(Towards) Multi-Order Hard X-Ray Imaging with Multilayer Zone Plates

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Thanks to:
Aike Ruhlandt[1], Sarah Hoffmann[1], Anna-Lena Robisch[1], Felix Schlenkrich[2], Alexey Zozulya[3], Thomas Hoinkes[4], Arno Rauschenbeutel[4]

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Former group members:
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Technical support:
Bastian Hartmann[1], Jan Goeman[1], Volker Radisch[2]

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Deutsche Forschungsgemeinschaft, SFB 755
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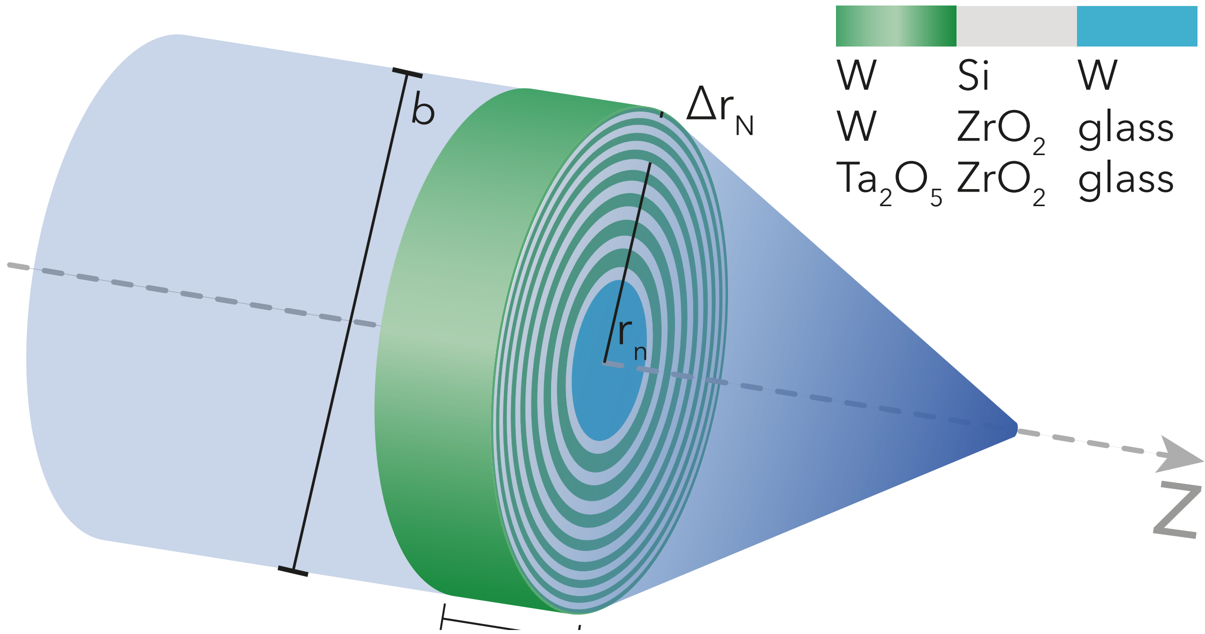


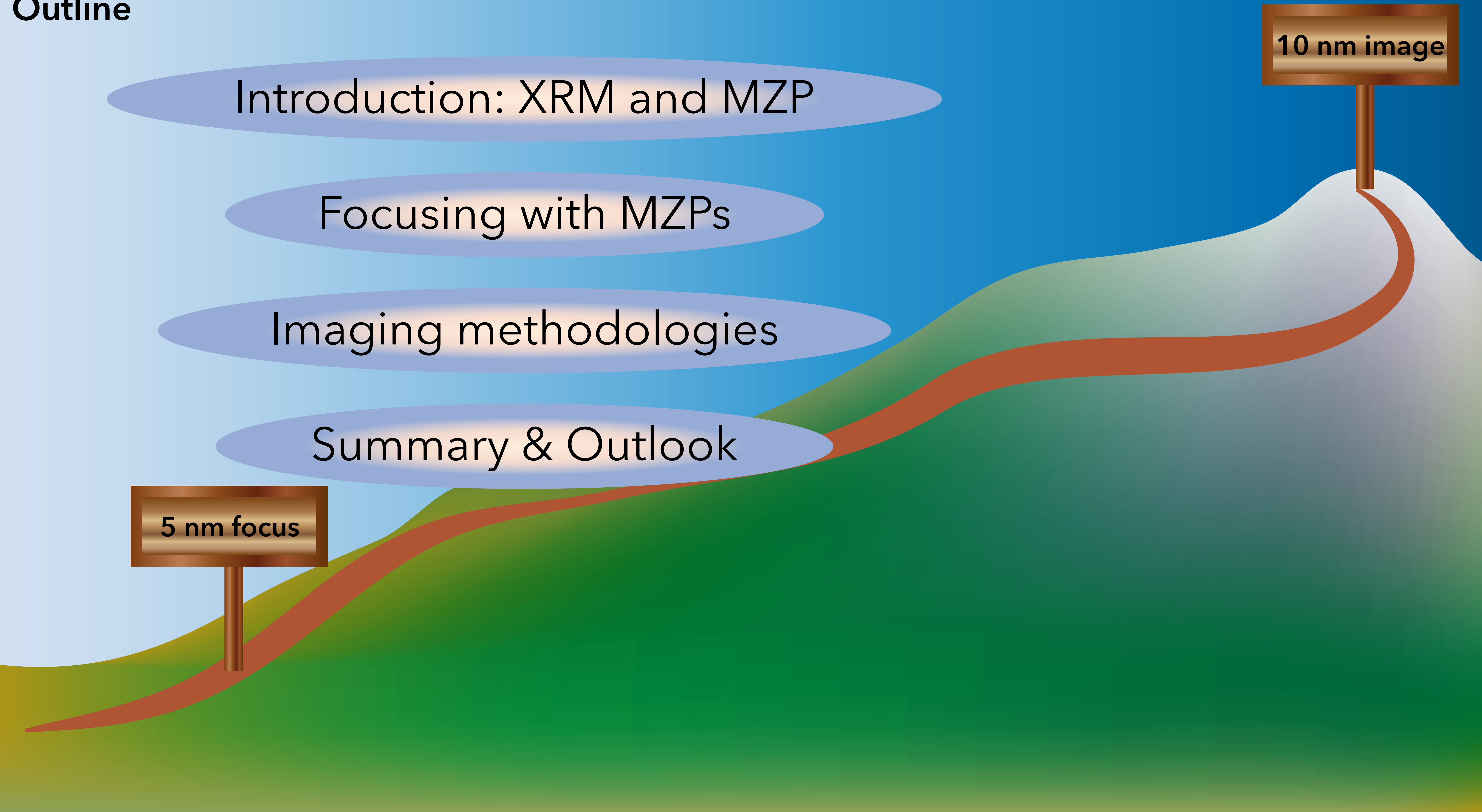
Outline

Introduction: XRM and MZP

Focusing with MZPs

Imaging methodologies

Summary & Outlook

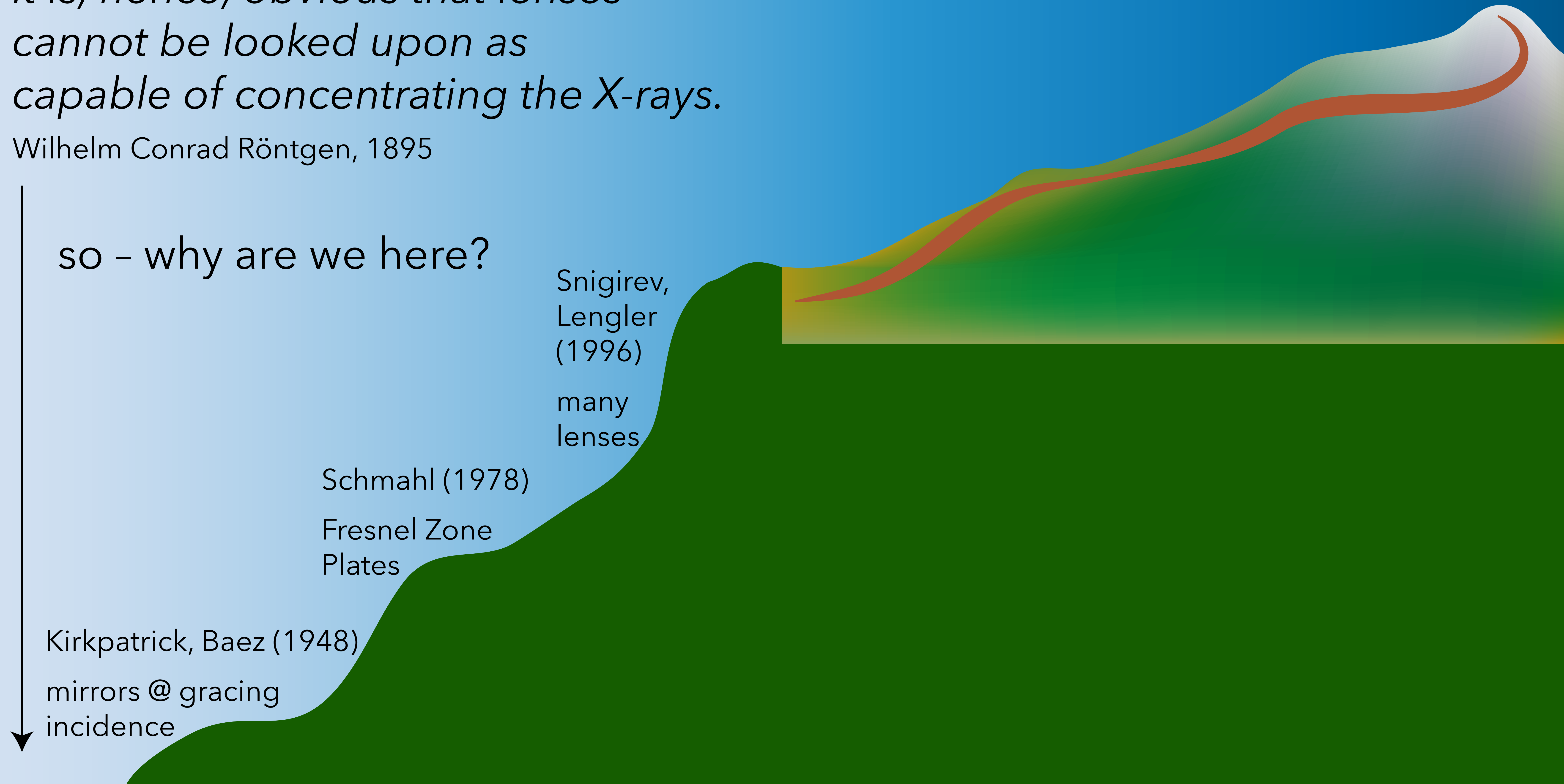


History

It is, hence, obvious that lenses cannot be looked upon as capable of concentrating the X-rays.

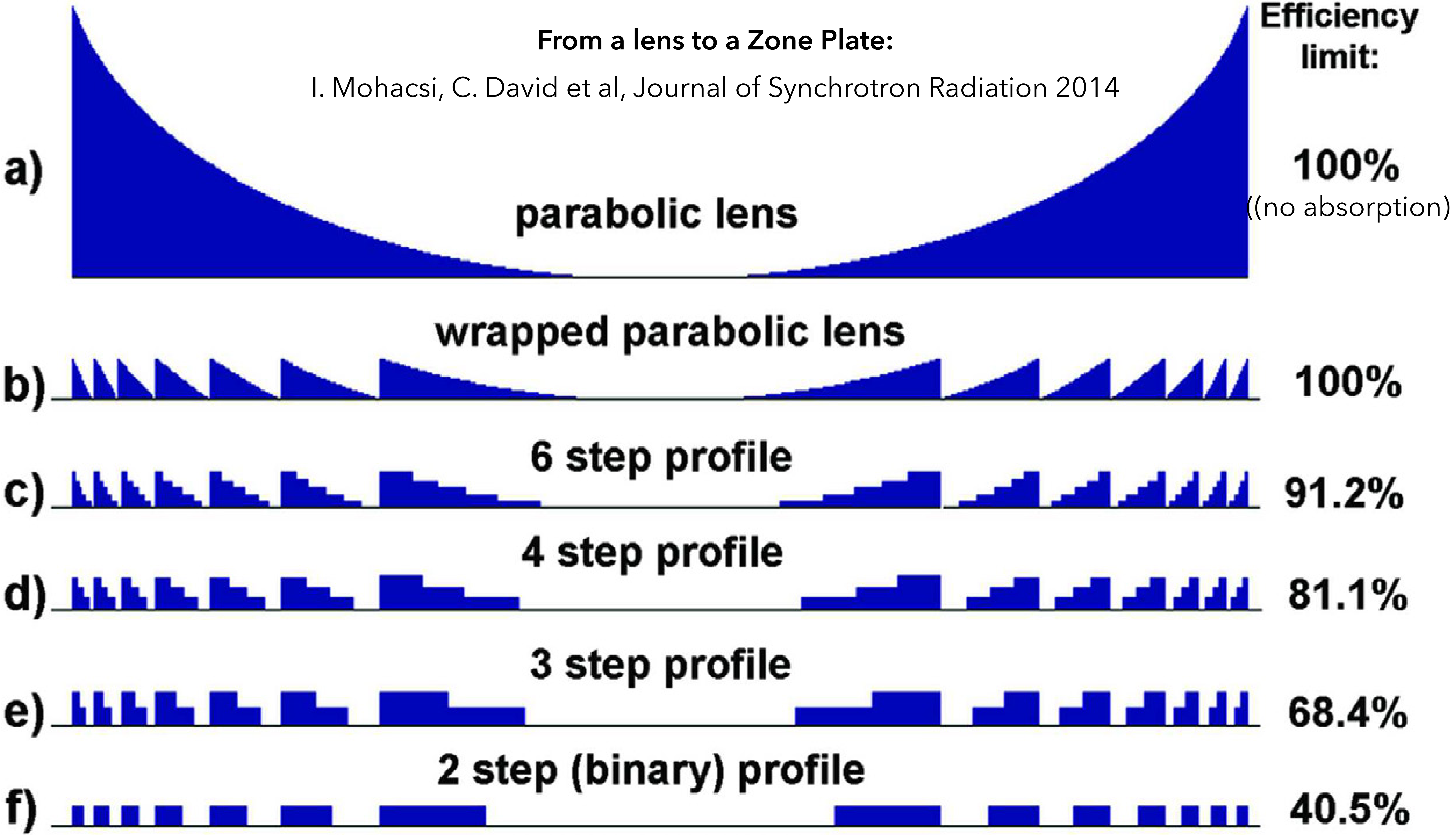
Wilhelm Conrad Röntgen, 1895

so – why are we here?



From a lens to a Zone Plate:

I. Mohacsi, C. David et al, Journal of Synchrotron Radiation 2014



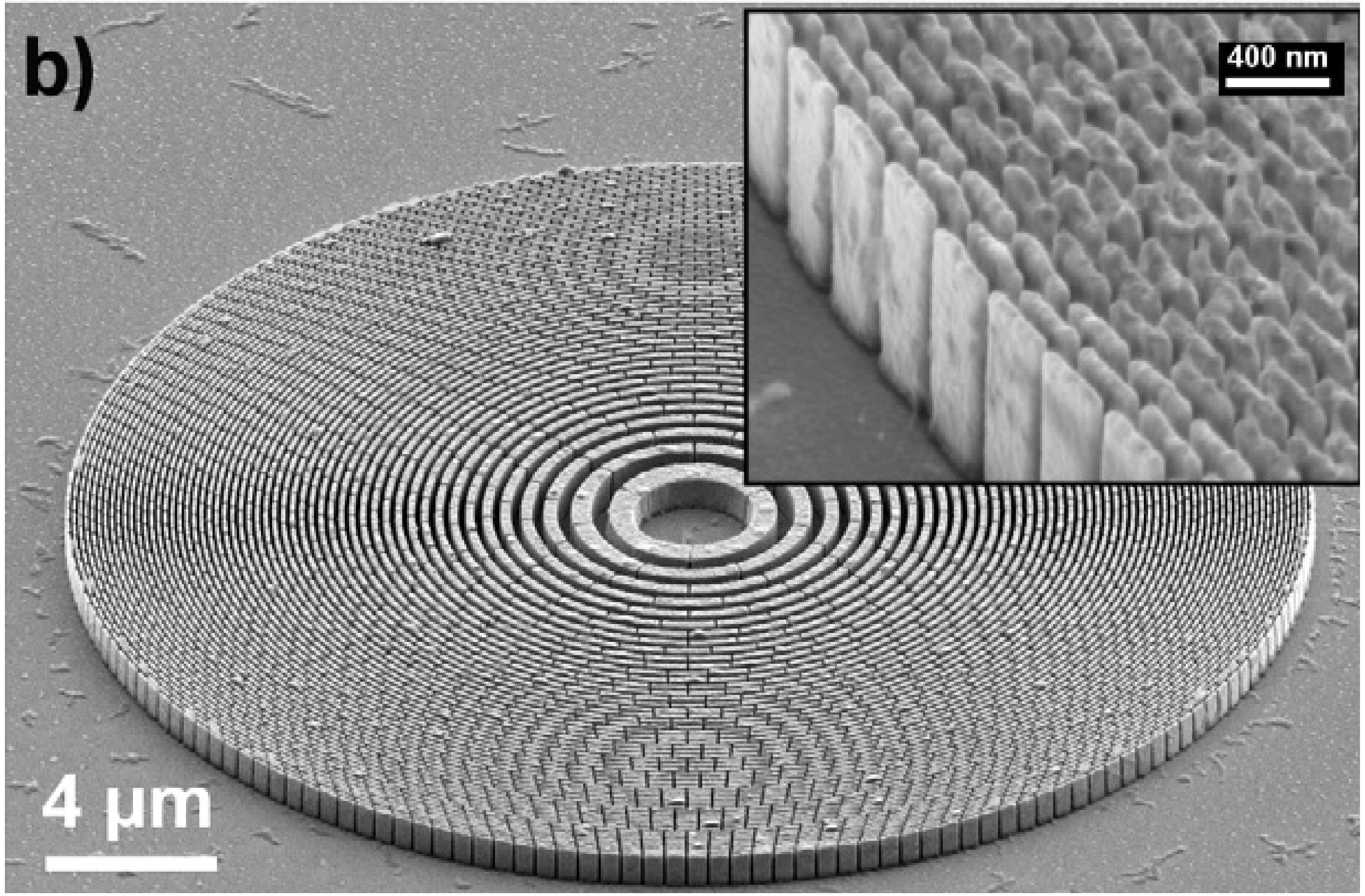


Figure 7. Scanning electron microscope image of (a) 0.5 μm and (b) 1 μm thick Fresnel zone plates fabricated by electroplating Au in PMMA molds that were produced by directly writing into the resist with 100 keV electrons. The insets present magnified inner and outer regions of the devices.

The “gold” standard:

E-Beam Lithography,

Electroplating

Aspect ratios of > 20 possible; phse-shifting FZPs for ~ 8 keV

S. Gorelick, C. David et al, Nanotechnology 2010

Focusing with MZPs

(Binary) Zone Plate Law:

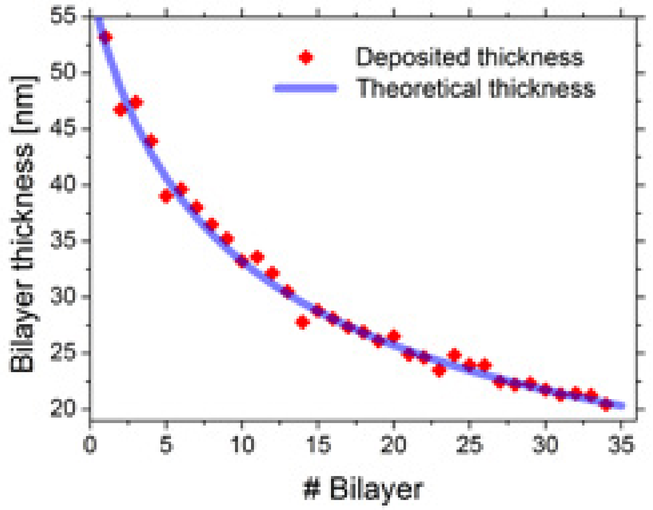
$$r_n = \sqrt{\left(\frac{n\,\lambda}{2}\right)^2 + n\,\lambda\,f}$$

zone radius for
given wavelength
and focal length

absorption vs. phase-shifting:

- efficiency varies as a function of optical thickness
- waveguiding effects for thin zones
- layer roughness / fluctuations
- optimal profile along optical axis

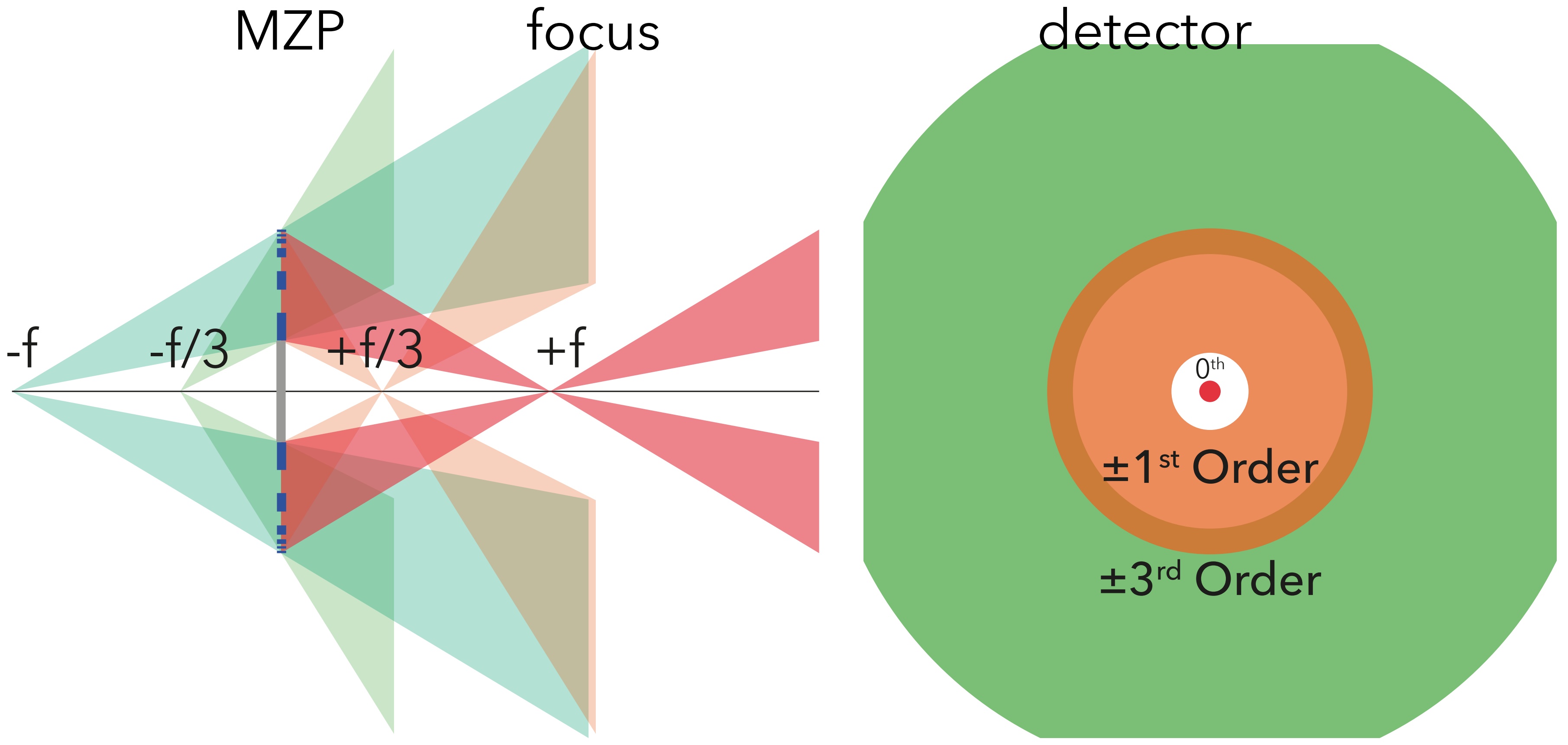
multiple orders / diverging orders:



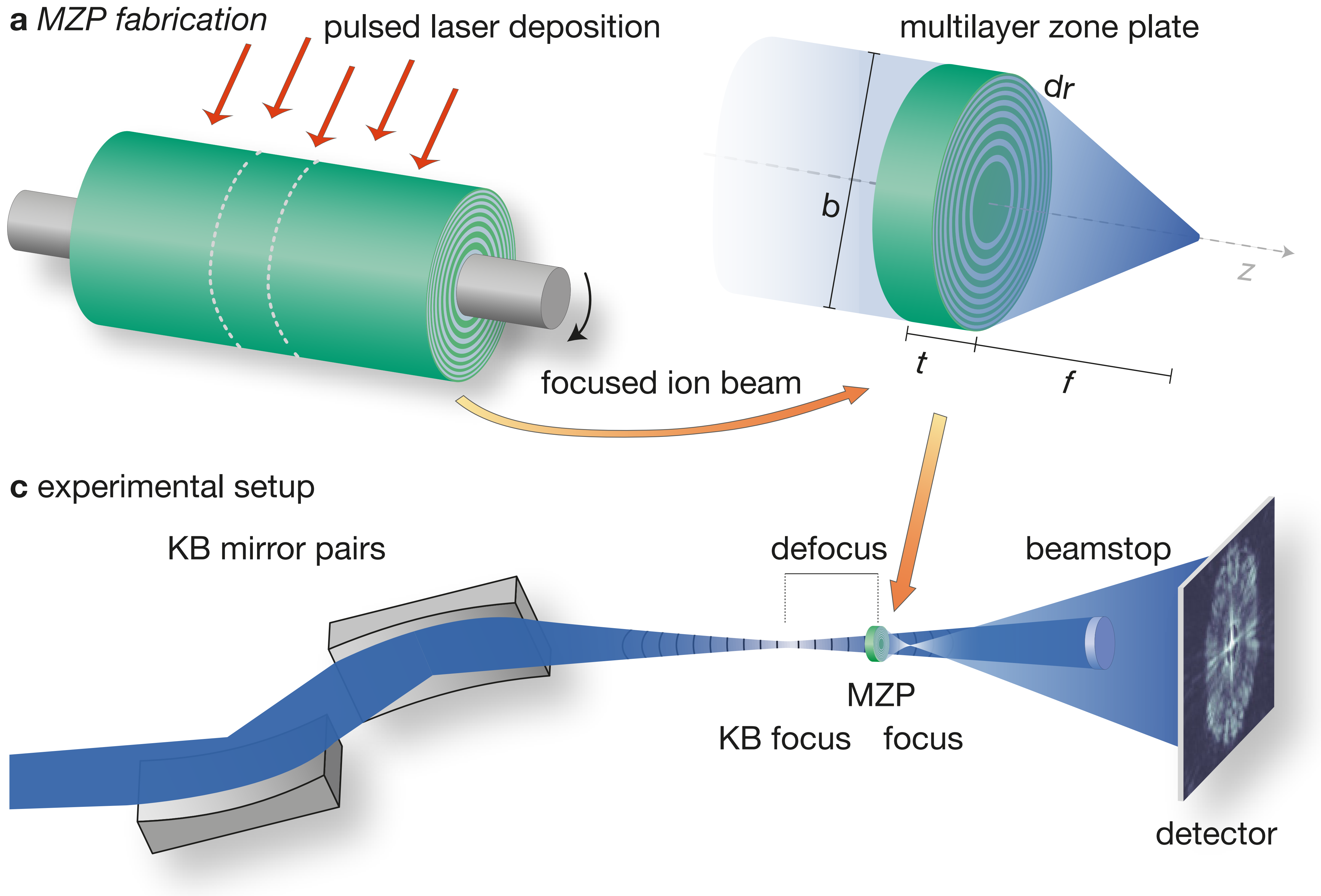
here: Ta_2O_5 / ZrO_2

n = 12 ... 81
f = 0.47 mm
λ = 0.00 nm
E = 18.0 keV

here:
hollow cone
illumination



Fabrication: Pulsed Laser Deposition and Focused Ion Beam



layer thicknesses: **3 nm** ... 30 nm

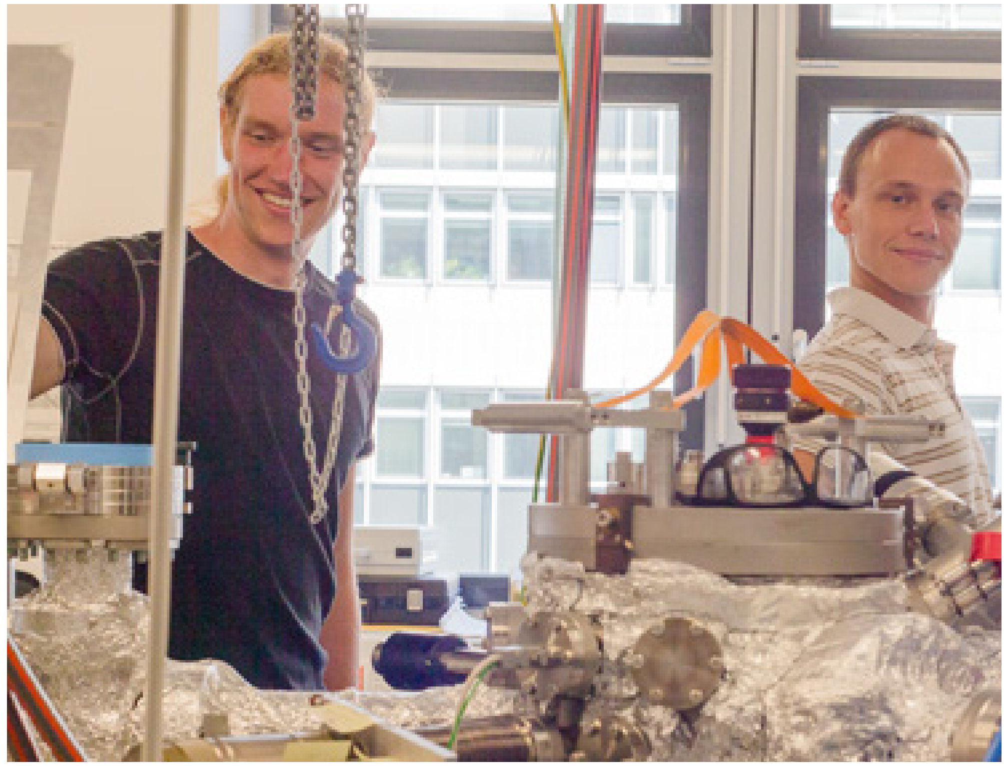
wire / glass core: < 1 μm ... 2 μm

layers: 50 ... 80 in x-rays

latest MZP size:
⌀ **9 μm** and growing

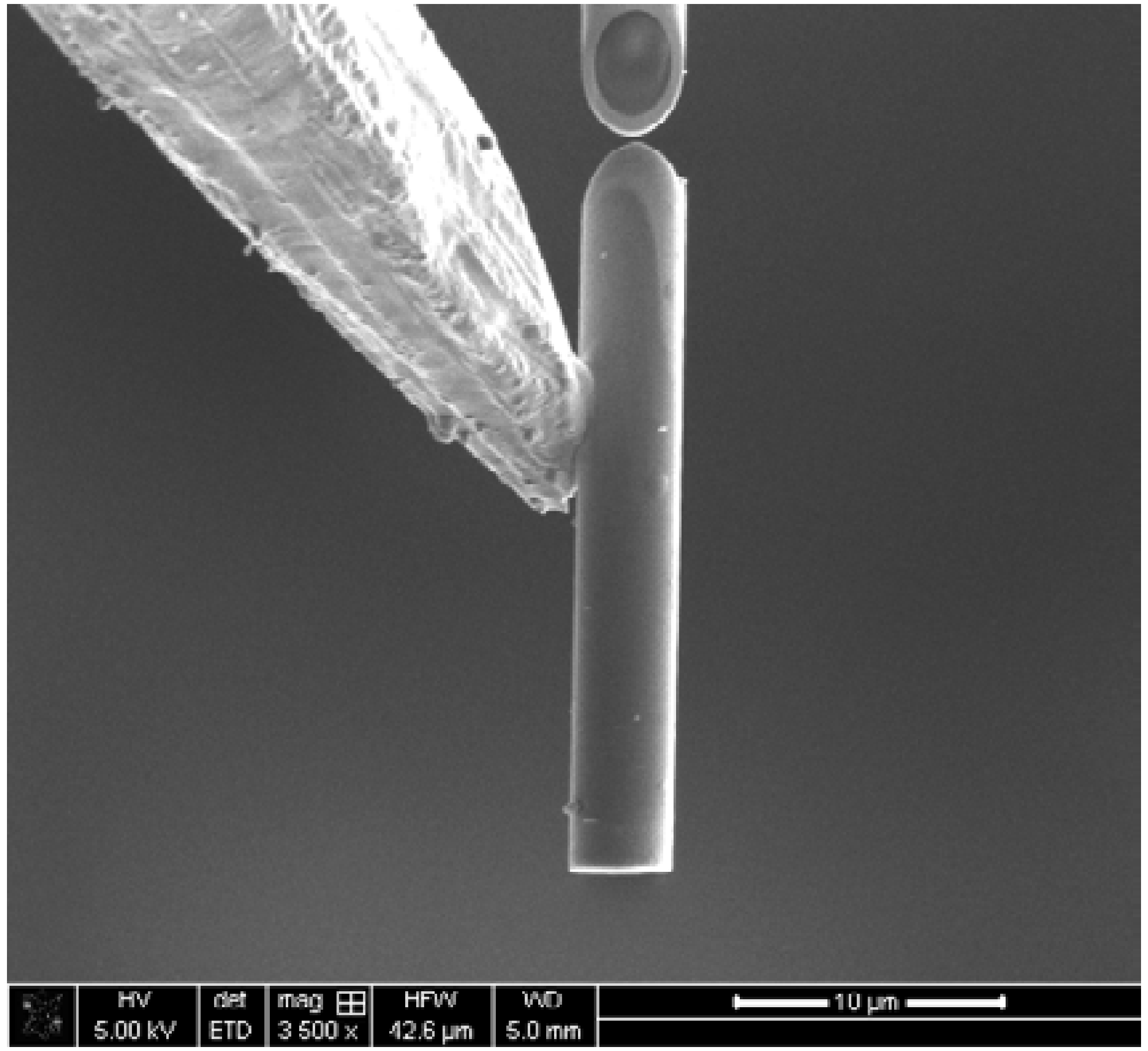
advantage PLD:
cumulative smoothening

so outer layers are
better than inner ones

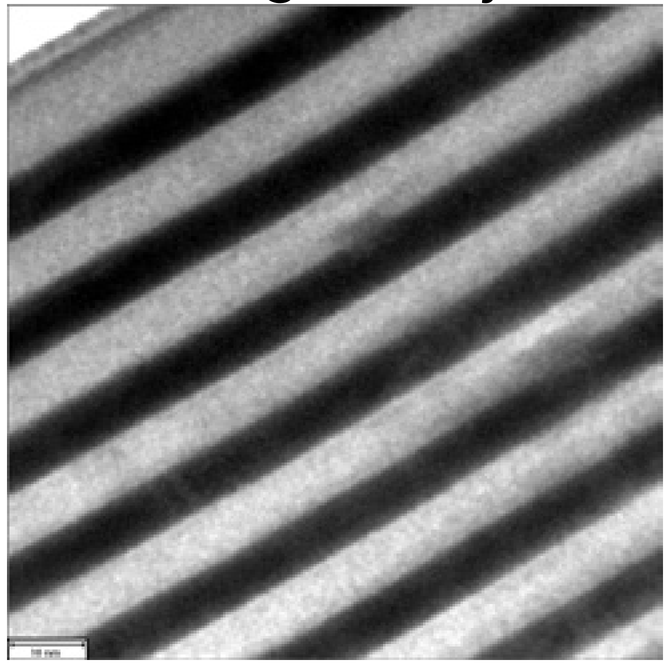


Döring et al., Optics Express (2013)

FIB handling



TEM image of layers



Poster on MZP fabrication:

Poster #4 on Tuedsay's session
C. Eberl et al.

PLD parameters:

Ta_2O_5 / ZrO_2

2.6 J / cm2 @ 10 Hz

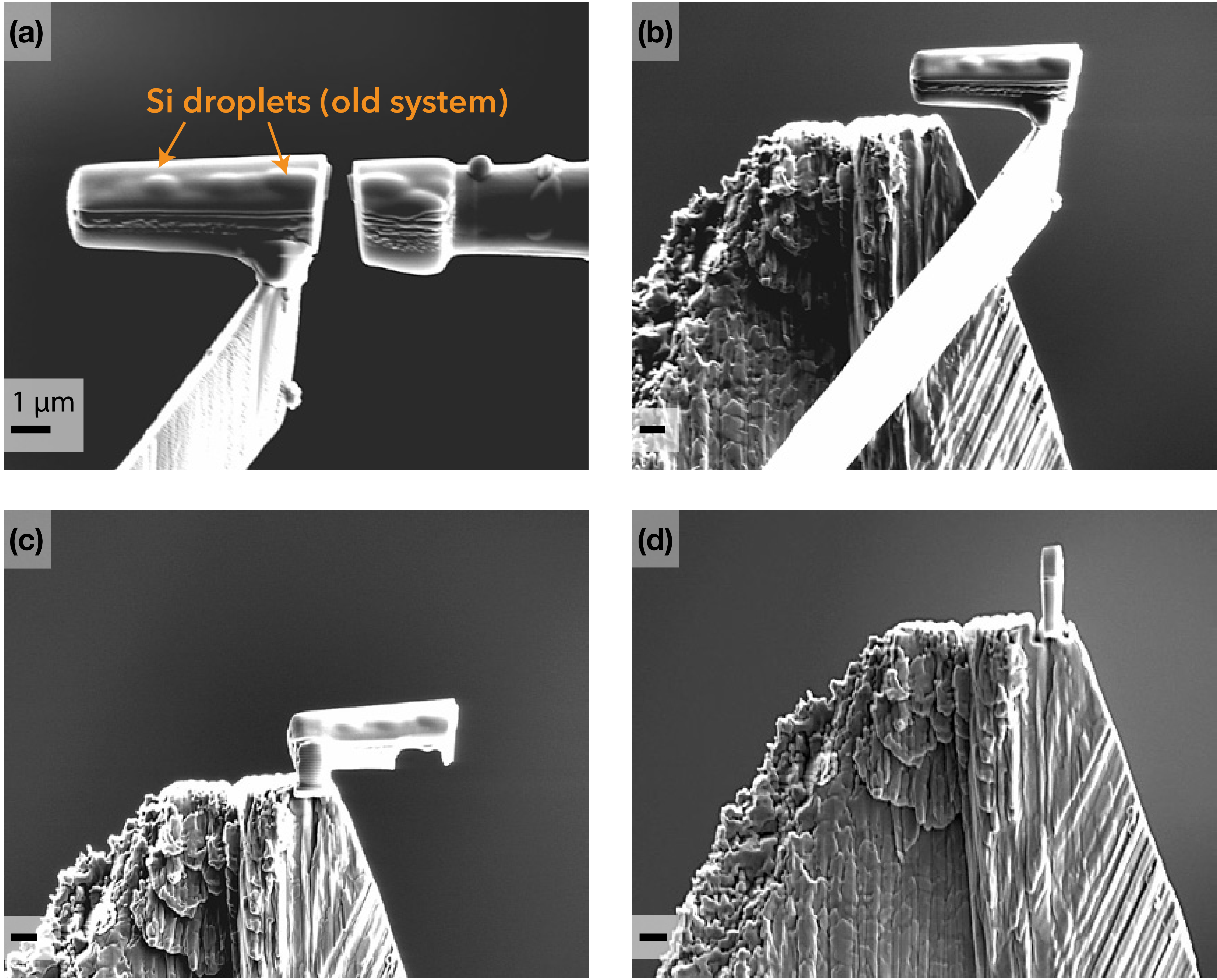
40 nm / 1000 pulses ~ 20 minutes

nearly constant, monitored rate

FIB slicing
of individual
an lens,

mounted on
a support wire

optical thickness ~ 10 μm



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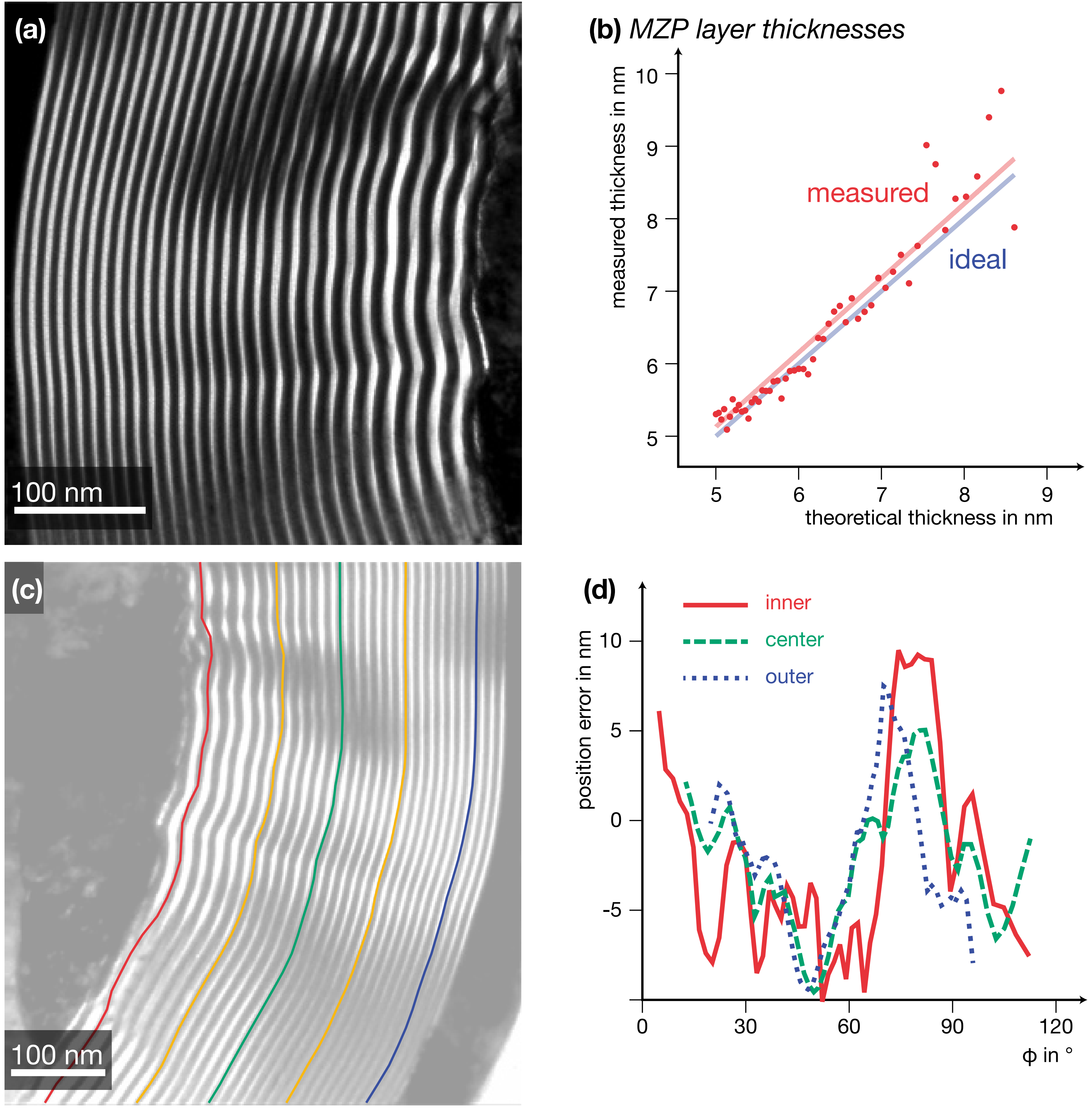
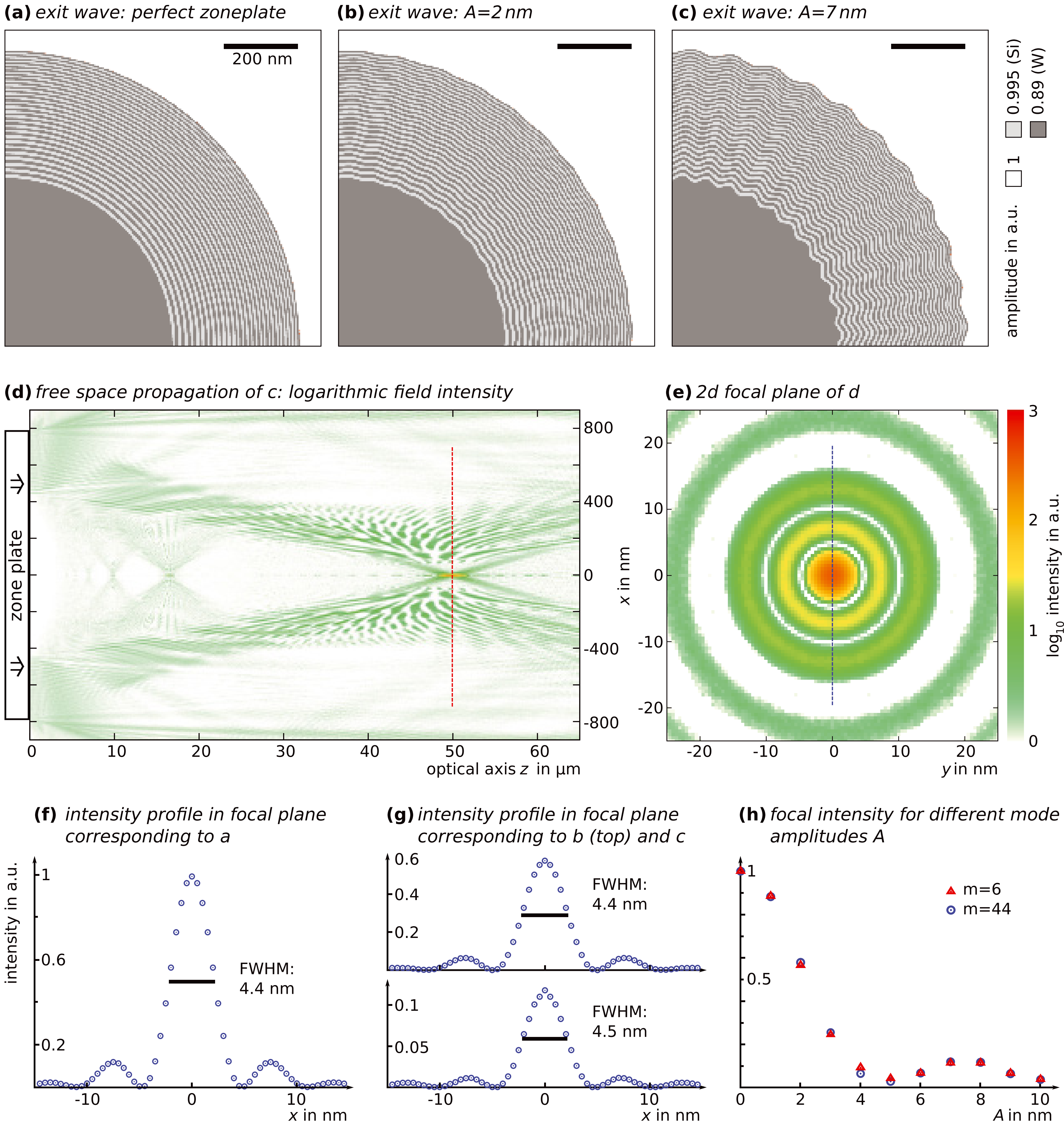
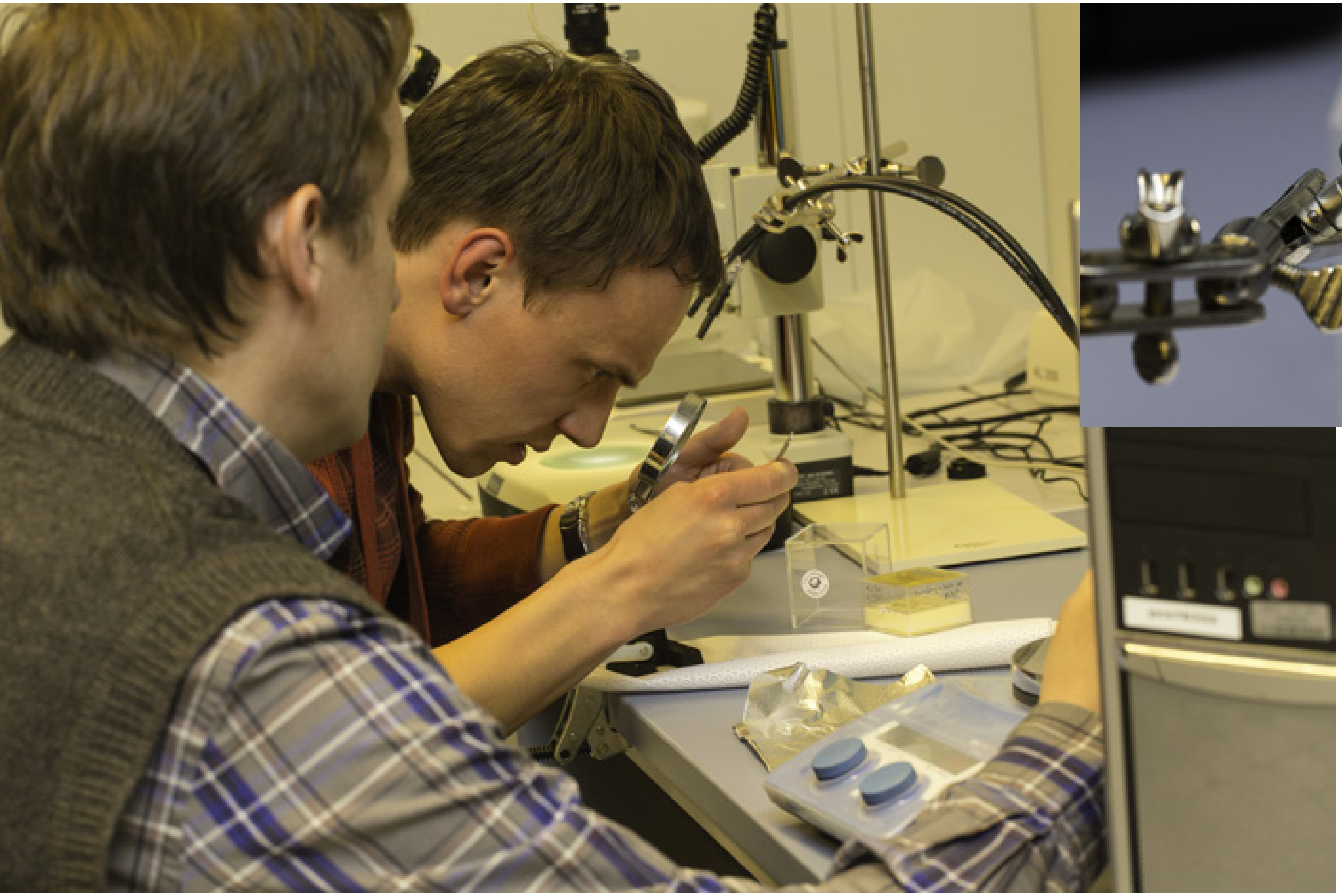
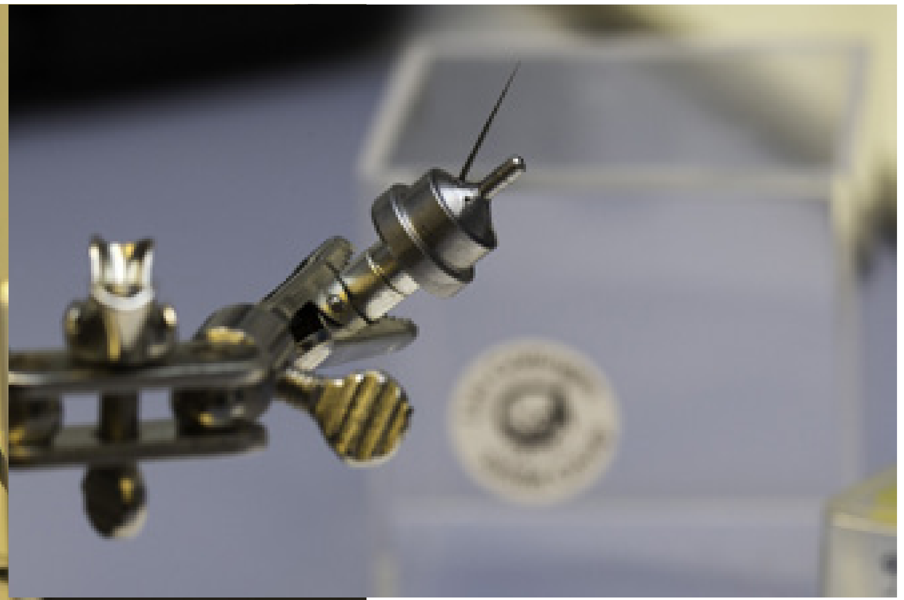
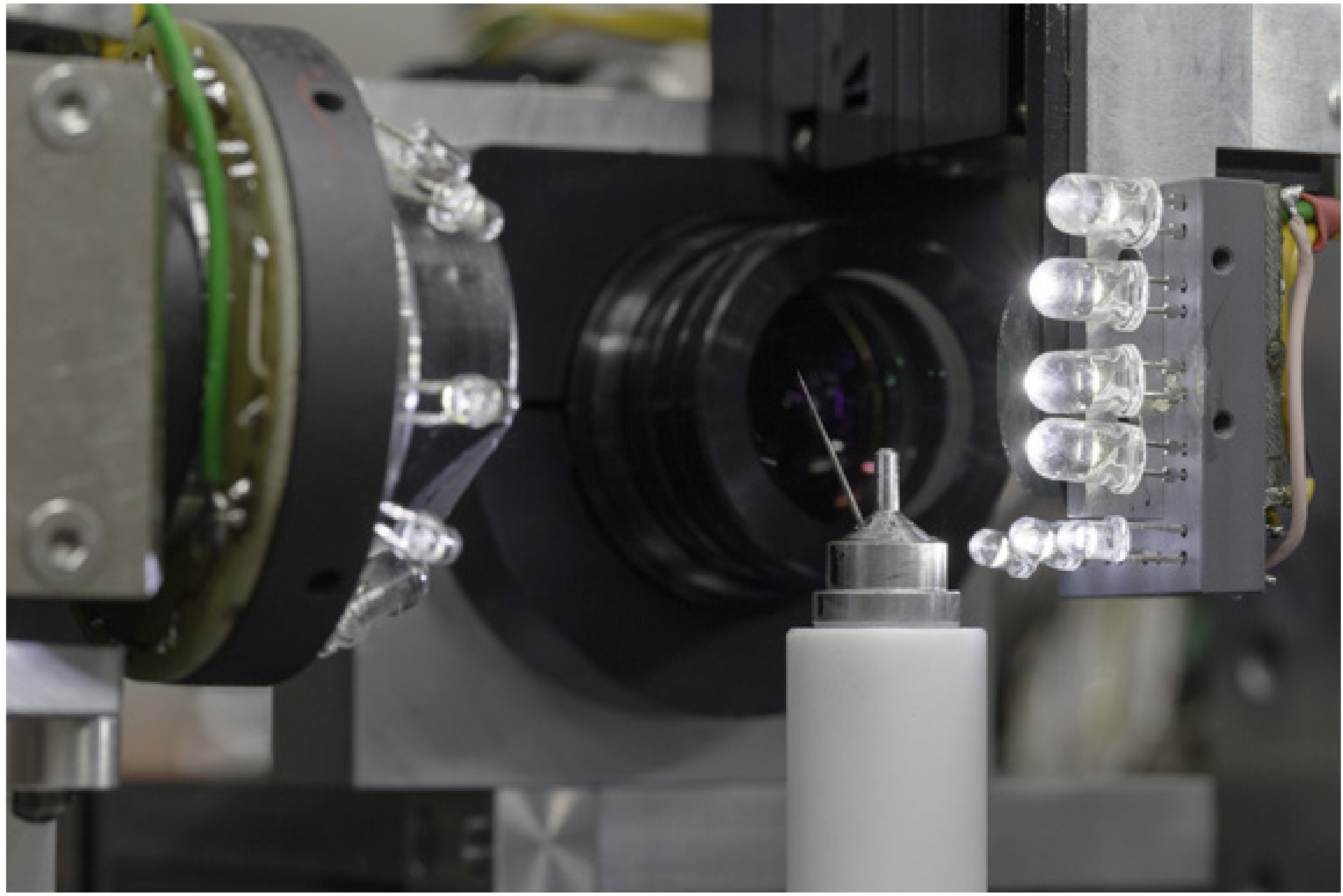


Rough wire – Let's simulate it! – Let's try it!

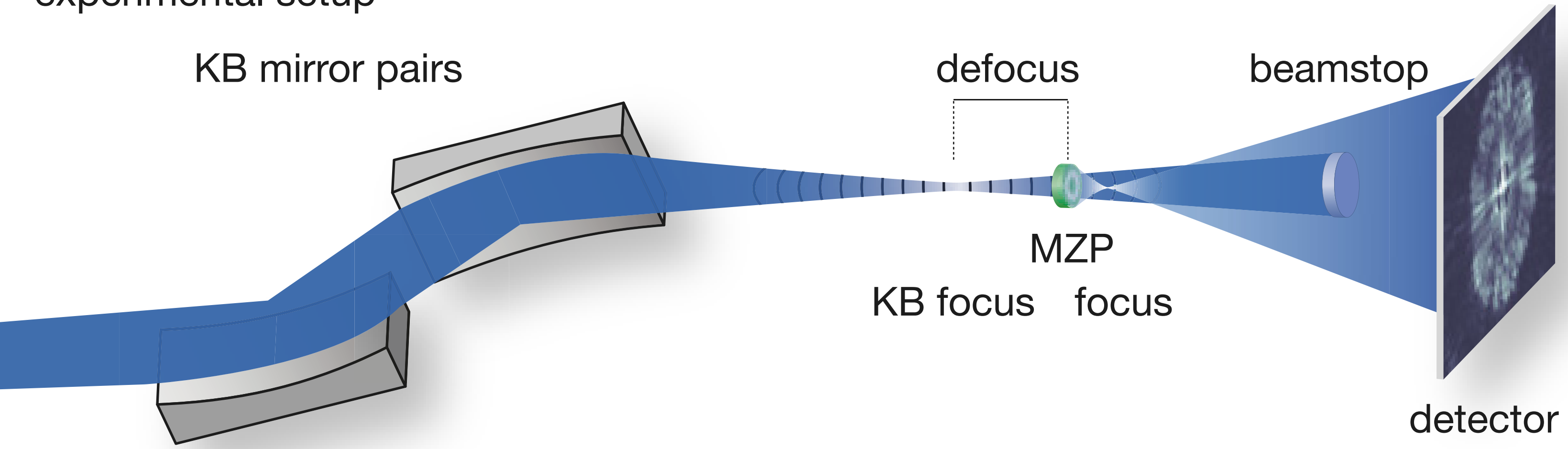


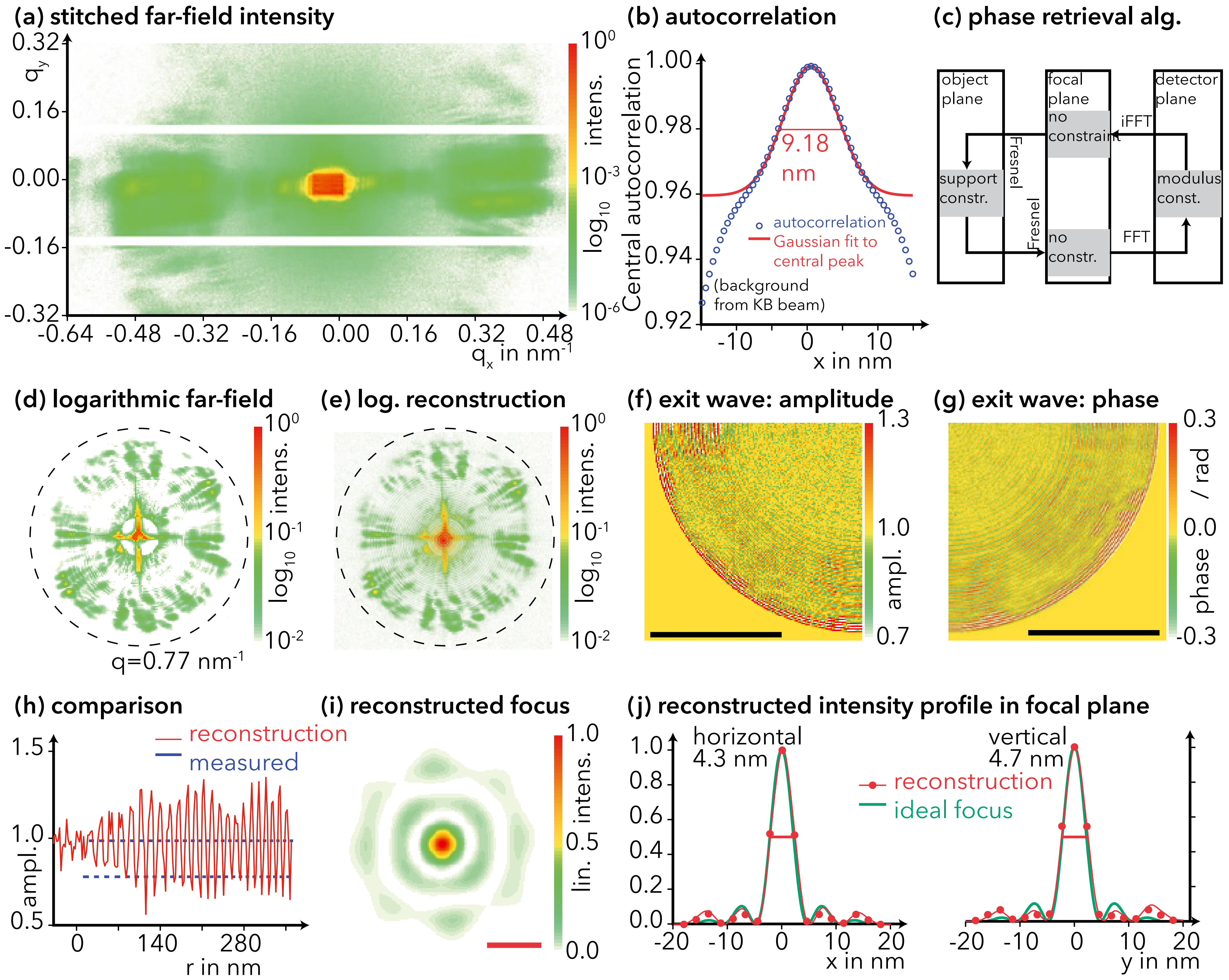






experimental setup





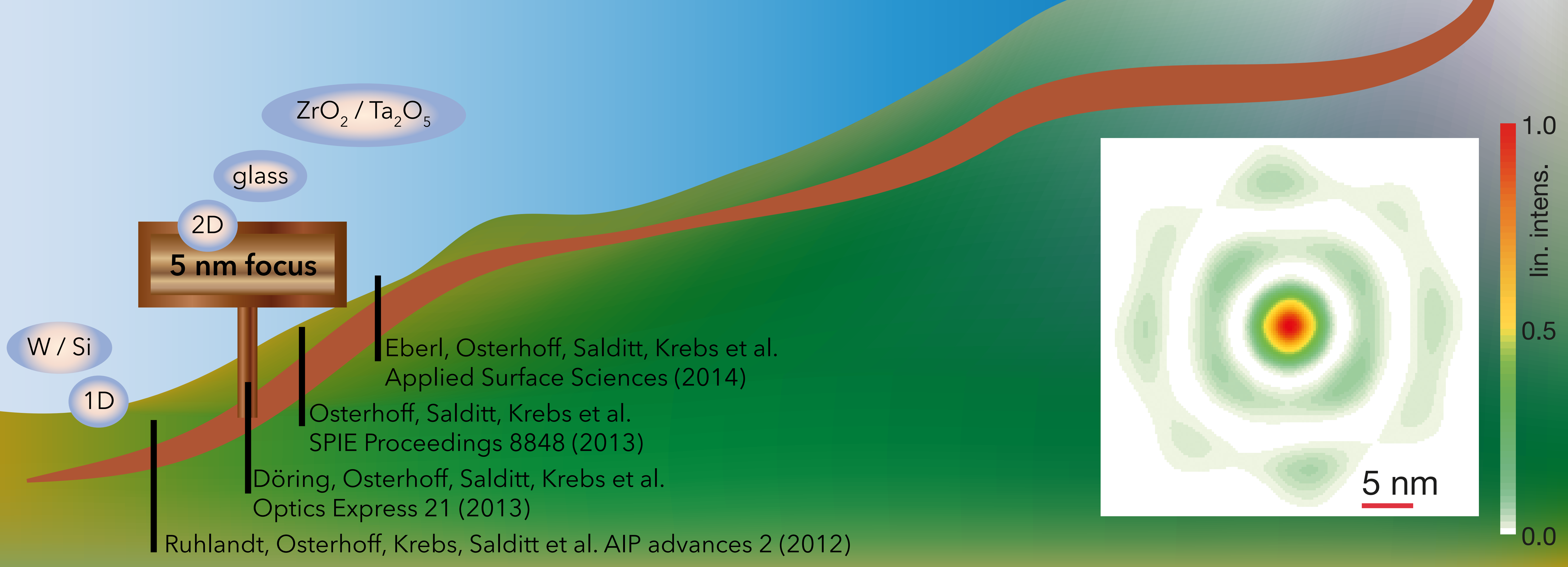
(a) stitched far-field intensity
(b) autocorrelation
(c) phase retrieval alg.
Central autocorrelation
9.18 nm
autocorrelation
Gaussian fit to central peak
(background from KB beam)
x in nm
q_y
q_x in nm^{-1}
$\log_{10}$ intens.
object plane
focal plane
detector plane
no constraint
iFFT
Fresnel
support constr.
modulus const.
no constr.
FFT
(d) logarithmic far-field
(e) log. reconstruction
(f) exit wave: amplitude
(g) exit wave: phase
q=0.77 nm^{-1}
ampl.
phase / rad
(h) comparison
(i) reconstructed focus
(j) reconstructed intensity profile in focal plane
reconstruction
measured
r in nm
lin. intens.
horizontal 4.3 nm
vertical 4.7 nm
ideal focus
y in nm

Summary: Focusing

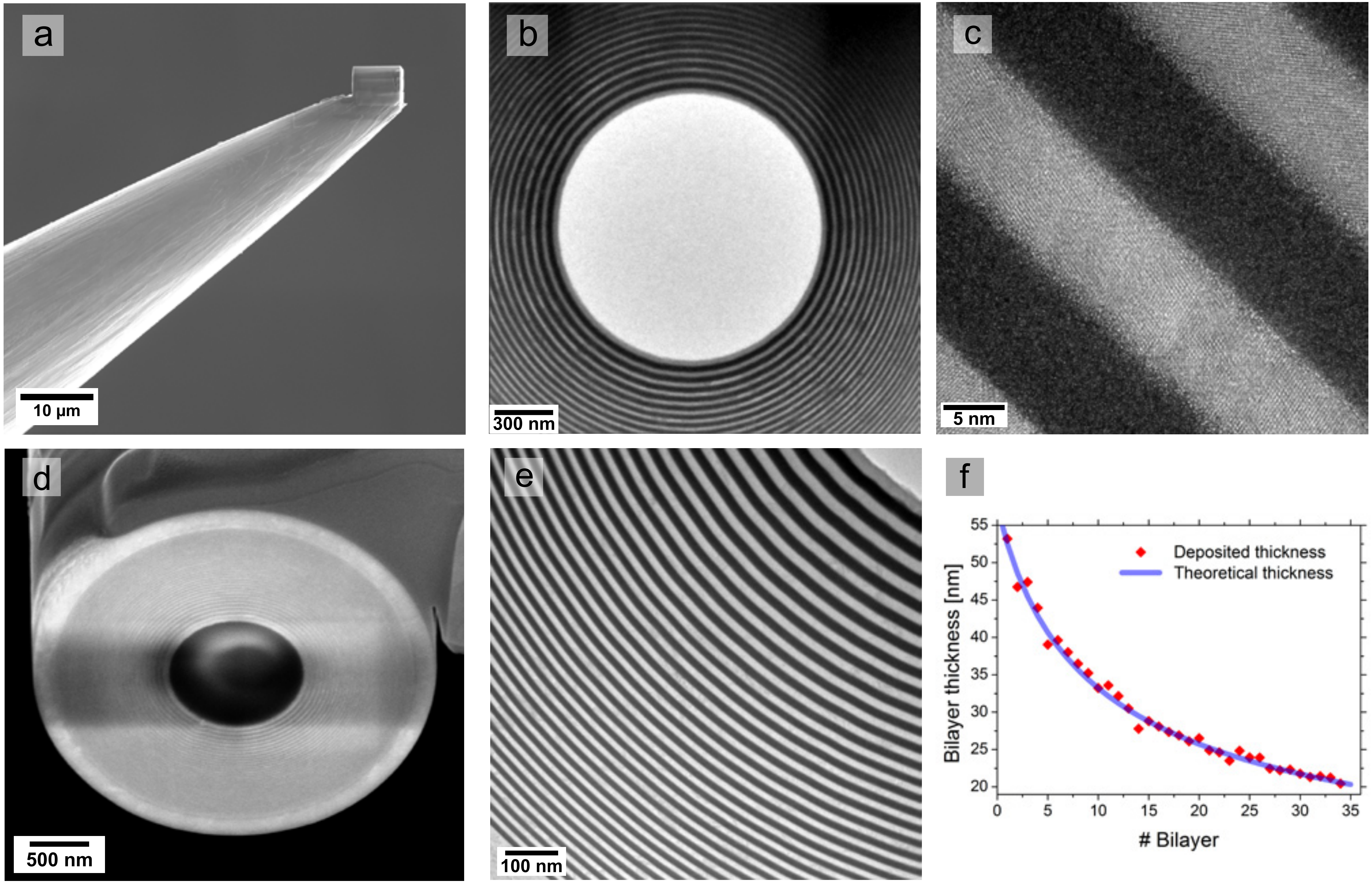
5 nm focusing @ 7.9 keV, 13.8 keV
10 nm @ 18.0 keV

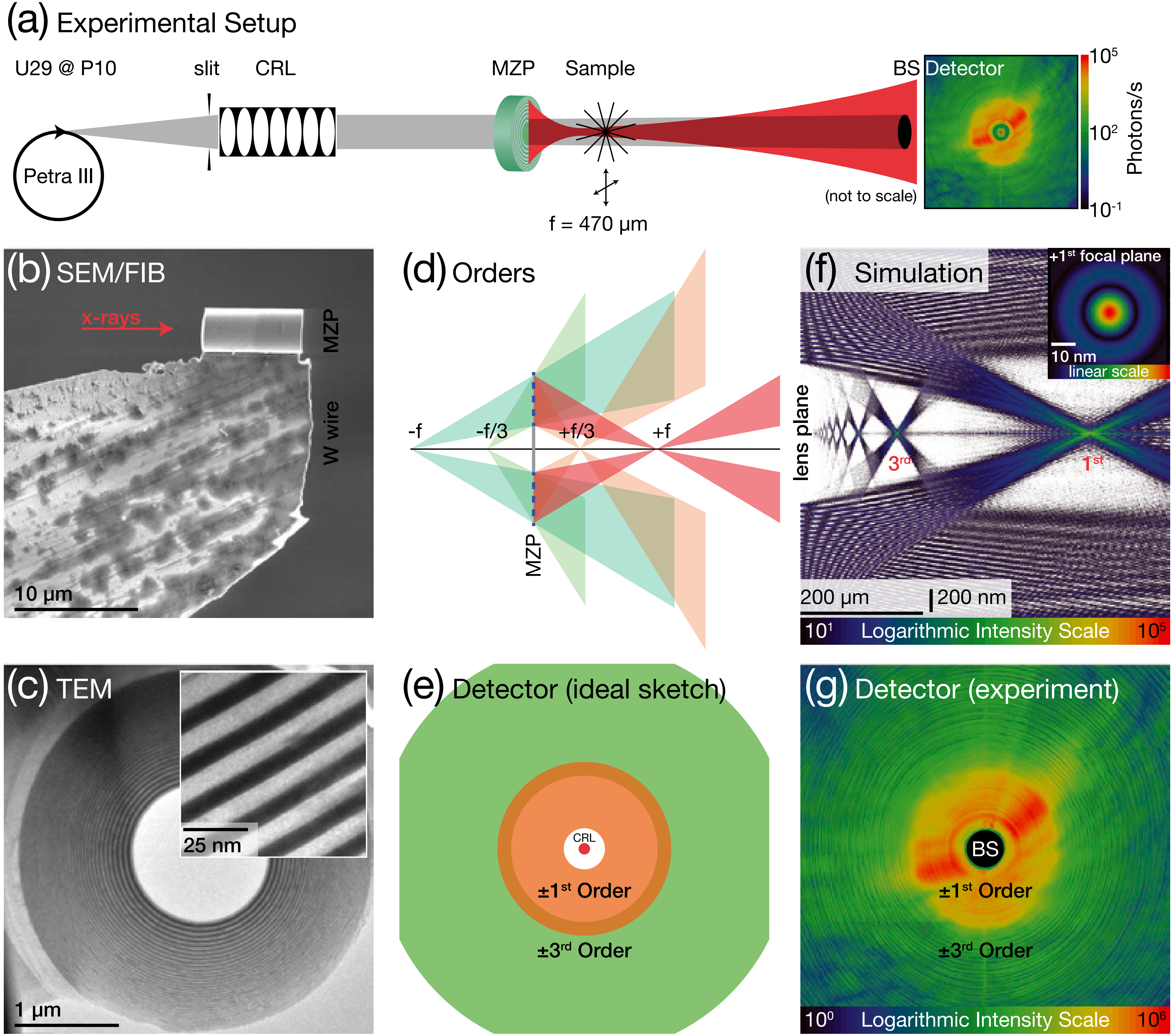
only indirect characterisation via phase-retrieval

inconsistent ptychography along optical axis

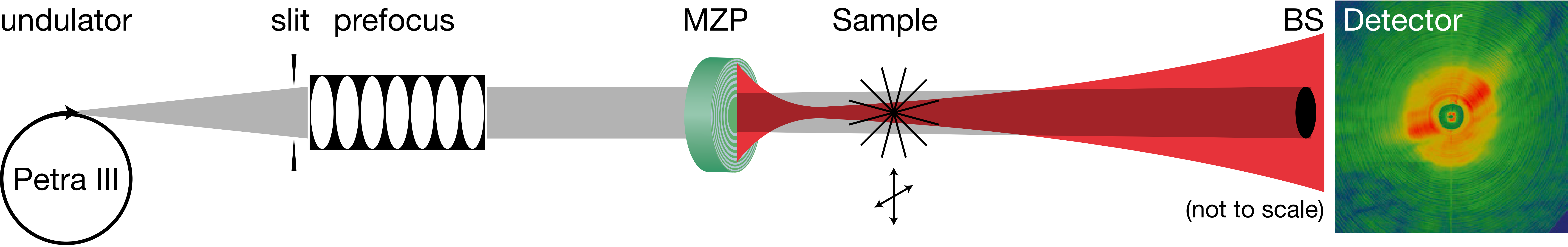


The new system: Ta_2O_5 / ZrO_2





(a) Experimental Setup
U29 @ P10
slit
CRL
MZP
Sample
BS
Detector
Petra III
f = 470 µm
(not to scale)
10^5
10^2
10^-1
Photons/s
(b) SEM/FIB
x-rays
MZP
W wire
10 µm
(c) TEM
25 nm
1 µm
(d) Orders
-f
-f/3
+f/3
+f
MZP
(e) Detector (ideal sketch)
CRL
±1st Order
±3rd Order
(f) Simulation
+1st focal plane
10 nm
linear scale
lens plane
3rd
1st
200 µm
200 nm
10^1 Logarithmic Intensity Scale 10^5
(g) Detector (experiment)
BS
±1st Order
±3rd Order
10^0 Logarithmic Intensity Scale 10^6



Experiments carried out at GINIX setup, P10 beamline, PETRA III (DESY, Hamburg, Germany)

E = 18.0 keV, no OSA

prefocusing with CRLs

MZP focal length: 470 µm

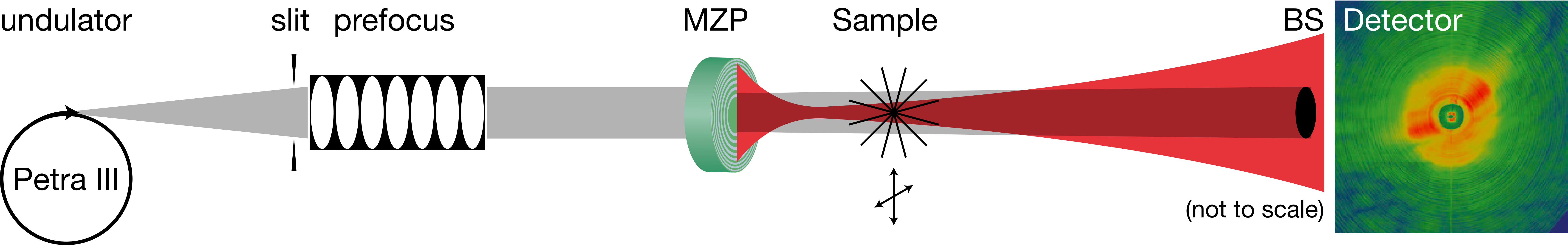
detector distance: ~ 5.1 metre (Pilatus 300k)

Samples:

W line aperture,

semiconductor nanowires,

Siemens Star (50 nm)



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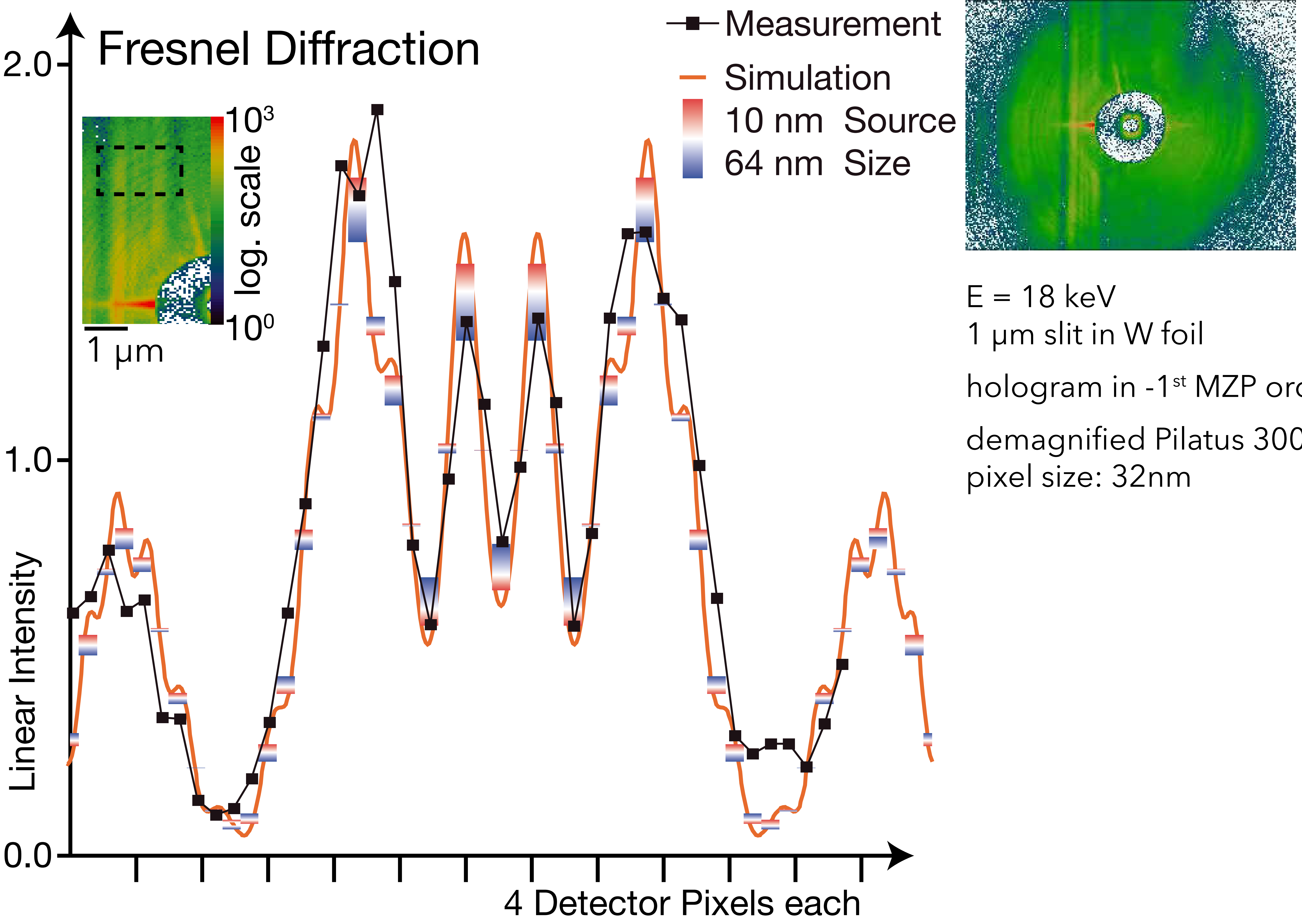
Imaging modes:

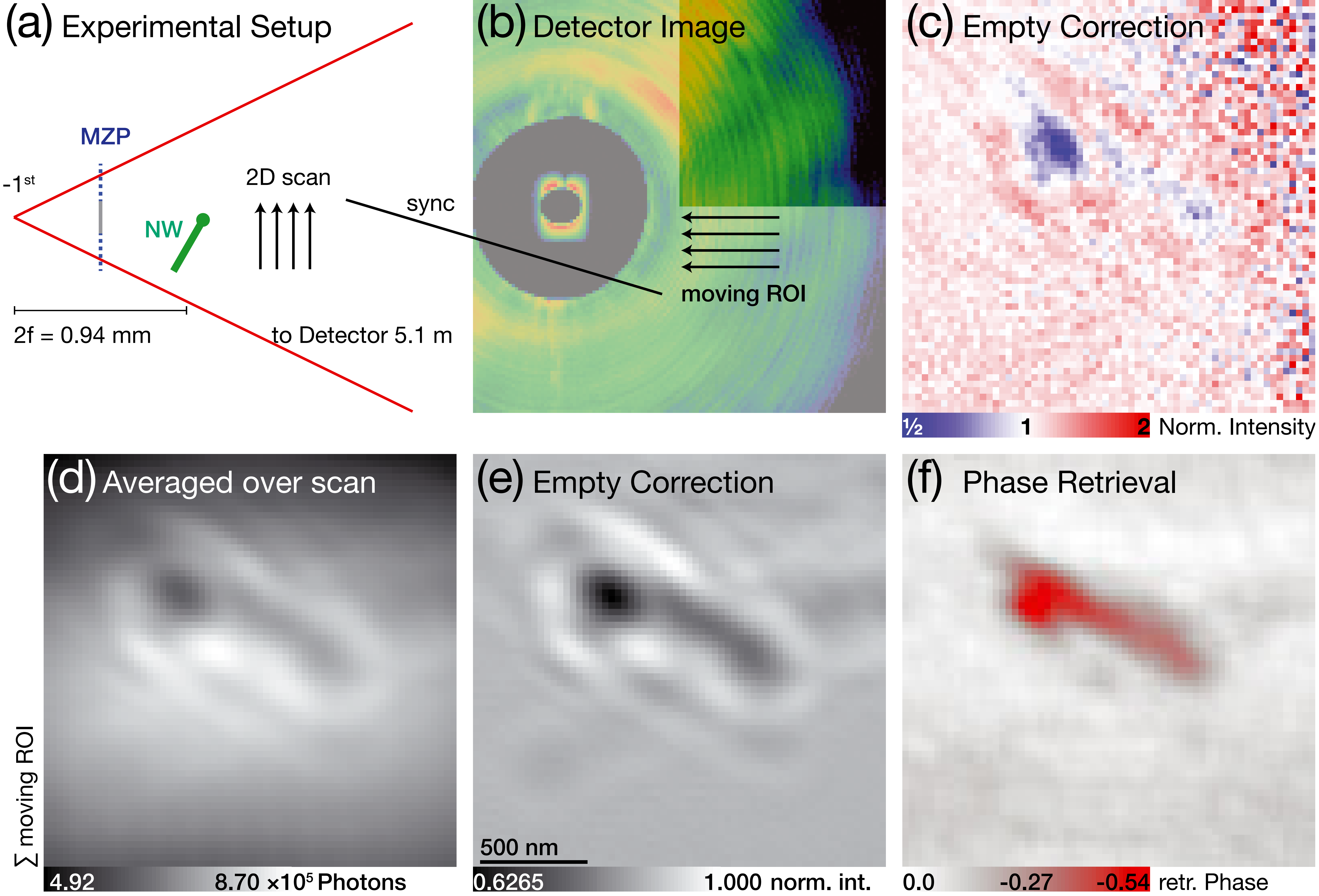
holography,

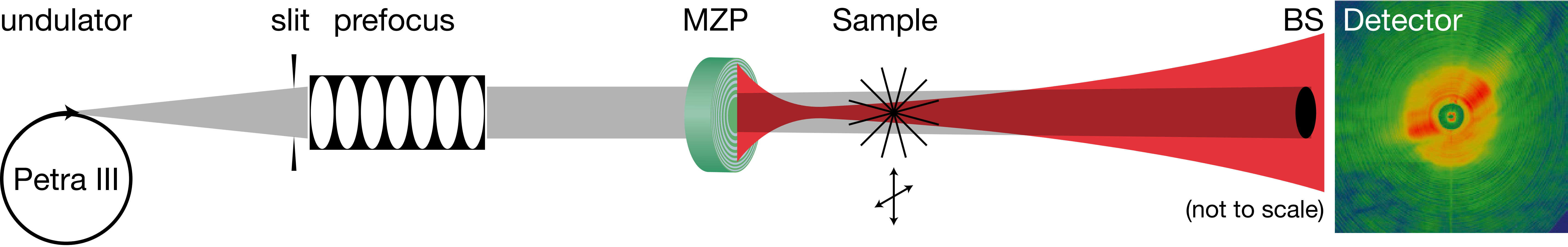
STXM,

STXM with holographic -1st order



E = 18 keV
1 µm slit in W foil
hologram in -1st MZP order
demagnified Pilatus 300k
pixel size: 32nm


(a) Experimental Setup
MZP
-1st
NW
2D scan
sync
2f = 0.94 mm
to Detector 5.1 m
(b) Detector Image
moving ROI
(c) Empty Correction
½
1
2
Norm. Intensity
(d) Averaged over scan
∑ moving ROI
4.92
8.70 ×10⁵ Photons
(e) Empty Correction
500 nm
0.6265
1.000 norm. int.
(f) Phase Retrieval
0.0
-0.27
-0.54
retr. Phase



Imaging modes:

holography,

STXM,

STXM with holographic -1st order

Problem:
for simple STXM analyis,
e.g. differential phase contrast,
resolution is limited by beam size;

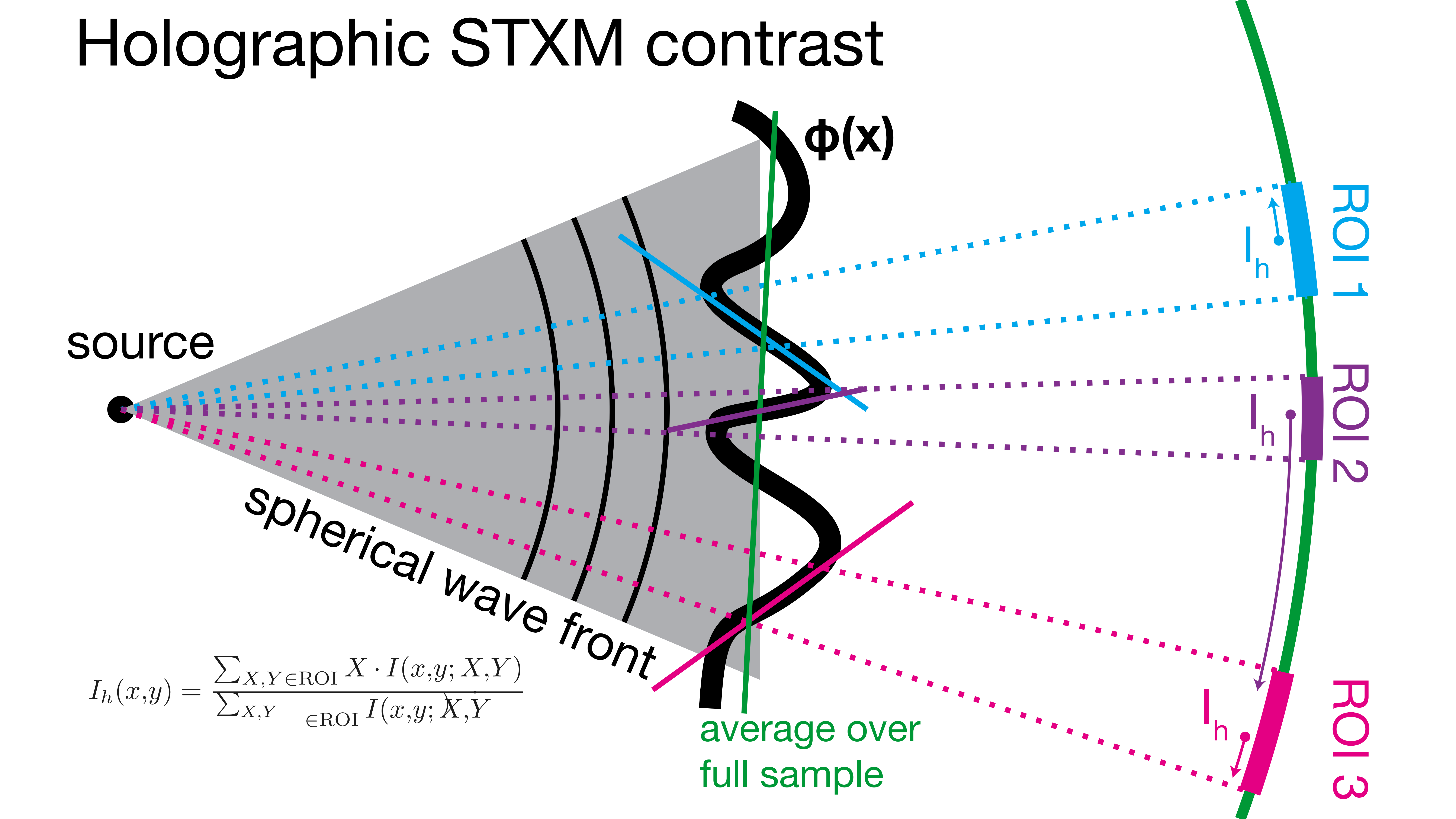
here: beam size of -1st order is
roughly 6 µm in +1st focal plane

X,Y: detector pixel coordinates

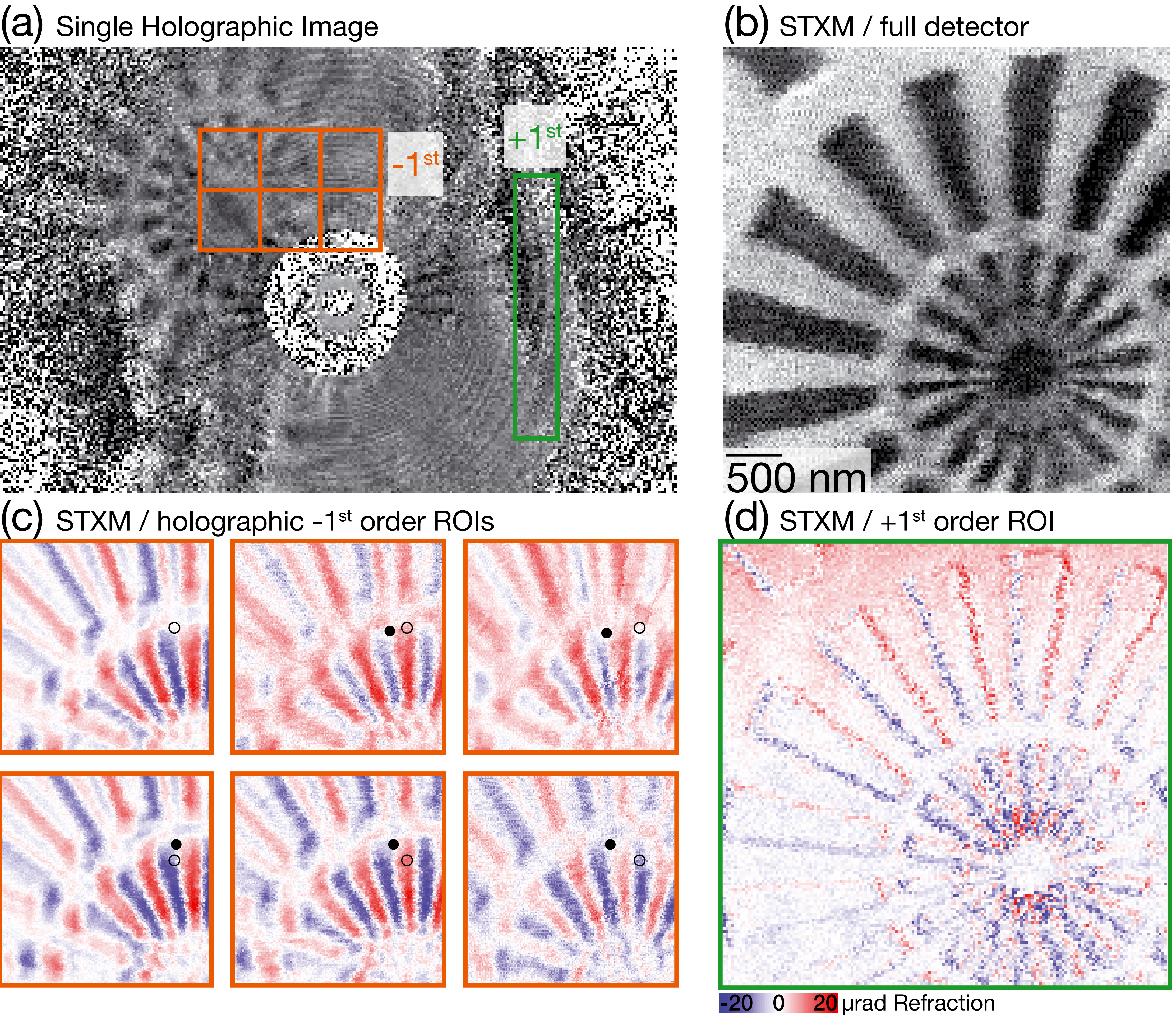
x,y: scan positions

Ih: centre-of-mass,
horizontal direction
~ differential phase contrast

$$I_h(x,y) = \frac{\sum_{X,Y \in \mathrm{ROI}} X \cdot I(x,y;X,Y)}{\sum_{X,Y \in \mathrm{ROI}} I(x,y;X,Y)}$$



Holographic STXM contrast
ϕ(x)
source
spherical wave front
average over
full sample
ROI 1
ROI 2
ROI 3
I_h
I_h
I_h
$I_h(x,y) = \frac{\sum_{X,Y\in\mathrm{ROI}} X \cdot I(x,y;X,Y)}{\sum_{X,Y\in\mathrm{ROI}} I(x,y;X,Y)}$



STXM parameters:

161 × 161 images, lines in fly-mode (continuous scan)

scan area: 4 µm × 4 µm

illumination time / point: 10 ms

beam sizes:

10 nm in +1st order (but sample not in focal plane)

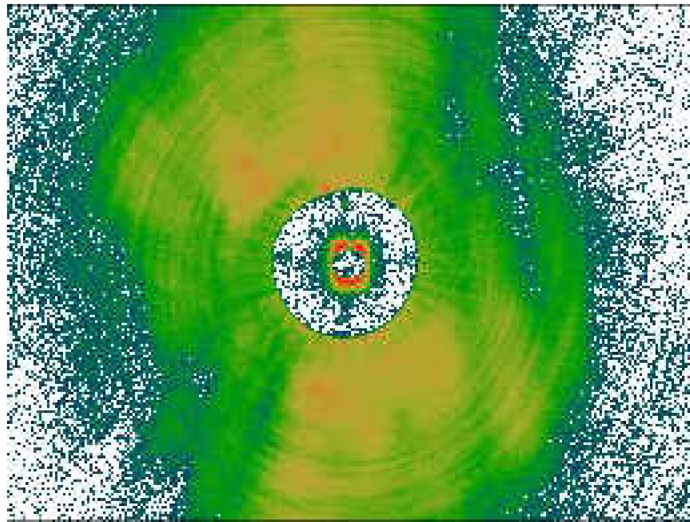
6 µm in -1st order

3 µm in 0th order

"step" size: 25 nm

finest details: 50 nm

but vibrations: ~ 50 nm

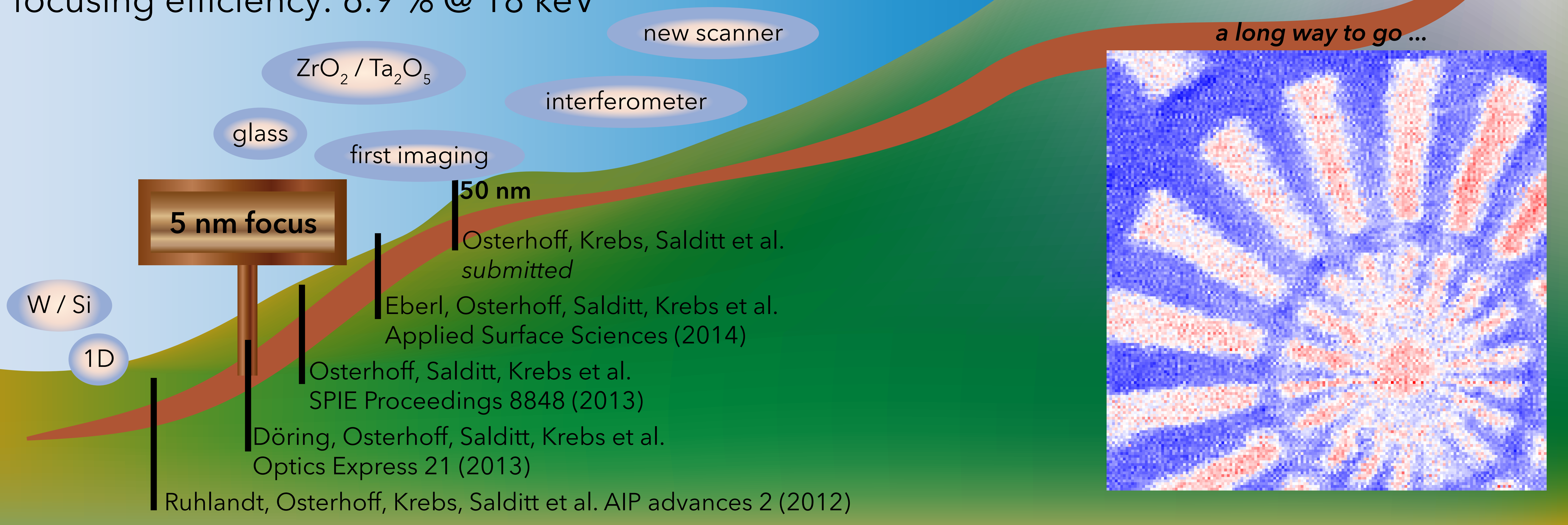


Summary: Imaging

- quantitative Fresnel edge diffraction
- holographic imaging of nanowire
- holographic STXM of Siemens star

resolution so far: 50 nm
currently limited by vibrations

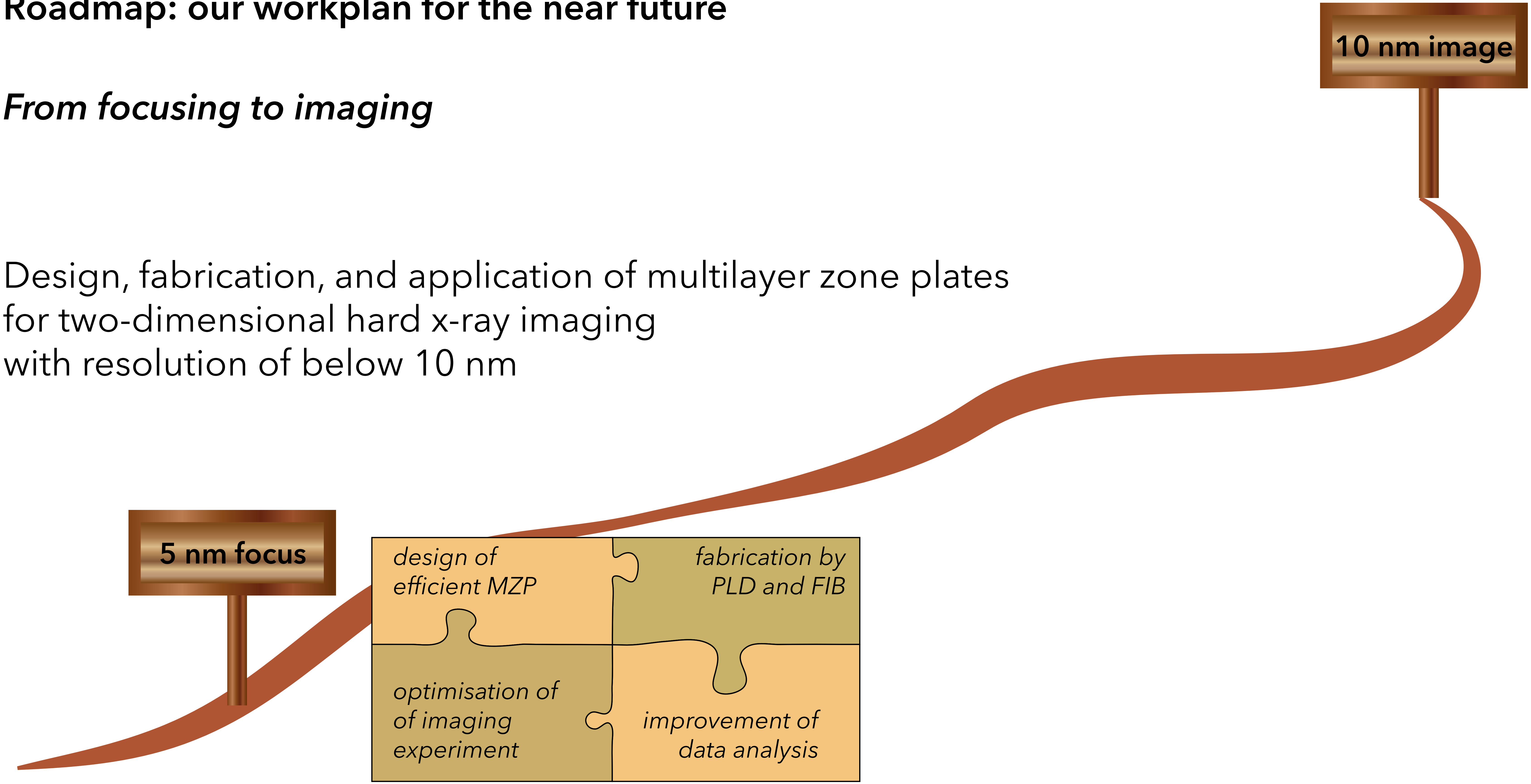
focusing efficiency: 6.9 % @ 18 keV



Roadmap: our workplan for the near future

From focusing to imaging

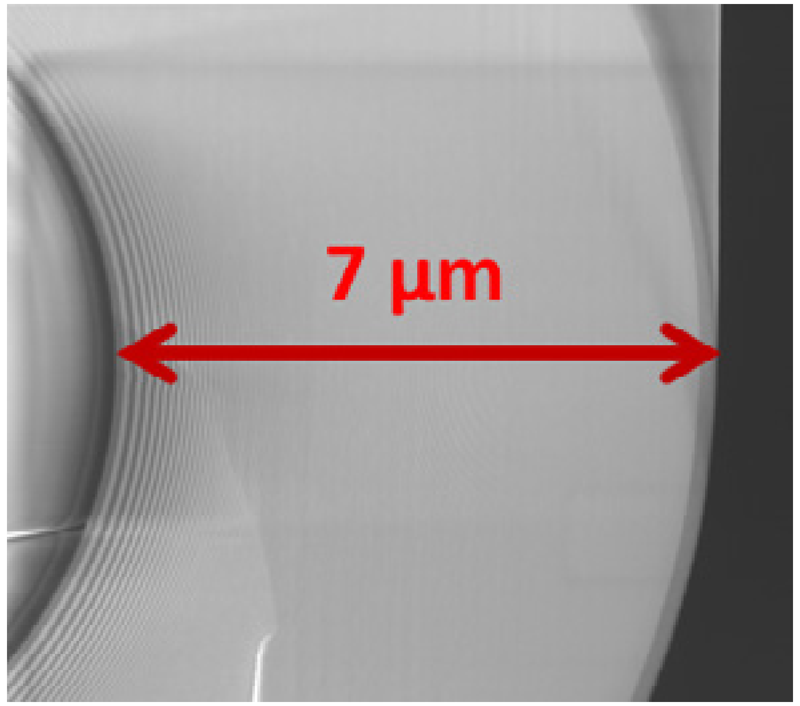
Design, fabrication, and application of multilayer zone plates
for two-dimensional hard x-ray imaging
with resolution of below 10 nm



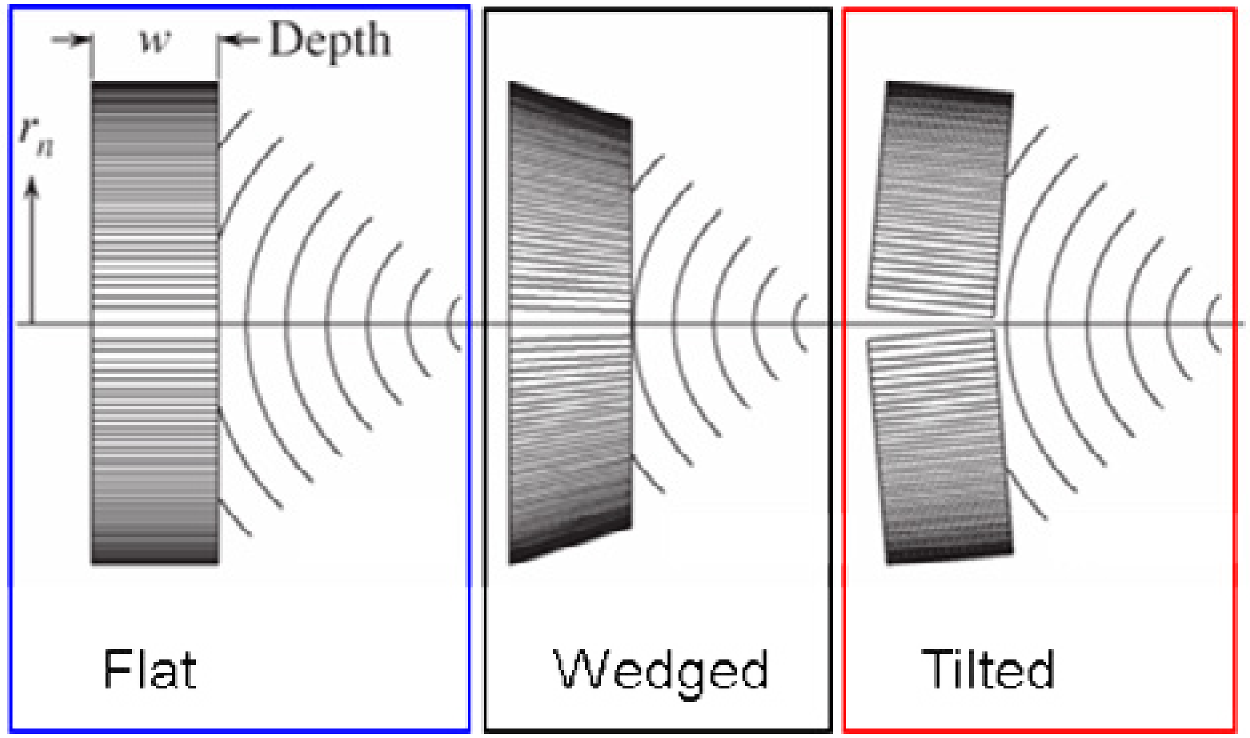
Roadmap: our workplan for the near future

Lens design and fabrication

efficiency by thick ML

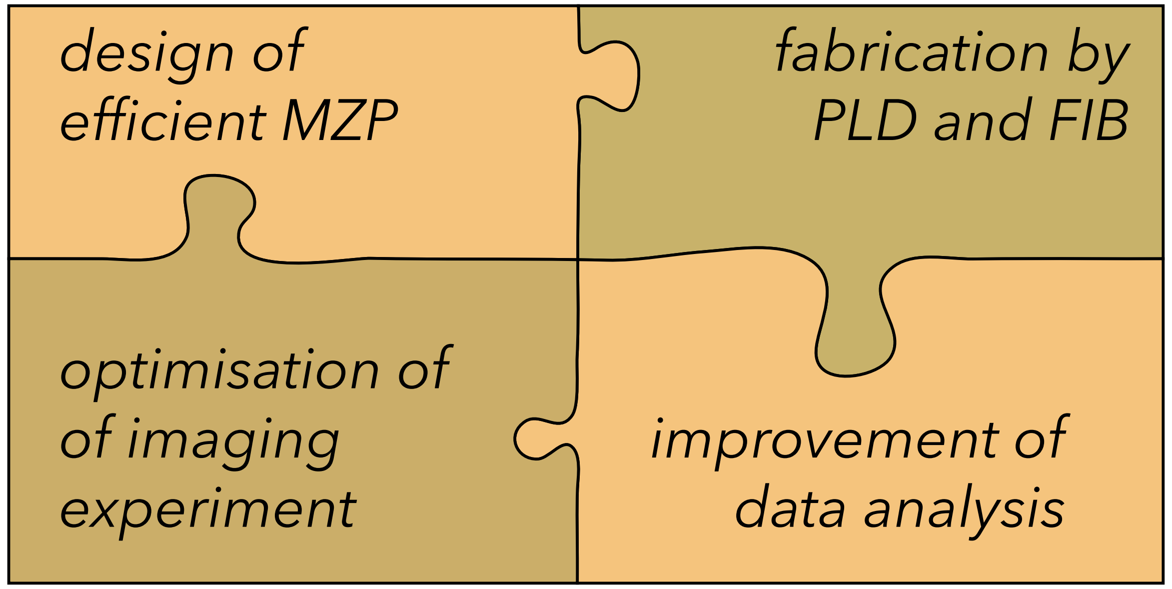


wedged / curved / tilted structures



(Kang, Maser 2006)

calculation of
optimal design

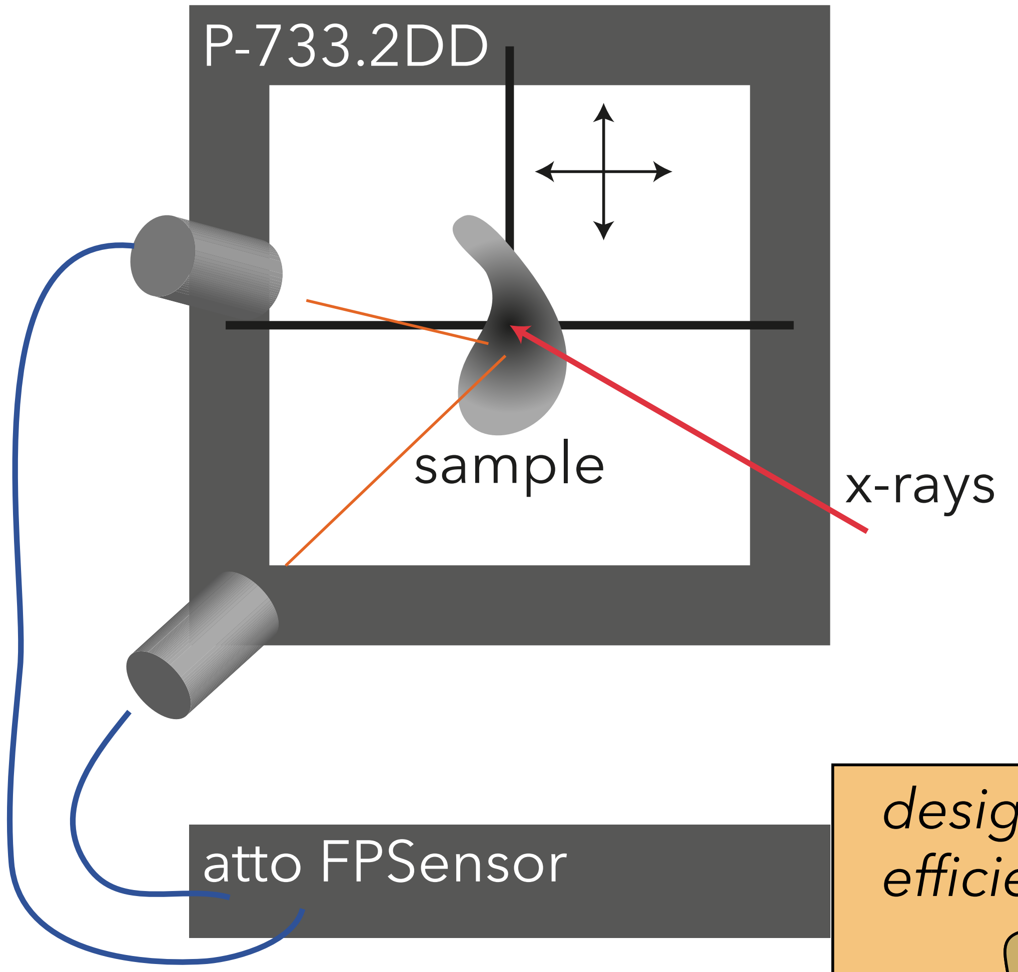


how to PLD
such a shape?

Roadmap: our workplan for the near future

Experiment and data analysis

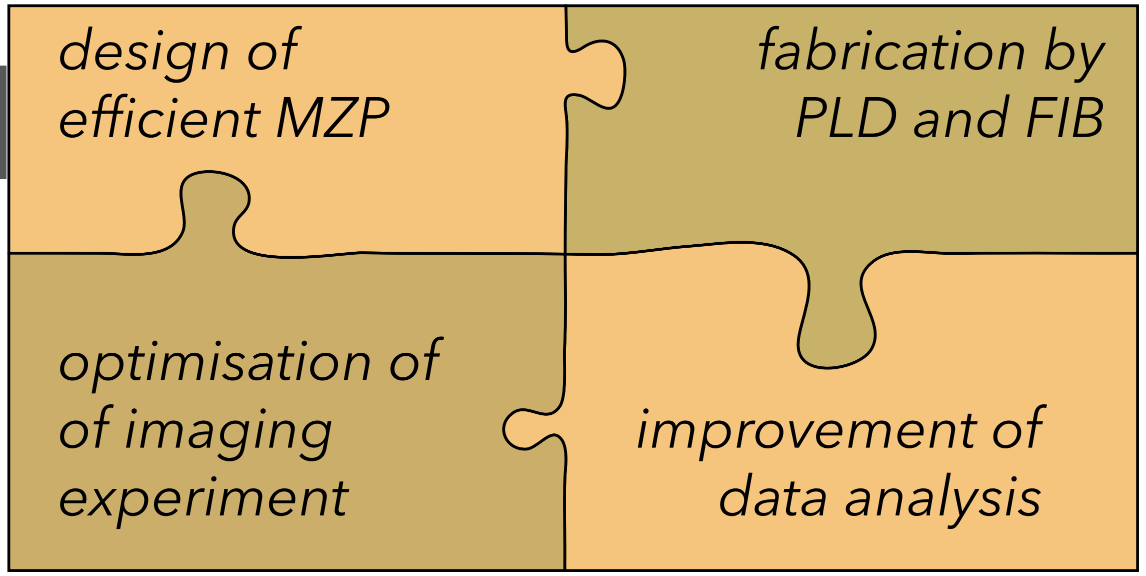
new scanner, interferometer



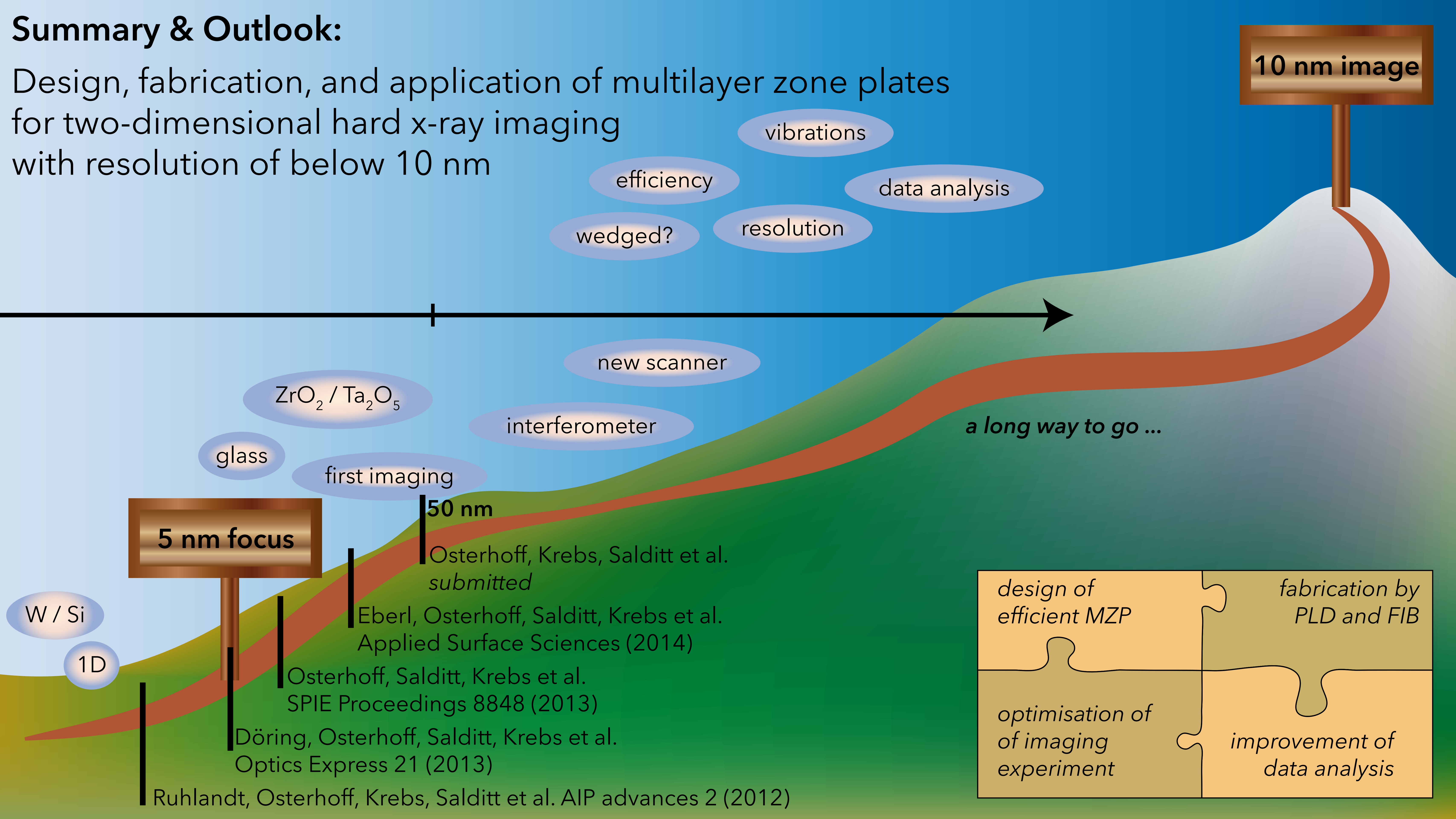
topics to be addressed:

+ include positions in STXM,
+ improved holographic reconstruction,
+ automatic analysis for scans,
+ ptychography

vibrations, drift;
fast scanning;
order sorting aperture



non uniform scanning,
interferometric control



Summary & Outlook:
Design, fabrication, and application of multilayer zone plates
for two-dimensional hard x-ray imaging
with resolution of below 10 nm
10 nm image
vibrations
efficiency
data analysis
wedged?
resolution
new scanner
ZrO_2 / Ta_2O_5
interferometer
a long way to go ...
glass
first imaging
50 nm
5 nm focus
Osterhoff, Krebs, Salditt et al.
submitted
Eberl, Osterhoff, Salditt, Krebs et al.
Applied Surface Sciences (2014)
W / Si
1D
Osterhoff, Salditt, Krebs et al.
SPIE Proceedings 8848 (2013)
Döring, Osterhoff, Salditt, Krebs et al.
Optics Express 21 (2013)
Ruhlandt, Osterhoff, Krebs, Salditt et al. AIP advances 2 (2012)
design of
efficient MZP
fabrication by
PLD and FIB
optimisation of
of imaging
experiment
improvement of
data analysis

(Towards) Multi-Order Hard X-Ray Imaging with Multilayer Zone Plates

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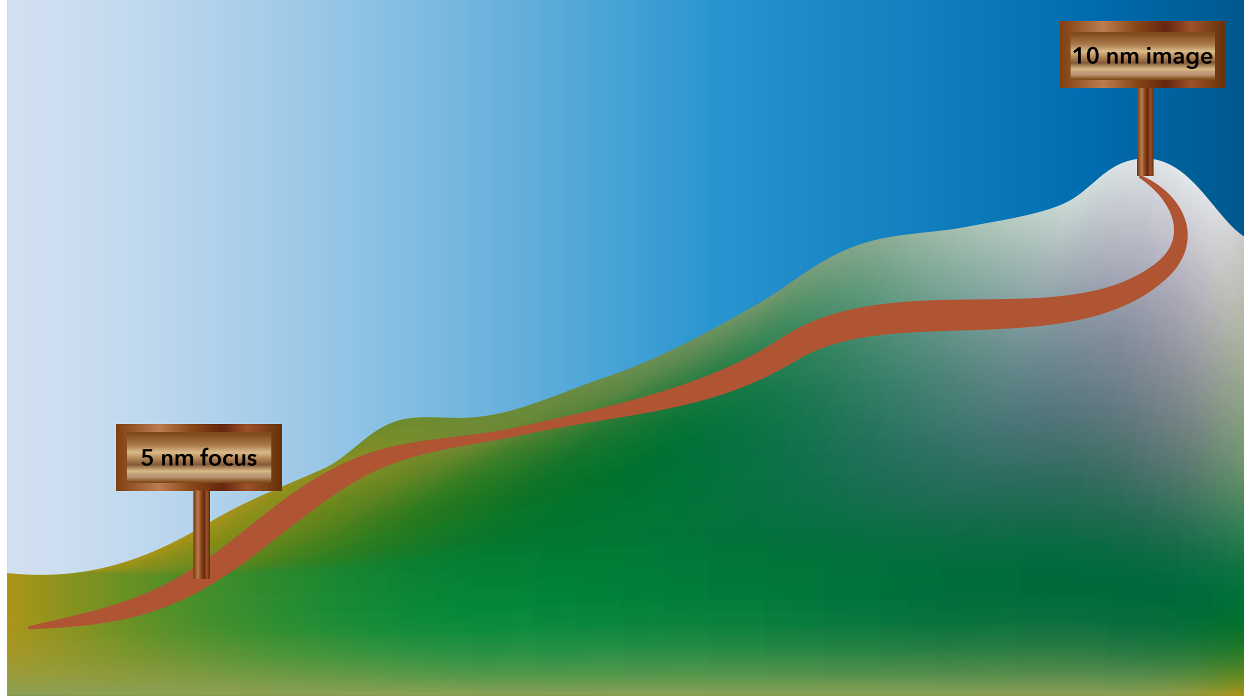

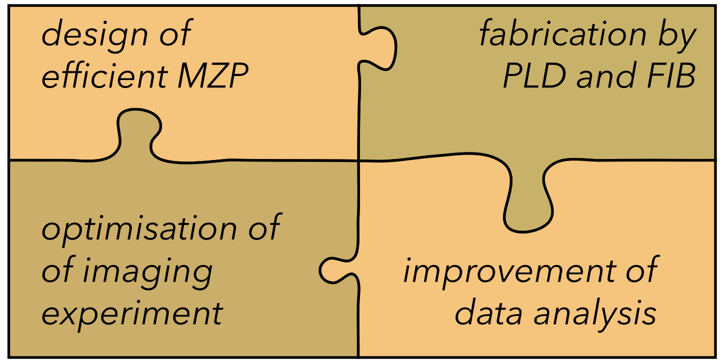

Poster on MZP fabrication:

Poster #4 on Tuesday's session
C. Eberl *et al*