Hard X-ray tomography on the nanometer length scale

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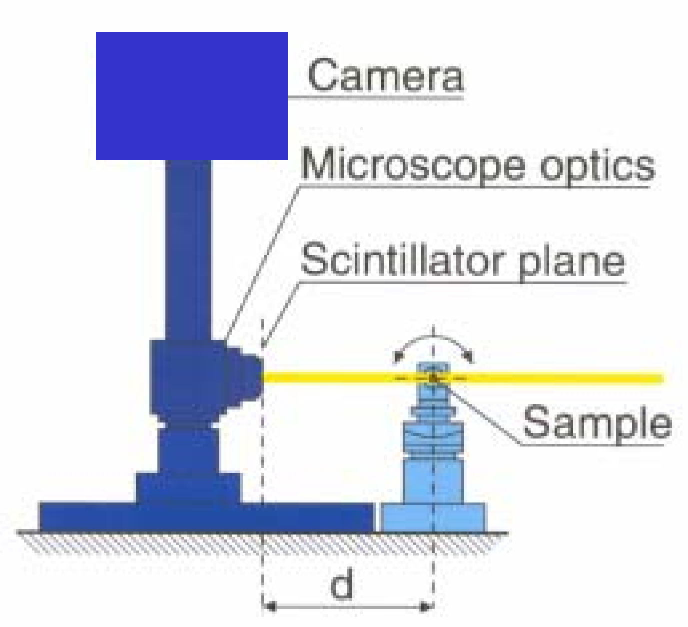
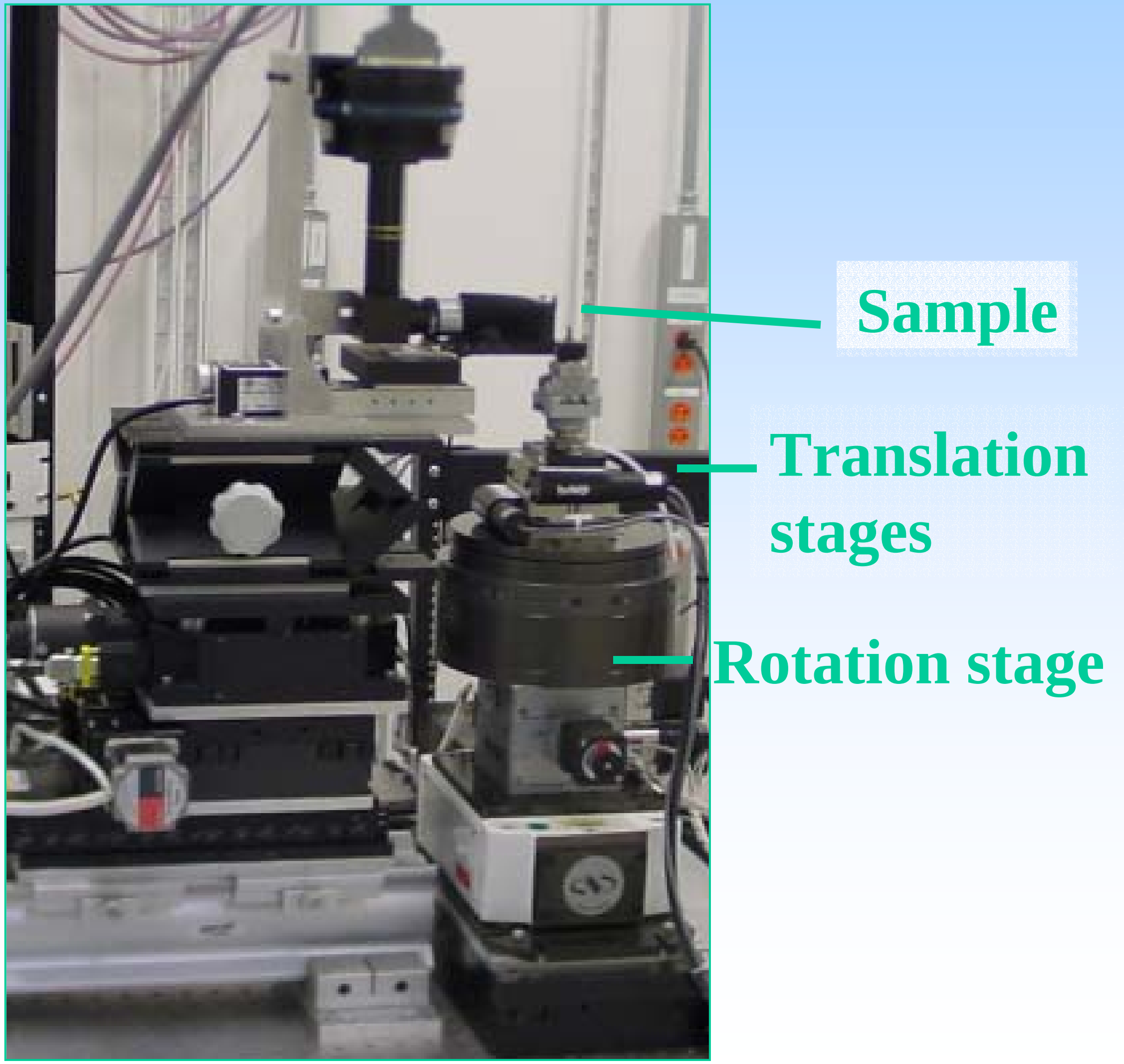
- Introduction
- Microscope at 34 ID-C
- Limits with current equipment
- Prospects for the future
  - A microscope with 50nm resolution

  Summary

Experimental set-up

Detector system | Tomography stage





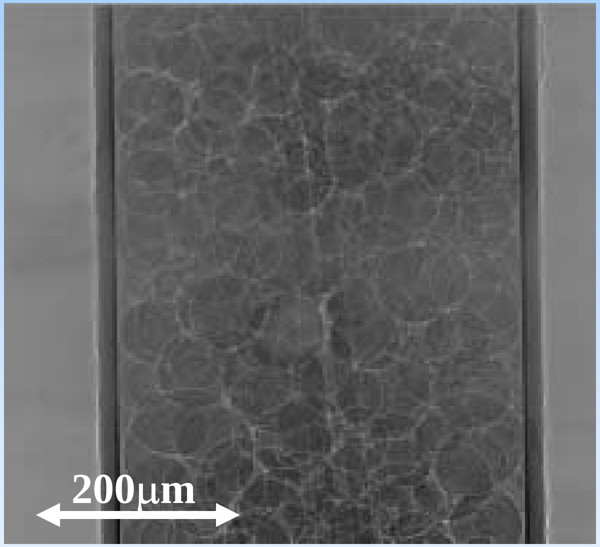
-Tomography and in-line phase contrast imaging

-High quality stages
Rotation Stage with air bearing (run out < 20nm)

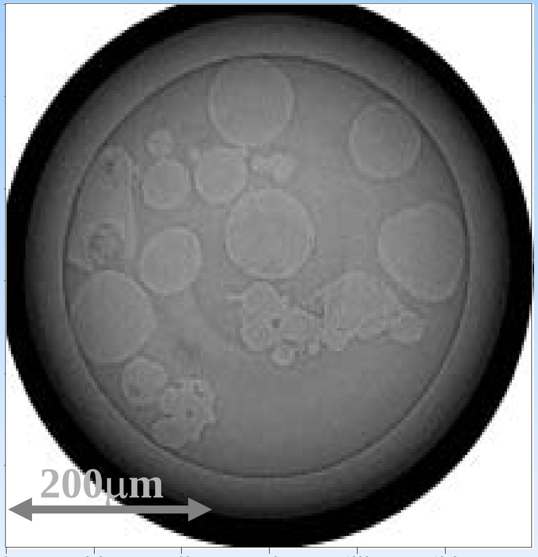
Scientific application

(coll. T. Terry, M. Harris, Purdue University)

Projection image



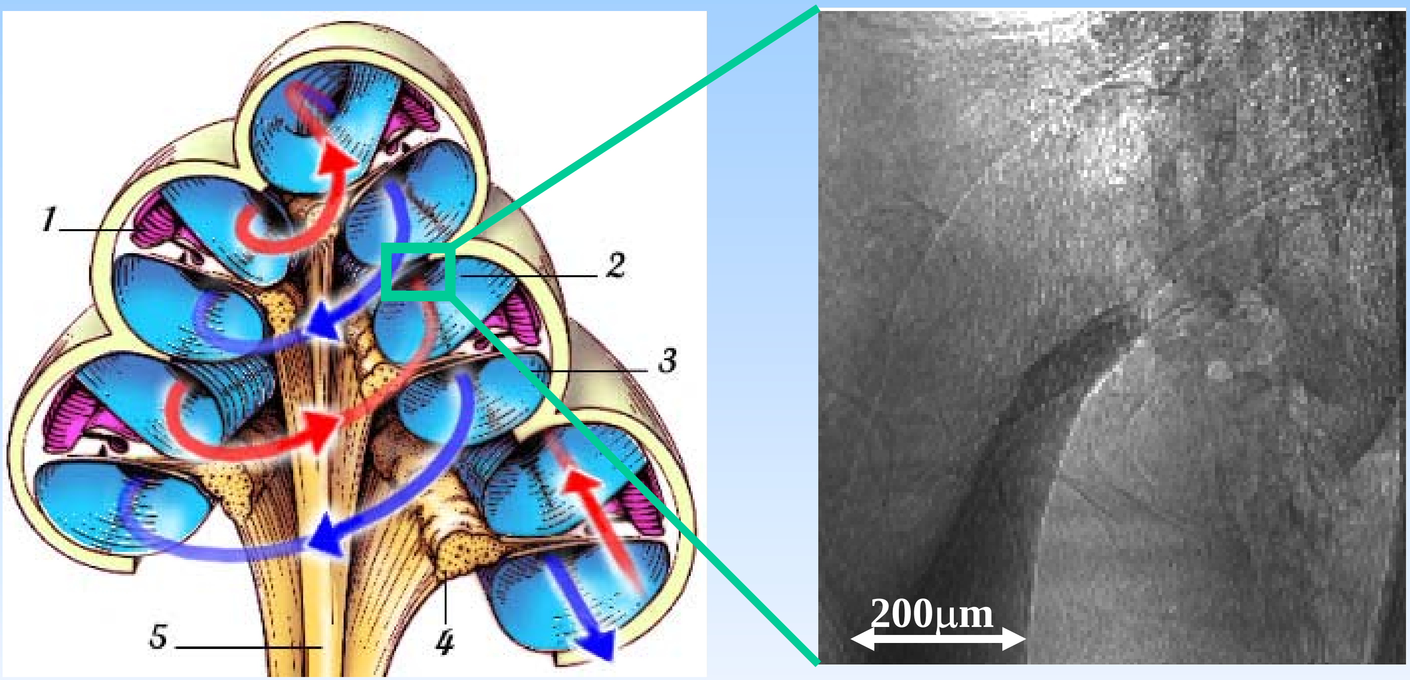
Reconstructed slice



- SiO_2 and ZrO_2 spheres
- Hollow spheres with high relative surface
- biomedical applications; chemical reactions
- Size distribution in function of the preparation procedure
- In-line phase contrast
- Features over several length scales; wall thickness
- More detailed study → microscope

Scientific application

(coll. C.-P. Richter, Northwestern University)



http://www.iurc.montp.inserm.fr/cric51/audition/english/cochlea/fcochlea.htm

- Better understanding of hearing process
- in function of frequency, diff. parts stimulated
- Dynamic study
- Again: features on different length scales
- Unique approach to the problem
- Challenging project

Magnified Imaging

Resolution mainly limited by detector

<u>How to obtain higher spatial resolution?</u>

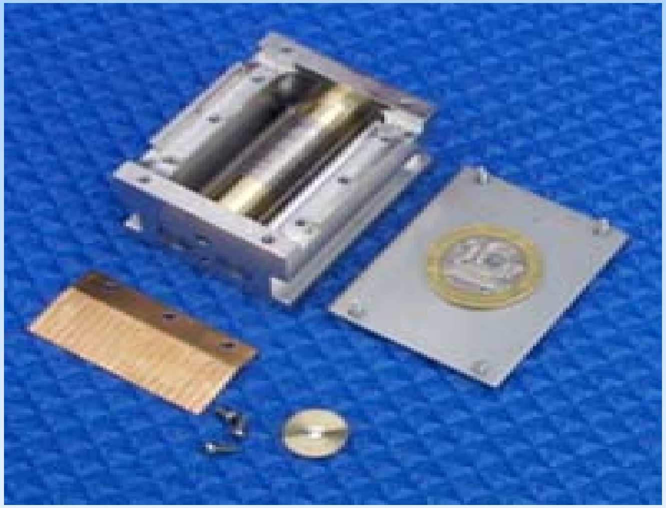
- lensless imaging (-> talk C. Jacobsen)

- imaging using hard X-ray optics
  - **Scanning techniques with focused beam**
  - **Fan beam geometry**
  - **<u>Full-field imaging</u>**

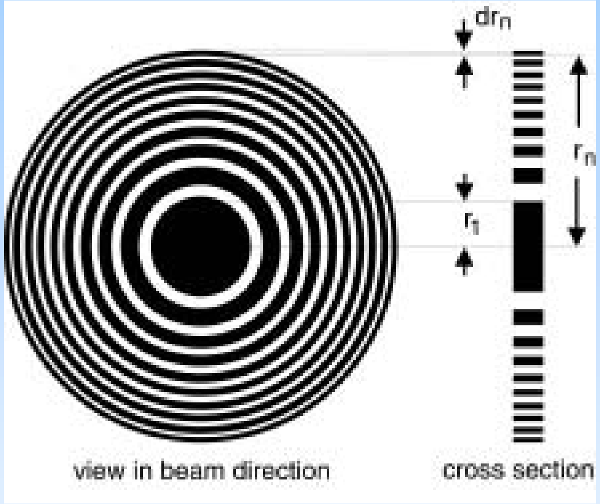
Magnified Imaging

Hard X-ray optics

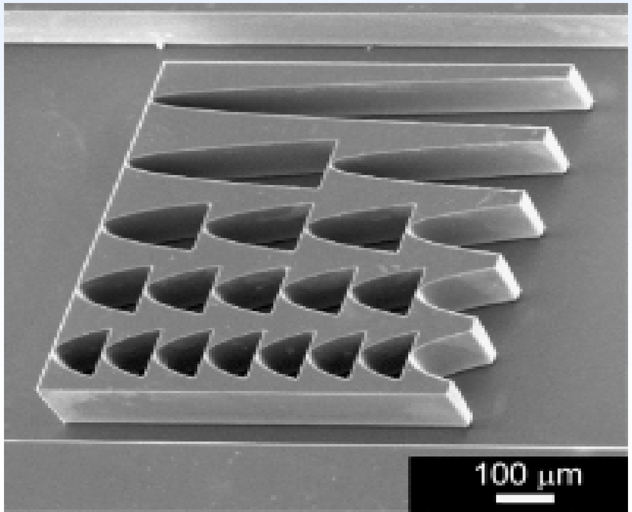
Compound Refractive Lens



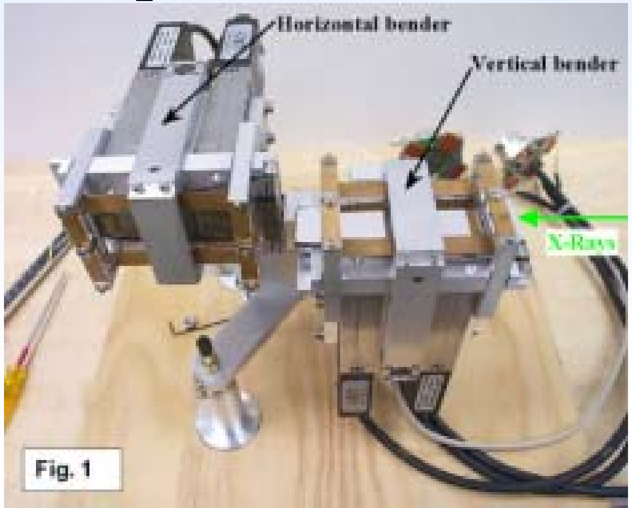
Fresnel-Zone Plate



Planar Lens



Kirkpatrick-Baez Mirror



Magnified Imaging

Phase 0

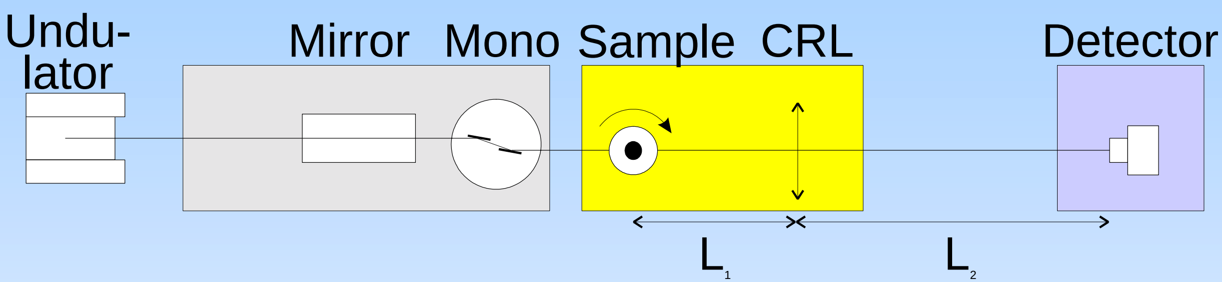
Preface

Experiments in the past with impact for current development

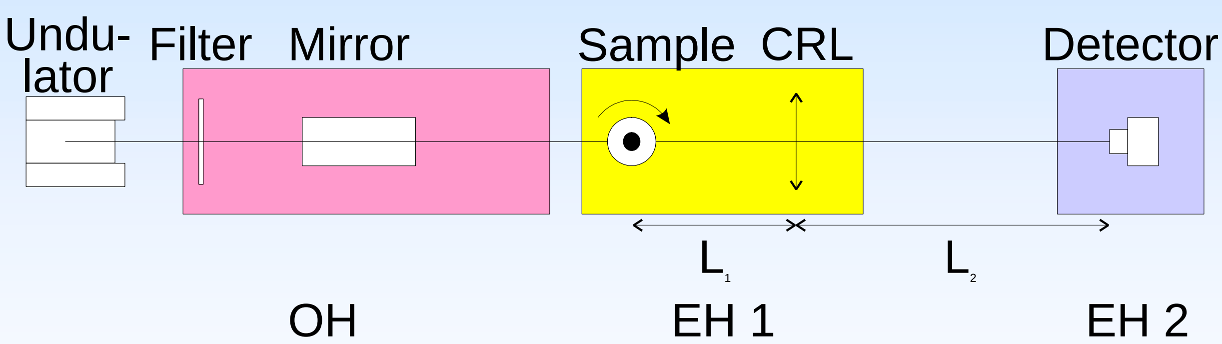
Hard X-ray full-field microscope

Experiments at ESRF ID 22

Monochromatic



Pink beam



$M=L_2/L_1$

$f=1\text{-}2$ m

$L_1= 2\text{-}5$ m

$L_2\sim 20$ m

-beamline nicely adapted (lenses with f~1-2m)

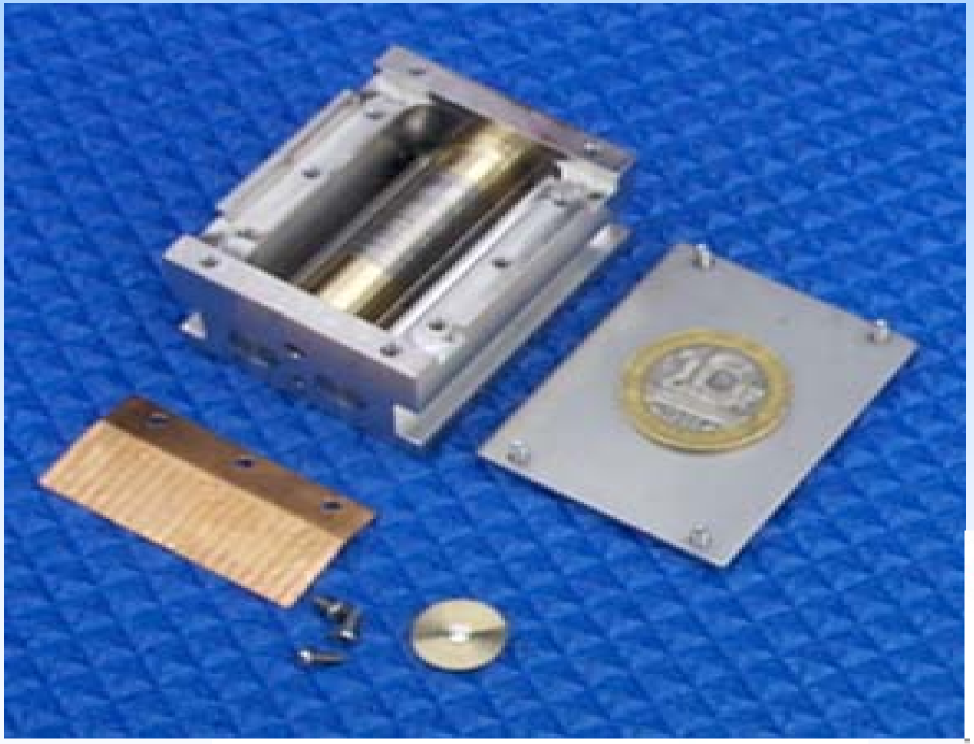
-$I_{pink}\sim 50xI_{mono}$;

$\Delta E/E=10^{-2}$(pink);10^{-4}(mono)

Parabolic Compound Refractive Lens

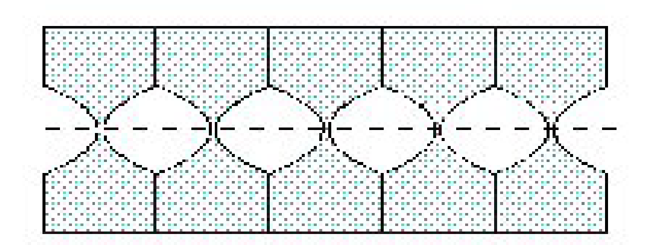
(C.G. Schroer, B. Lengeler, RWTH Aachen)

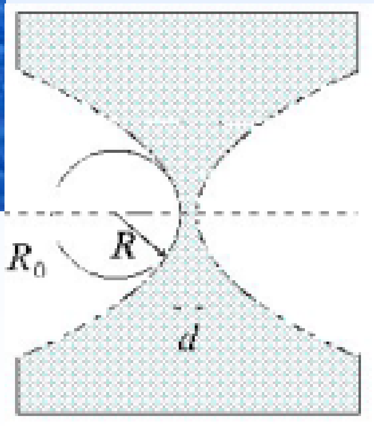
Set of parabolic lenses (typ. 50-200)



Focal length f can be varied with energy E and number of lenses N

f=2 m with 50 lenses at 20 keV
- effective aperture ~ 0.2 mm
- diffr. limit 0.3 micron
- transmission 3 %



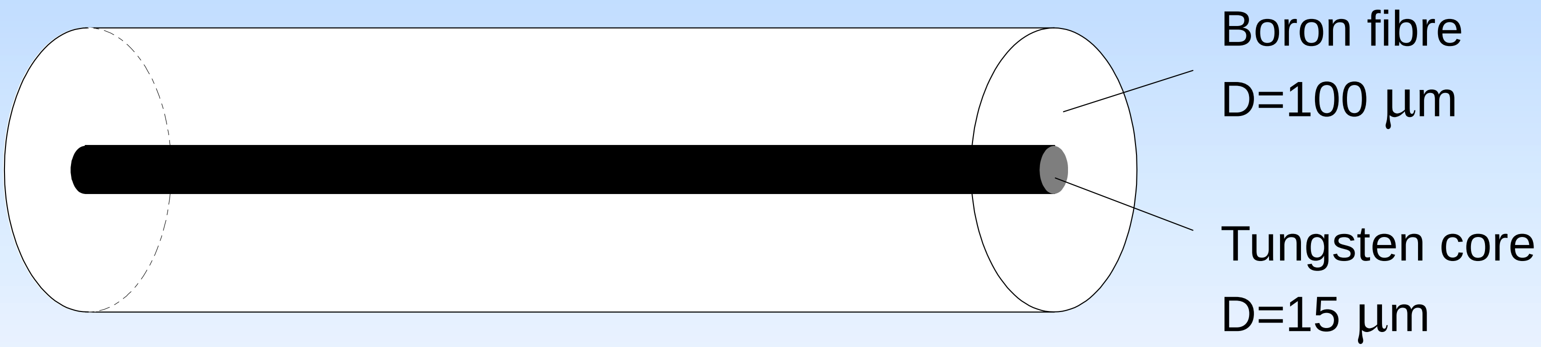


R: 0.2 mm
R_0: 0.45 mm
d: 0.01 mm
material: Al

X-ray magnified tomography

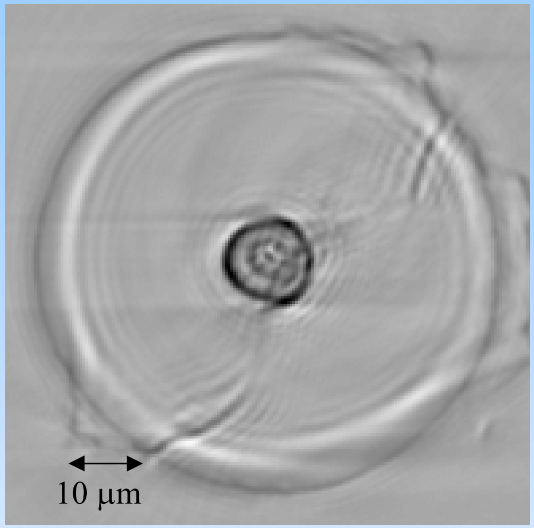
Sample

Boron fiber with tungsten core

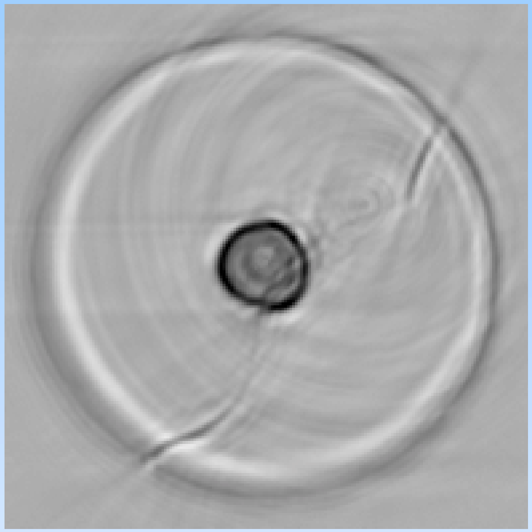


Combined absorption-contrast (tungsten) and phase-contrast (boron) object

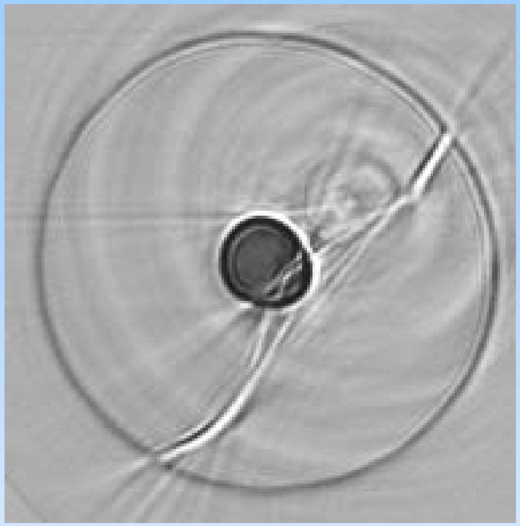
Tomography: phase contrast and pink beam



3.4x
mono



3.4x
pink



10x
pink

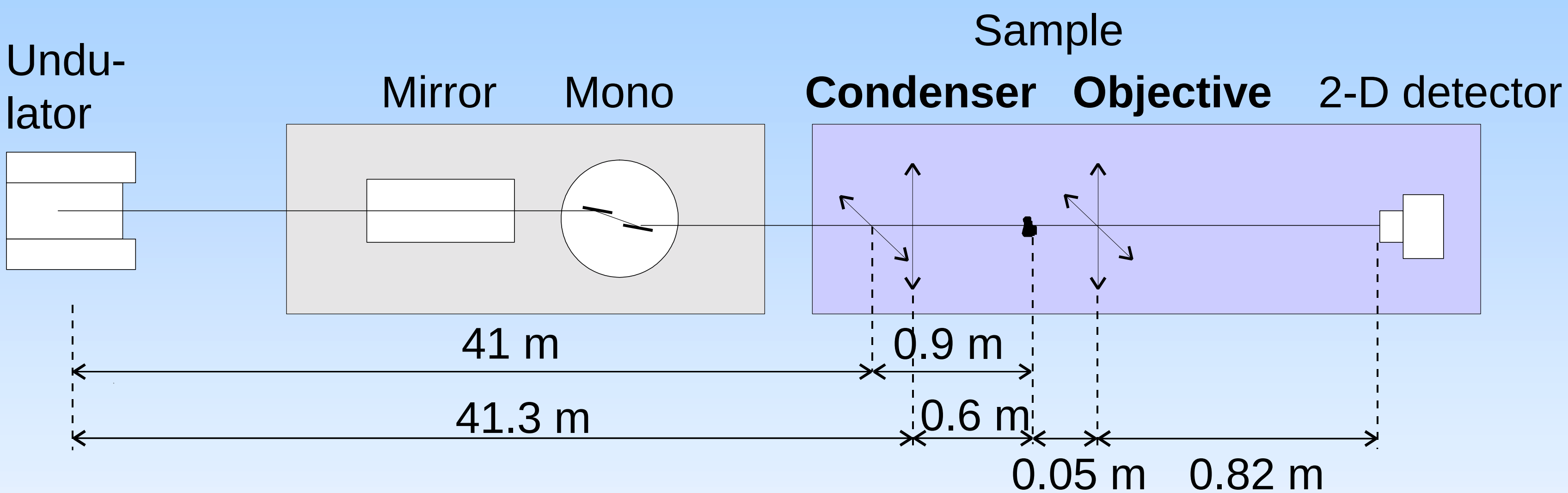
Al parabolic CRL; E=19.7 keV

- no significant difference pink – mono (3.4x)
- resolution 10x ~ 1µm
- study of mechanism of image formation
- beryllium lenses overcome actual limit

Ref.: C. Rau et al., NIM A, (2001).
T. Weitkamp et al., SPIE proc., (2002).
C. Schroer et al., SPIE proc., (2002).

KB-FZP microscope

C. Rau, U. Neuhäusler, G. Schneider, O. Hignette, O. Leupold



Condenser (O. Hignette, G. Rostaing, ESRF)

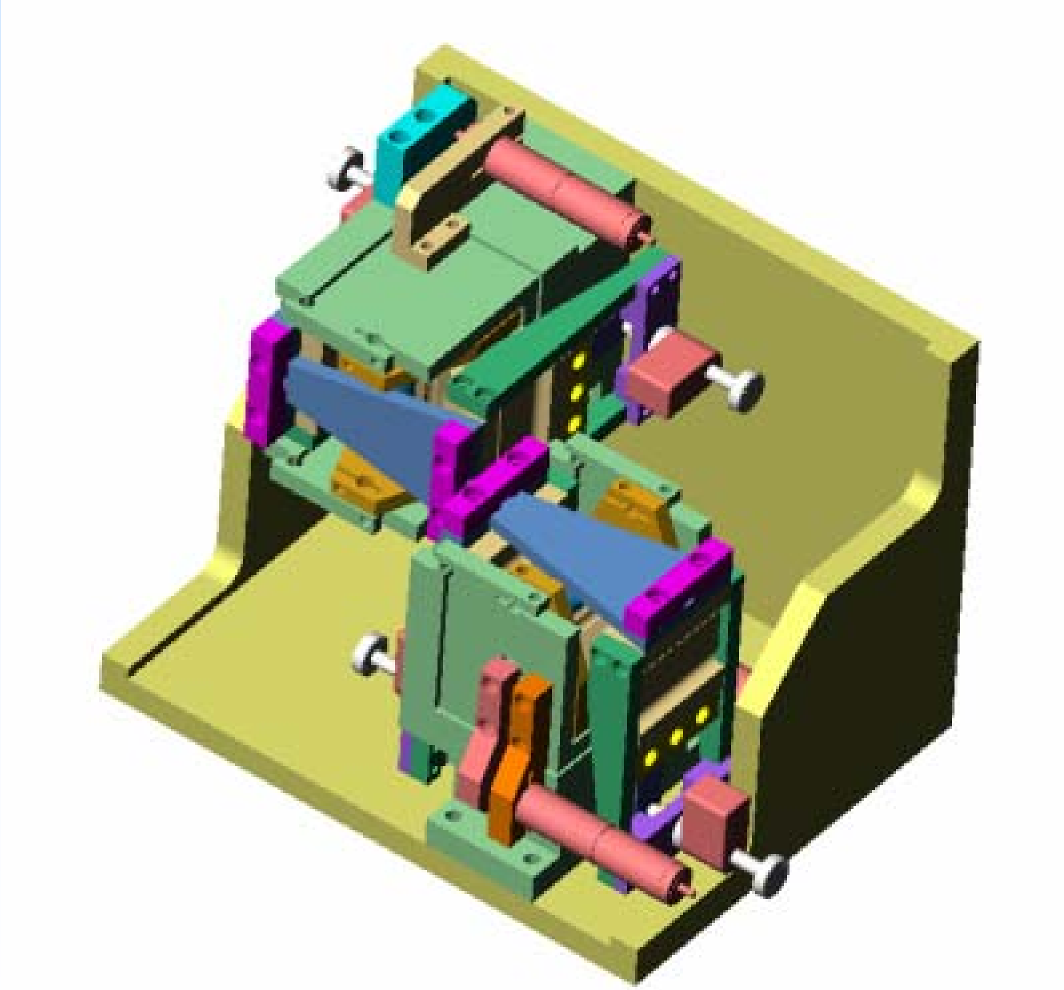
KB multilayer mirror: high reflectivity, matches Objective aperture

Objective (M. Panitz, IRP Göttingen):

micro-FZP: high resolution, reasonable efficiency

Kirkpatrik-Baez System
(O. Hignette, A. Rostaing, ESRF Grenoble)

Design of the system[1]



Ni-Be_4C multilayer
d=24 Å

f_{hor}=0.9 m f_{vert} =0.6 m

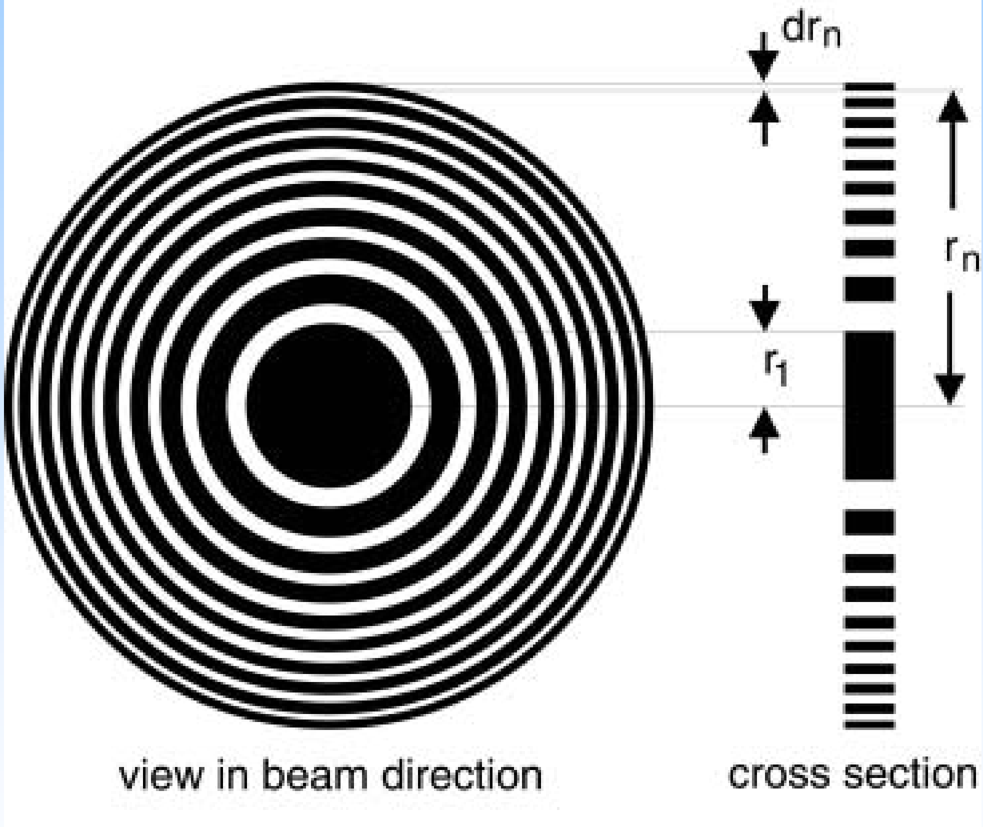
Reflectivity 33 %

[1] courtesy O. Hignette

Micro Fresnel-Zone plate

(M. Panitz, G. Schneider, M. Peuker, G. Schmahl: Uni Götttingen)

Scheme[1]



[1]courtesy U. Neuhäusler

Material	**Au**
r_n	**65 μm**
dr_n	**70 nm**
δ	**85 nm**
Thickness	**500 nm**
Efficiency at 13 keV	**2 %** (10% at 4 keV)
Gain	**2×10^4**
f at 13keV	**4.7 cm**

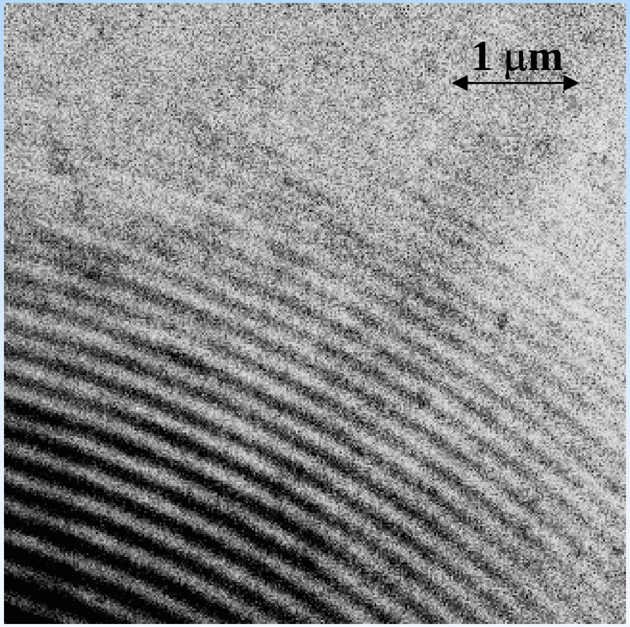
focal length $f=2\cdot r_n\cdot dr_n/\lambda$

resolution $\delta= 0.61\cdot \lambda /N.A.=\mathbf{1.22\cdot dr_n}$

High-resolution imaging

Imaging with sub-100nm lateral resolution with a combined KB[1]-FZP[2] microscope

- KB system as a condenser: high flux
- micro-FZP: high resolution 85 nm
- exposure time: < 1min, E= 13 keV



Significant improvement

- Nano-tomography
- Zernike phase-contrast

[1] O. Hignette, ESRF
[2] M. Panitz, IRP Göttingen

Why are these experiments important?

- Proof of principle
- Tomography possible
- Use of pink beam
- Magnified Imaging of phase contrast objects
- High resolution with FZPs

Phase I

The Present

Realization of the microscope

Microscope at UNICAT/APS

<u>UNICAT</u>: 4 different partners (UIUC, ORNL, NIST, UOP)

3 beamlines in sector 33&34

Methods: USAXS, XRD, Spectroscopy, …

Tomography setup at UNICAT/APS

- Tomography
- KB-FZP microscope

Sub-100nm tomography

Nano-Tomography available for large scientific community

development Instrument – valuable Experiments

Layout Microscope 34 ID

- Hard X-rays
    - Penetration depth , sample handling, depth of focus

- Resolution <100nm
    - Use KB-FZP system

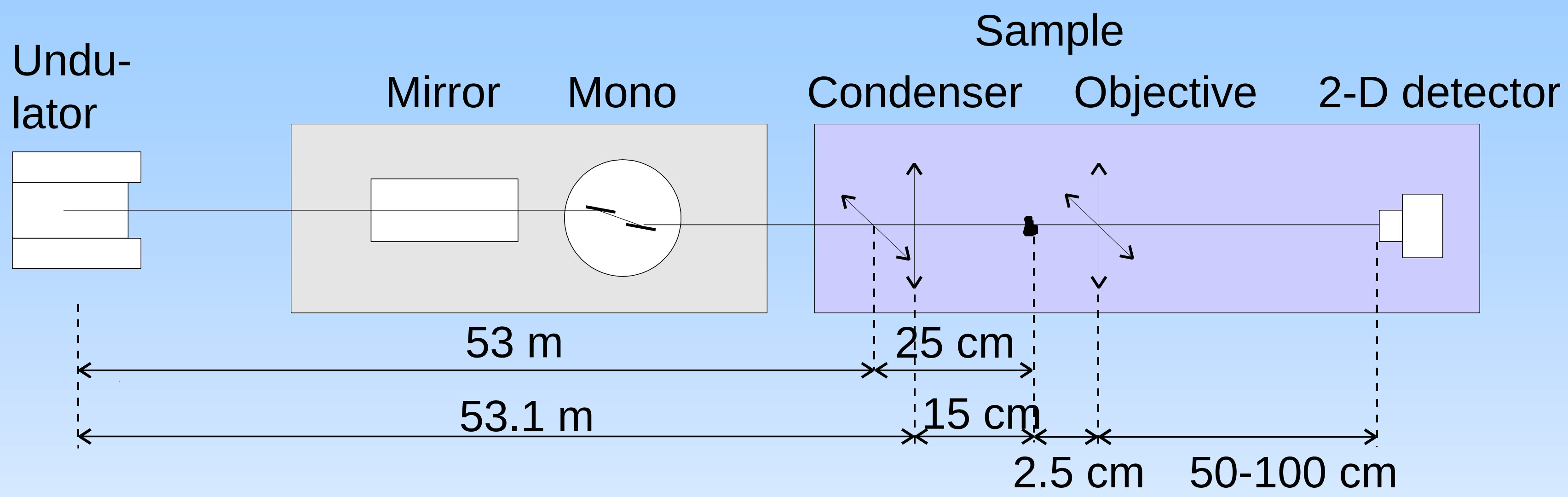
-Applications in Materials Science and Biomedicine

- Flexible design
- Evolutionary improvement of setup
- Technical development accompanied by scientific experiments

Microscope at 34 ID

- Energy range 6-12 keV (currently 9keV)
  - Depth of focus of FZP: 50-100μm
  - →100μm-diameter samples
  - → fits to Pixel array of CCD
  - Elements like Cu, Zr, Au, Ag can be studied using absorption contrast
  - Biomaterials have to be studied by making use of phase contrast
  - Detector efficient at this energy

Microscope at 34 ID-C

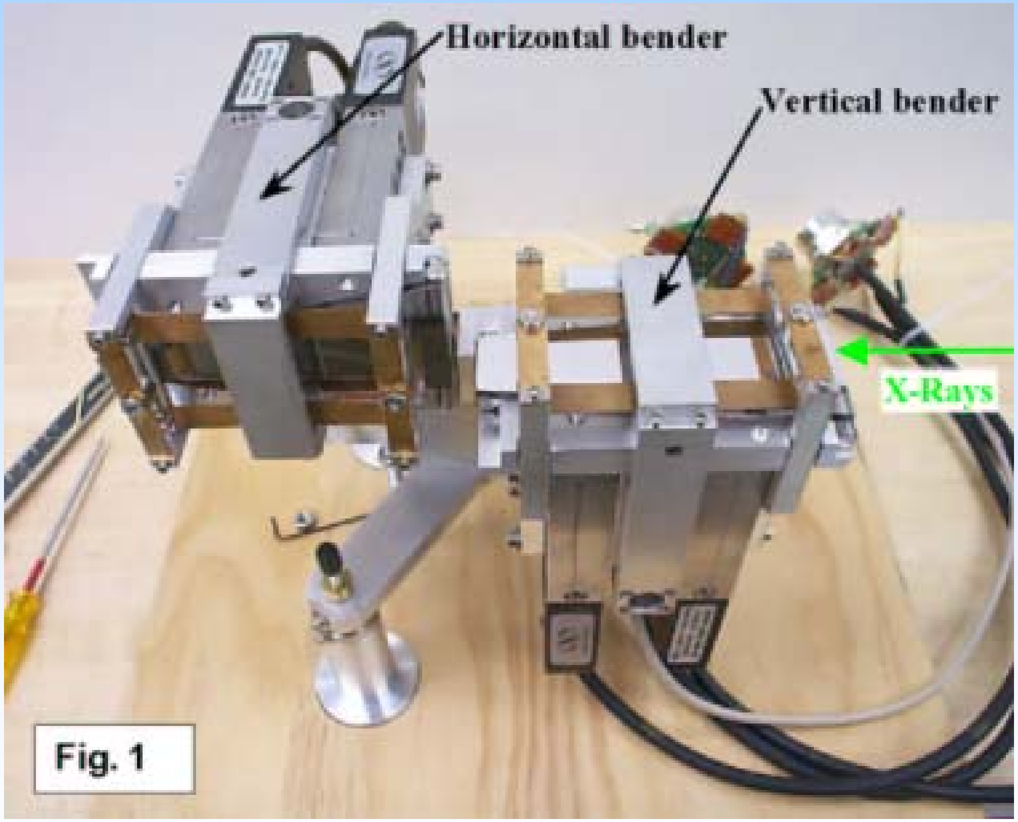


-Experiments in “parasitic mode”(E-Station main user)
-> amount of beamtime

Kirkpatrik-Baez System

(P. Eng, CARS, University of Chicago/J. Qian, R. Conley,L. Assoufid, APS)

Photo of the system[1]



Pt-coated Si crystal

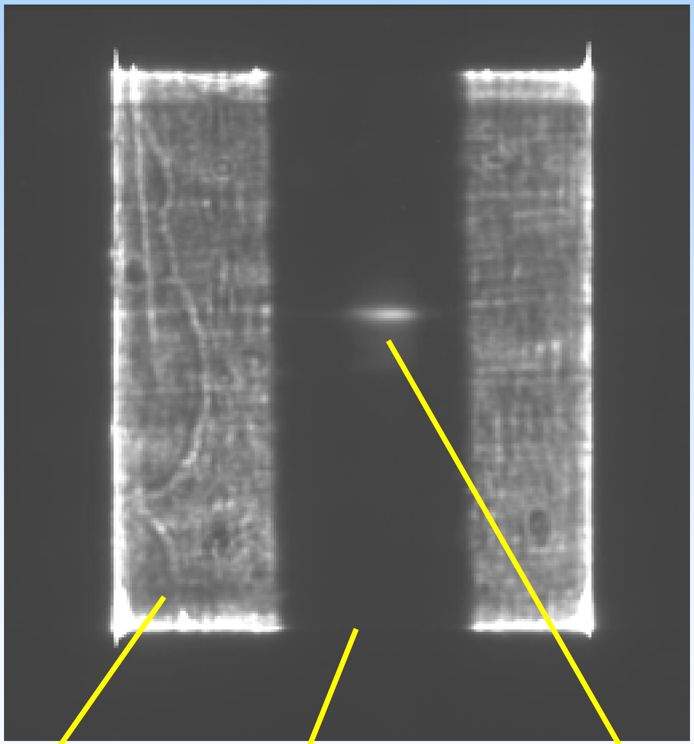
f_{hor}=0.25 m f_{vert} =0.15 m

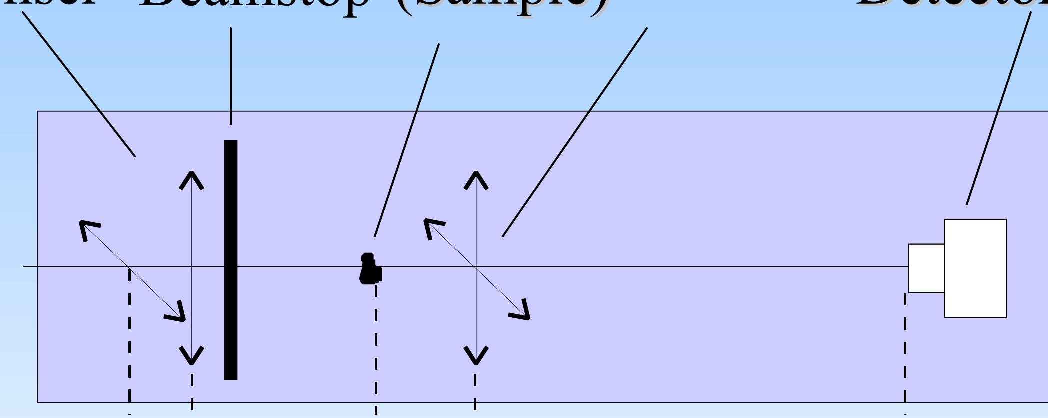
Reflectivity >80 % (theoretical)

Roughness < 1Å

[1] courtesy P. Eng, CARS, U. of Chicago

Microscope at 34 ID





to source: 53 m

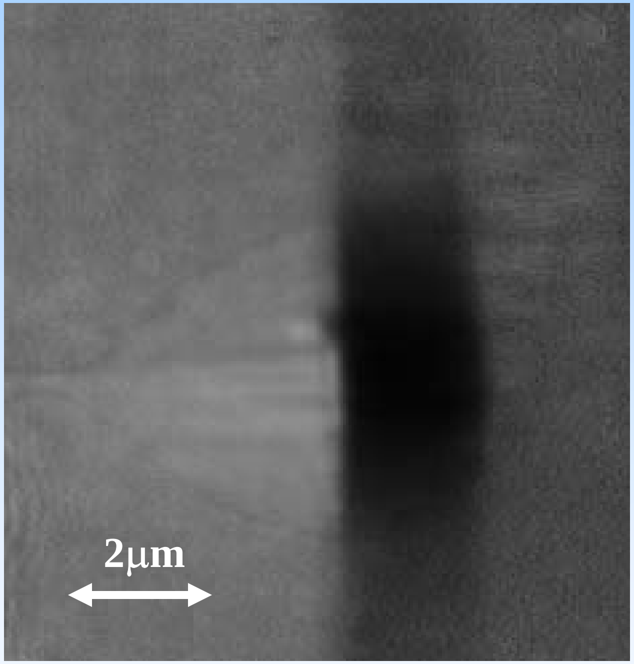
Intermediate Spot>4x5μm^2

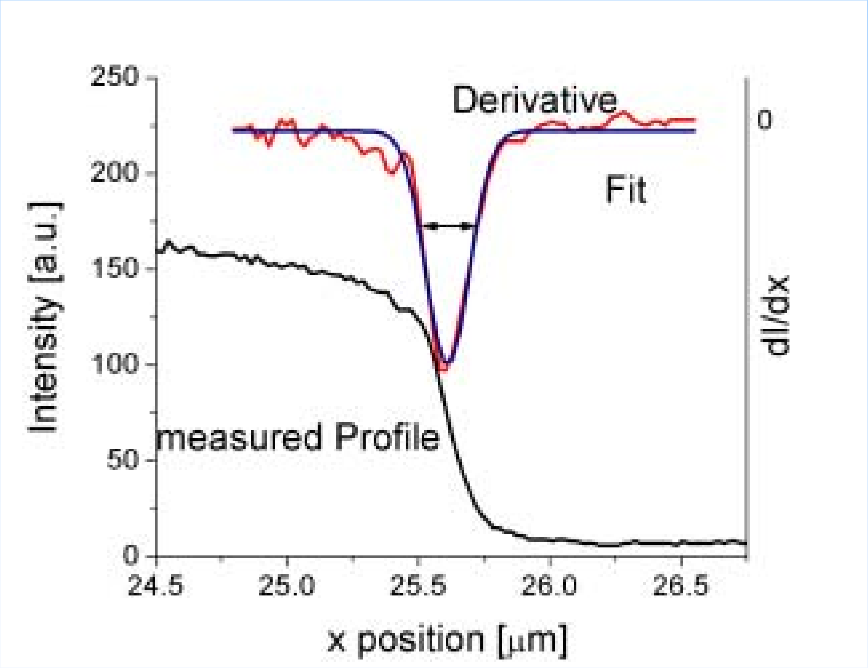
E = 9 keV

t_{exp}= 30 sec.

Microscope at 34 ID

Profile of an GaAs-edge 20x X-ray magnification





Measured Resolution 165 nm 20x X-ray magnification

135 nm 30x X–ray magnification

… nm 40x X-ray magnification

Phase II-IV

The Near and far future

Next developments

and

potential of microscope

Microscope at 34 ID

Limits of current setup

Resolution limit:

Higher order of ZP

Resolution $\delta=1.22dr_n/\mathbf{m}$ m:order

Efficiency $\eta \sim 1/m^2$ m is impair

Finer outer zones

dr_n of 40nm (Phase II)

Reaching 10nm resolution? (Phase III)

Zone plates with finer structure?

Microscope at 34 ID

Even finer structures possible?

Problem aspect ratio:
Etching techniques limited (> 1:20??)
→Deposition techniques: sputtering

Way of fabrication:
Circular lenses
Linear lenses

Thin lens approximation valid?

Prospects for the future

A microscope with 50nm resolution

- Depth of focus
- Field of View
- Intensity
- Contrast
- Mechanical Stability

Depth of focus

<u>Depth of Focus:</u>

$\sim\delta^2/\lambda$

$DOF = 2(dr_n)^2/\lambda$

$\rightarrow$ DOF 15-30μm for E = 6-12 keV

higher energies - contrast
Only partially sharp imaging – O.K. for Tomography
Smaller samples – handling, preparation
Field of view of Detector

Field of view

<u>Field of view:</u>

- 2048 Pixel with 12.5 nm size → 25 μm field of view
- Larger Detector Array (e.g. 4k x 4k)
- Tomography with 360º rotation?
- Partial Tomography?

- More general (valid also for higher resolution):

  combine low and high resolution tomographic data

  high resolution around rotation center

→ fits also with DOF problem: only central part of sample can be imaged with high resolution

Intensity

<u>Intensity:</u>

Why important?

Not only smaller but also
thinner features (3D) → contrast decrease→ N_{Ph}^{2}

with constant detector resolution and field of view:

Increase magn. → illum. Detector Area^2→ N_{Ph}^{2}

$\delta \sim N_{Ph}^{4}$

Microscope at 34 ID

Intensity:

Use of Pink beam 50-100 higher Intensity:

-Condition for FZP (chromatic aberration):

$N < E/\Delta E = 10^2$ N: Number of Zones

-but also 100 Zones minimum required for functioning of FZP

For $dr_n = 40nm$ @ 9 keV

$D = 4N\, dr_n$	$D = 16\ \mu m$	D: diameter of Zone Plate
And		dr_n : finest Zone width
$f = 4N\, (dr_n)^2 / \lambda$	$f = 5\ mm$	N: number of Zones
		f: focal length

- ***In principle*** possible- ***Real*** quality and feasibility of such a FZP?

- Perhaps intermediate solution? $N=500 \longrightarrow \Delta E/E = 2*10^{-3}$

Contrast

<u>Contrast:</u>

Number of counts necessary to visualize a given contrast:

Rose Signal-to-Noise Ratio

$\Delta SNR_{Rose} = C * \mathrm{Sqrt}\,(N_q)$

C: contrast

N_q: mean number of quanta/Pixel

SNR_{Rose} should be at least 5

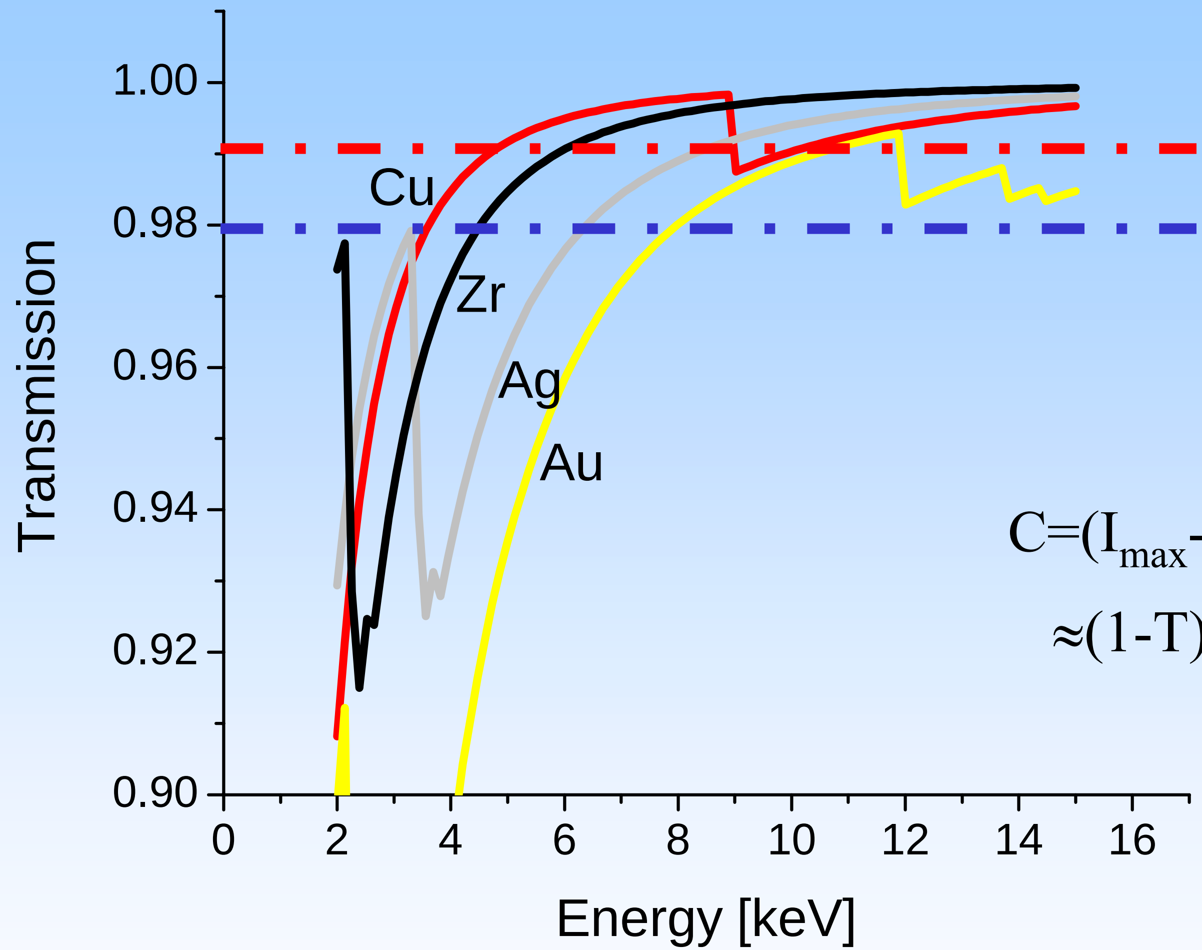
(for the table 10)

With actual equipment

C	**N_q**	Field of View	t_{exp}
1%	**10^6**	**30x20 μm^2 (2400x1600 Pixel)**	minutes

Space for improvements (pink beam, efficiencies,…)

Absorption/Transmission* of different elements with d=50nm as a function of E



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

$\approx(1-T)/2$

- Go to lower energies? May be, but
    - Problem DOF
    - Considering “free standing sample” - Penetration depth

→ hard X-rays = phase contrast

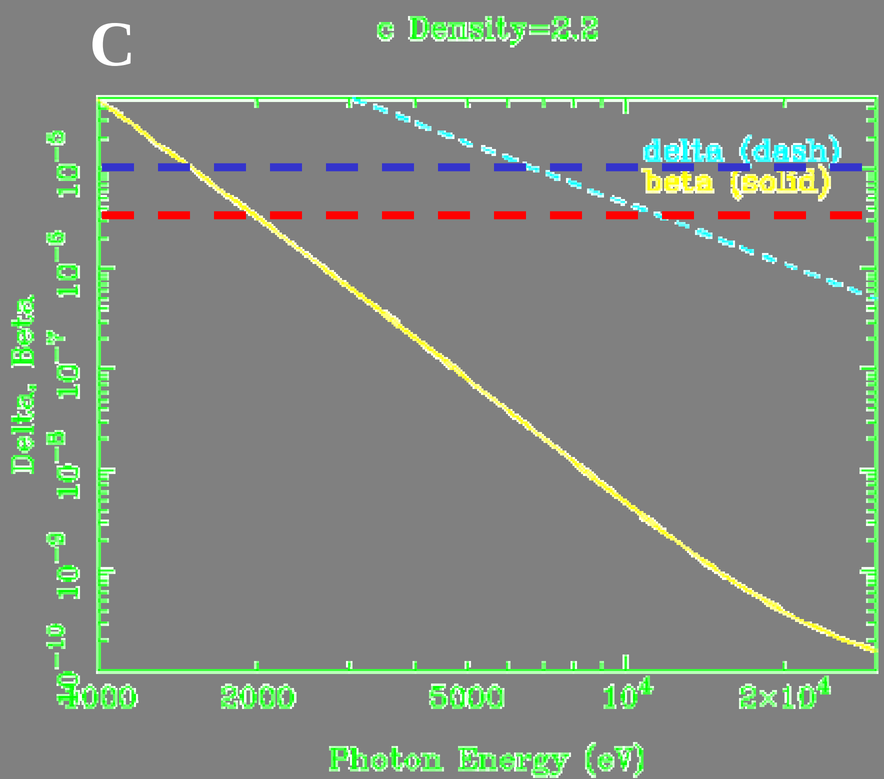
*http://www-cxro.lbl.gov

Phase Contrast - Refractive Index

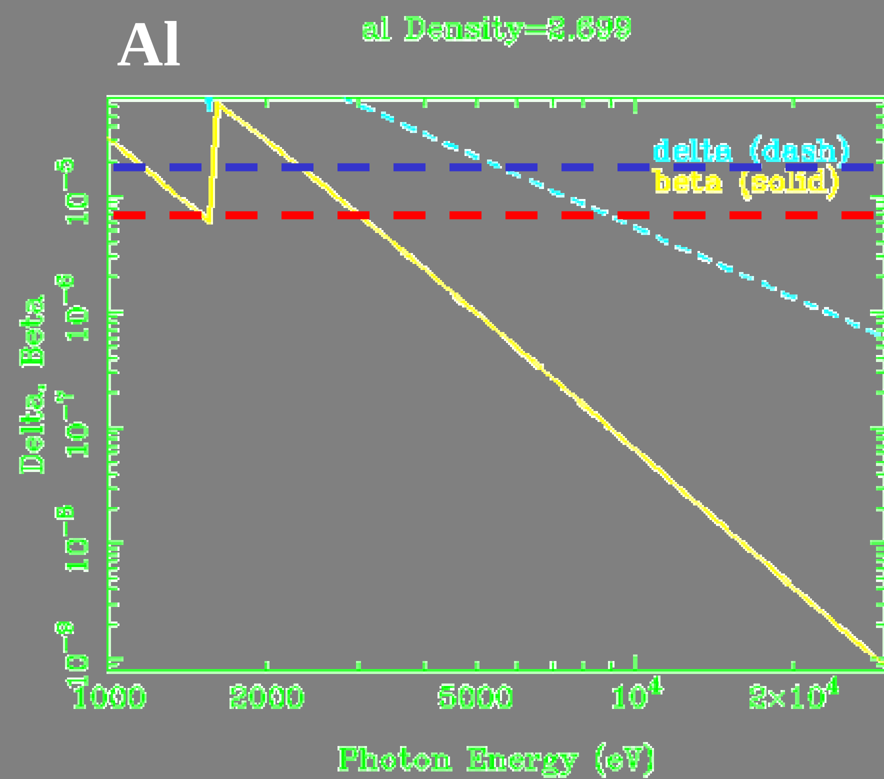
For very thin samples ($\Delta\Phi \ll 1$) and supposing pure phase contrast object $\Delta\Phi \sim C$

$$\Delta\Phi = (2\pi/\lambda)\,\delta\, l$$

C



Al



Sufficient contrast in interesting energy range

*http://www-cxro.lbl.gov

Microscope at 34 ID

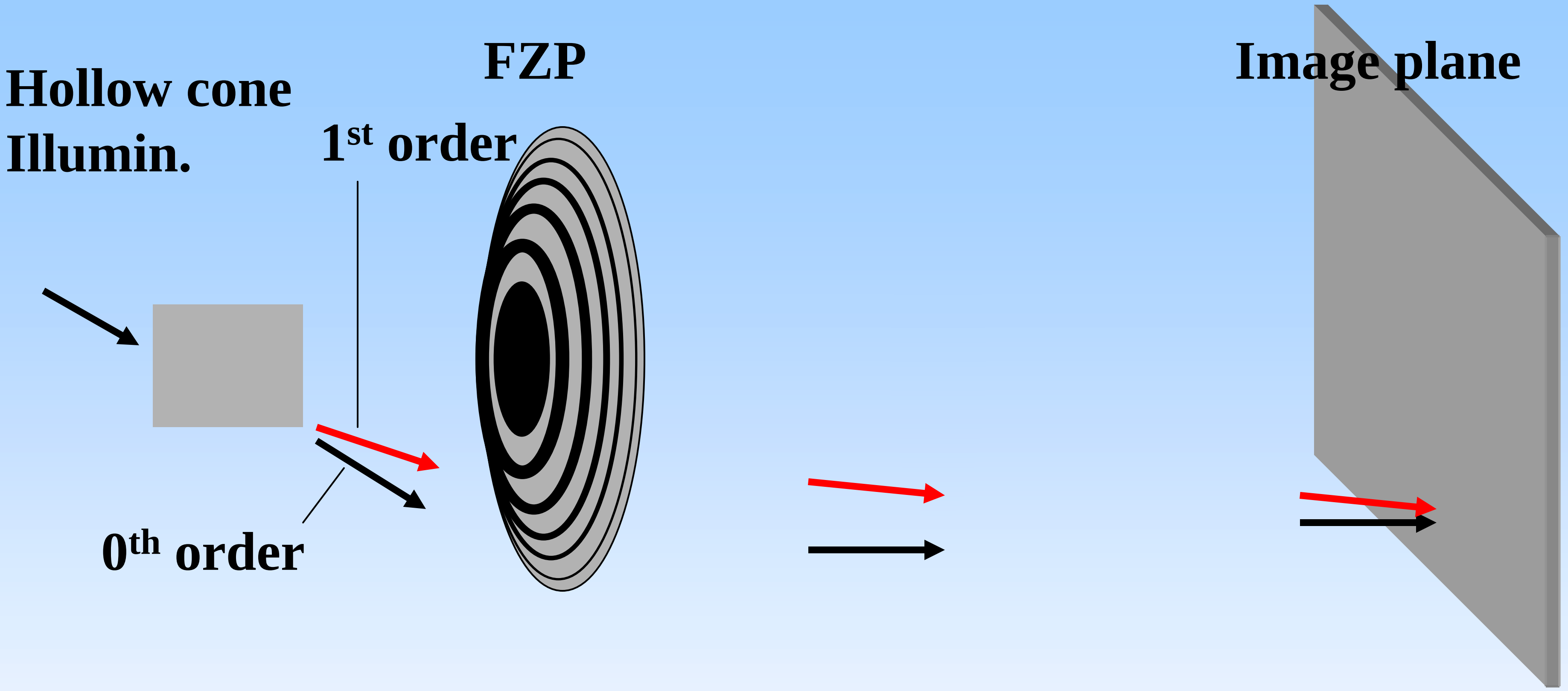
Phase Contrast:
Making use of Zernike Phase contrast method:

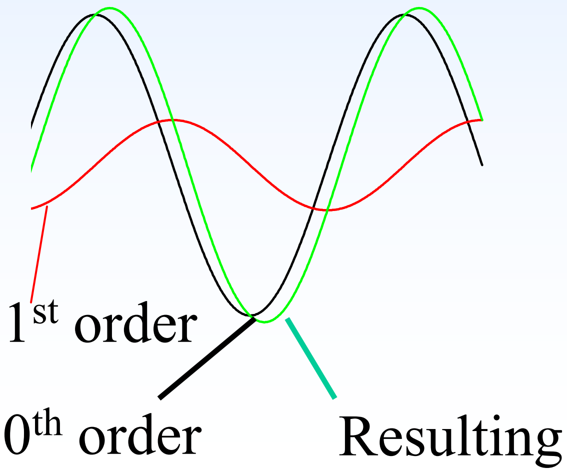
Basic Idea:

-Transform a phase information into an amplitude information
-Diffracted beam(1st order) phase shifted in respect to undiffracted beam(0th order).

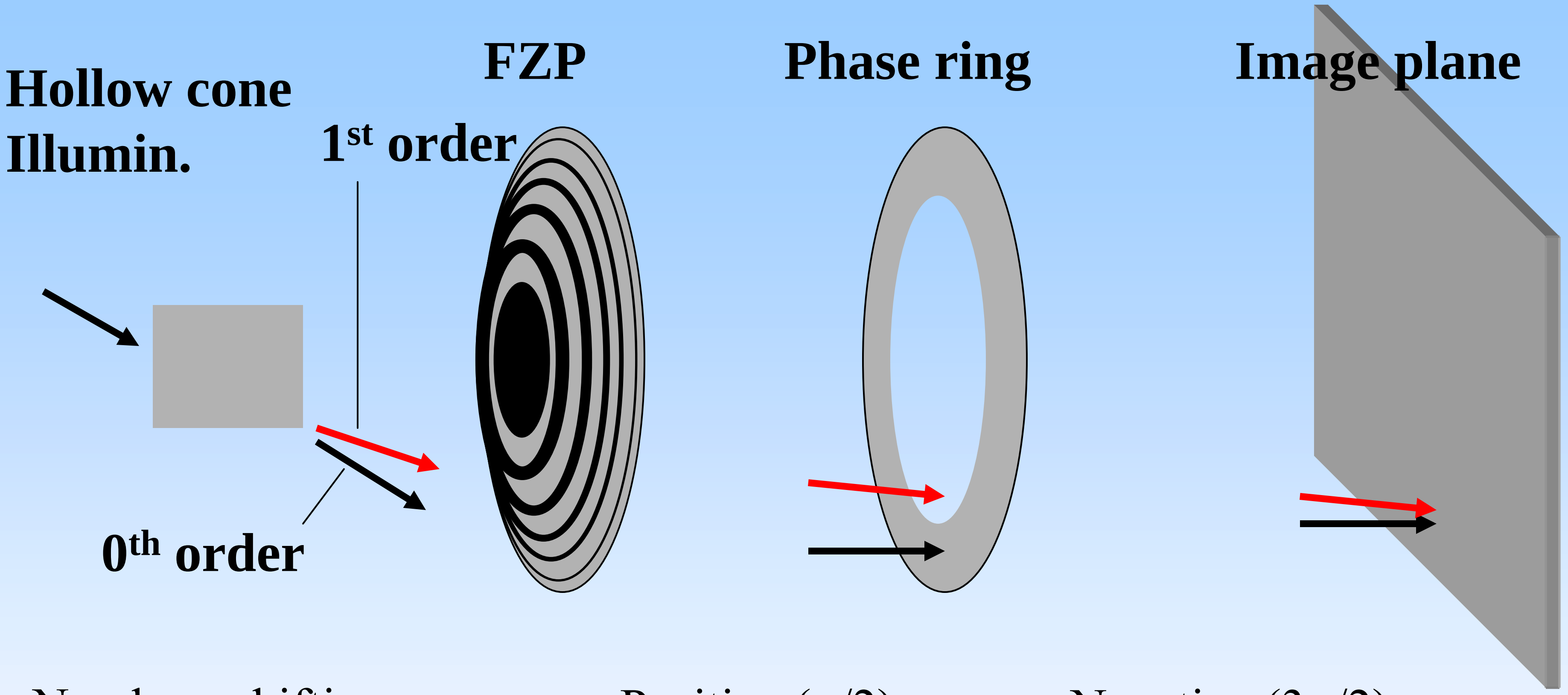
-1st and 0th order spatially separated in back focal plane
-Phase ring in back focal plane:
phase shift ($\pi/2$ or $3\pi/2$) and attenuation of 0th order
-In image plane increased contrast can be achieved

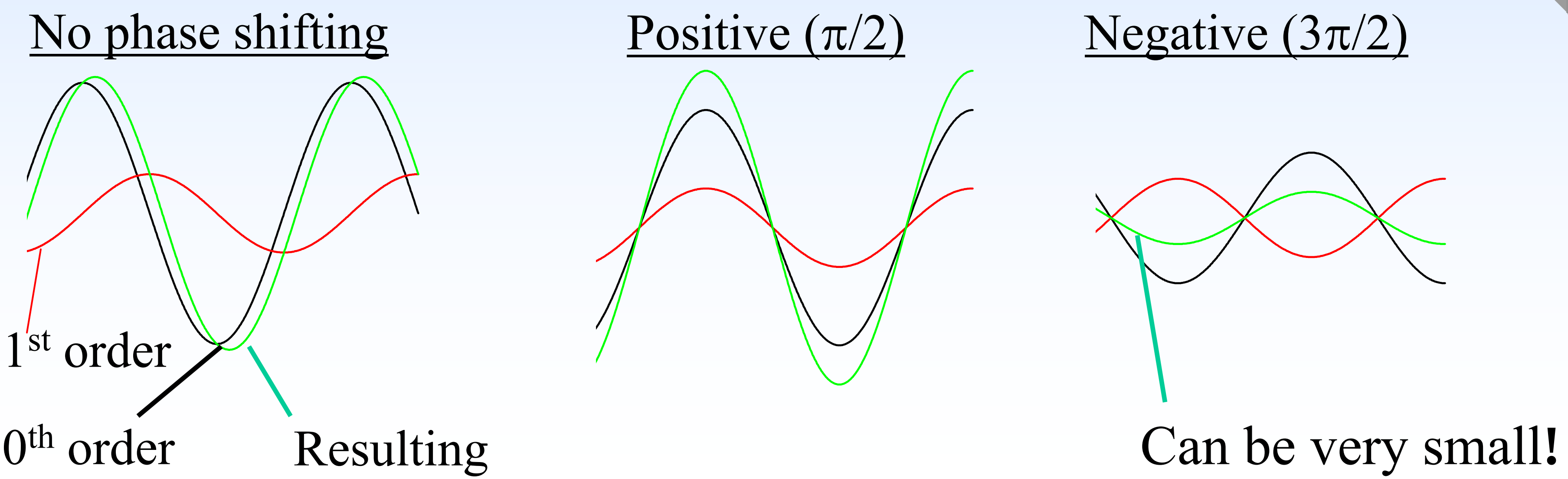
Zernike Phase Contrast





Zernike Phase Contrast





Summary

Development of sub-100 nm Microscope at 34 ID-C

-KB-FZP optic

-Zernike Phase Contrast

Current project for user-friendly scientific applications

In future 50nm imaging/tomography

- min.exposures/optimization

Ultimate resolution 10nm?

Relation to other methods?

Lensless imaging? let's listen to the next talk!

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