07 December2018

Transmission Electron Microscopy

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K. Urban, Umbrella Winter School: *Methods for Material Characterization*, Kfar Blum/Israel, 2018

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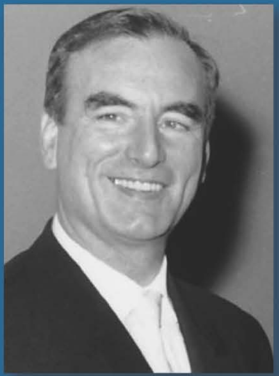
1931: (Conventional) Transmission Electron Microscope (CTEM)



Ernst Ruska

1937: Scanning Transmission Electron Microscope (STEM)

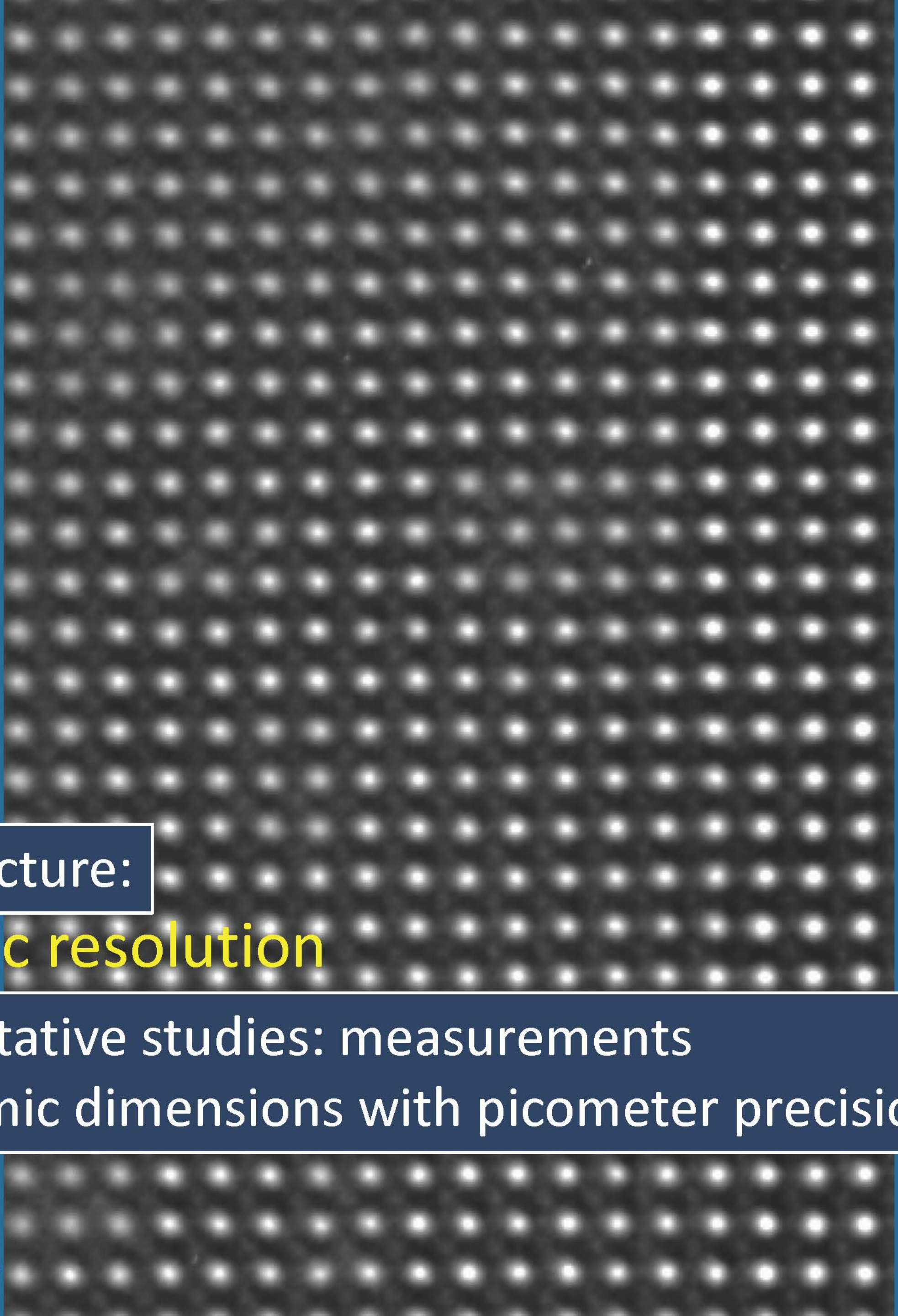
Manfred v. Ardenne

1997: Aberration corrrected optics

Max. Haider

Harald Rose

Knut Urban



1 CTEM

2 STEM

This lecture:

atomic resolution

quantitative studies: measurements
in atomic dimensions with picometer precision

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Transmission Electron Microscopy
CTEM

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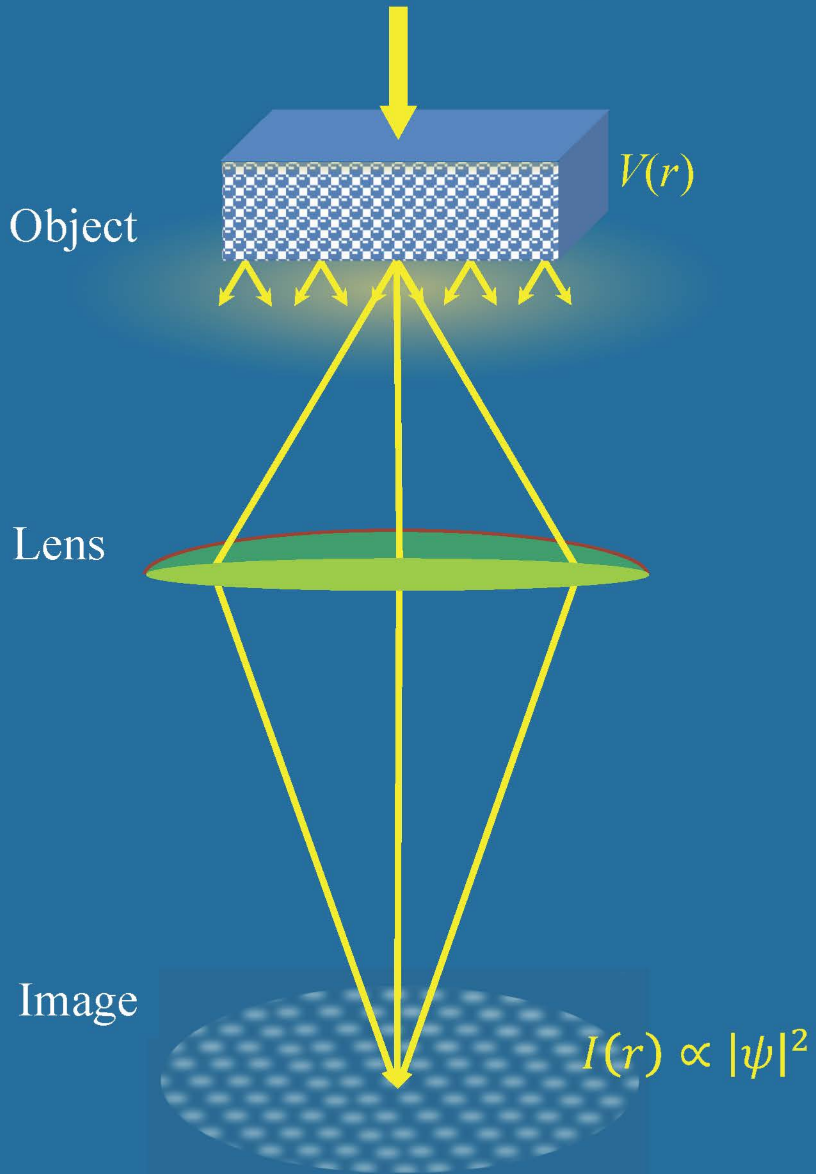
Part I

How is an atomic image formed
in the transmission electron microscope?

The principles of imaging

$$\psi(r) = exp(2\pi i k_0 r)$$



$$\rho(r) = \sum_i \rho_i(r) * \delta(r - r_i)$$

unknown structure

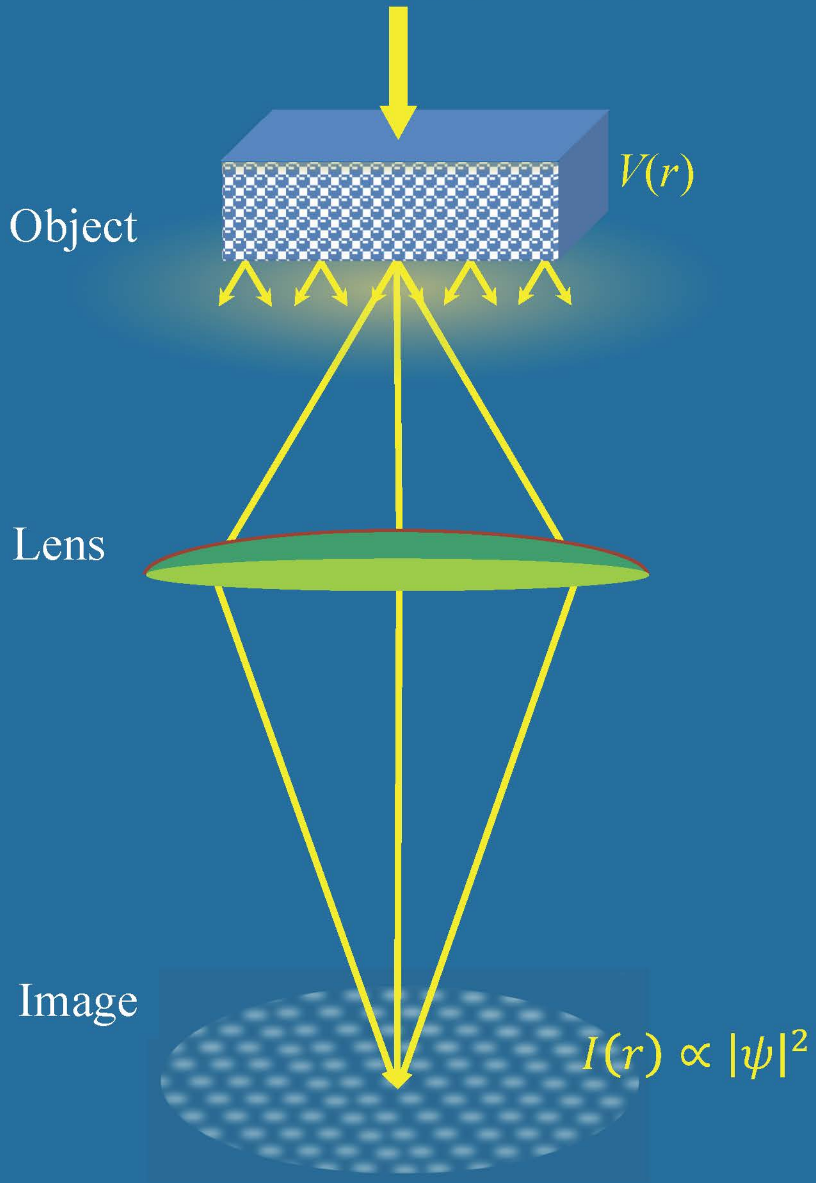
exit-plane wave function

$$\psi(r) = \int_g \psi(g)\, exp\{2\pi i(k_0 + g)r\}dg$$

g - spatial frequency

The principles of imaging

$$\psi(r) = exp(2\pi i k_0 r)$$



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exit-plane wave function

$$\psi(r) = \int_g \psi(g) \, exp\{2\pi i(k_0 + g)r\} dg$$

g - spatial frequency

lens aberrations

aberration-induced phase shifts

$$\psi(g) exp\{-2\pi i \chi(g)\}$$

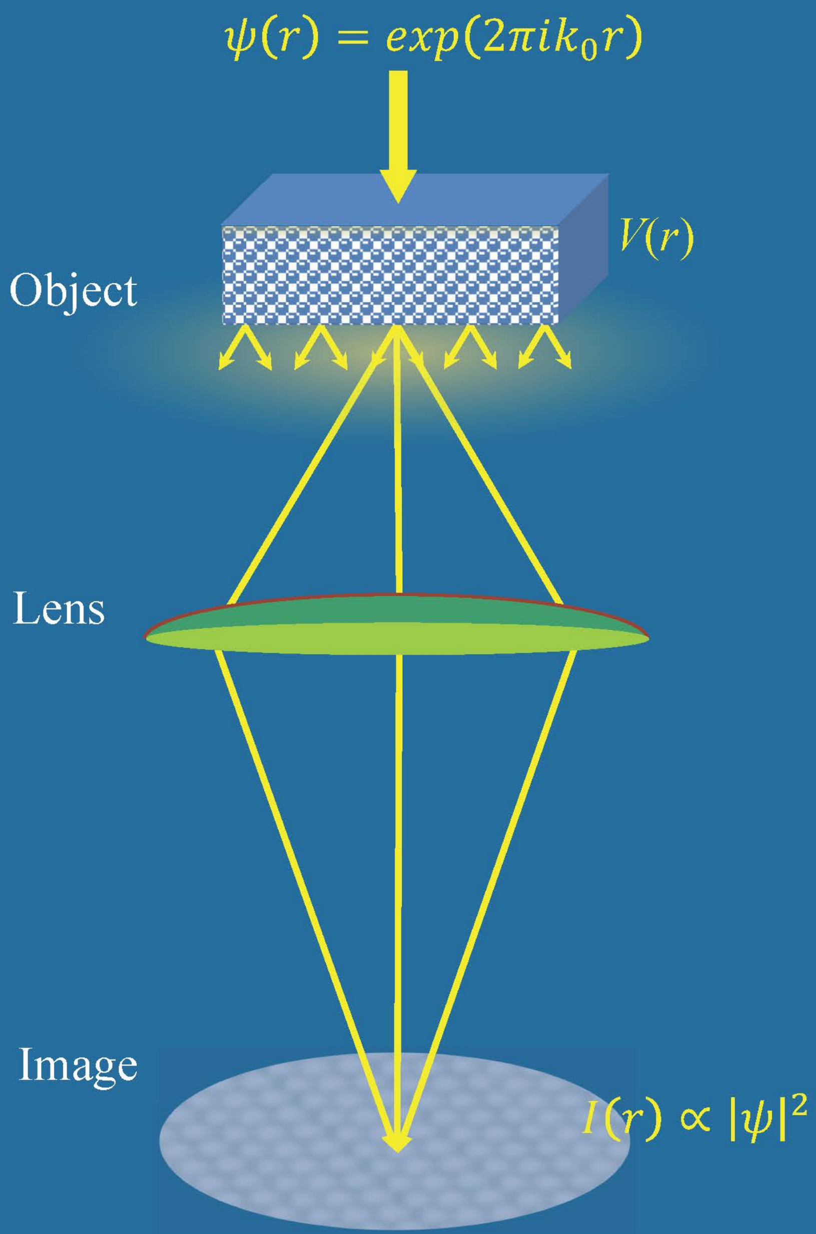
$$\chi(g) = \frac{1}{4} C_S \lambda^3 g^4 + \frac{1}{2} Z \lambda g^2 + \cdots$$

aberration function

spherical aberration

defocus

The principles of imaging



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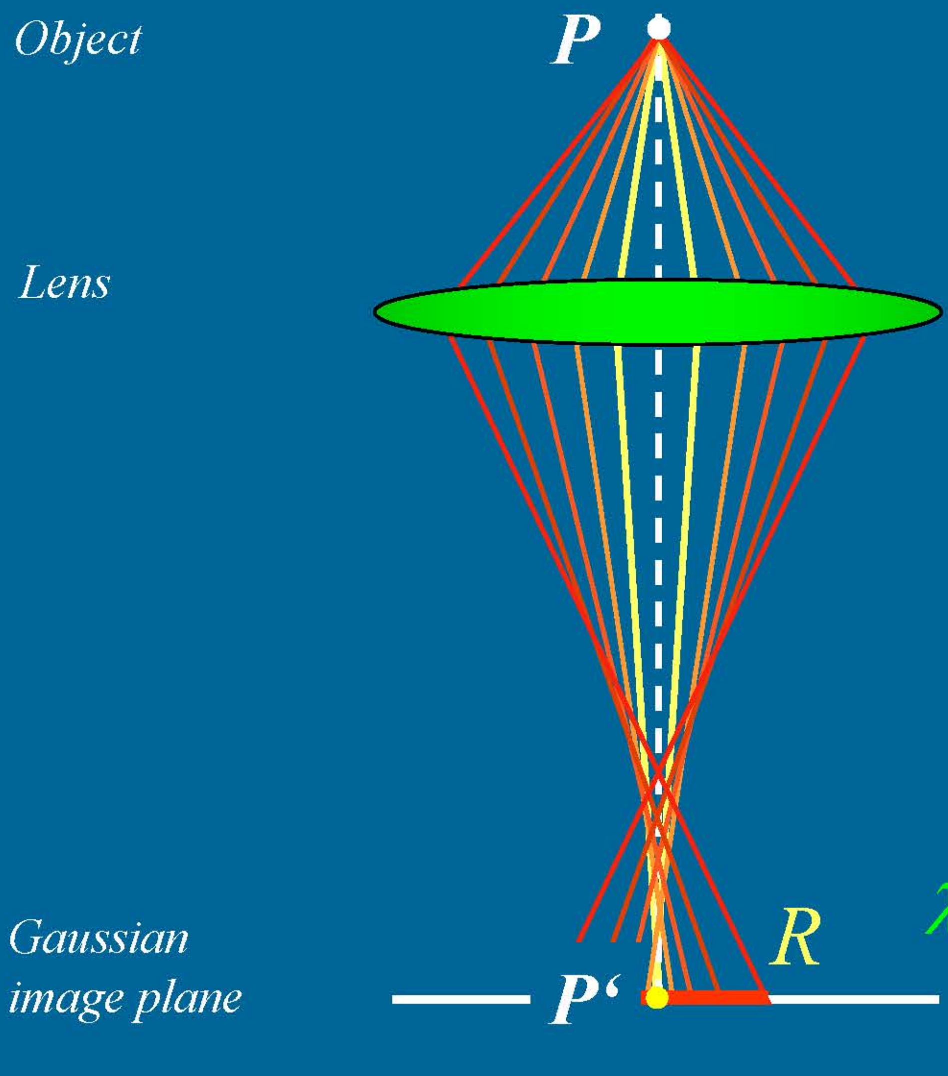
spherical aberration

defocus

Spherical aberration

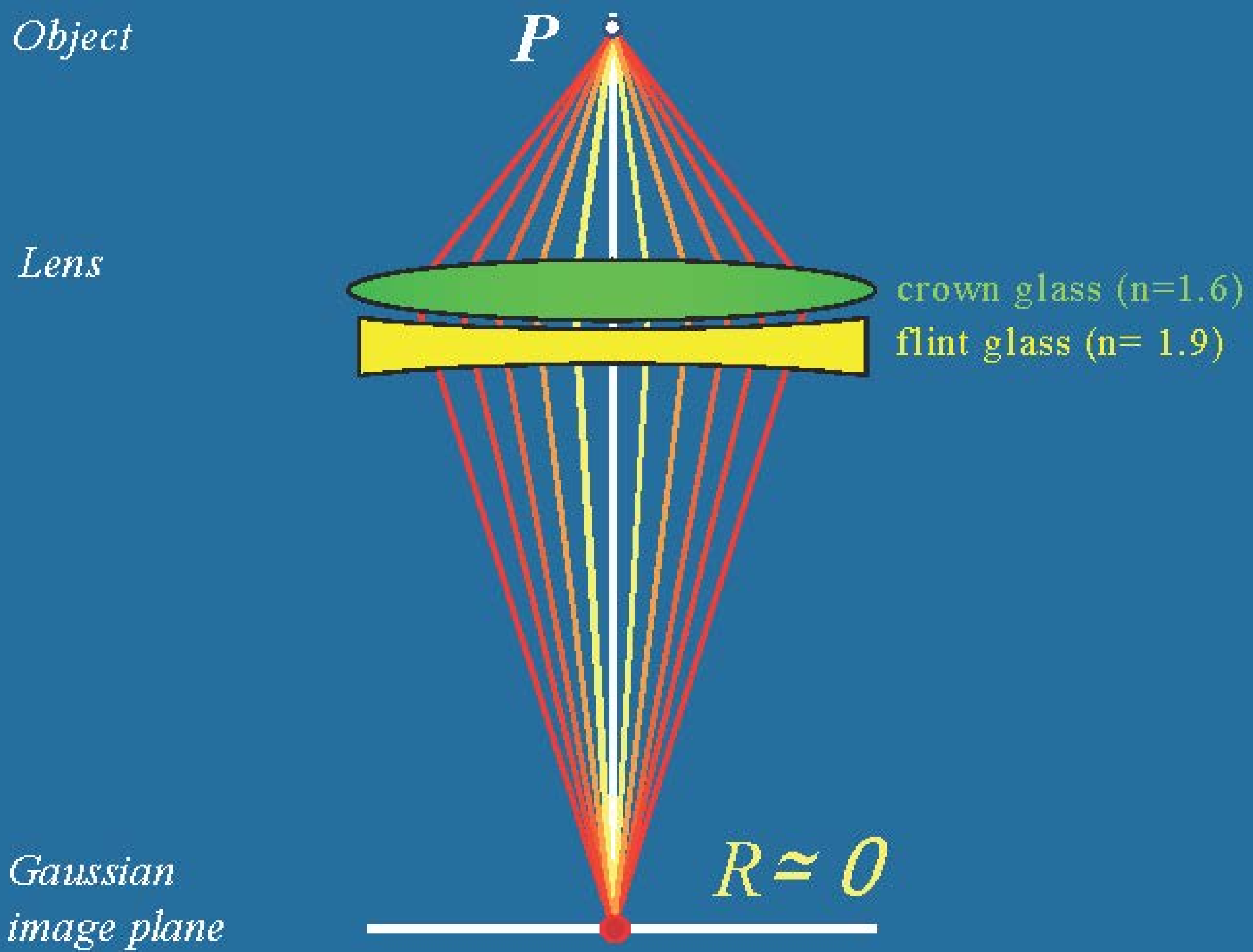


Point spread function

$$R \propto \left| \frac{\partial \chi}{\partial g} \right|_{\max}$$

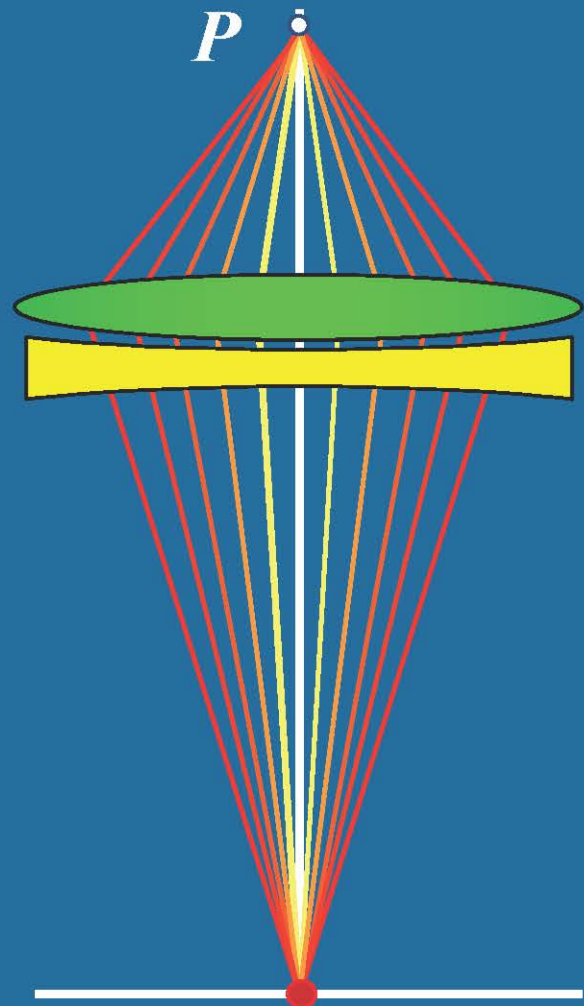
$$\chi(\mathbf{g}) = \tfrac{1}{4} C_S \lambda^3 \mathbf{g}^4 + \tfrac{1}{2} Z \lambda \mathbf{g}^2 + \ldots$$

Spherical aberration **in light microscopy**

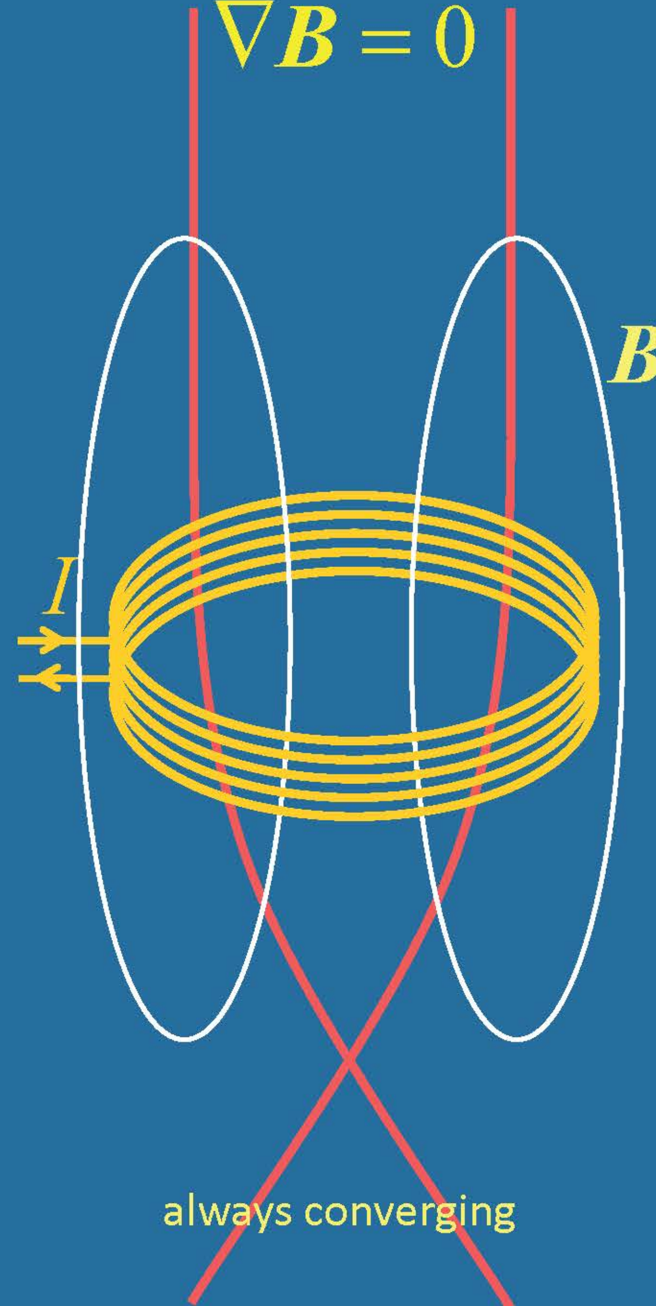


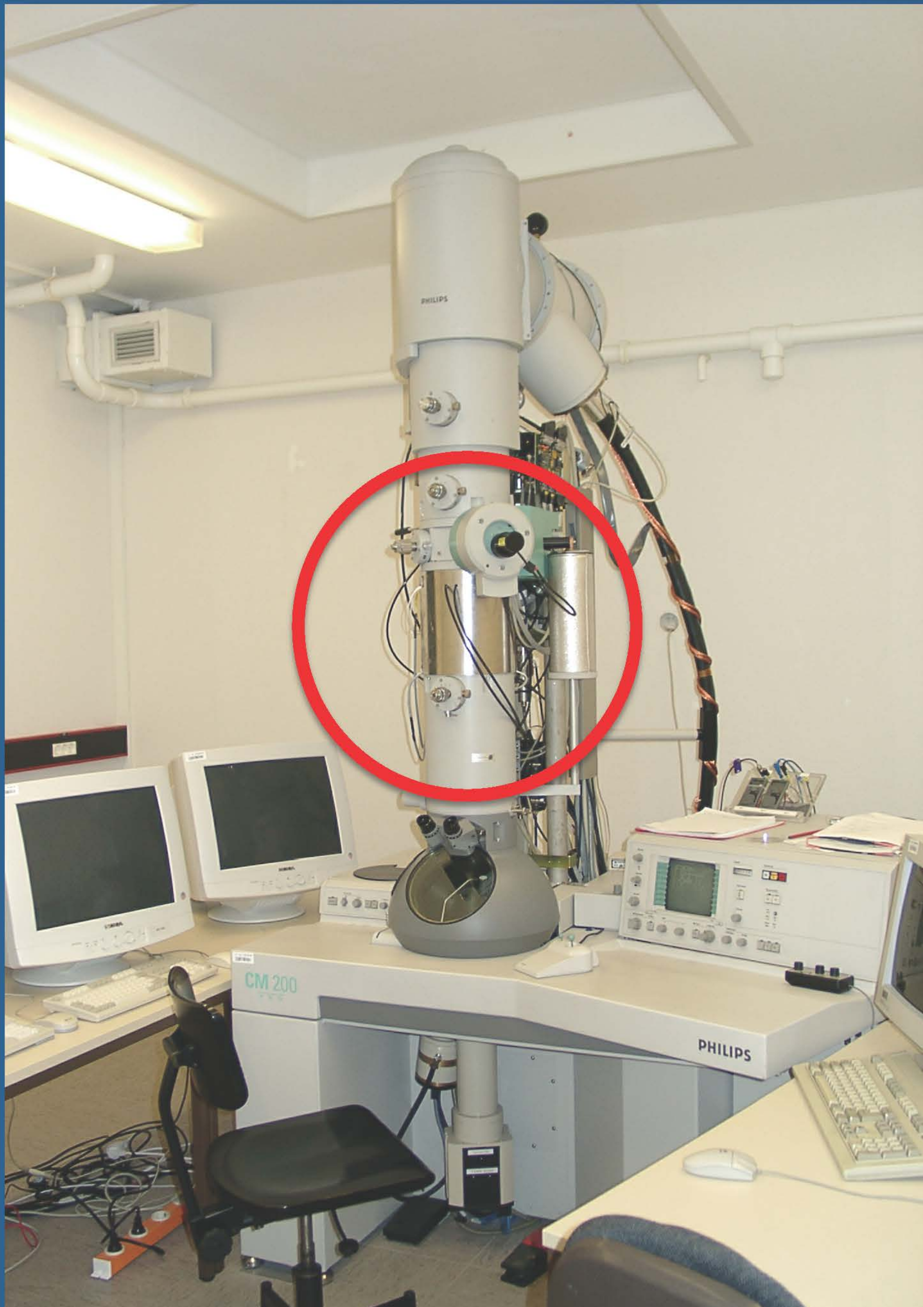
Ernst Abbe, 1875

Spherical aberration **in electron microscopy**



Gauss‘ law of magnetism





1997

the world‘s first
aberration-corrected electron microscope

M. Haider, H. Rose, K. Urban et al. ***Nature* 392**, 768 (1998)

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2004

FEI
thermoscientific

HITACHI

JEOL

nion

2004

As of today: about 800 aberration-corrected electron microscopes

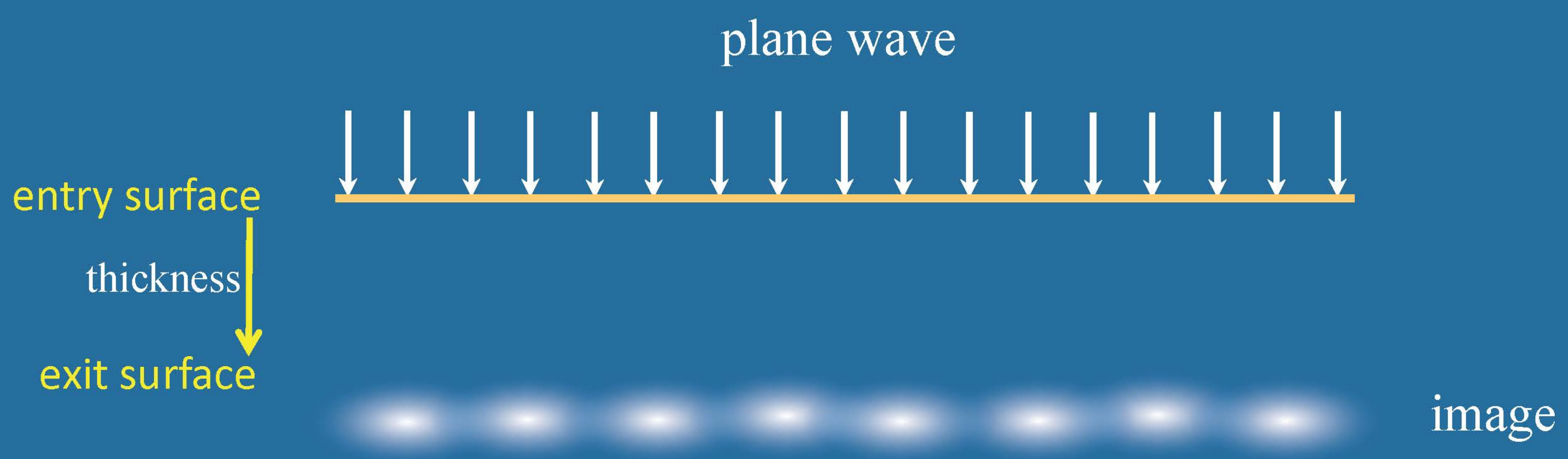
All - CTEM and STEM - have our double-hexapole correction system
(except for @ 25 Nion instruments)

Part II

How do the atoms produce contrast?

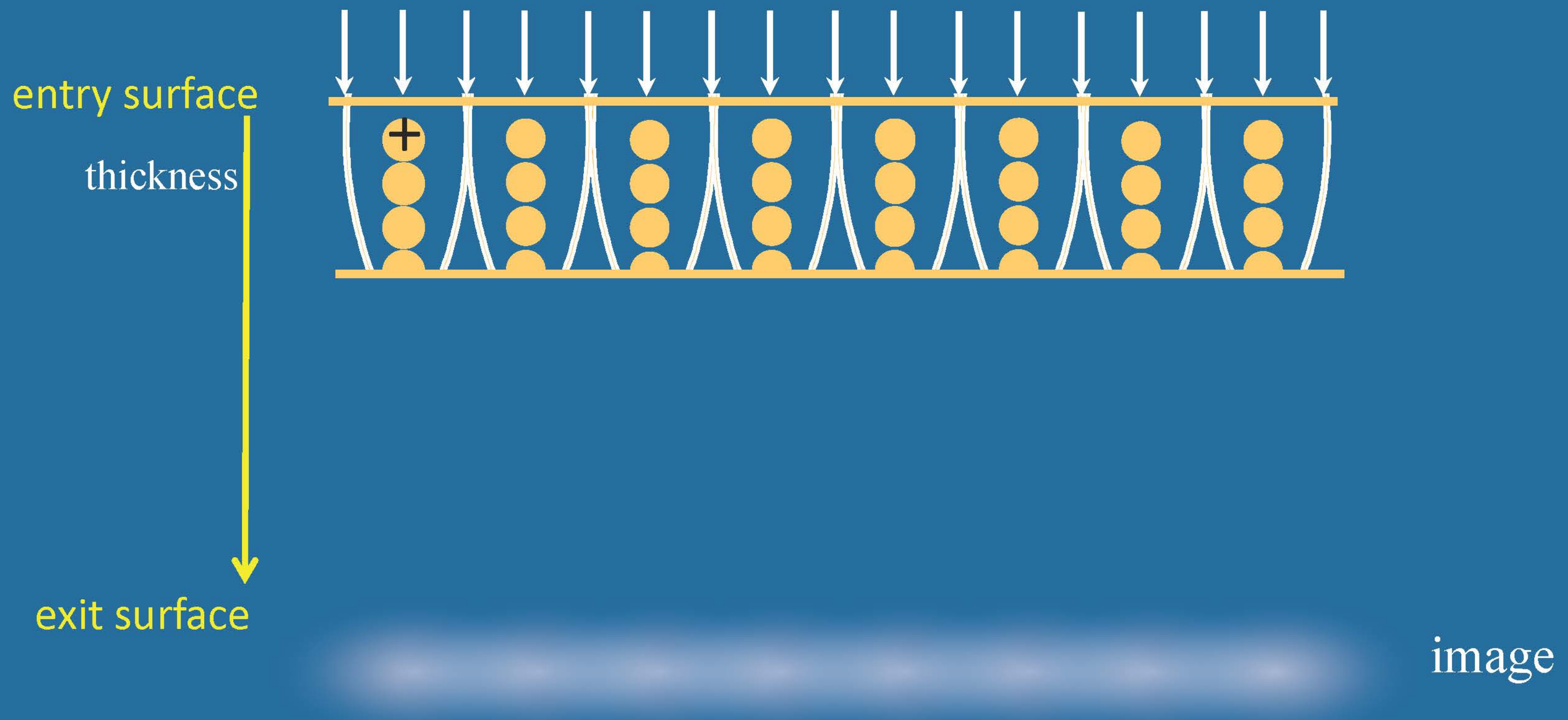
Amplitude contrast in atomic imaging

Electron diffraction channelling



Amplitude contrast in atomic imaging

Electron diffraction channelling



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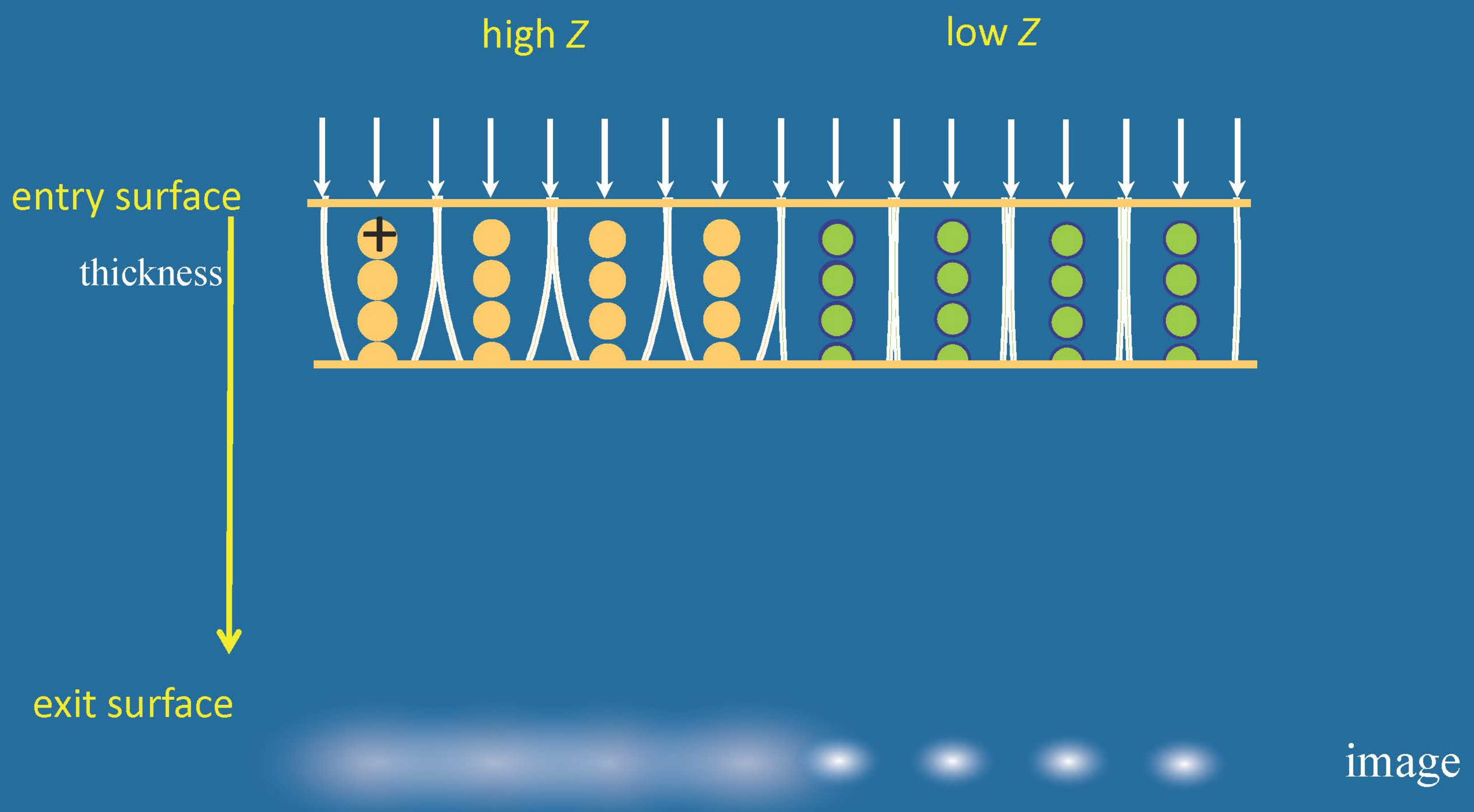
<u>Amplitude</u> contrast in atomic imaging

Electron diffraction channelling



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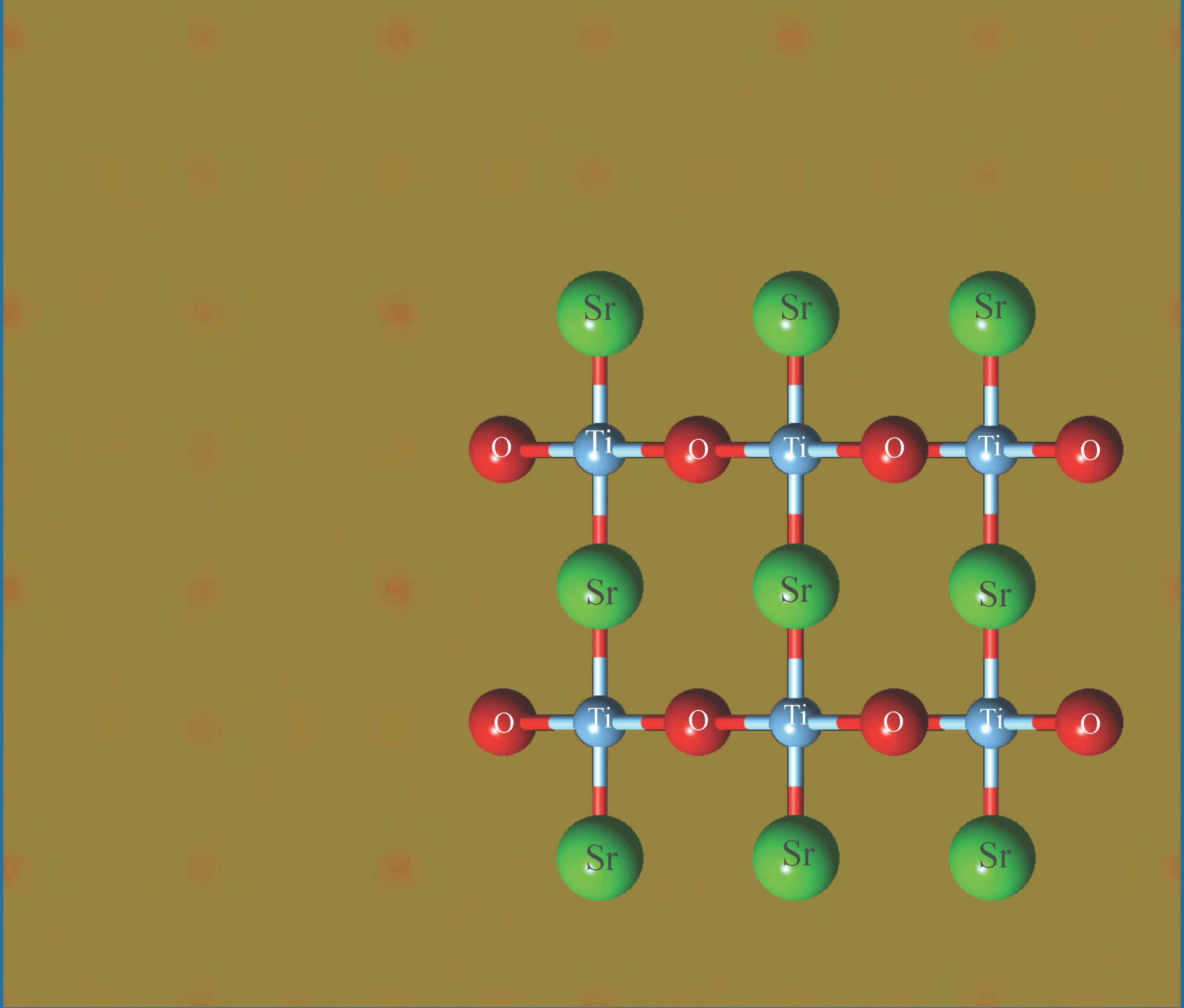
increasing video time t = increasing depth in crystal d

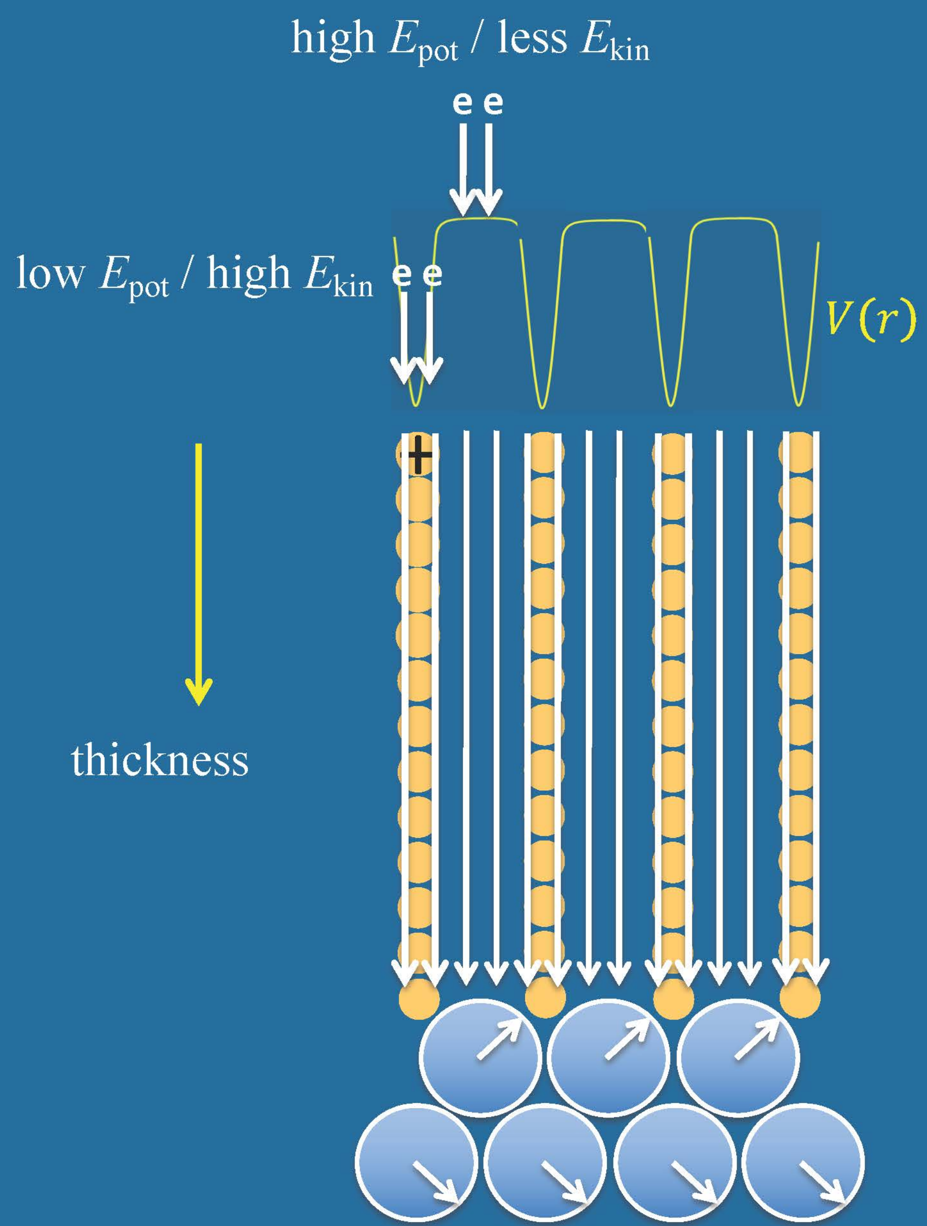


Sr (Z = 38)

Ti (Z = 22)

O (Z = 8)

Phase contrast in atomic imaging



Electrons moving at the atoms
are faster:
their phase is more advanced

Phase Contrast

Phase (differences) cannot be seen

Zernike's technique
allows to convert phase contrast
into amplitude contrast

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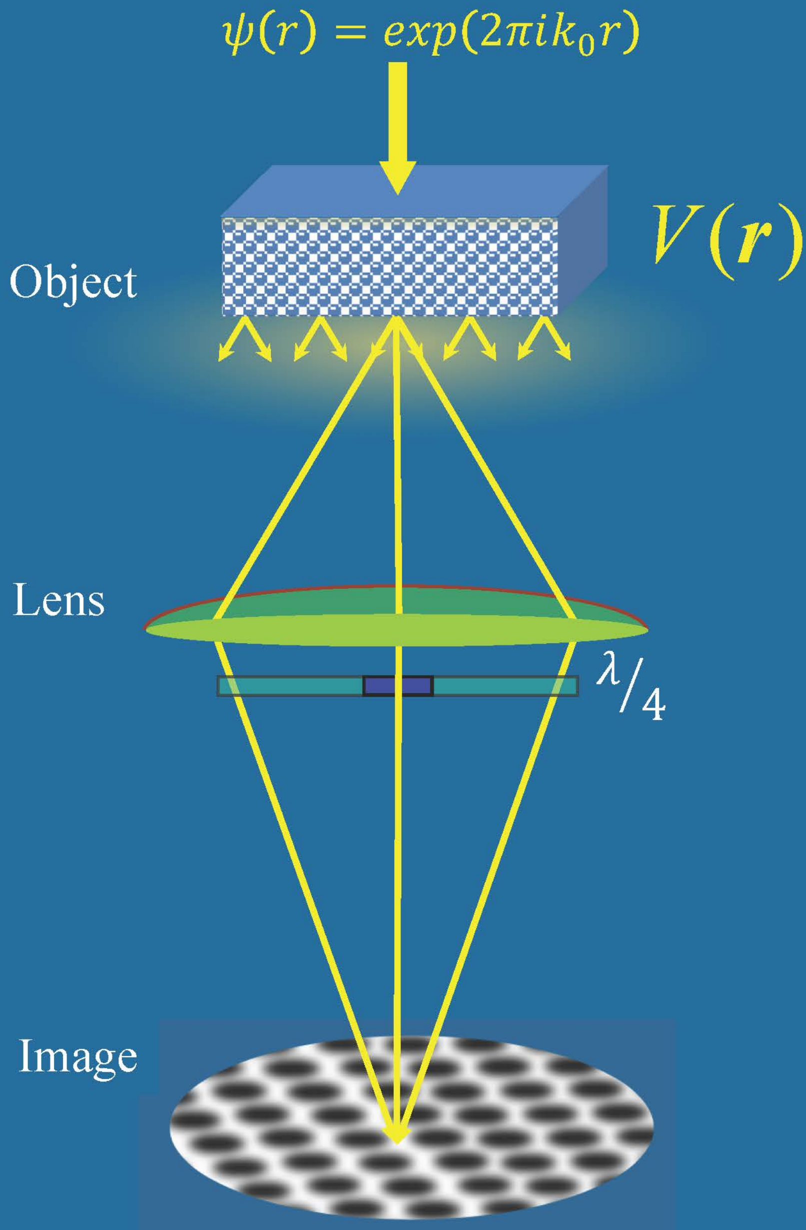
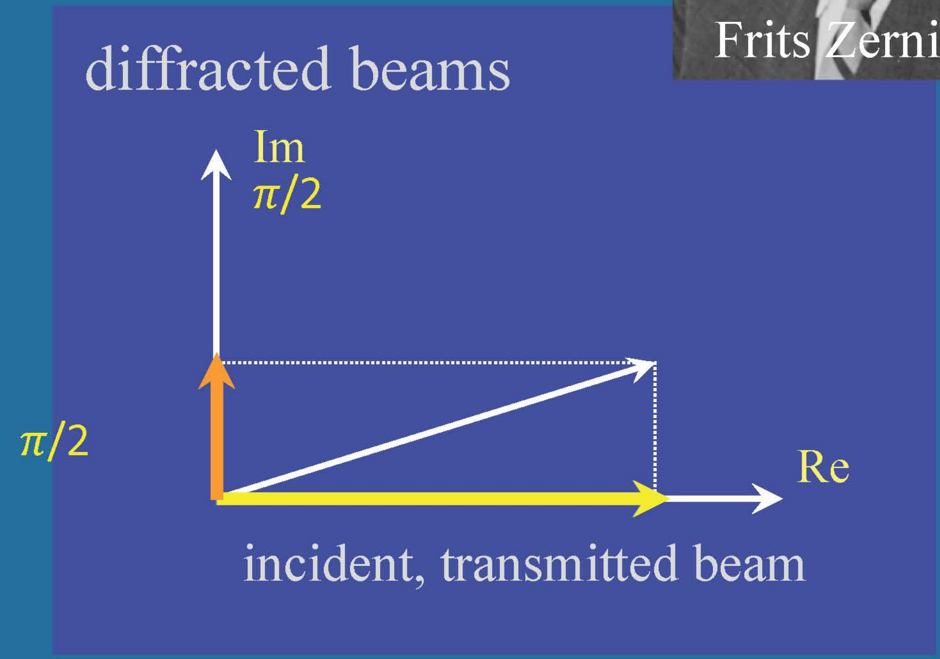
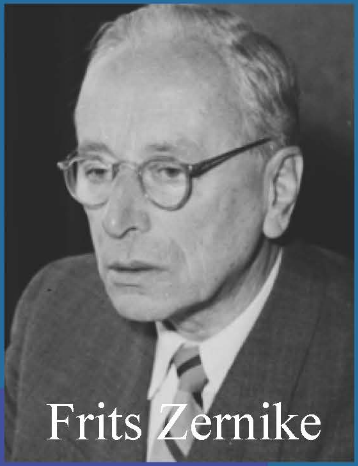
Zernike technique in the light microscope (1930)



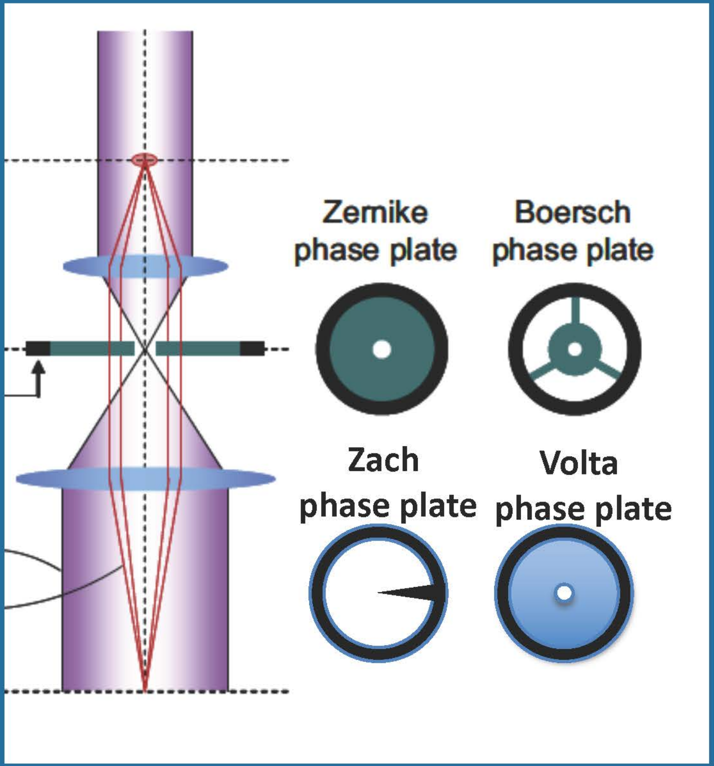




dark contrast on a bright background

Zernike technique in the electron microscope

typically by 5 orders smaller λ



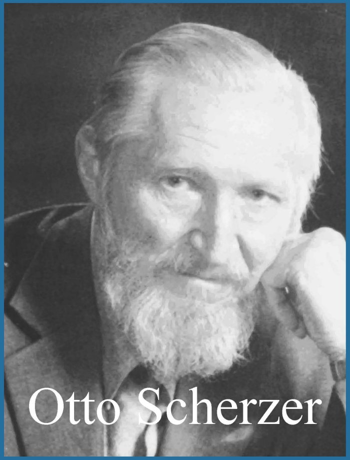
W.-H. Chang et al., Structure **18**, 17 (2010)
R. Danev et al., PNAS 111, 15635 (2014)

Applications of phase plates mainly in biology

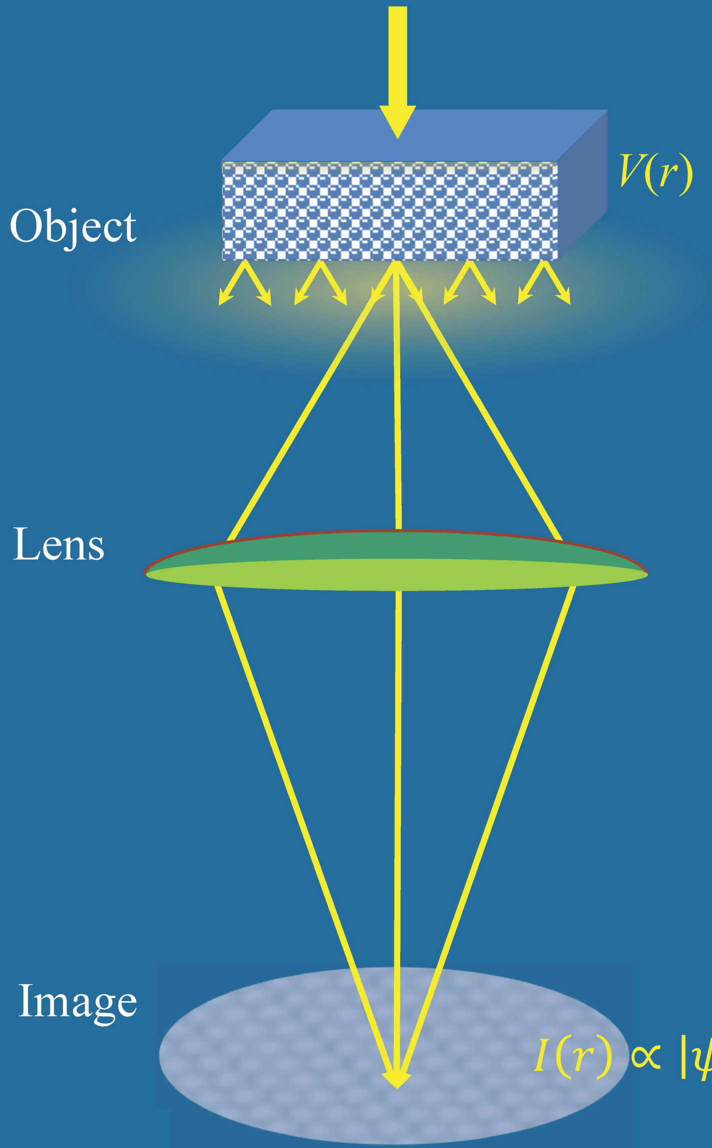
In materials science:
defocus technique,
i.e.
the lens has two functions:

1) to image
2) and to provide contrast

Zernike technique in the electron microscope (1949)



$$\psi(r) = exp(2\pi i k_0 r)$$



exit-plane wave function

$$\psi(r) = \int_g \psi(g)\, exp\{2\pi i (k_0 + g) r\} dg$$

lens aberrations

aberration-induced phase shifts

$$\psi(g) exp\{-2\pi i \chi(g)\}$$

$$\chi(g) = \frac{1}{4} C_S \lambda^3 g^4 + \frac{1}{2} Z \lambda g^2 + \cdots$$

spherical aberration

defocus

$$I(r) \propto |\psi|^2 \propto sin 2\pi \chi(g)$$

contrast transfer function

Scherzer 's theory for phase contrast (1949)

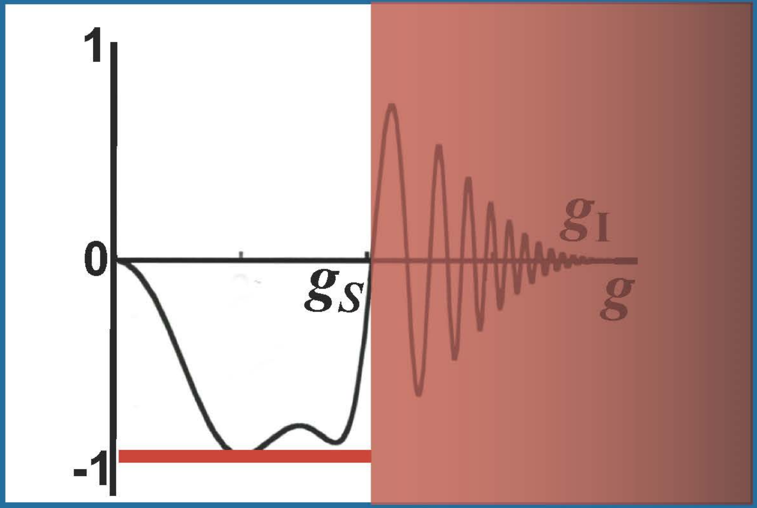
$$Z_S = -\left(\frac{4}{3}C_S\lambda\right)^{\frac{1}{2}}$$

$$g_S = 2(3C_S\lambda^3)^{-\frac{1}{4}}$$

$$d_S = g_S^{-1}$$

- extend region of close to "1" to large ***g***
- first "zero" at $\boldsymbol{g}_S$ as large as possible

Contrast $\propto \sin 2\pi\chi(g)$



Scherzer's contrast transfer function

the basis of "high resolution" for more than half a century

but there are issues:

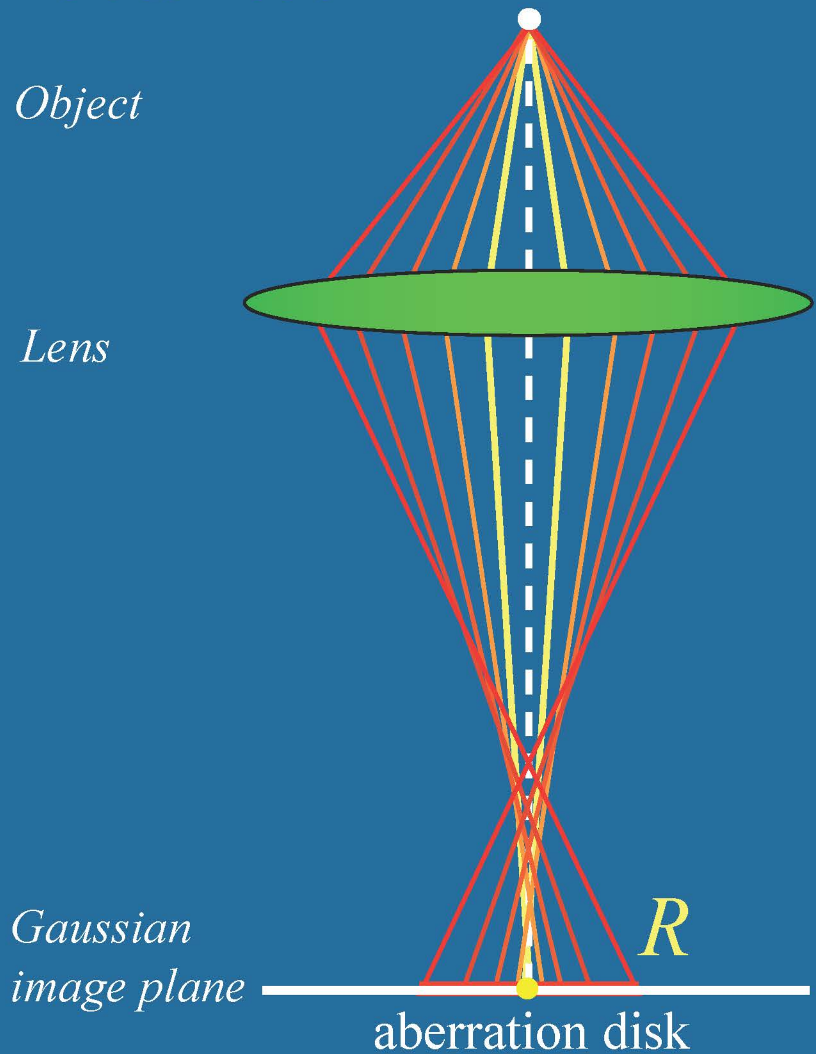
- *information is wasted*
- *severe delocalization*

$$\chi(g) = \frac{1}{4}C_S\lambda^3 g^4 + \frac{1}{2}Z\lambda g^2 + \cdots$$

spherical aberration — fixed

defocus — variable

Delocalization



Point spread function

$$R \propto \left|\frac{\partial \chi}{\partial g}\right|_{\max}$$

$$\chi(\mathbf{g}) = \tfrac{1}{4} C_S \lambda^3 \mathbf{g}^4 + \tfrac{1}{2} Z \lambda \mathbf{g}^2 + \ldots$$

aberration function

inserting the Scherzer value for Z

$$R = \left|\frac{\delta\chi}{\delta g}\right|_{max} = |C_S\lambda^3 g^3 + Z\lambda g|_{max} \approx 3d_S$$

delocalization 3 x the resolution

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Jülich theory for phase contrast in the aberration correted CTEM

M. Lentzen, K. Urban *et al.*, *Ultramicroscopy* **92**, 233 (2002)
C.L. Jia, M. Lentzen & K. Urban, *Science* **299**, 870 (2003)

$$\chi(g) = \frac{1}{4} C_S \lambda^3 g^4 + \frac{1}{2} Z \lambda g^2 + \cdots$$

spherical aberration — variable

defocus — variable

Jülich theory for phase contrast (2002)

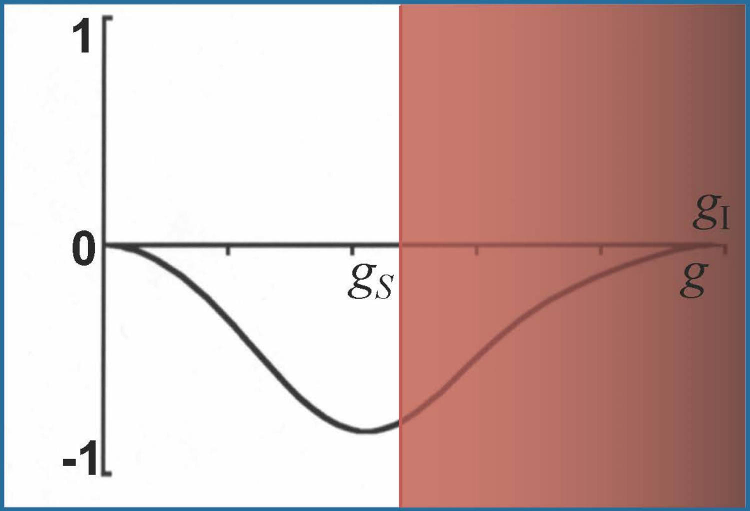
M. Lentzen, K. Urban *et al., Ultramicroscopy* **92**, 233 (2002)
C.L. Jia, M. Lentzen & K. Urban, *Science* **299**, 870 (2003)

$$Z_{opt} = -\frac{16}{9}(\lambda g_I^2)^{-1}$$

$$C_{S,opt} = +\frac{64}{27}(\lambda^3 g_I^4)^{-1}$$

$$R_{opt} = \frac{16}{27} g_I^{-1}$$

Contrast $\propto sin 2\pi\chi(g)$



Optimization of contrast transfer exploiting *variable* C_S and *variable Z:*

$$\chi(g) = \frac{1}{4} C_S \lambda^3 g^4 + \frac{1}{2} Z \lambda g^2 + \cdots$$

spherical aberration — variable

defocus — variable

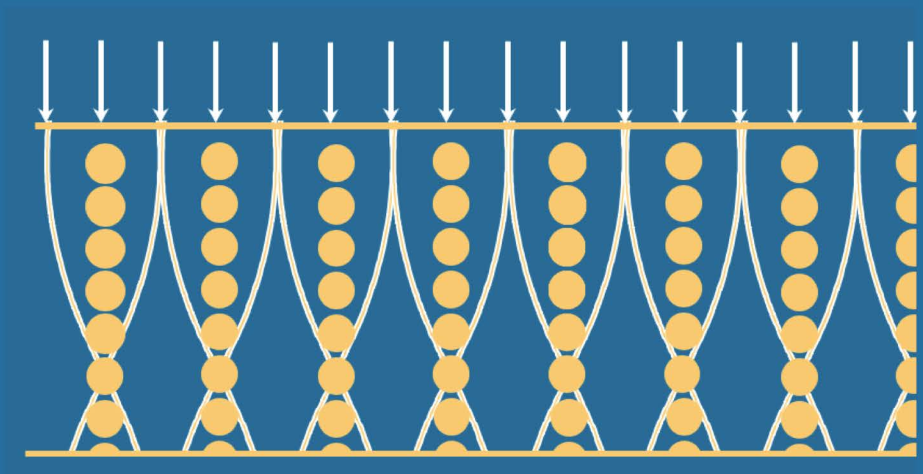
1) Information transfered up tp $\mathbf{g}_I$

2) No (!) Contrast delocalisation

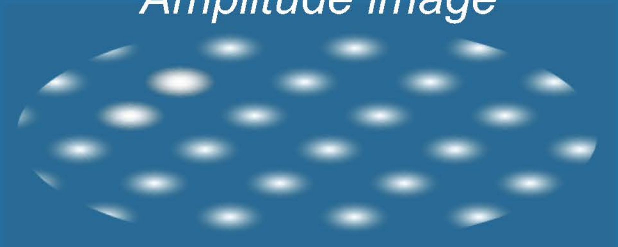
Amplitude contrast and phase contrast

One problem is remaining

electron diffraction channelling



Amplitude image

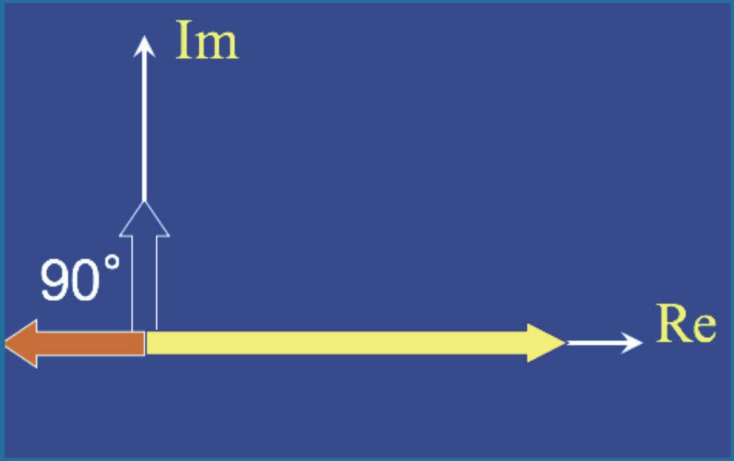


bright contrast

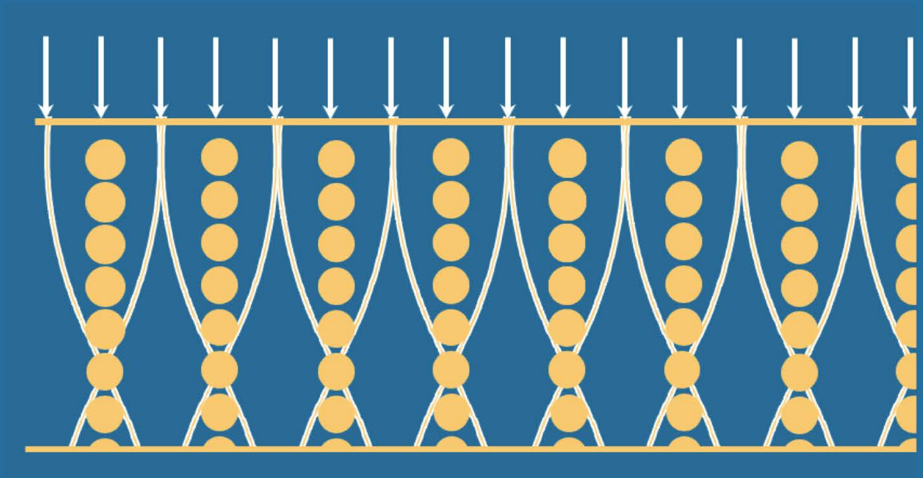
Amplitude contrast and phase contrast

$$Z_{opt} = -\frac{16}{9}(\lambda g_I^2)^{-1}$$

$$C_{S,opt} = +\frac{64}{27}(\lambda^3 g_I^4)^{-1}$$



electron diffraction channelling

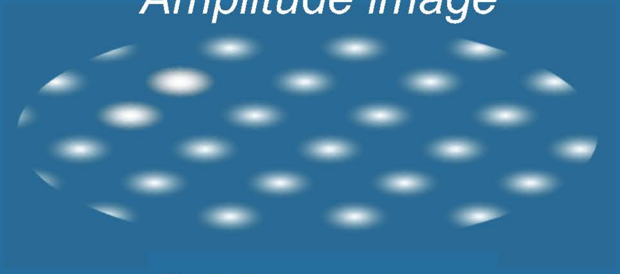


weakening each other

Phase image

dark contrast

Amplitude image



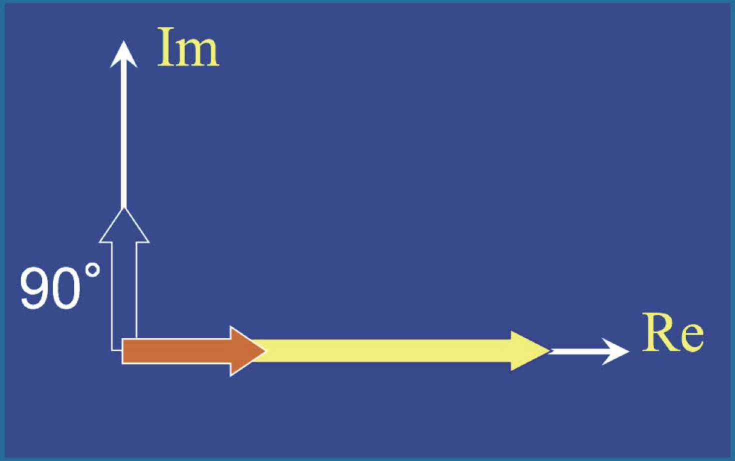
bright contrast

The trick of NCSI (negative spherical aberration imaging)

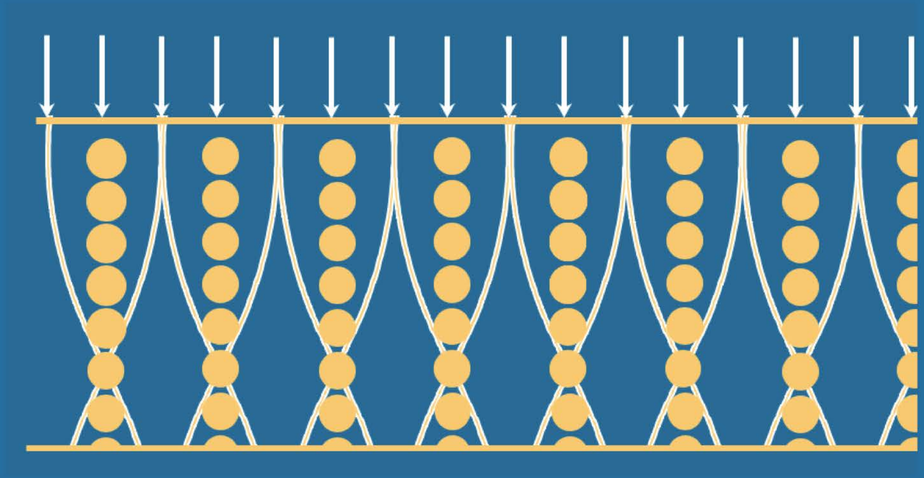
C.L. Jia, M. Lentzen & K. Urban, **Science 299**, 870 (2003)

$$Z_{opt} = +\frac{16}{9}(\lambda g_I^2)^{-1}$$

$$C_{S,opt} = -\frac{64}{27}(\lambda^3 g_I^4)^{-1}$$

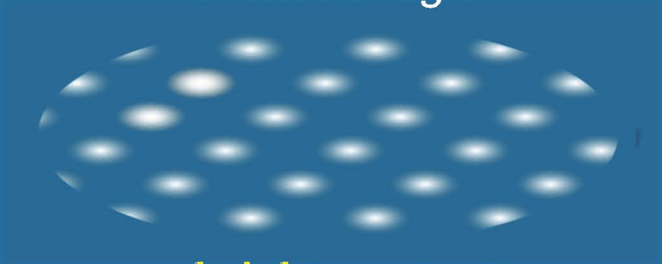


electron diffraction channelling



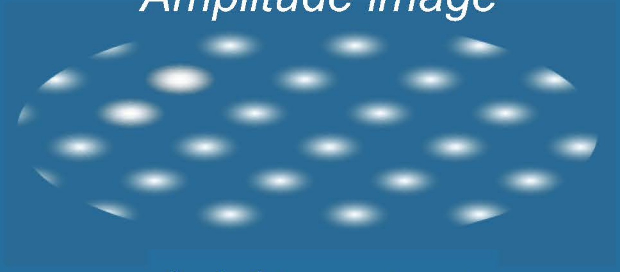
<u>enhancing</u> each other

Phase image



bright contrast

Amplitude image

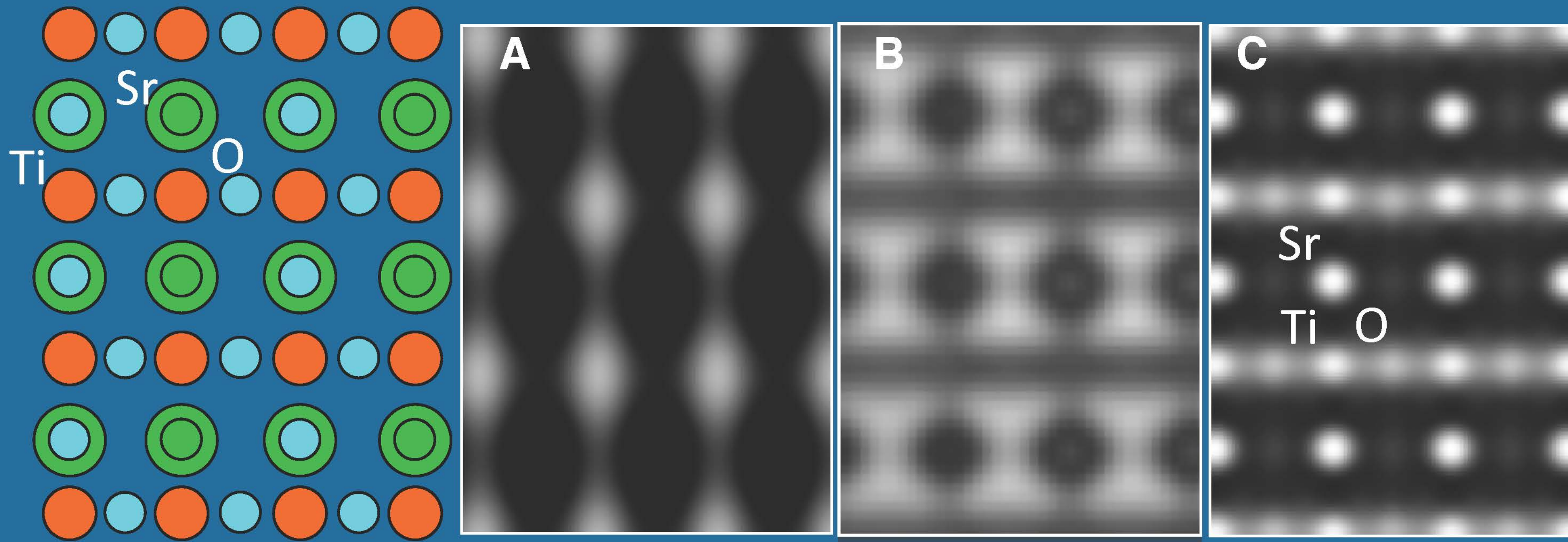


bright contrast

The trick of NCSI (negative spherical aberration imaging)

C.L. Jia, M. Lentzen & K. Urban, **Science 299**, 870 (2003)

Comparison of imaging modes for $SrTiO_3$



uncorrected | aberration corrected | corrected NCSI

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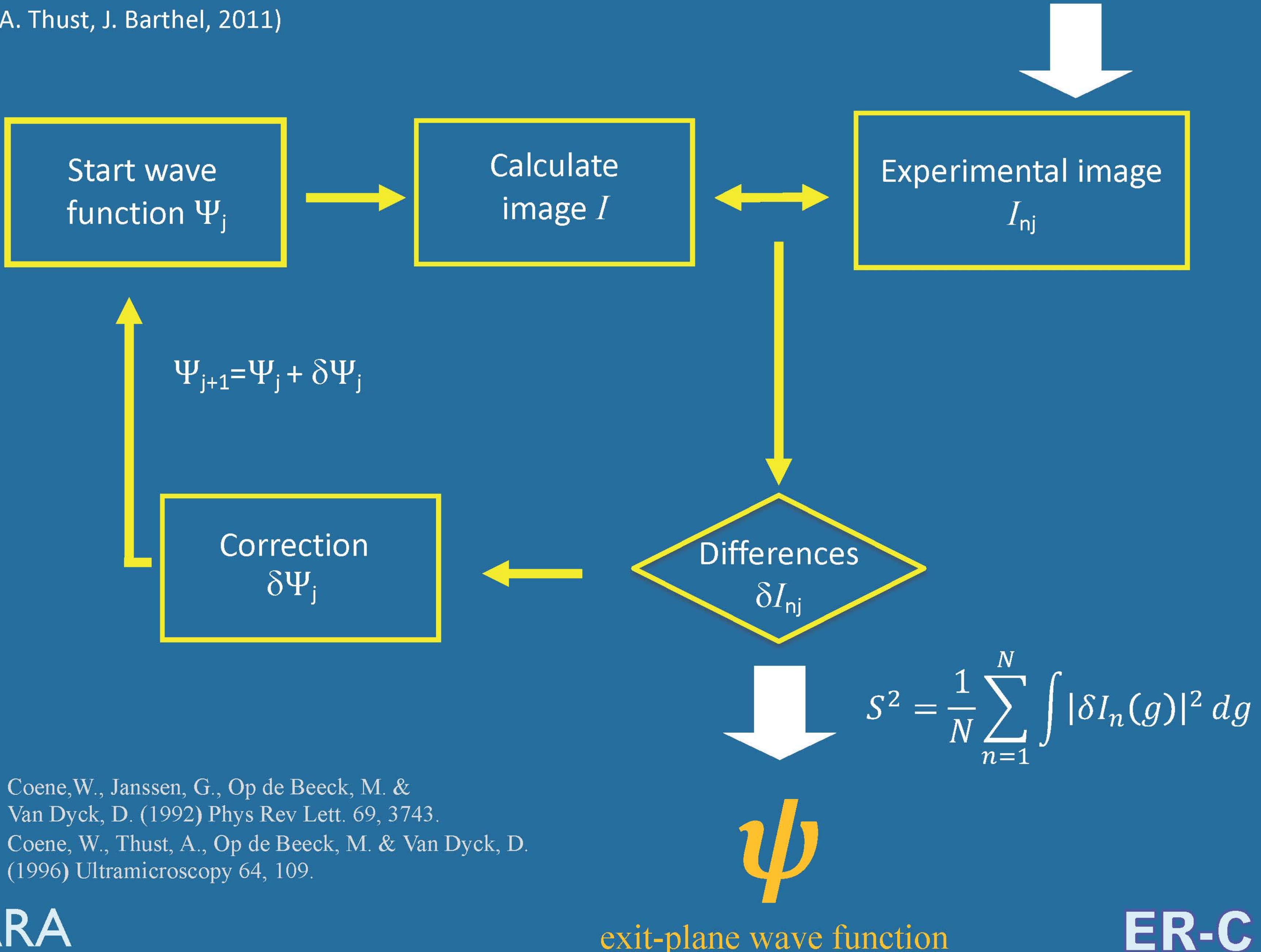
Part III

From the images to the unknown structure

Reconstruction of exit-plane wave function by TrueIMAGE™

(A. Thust, J. Barthel, 2011)



Coene,W., Janssen, G., Op de Beeck, M. & Van Dyck, D. (1992) Phys Rev Lett. 69, 3743.

Coene, W., Thust, A., Op de Beeck, M. & Van Dyck, D. (1996) Ultramicroscopy 64, 109.

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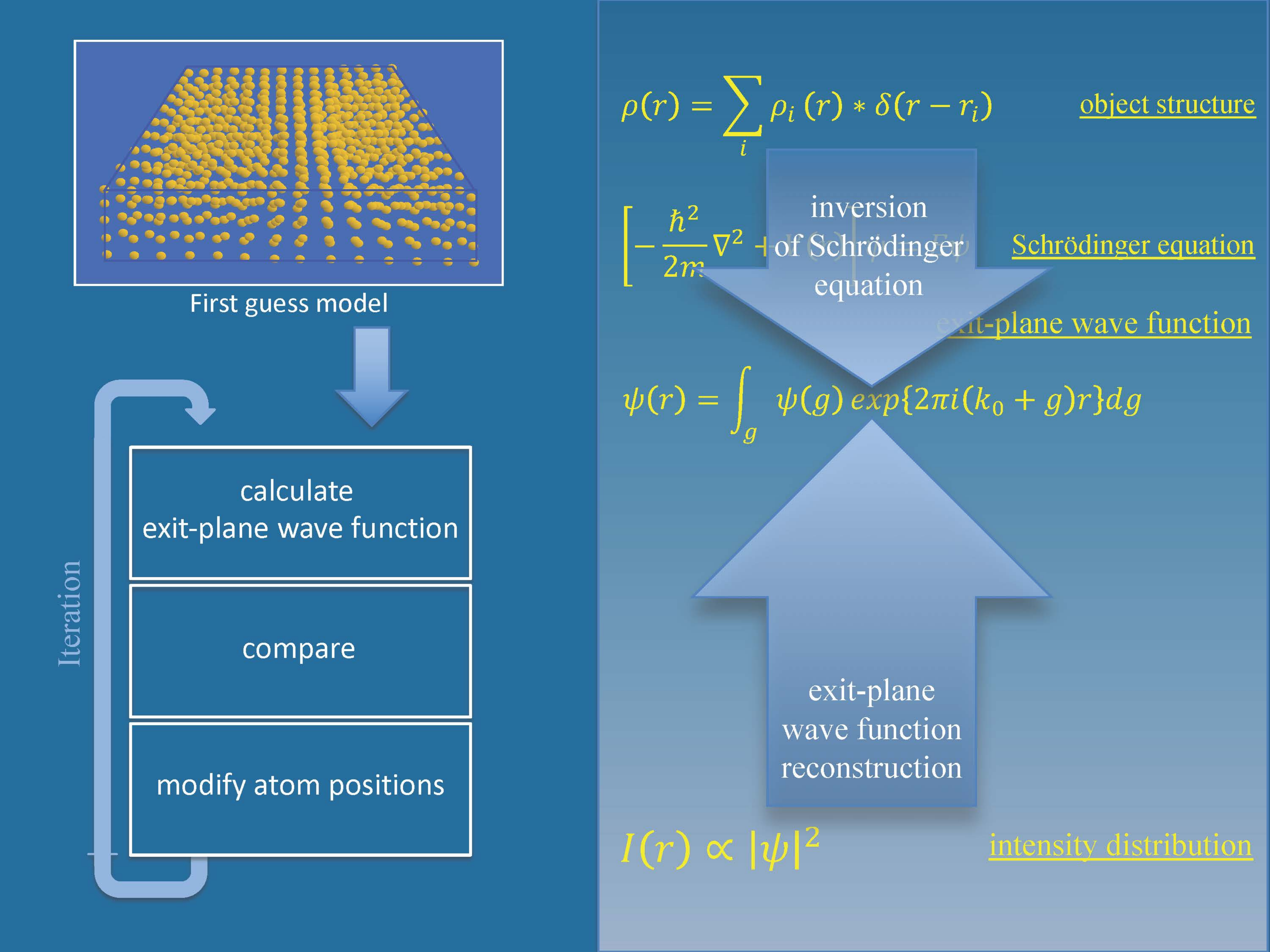
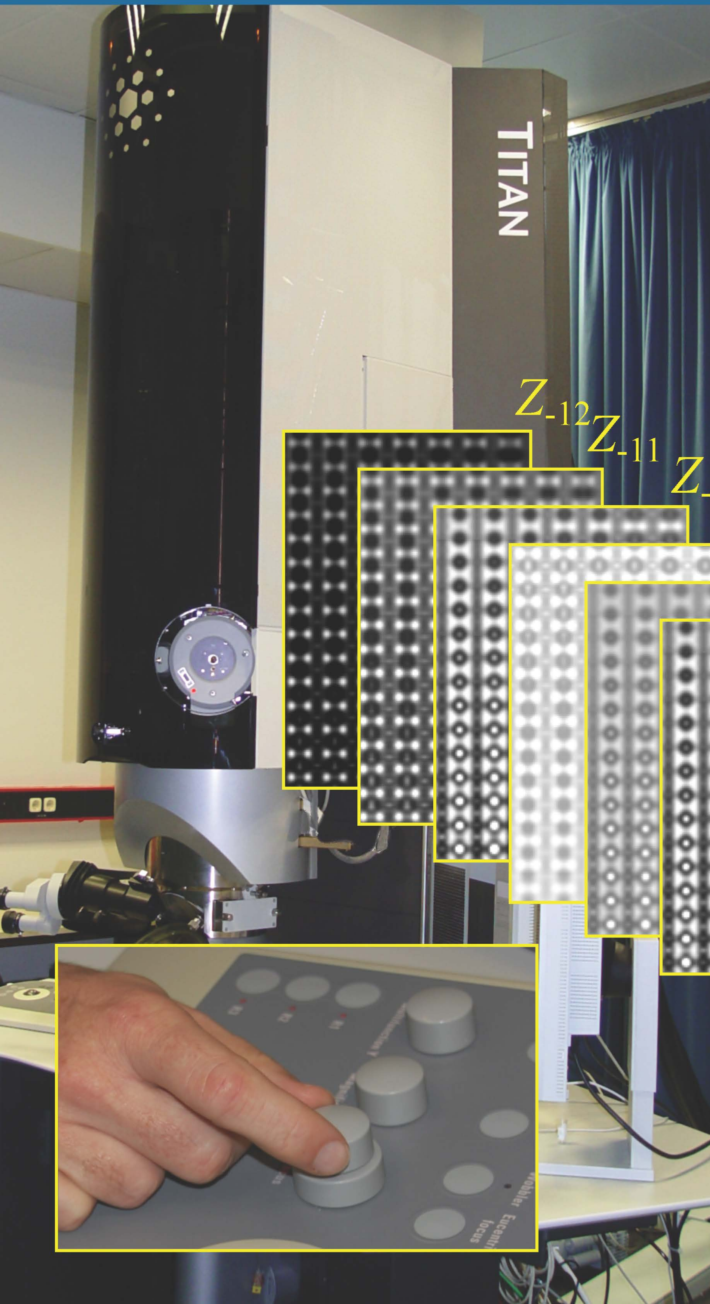
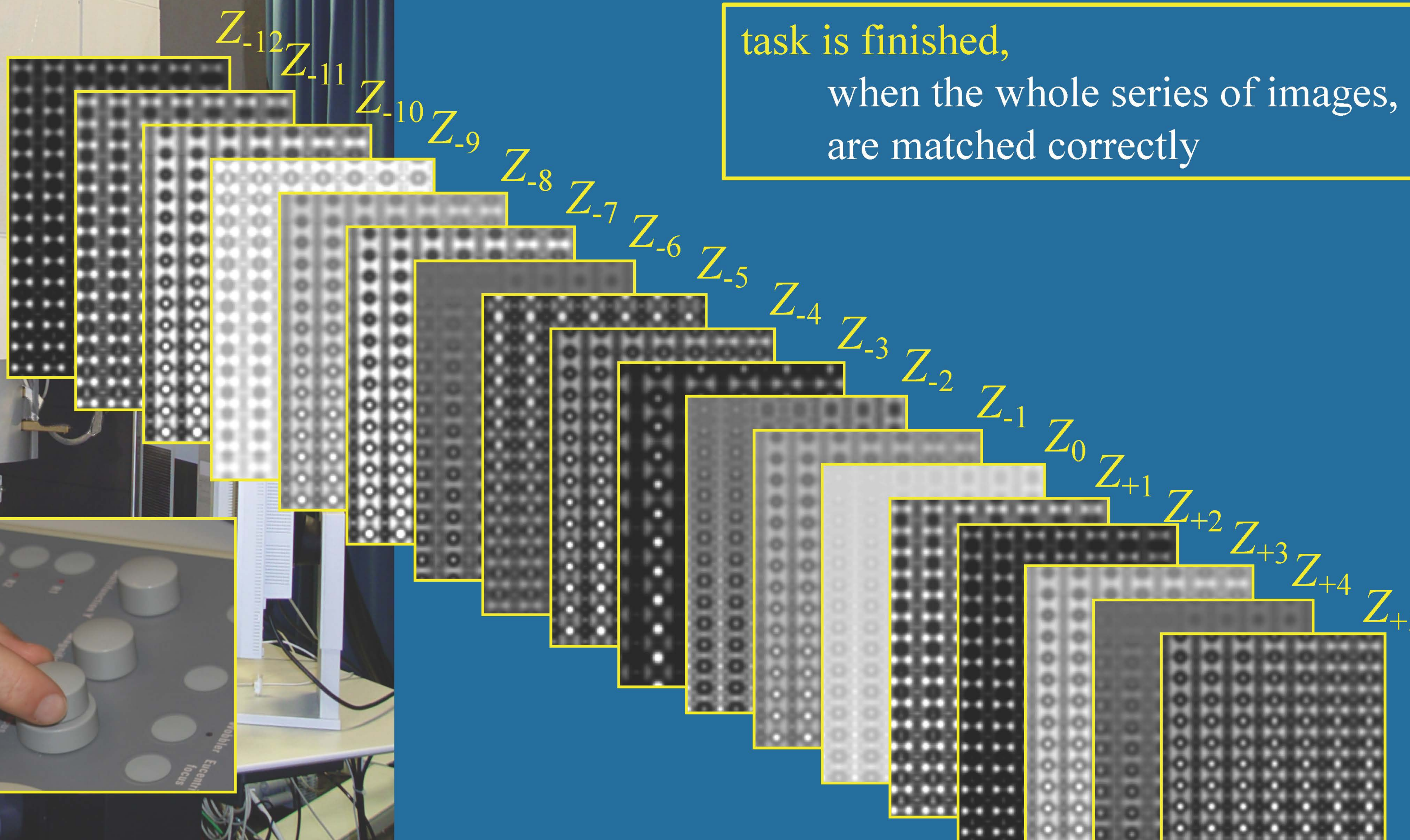

First guess model
calculate
exit-plane wave function
compare
modify atom positions
Iteration
$\rho(r) = \sum_i \rho_i(r) * \delta(r - r_i)$
object structure
$\left[-\frac{\hbar^2}{2m}\nabla^2 + \right.$
inversion
of Schrödinger
equation
Schrödinger equation
exit-plane wave function
$\psi(r) = \int_g \psi(g)\, exp\{2\pi i(k_0 + g)r\}dg$
exit-plane
wave function
reconstruction
$I(r) \propto |\psi|^2$
intensity distribution

Traditional: *Focal series*

focal series of images:

$$\chi(g) = \frac{1}{4} C_S \lambda^3 g^4 + \frac{1}{2} Z \lambda g^2 + \cdots$$





task is finished,
when the whole series of images,
are matched correctly

Traditional: ***Focal series***

focal series of images:

$$\chi(g) = \frac{1}{4} C_S \lambda^3 g^4 + \frac{1}{2} Z \lambda g^2 + \cdots$$

interferometric imaging

Disadvantages:

- manipulates the contrast transfer function
- optical instabilities
- sample drift
- diminishes – in effect – the resolution