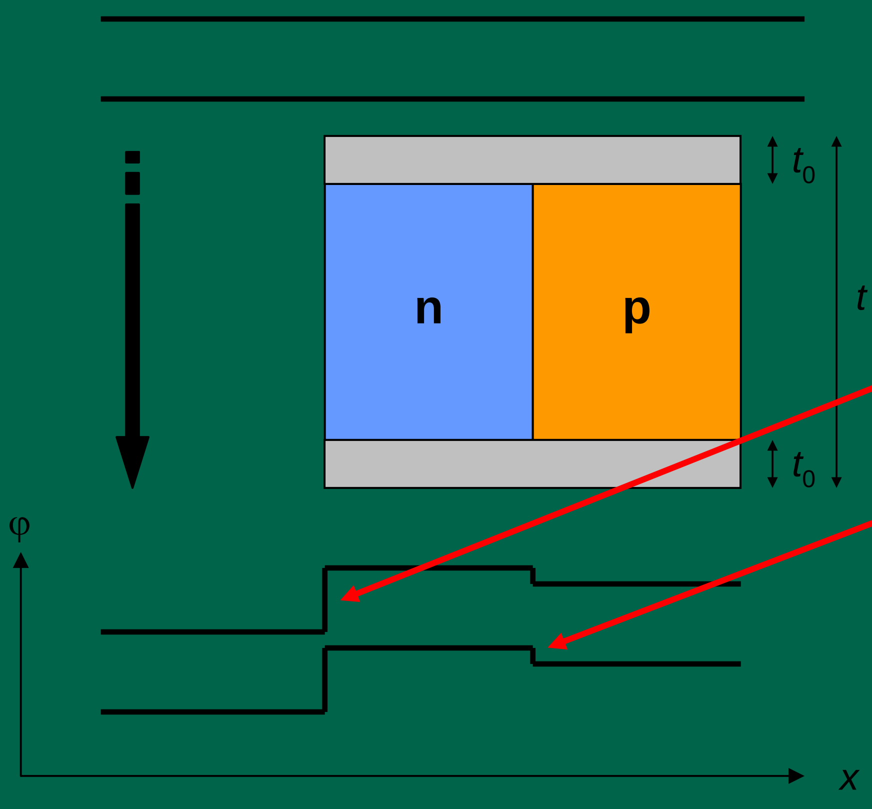
HREM - „of-axis” holography (cont.)



Applications: observations of quantum doth, quantum wells

Phase-Modulation at pn-Junctions

Phase-Modulation of Electron Wave



Phase-Shift at pn-Junction

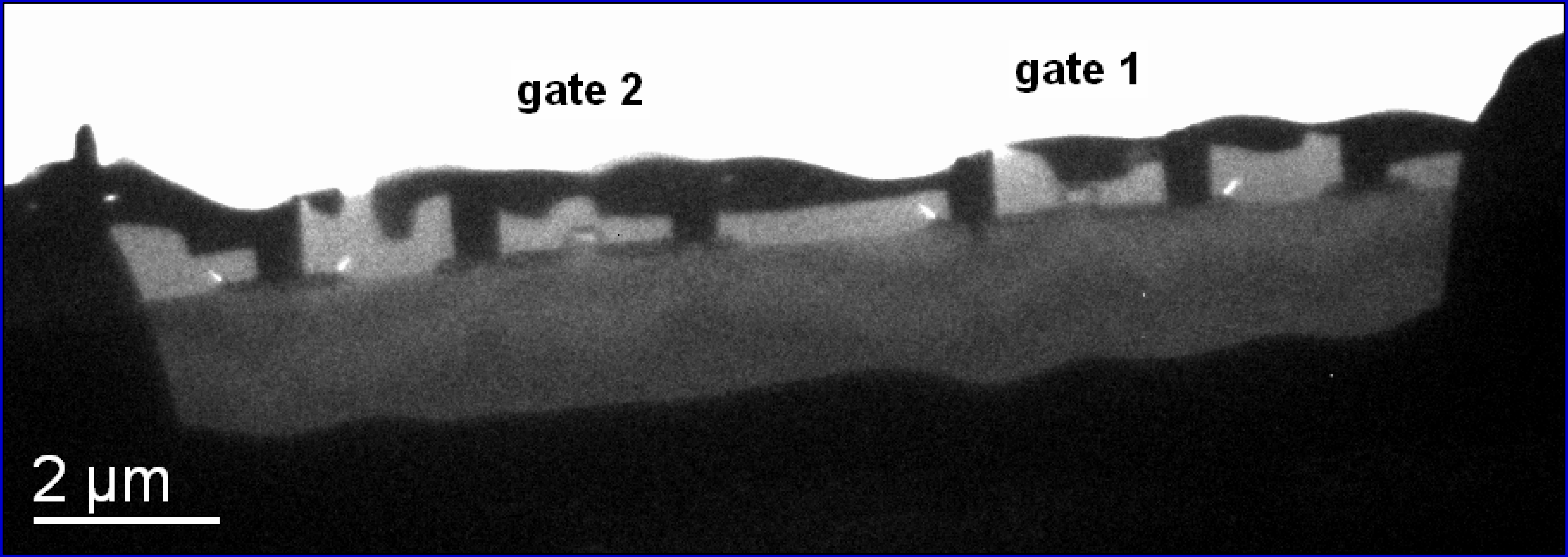
$$\varphi = \sigma \cdot (V_0\, t + \Delta V_{pn}\, (t - 2\, t_0))$$

σ	Interaction constant (0.00729 V^{-1} nm^{-1} for 200 kV)
V_0	Mean inner potential (≈ 12 V for Si)
t	*Specimen thickness*
ΔV_{pn}	Potential variation at pn-junction (≈ 0.7 ... 1.2 V)
t_0	Thickness of dead layers

Only valid for kinematic conditions!

W.D. Rau et al., PRL 1999

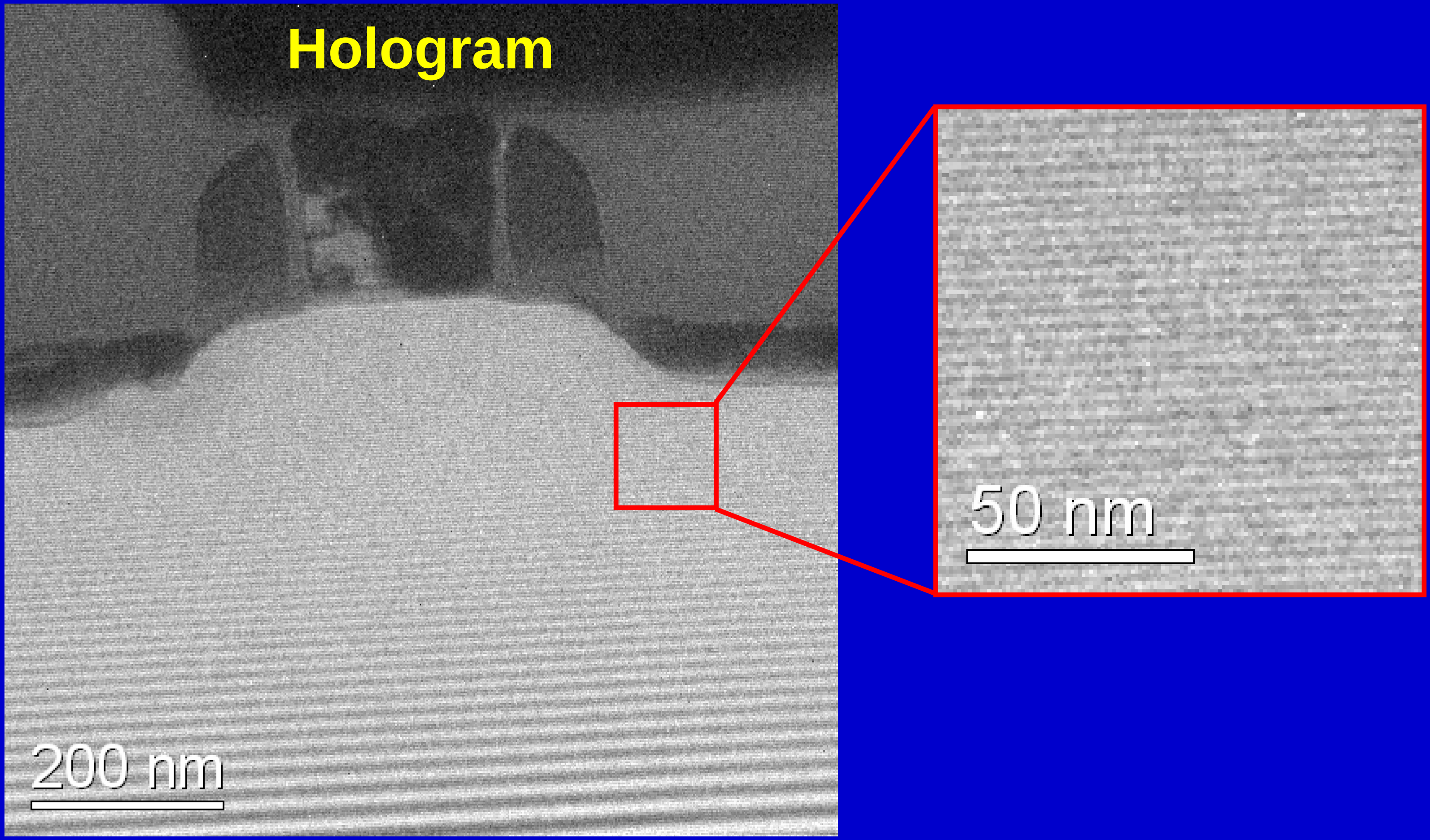
TEM-Image of FIB-Lamella



Sample: SEMATECH #16, 250 nm Gate Length

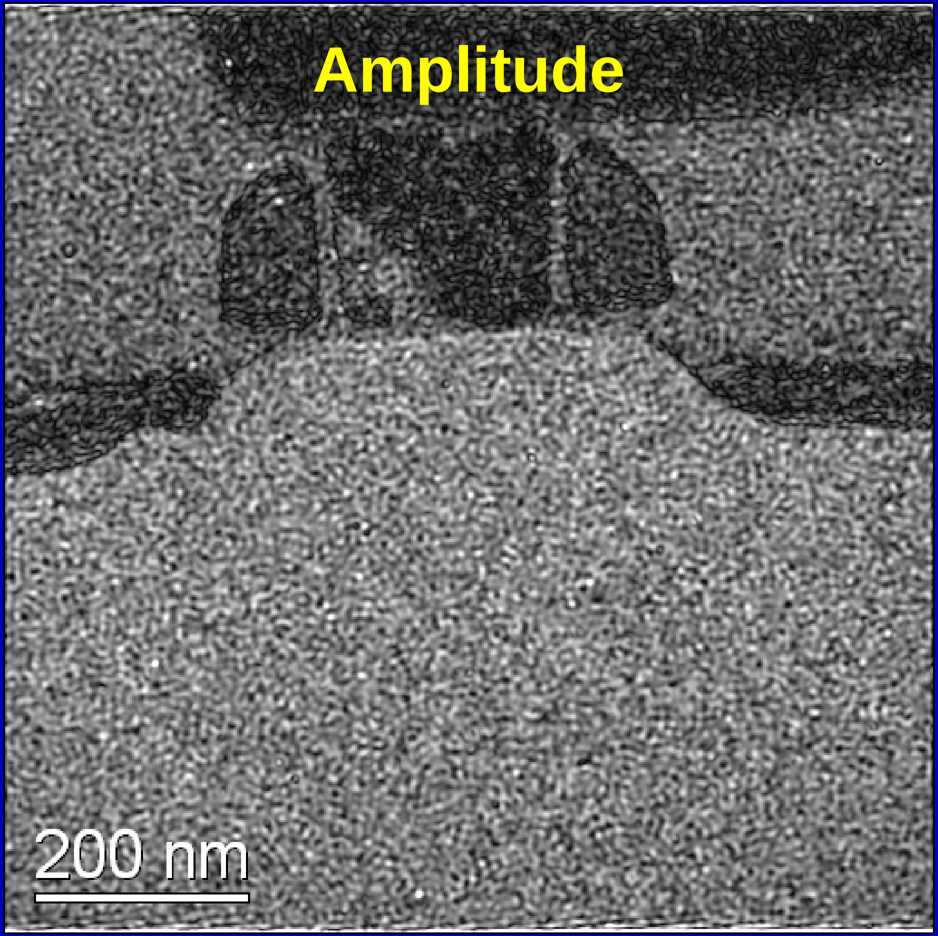
Thickness of Lamella: 200 nm

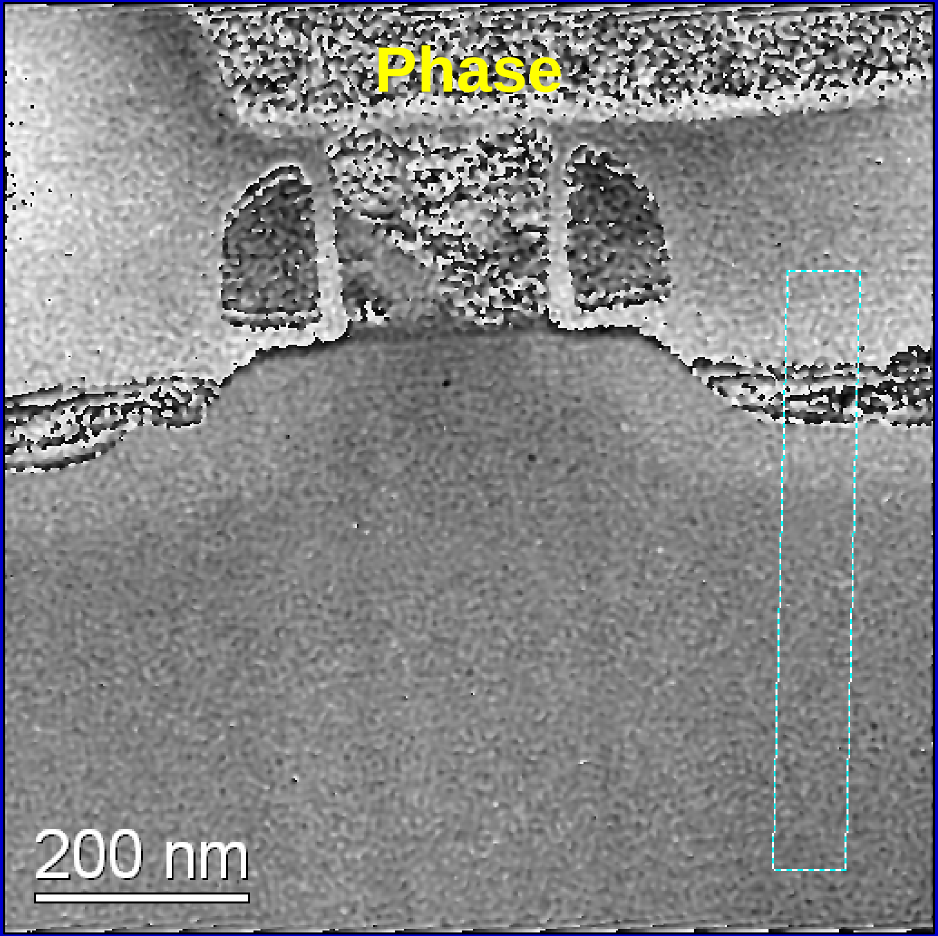
n-MOSFET-Hologram



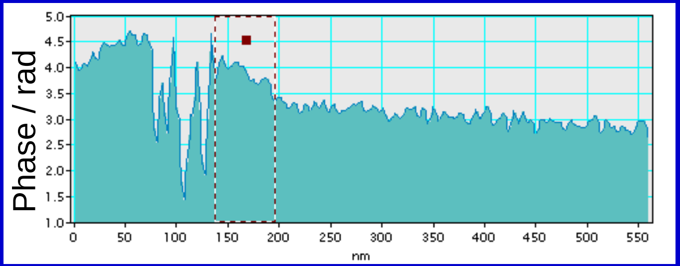
Sample: SEMATECH #16, 250 nm Gate Length, Gate 1
Microscope: Philips CM200FEG ST/Lorentz, U_A = 200 kV
Biprism Voltage: U_F = 160 V, Field of View: w = 860 nm
Fringe Spacing: s = 3.8 nm, Fringe Contrast in Reference-Hologram: μ = 0.05

n-MOSFET



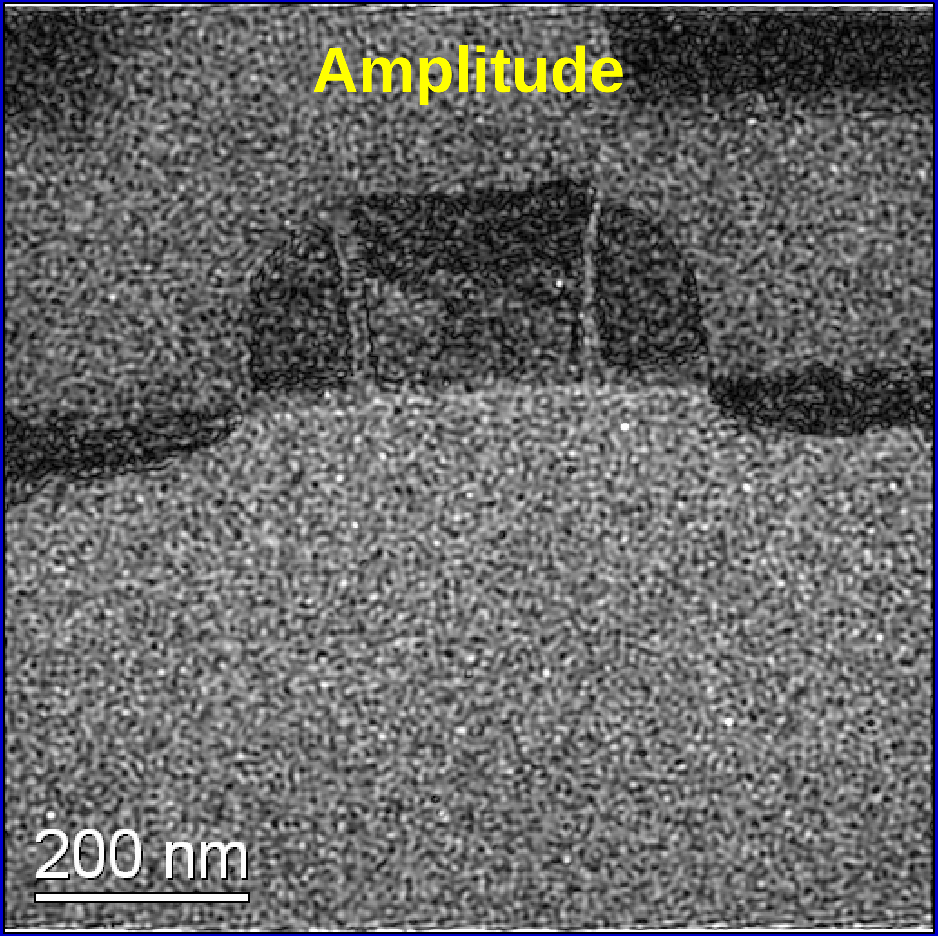


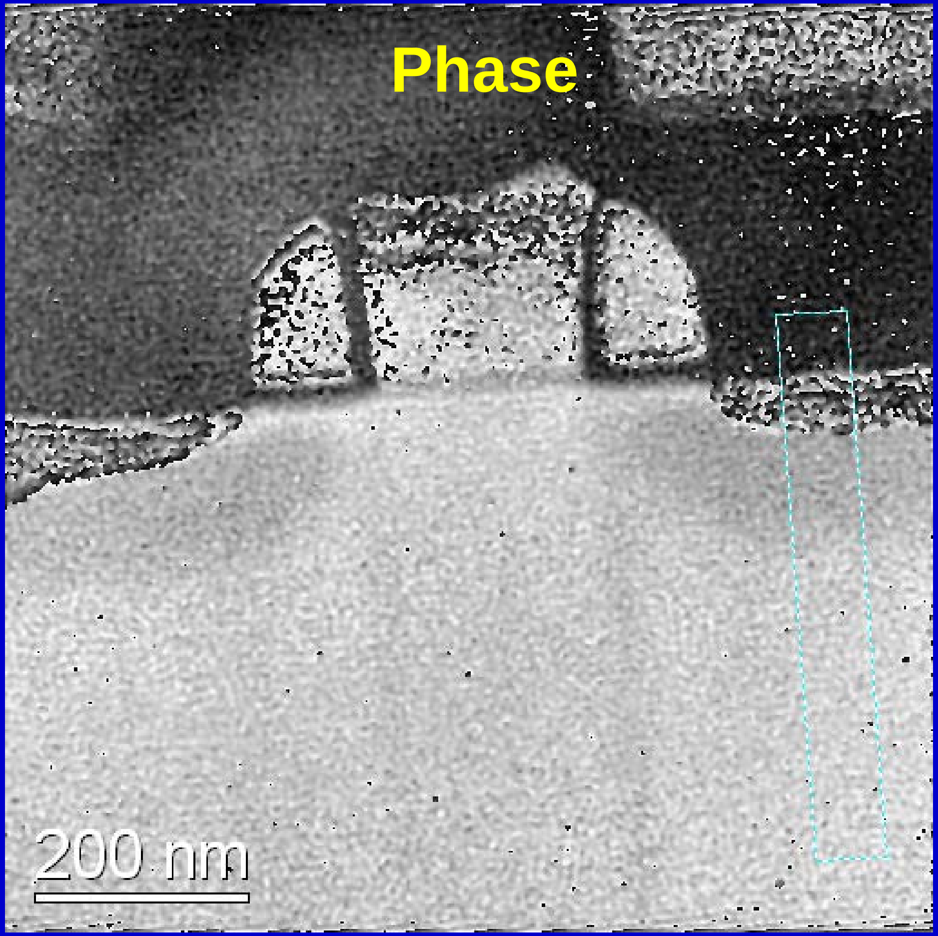
Sample: SEMATECH #16, 250 nm Gate Length, Gate 1



Approximation for Depletion Region Potential: $\Delta V_{pn} \approx 0.5$ V

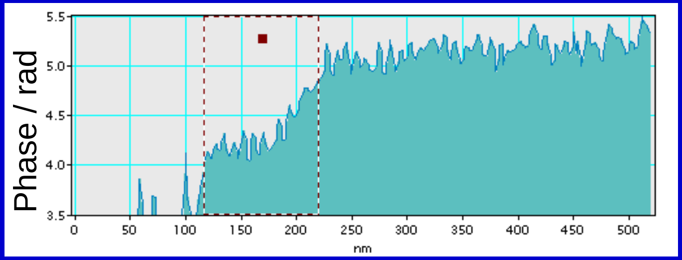
p-MOSFET



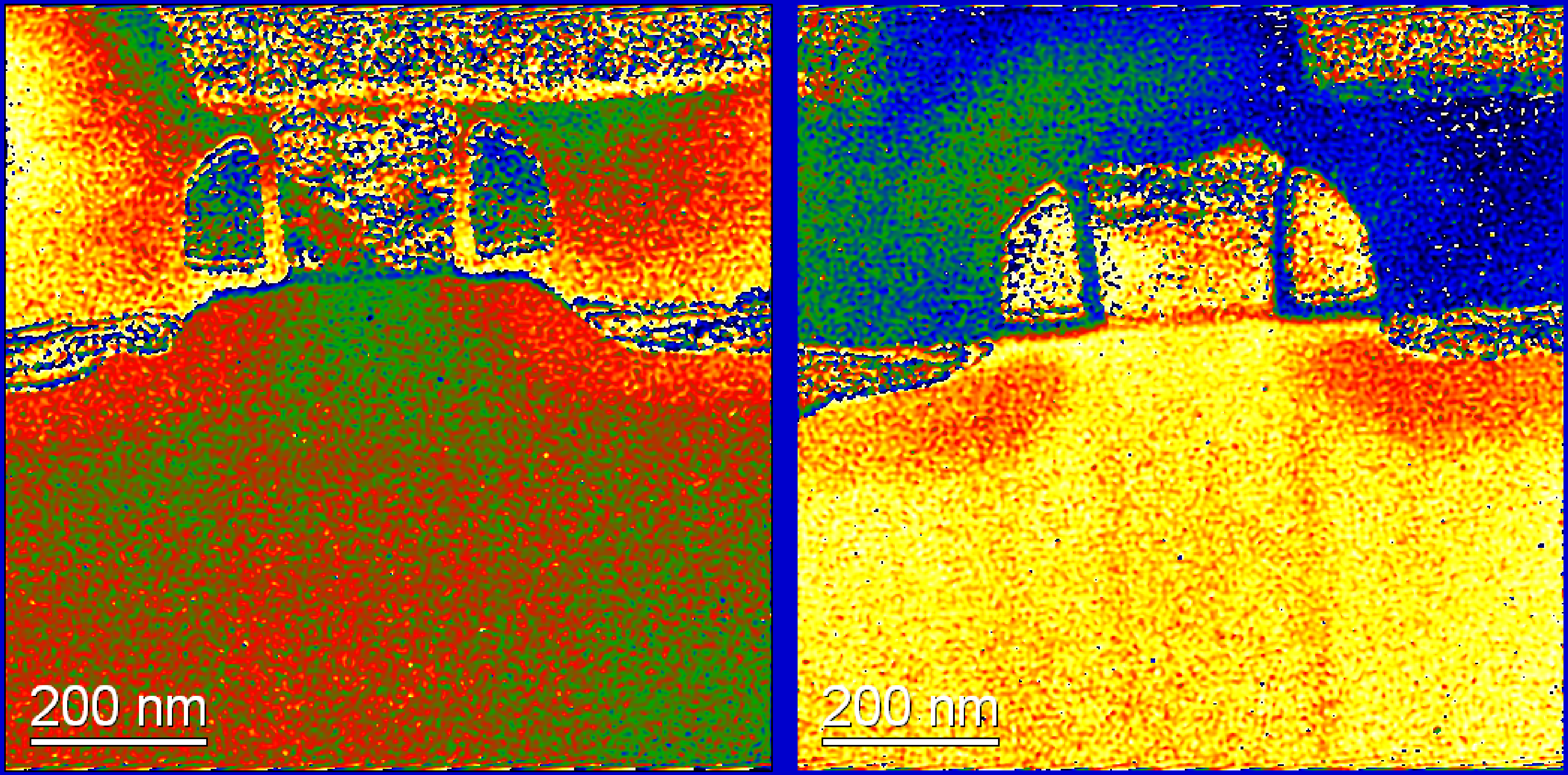


Sample: SEMATECH #16, 250 nm Gate Length, Gate 2

Approximation for Depletion Region Potential: $\Delta V_{pn} \approx -0.7$ V



Comparison

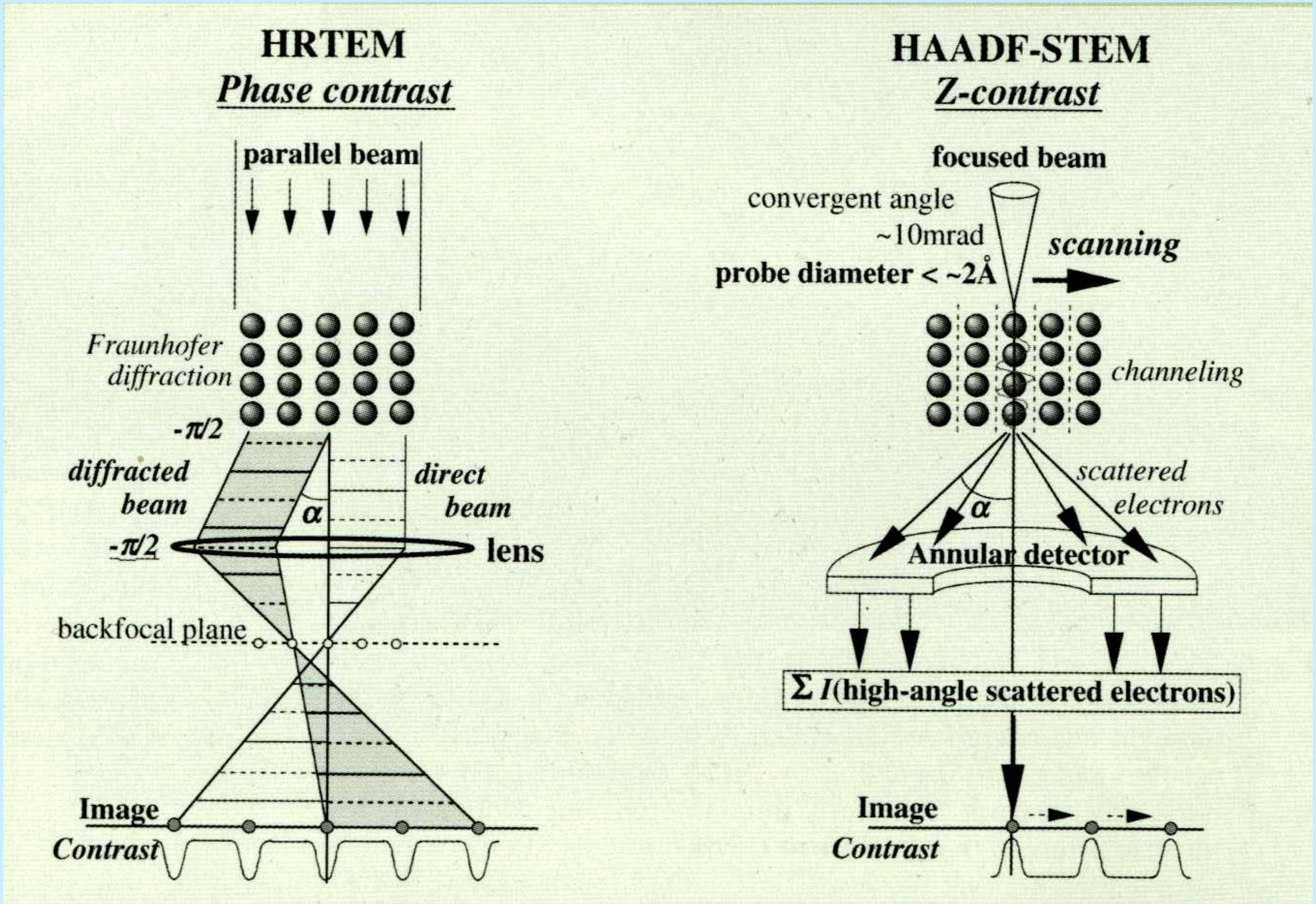


n-MOSFET

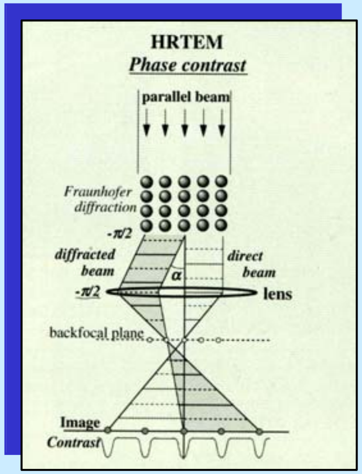
p-MOSFET

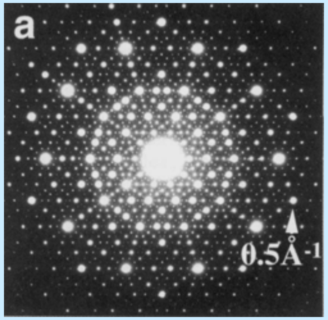
Sample: SEMATECH #16, 250 nm Gate Length

„HREM” ⇒ HAADF-STEM

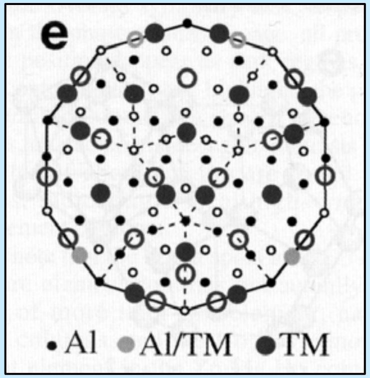


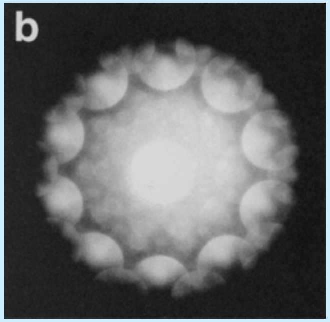
„HREM” ⇒ HAADF-STEM

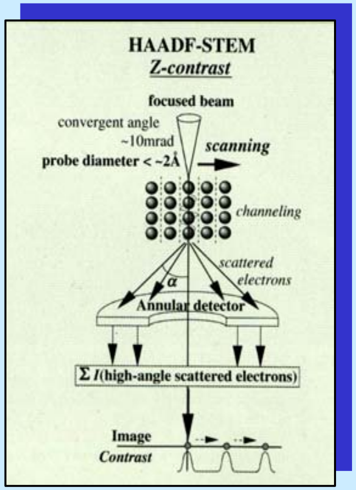


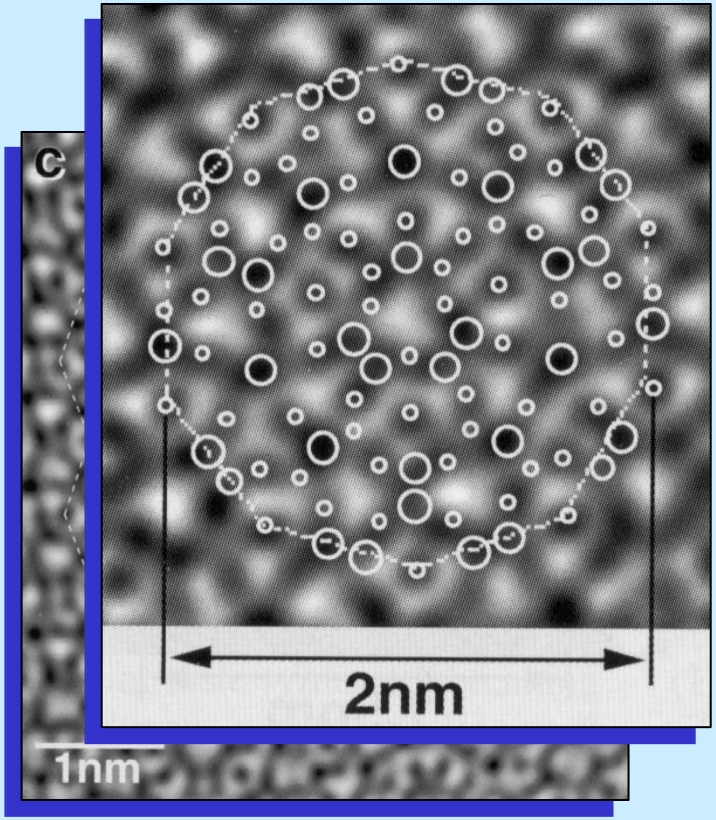


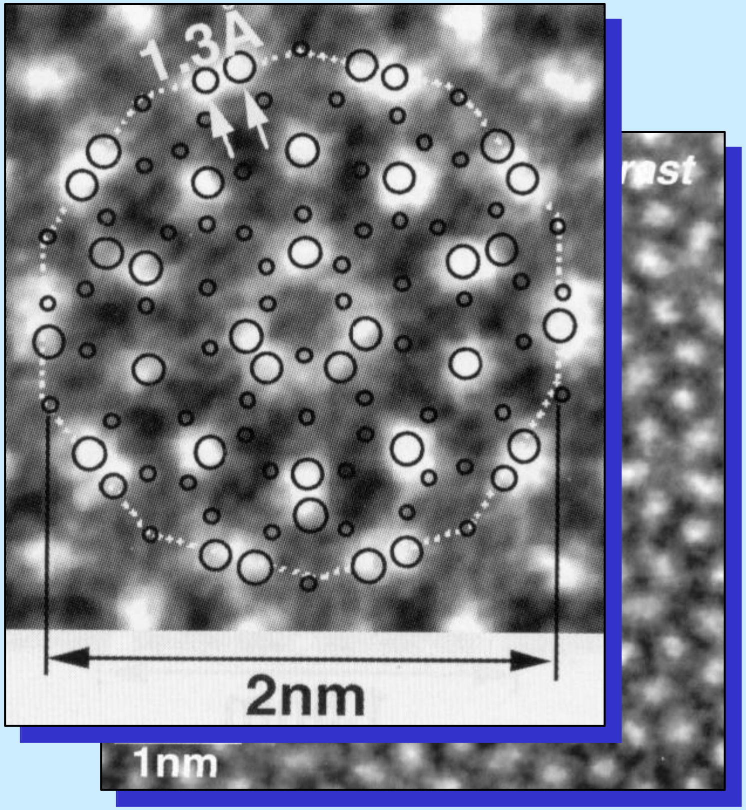
$Al_{72}Ni_{20}Co_8$











Structure – Image relationship

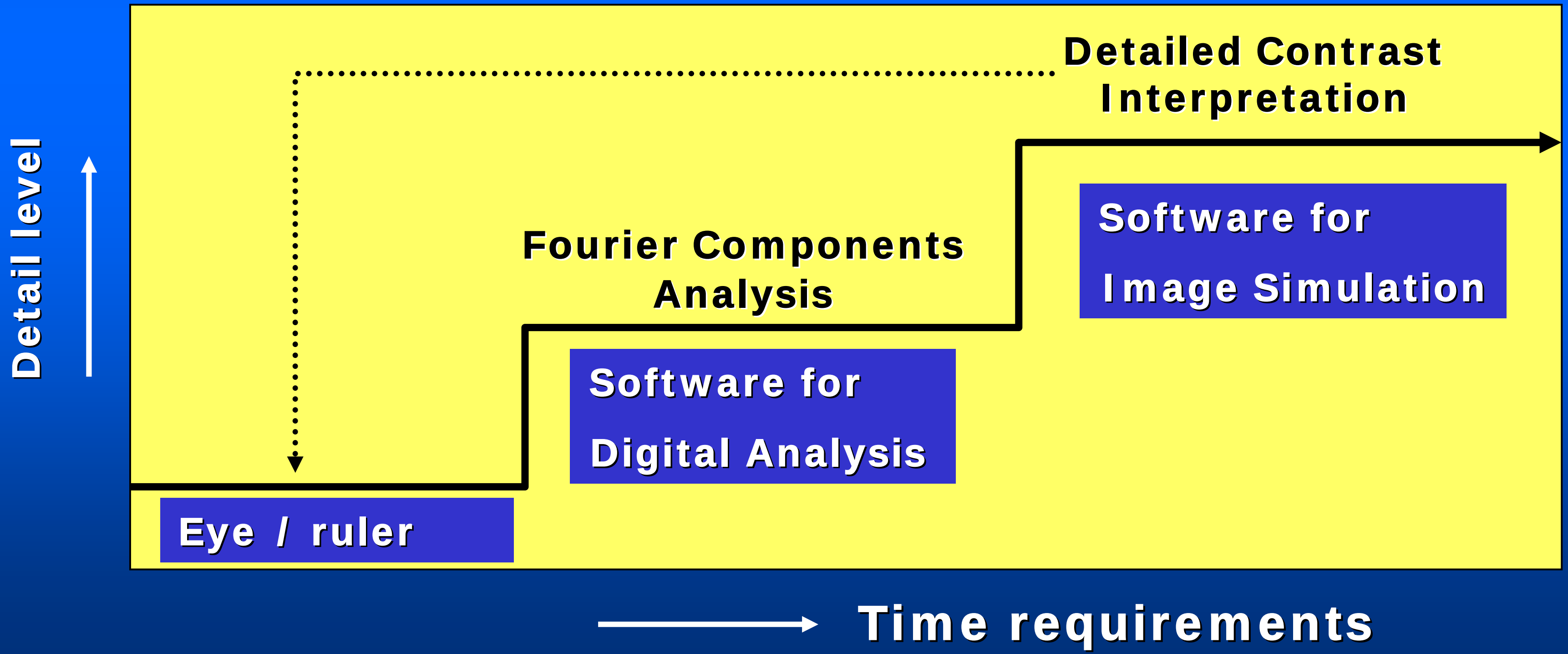
- Only for very thin crystals (kinematic scattering) and under proper recording conditions (Scherzer defocus) **HREM image** contrasts **may be DIRECTLY interpreted** in terms of position of atomic columns

- Otherwise, HREM image contrast interpretation must be done by **MATCHING experimental and CALCULATED /** SIMULATED **images**

- Although a direct retrieval of the structure from **HREM experimental images** is usually impossible, though these images **always contain rich crystallographic information**

HREM image interpretation

- Useful tools :
  - Electron Microscopy Simulation Software
  - Structure Modeling tools (complex supercells)
  - Image Processing (Fourier Analysis)



Detail level

Eye / ruler

Software for Digital Analysis

Fourier Components Analysis

Software for Image Simulation

Detailed Contrast Interpretation

Time requirements