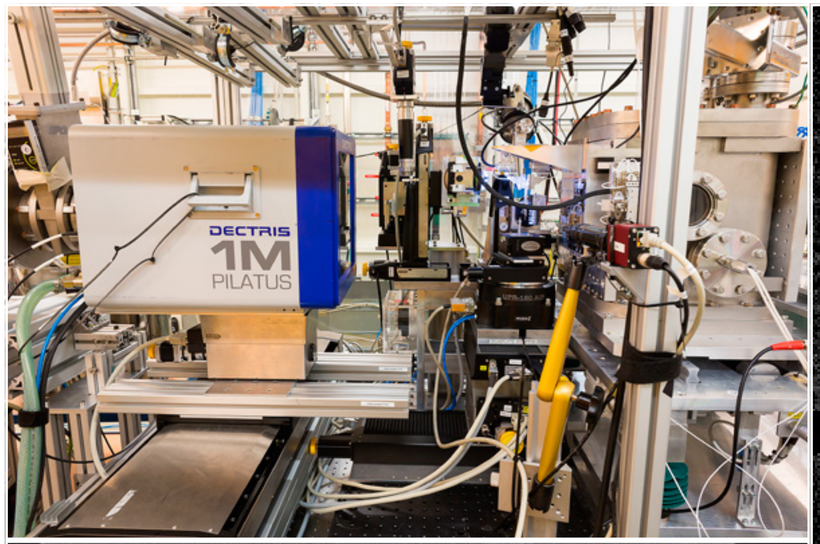
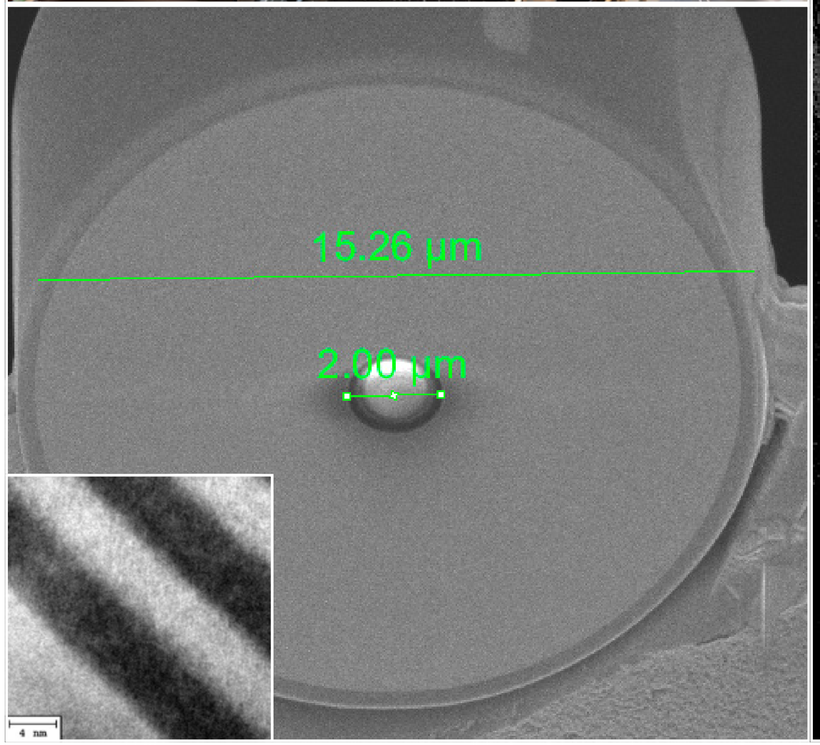
Hard X-Ray MZP-Imaging at GINIX

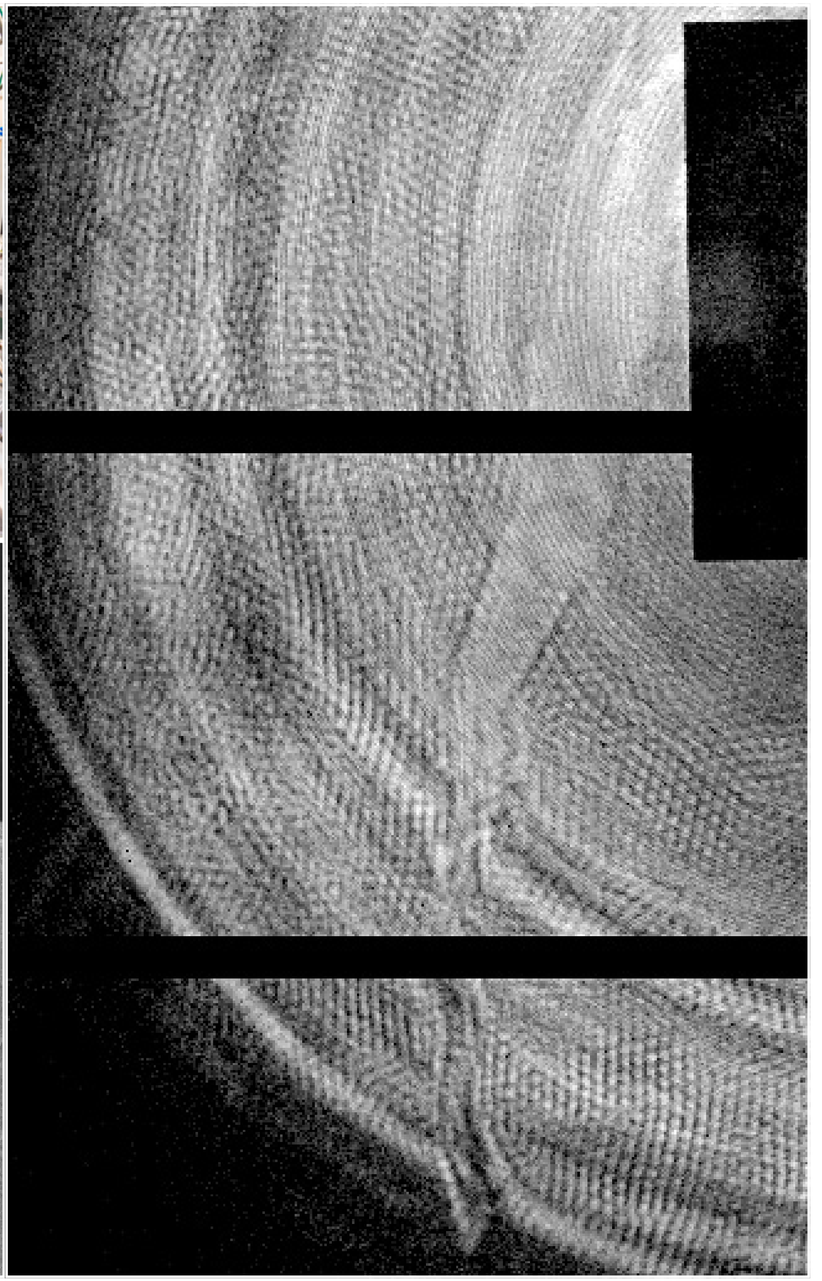
Markus Osterhoff
IRP / SFB 755

1. **GINIX**
2. **MZP Focusing**
3. **MZP Instrumentation**
4. **MZP Imaging**

Markus Osterhoff
Christian Eberl
Jakob Soltau
Hans-Ulrich Krebs







X-Ray Microscopy:
Past, Present, and Future

Symposium on the occasion of
Günter Schmahl's 80th Birthday

Göttingen, 21st & 22nd October, 2016

GINIX

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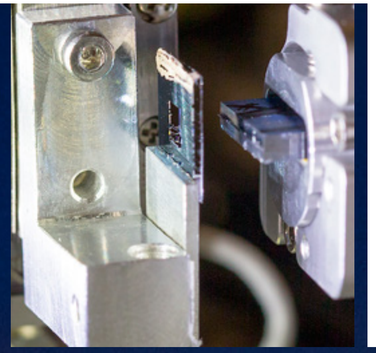
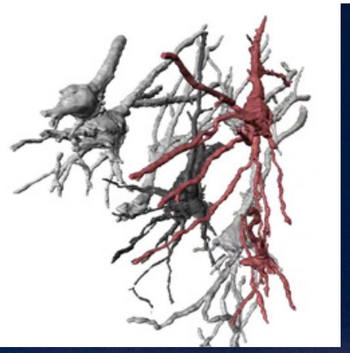
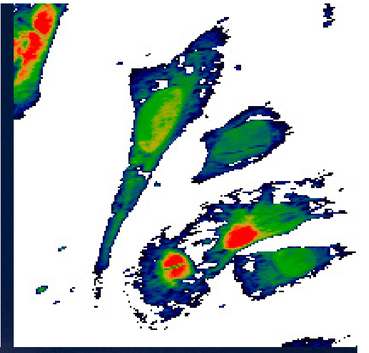
Göttingen Instrument for Nano-Imaging with X-rays

Flexible set-up: waveguide-based holo-tomography, scanning S/WAXS, fluorescence, and microfluidics

operated by AG Salditt, at P10 beamline (PETRA III, DESY)







GINIX: Overview

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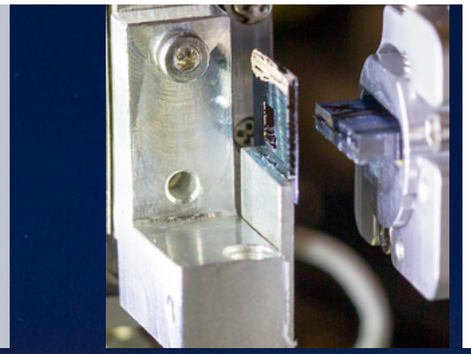

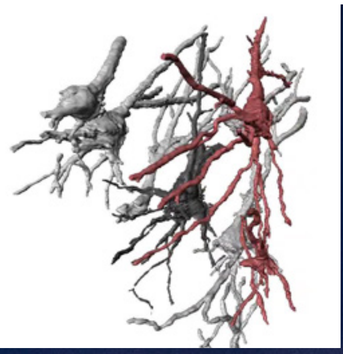

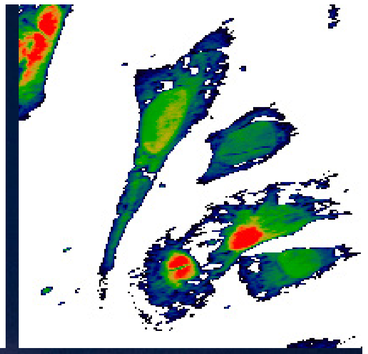

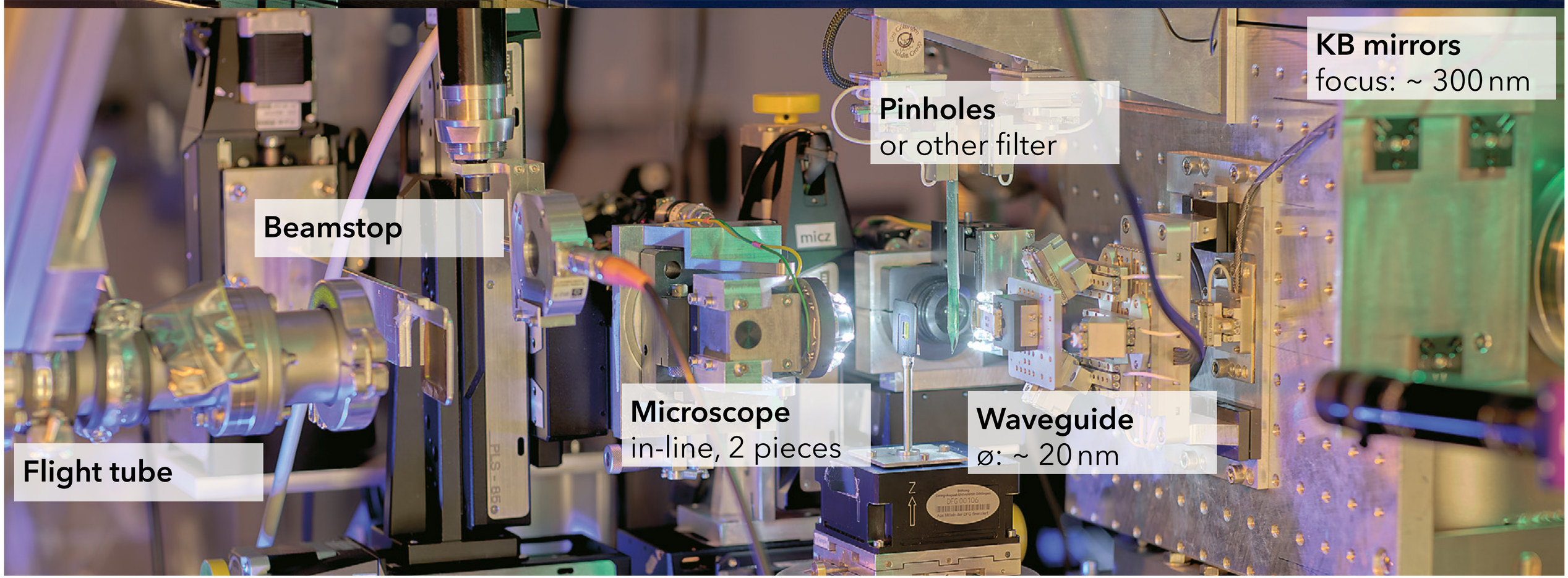

GINIX: Waveguide-based Imaging

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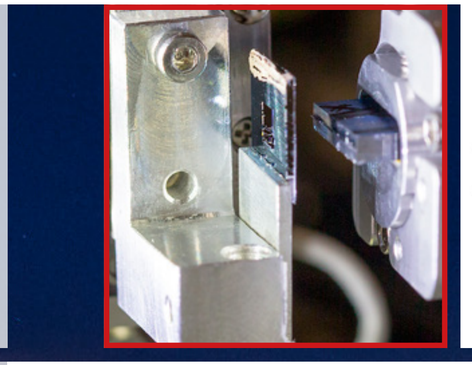
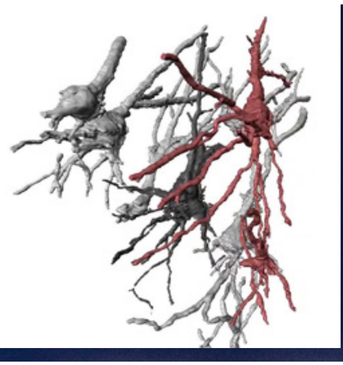
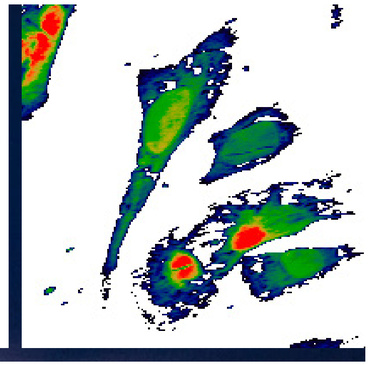






Ultra-small pinholes as coherence filter

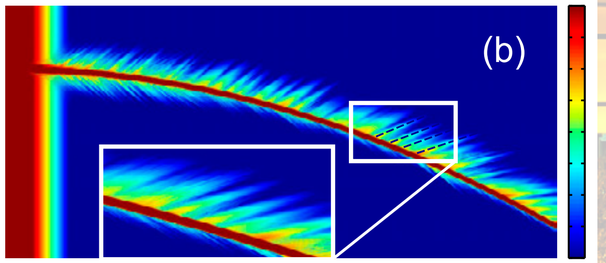
guiding channel (10…100 nm) in silicon wafer

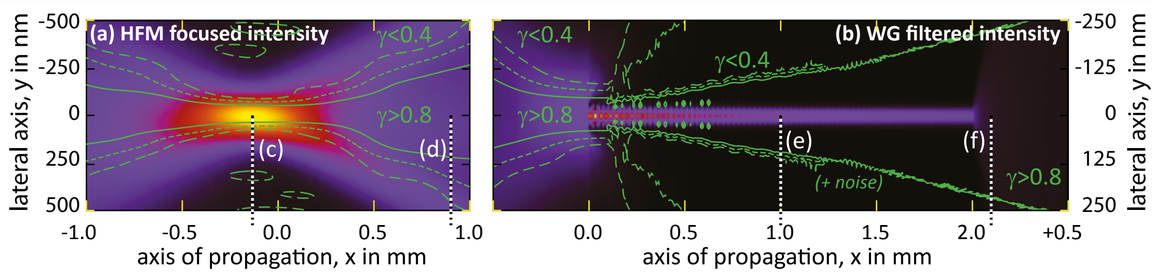
length: sub- to several mm

coherence filtering; cleanup of KB beam

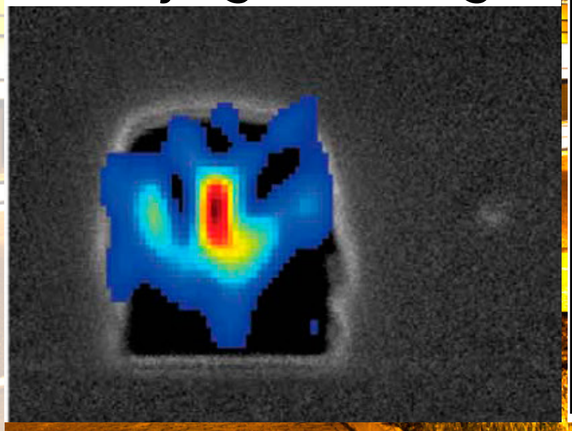
quasi-point source for imaging

advanced: beam-splitter; x-ray bender

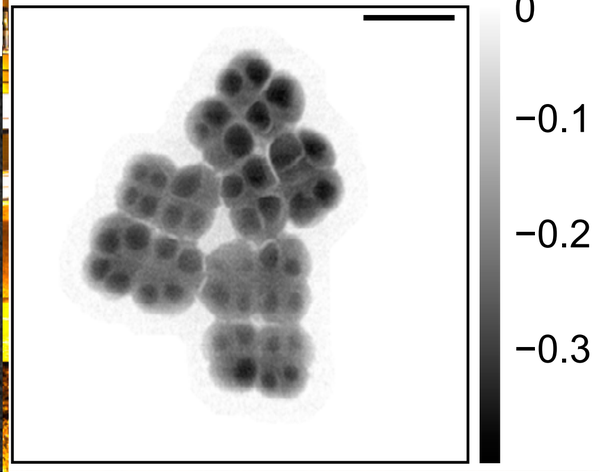




reconstructed wavefield overlaying SEM image



Resolution: 53 nm



T. Salditt, Physical Review Letters, 2015
M. Osterhoff, New Journal of Physics, 2011
S. Hoffmann, Acta Crystallographica A, 2016
M. Bartels, Physical Review Letters, 2015

GINIX: Holographic Tomography

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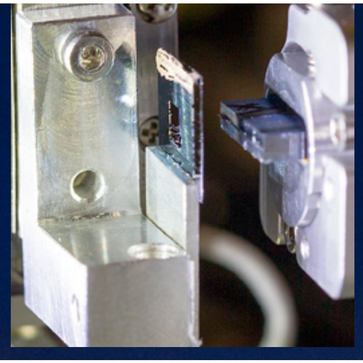
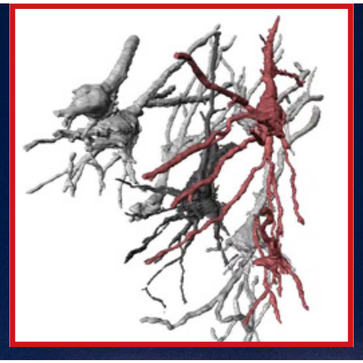
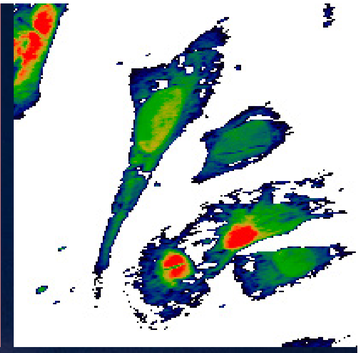
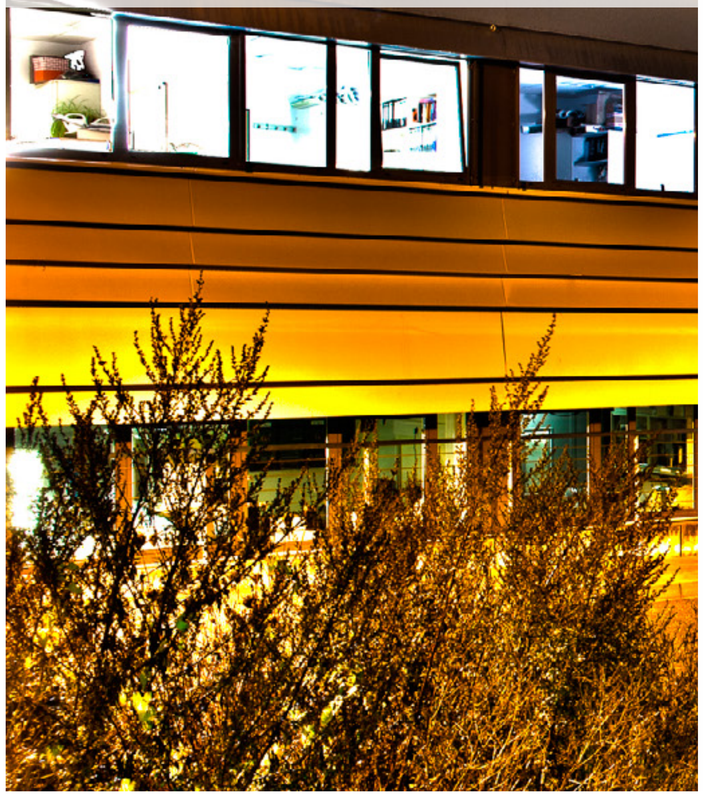
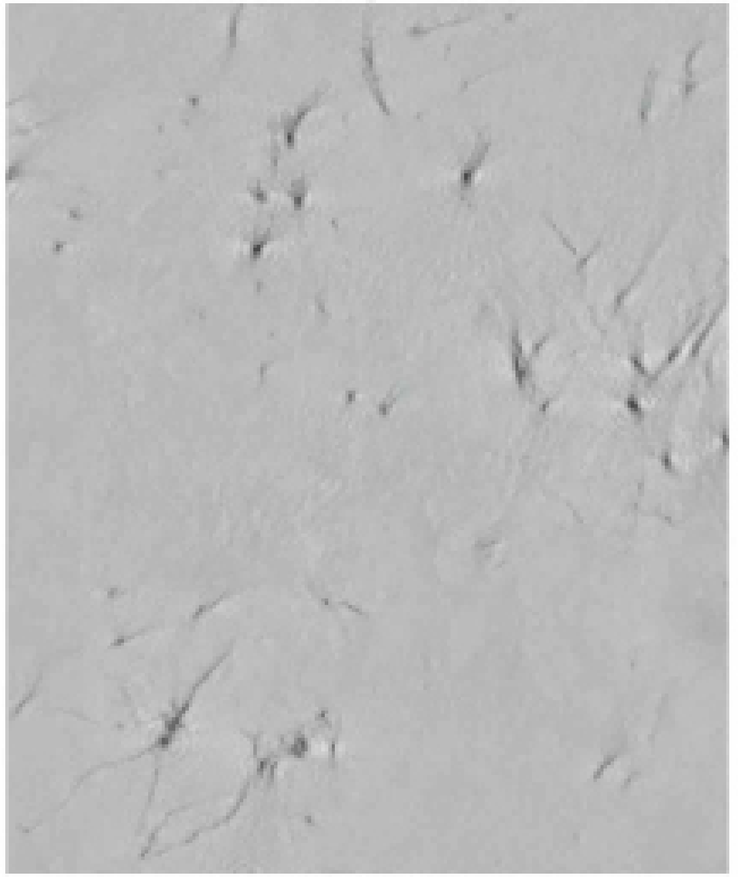


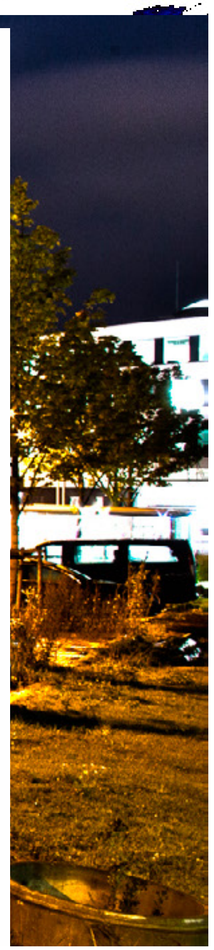




Silver-stained neurons: determine the connectivity







GINIX: Scanning SAXS / WAXS

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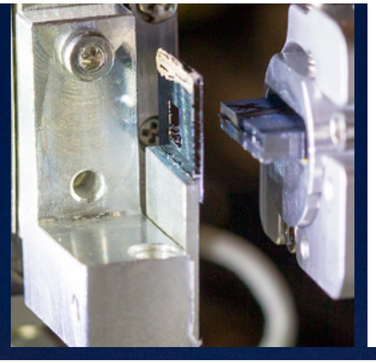
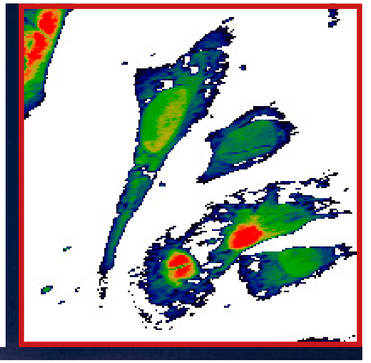


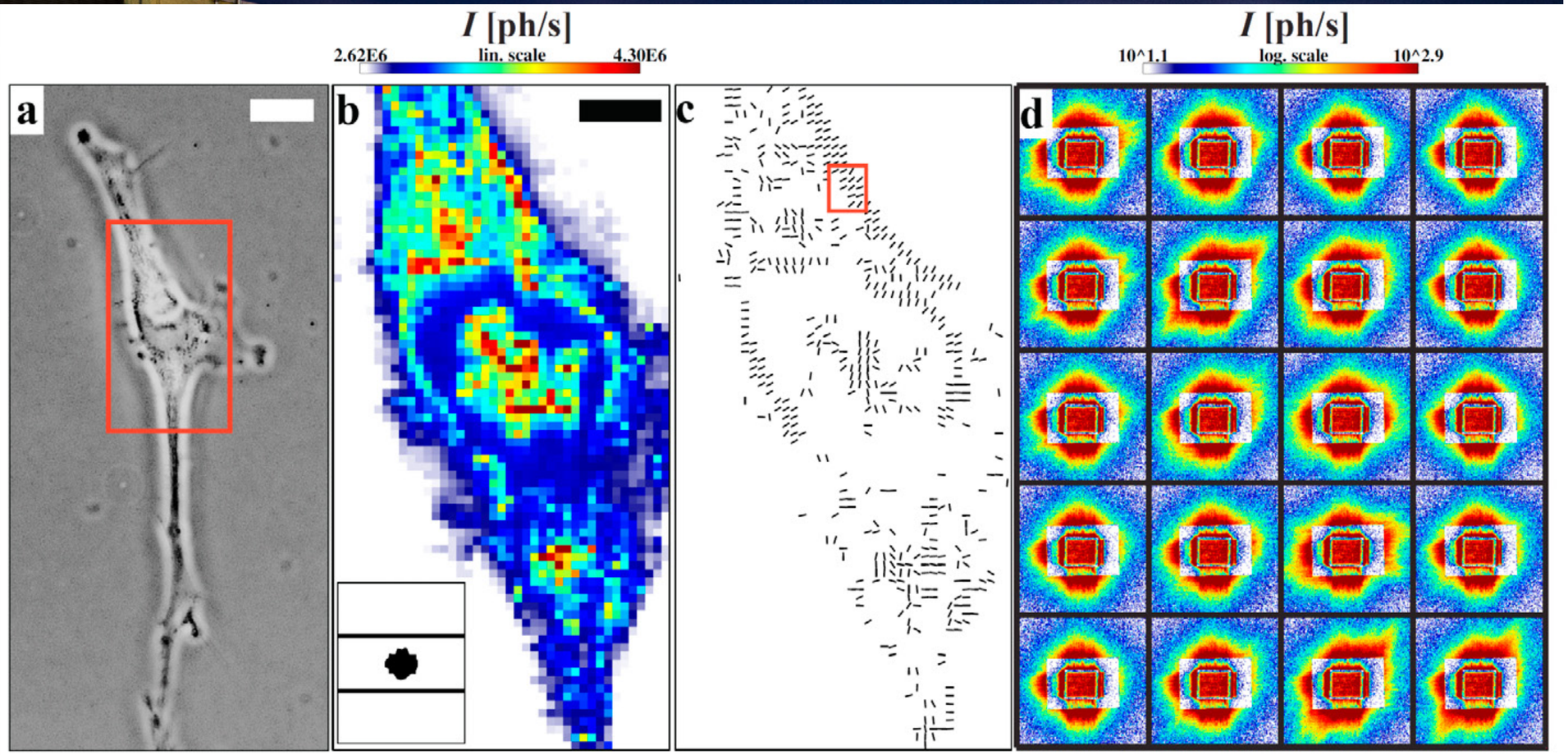


Reciprocal information with spatial resolution

human Mesenchymal Stem Cells

comparing:
visible 40×,
X-ray darkfield,
orientations,
composite

reciprocal ~ 0.05 nm^{-1},
real-space ~ 200 nm



MZP focusing

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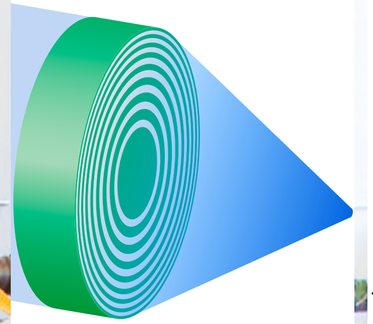
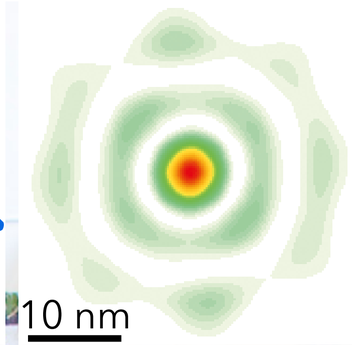
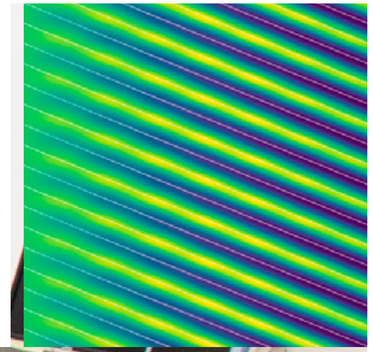
Multilayer Zone Plates fabricated by Pulsed Laser Deposition

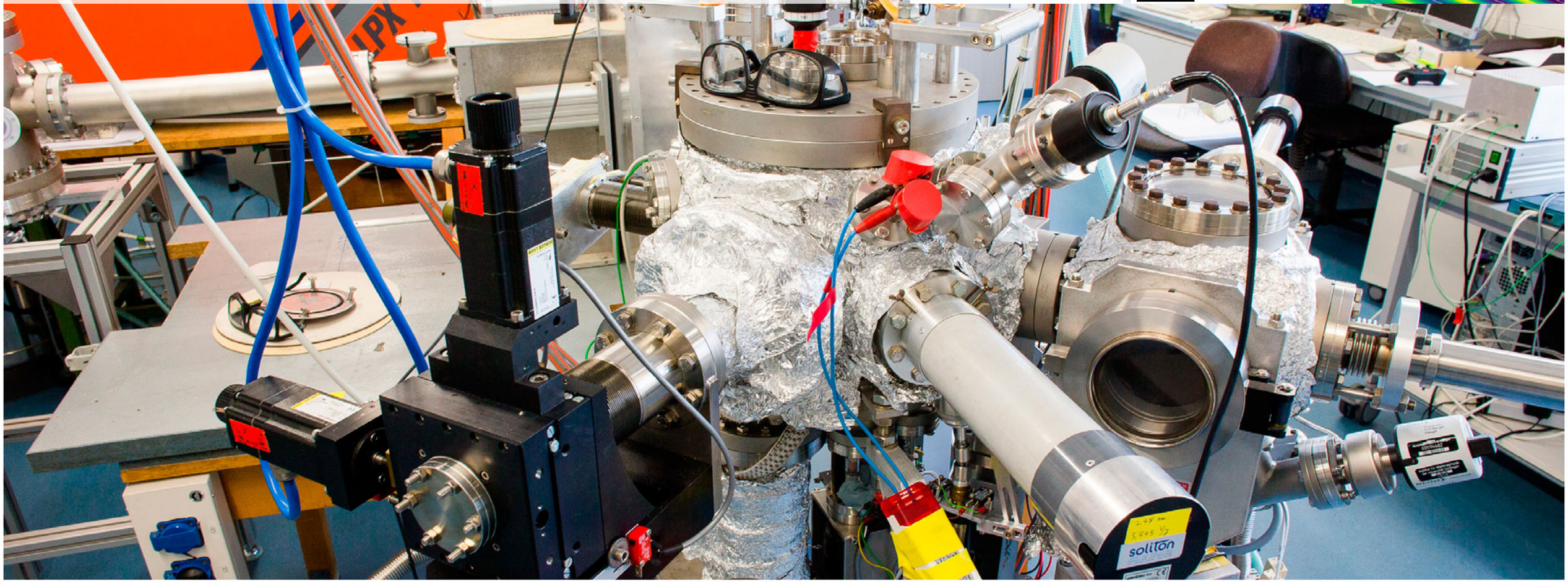
Basically: thick Fresnel Zone Plates.

So: Thank you for all your pioneering groundwork – and thanks for new technology.









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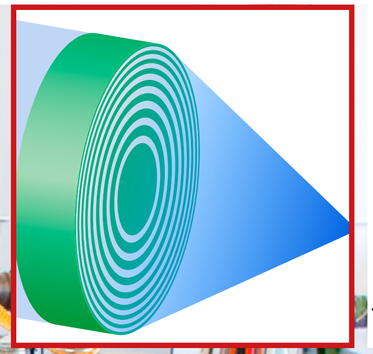

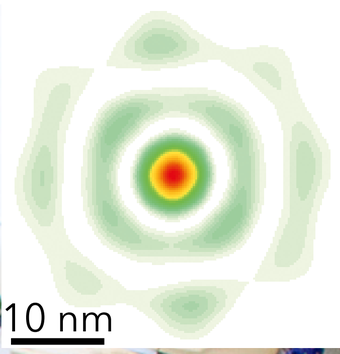

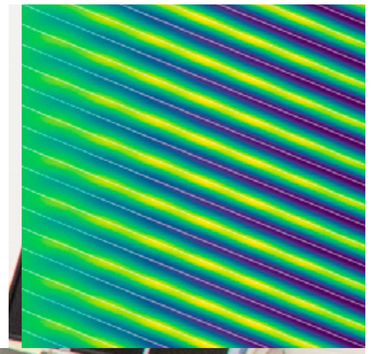

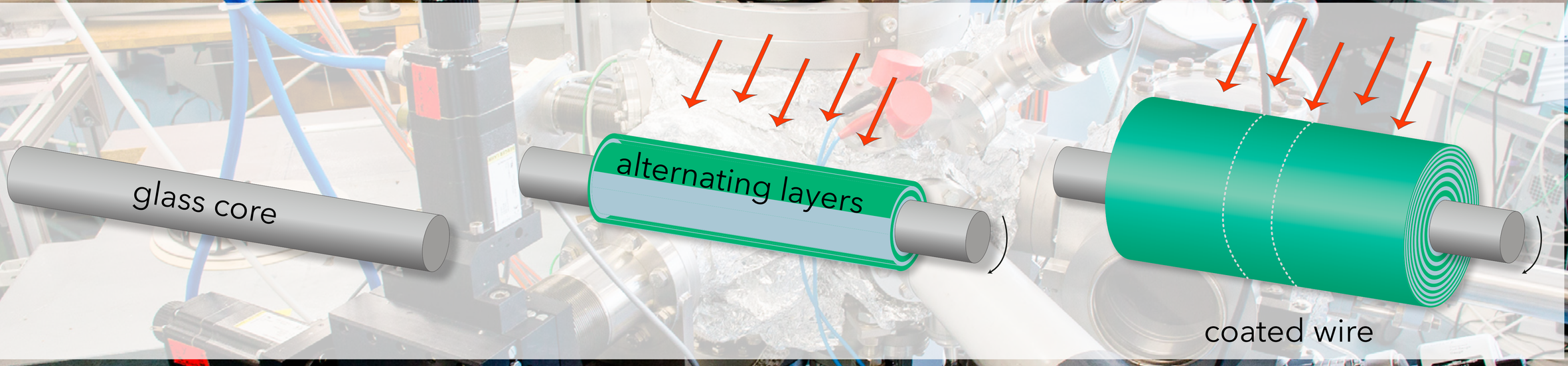

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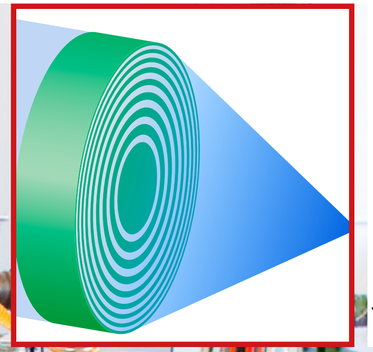
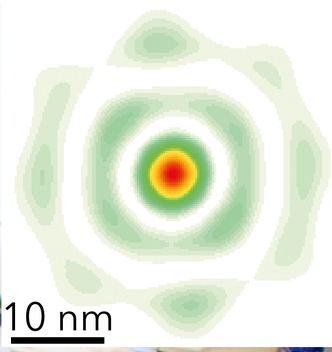
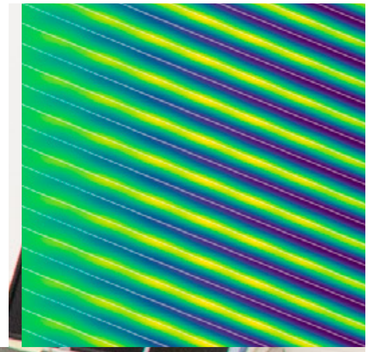






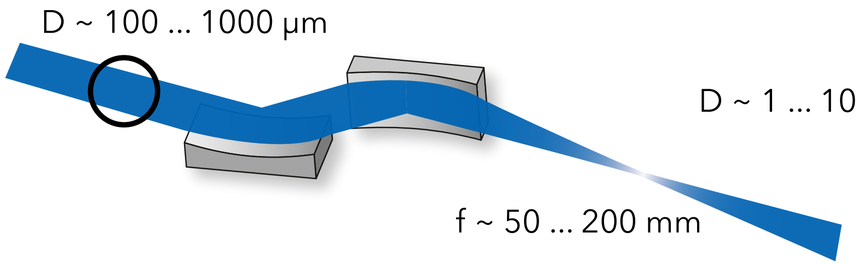
Combined Optics – Motivation

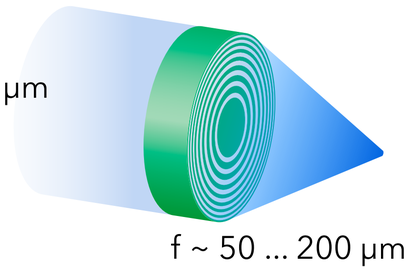
Kirkpatrick-Baez mirrors:

large absolute aperture,
long focal distance

Multilayer Zone Plates:

small absolute aperture for
very short focal distances possible





N.A. ~ D/f

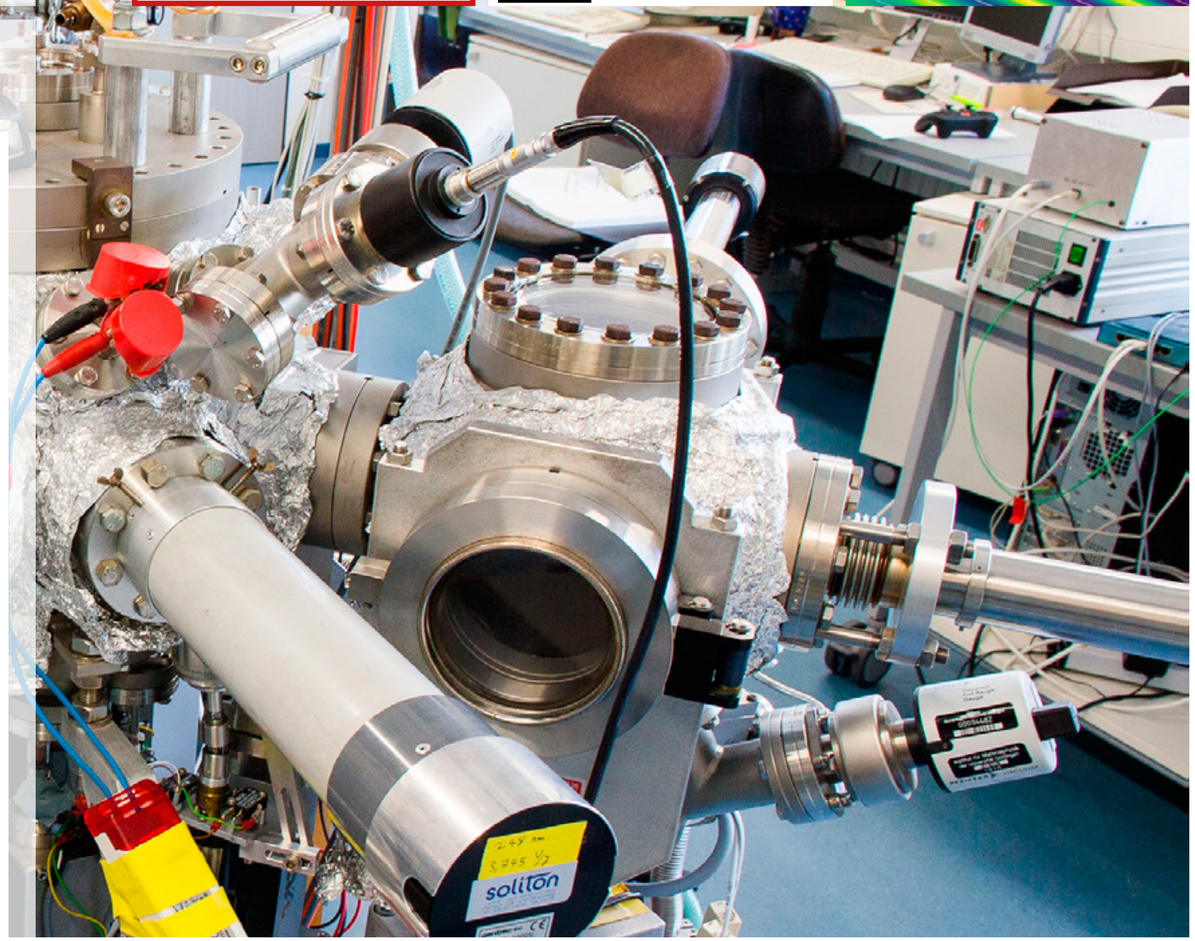
~ 0.005

high acceptance
large spot

~ 0.05

low acceptance
small spot

? high acceptance
small spot ?



MZP focusing – Design

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Basically: thick Fresnel Zone Plates.

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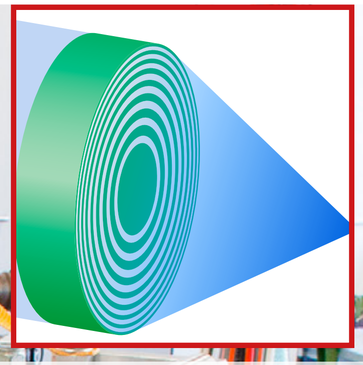

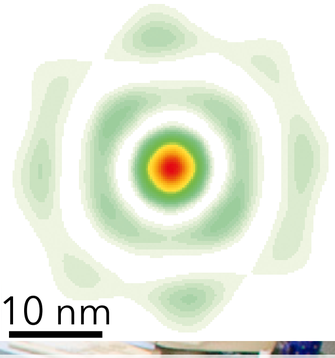

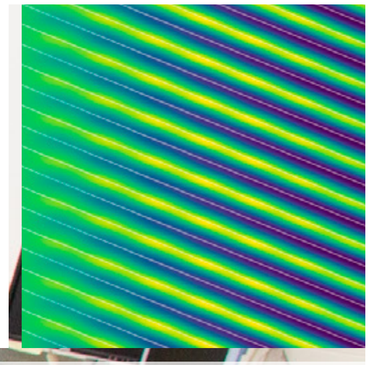

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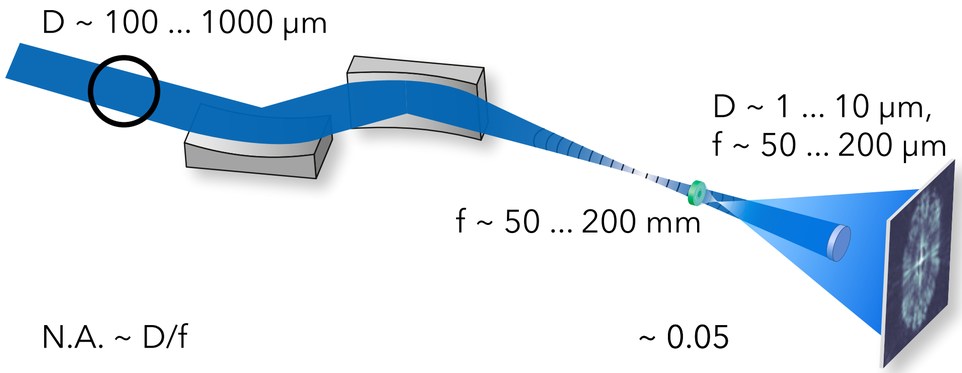


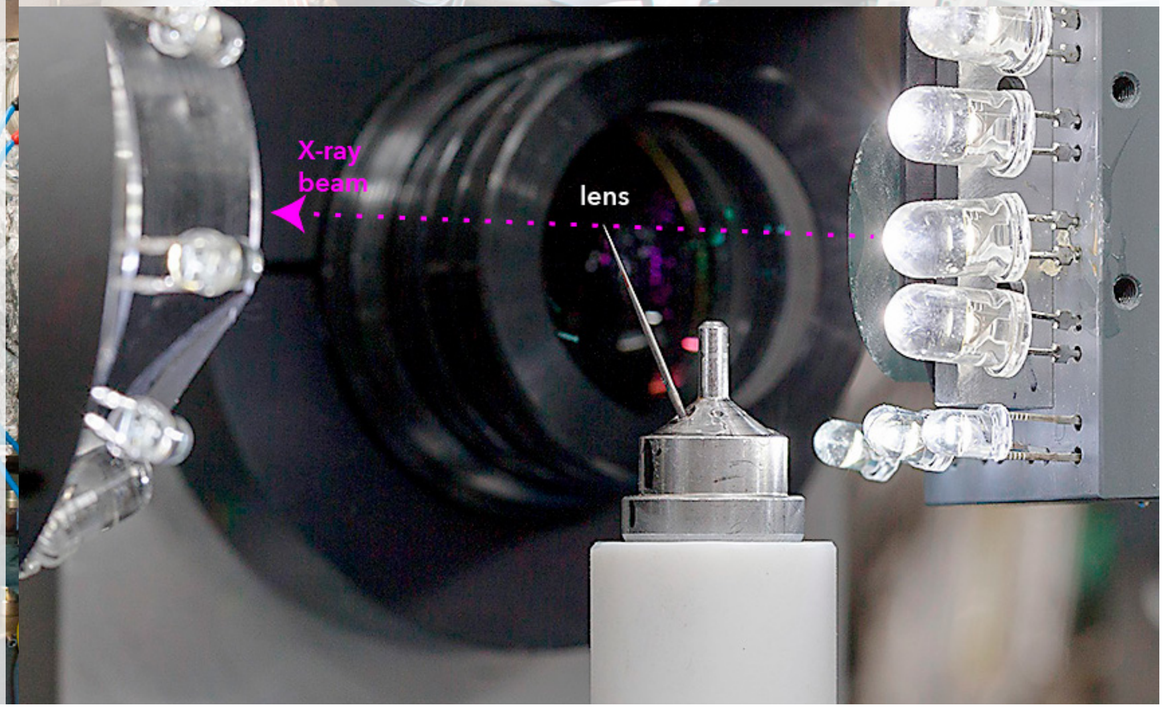
N.A. ~ D/f ~ 0.05

focal size @ 12.4 keV ~ 2.5 nm (Airy disk)

high acceptance
small spot

KB: collect incoming flux
MZP: high-NA focus

few layers: high bandwidth



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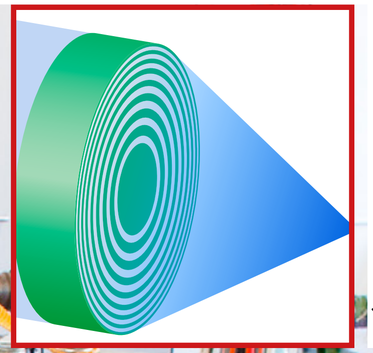

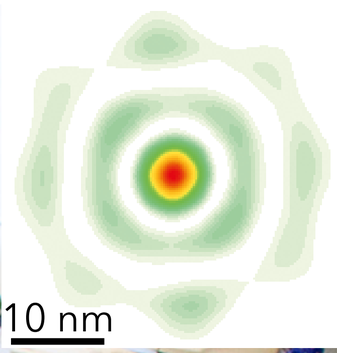

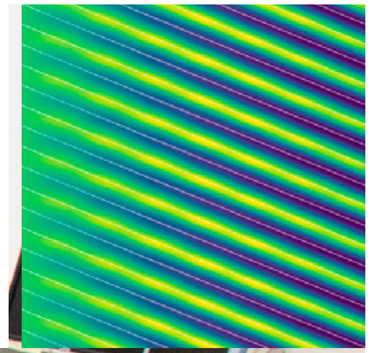

MZP-Parameters

Energies: 7.9 keV, 13.8 keV, 18 keV, 60 … 100 keV

Focal length: 50 µm, 0.5 mm, 5 mm

Apertures: 1.5 … 15 µm

Layers: 50 – 250

Instruments: GINIX @ P10, PETRA III; ID-31 @ ESRF

MZP focusing – Characterisation

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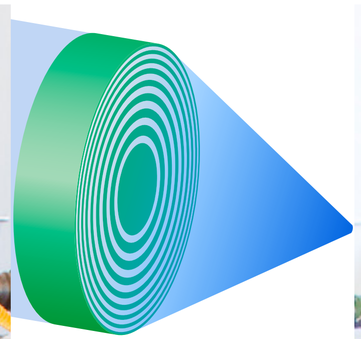
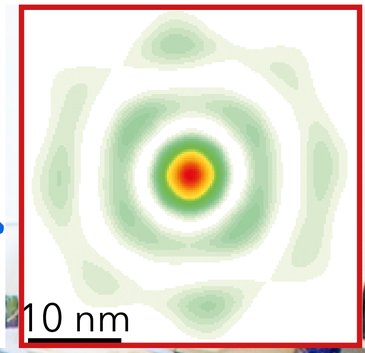
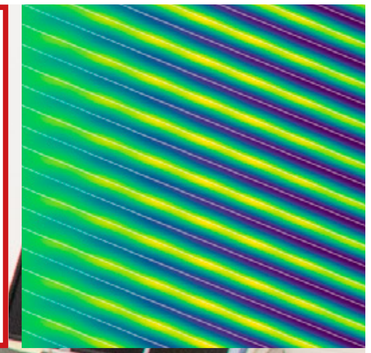




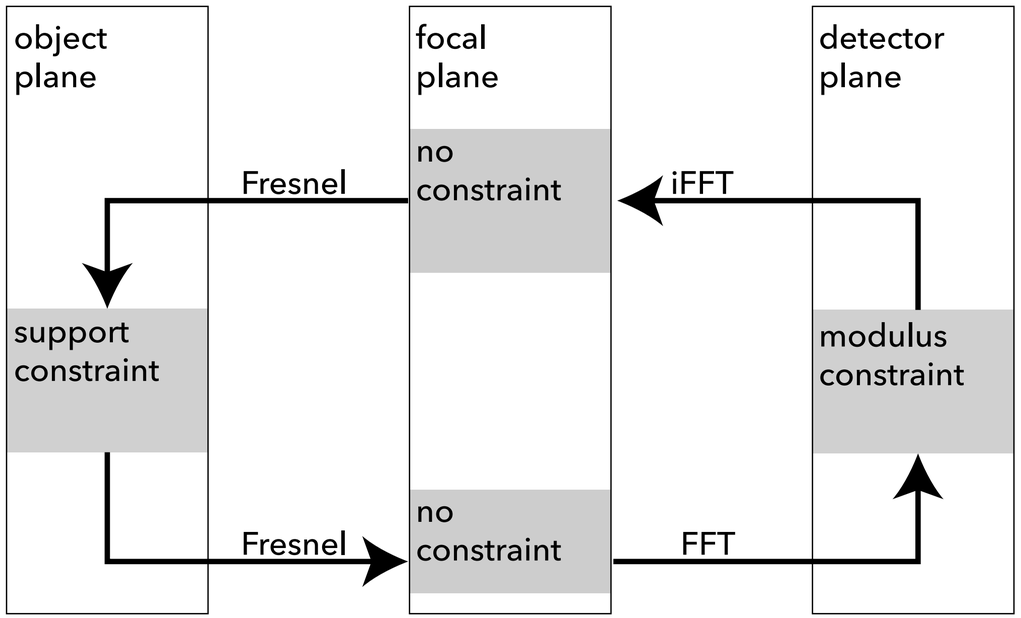


Focus characterisation – indirect, based on far-field intensity

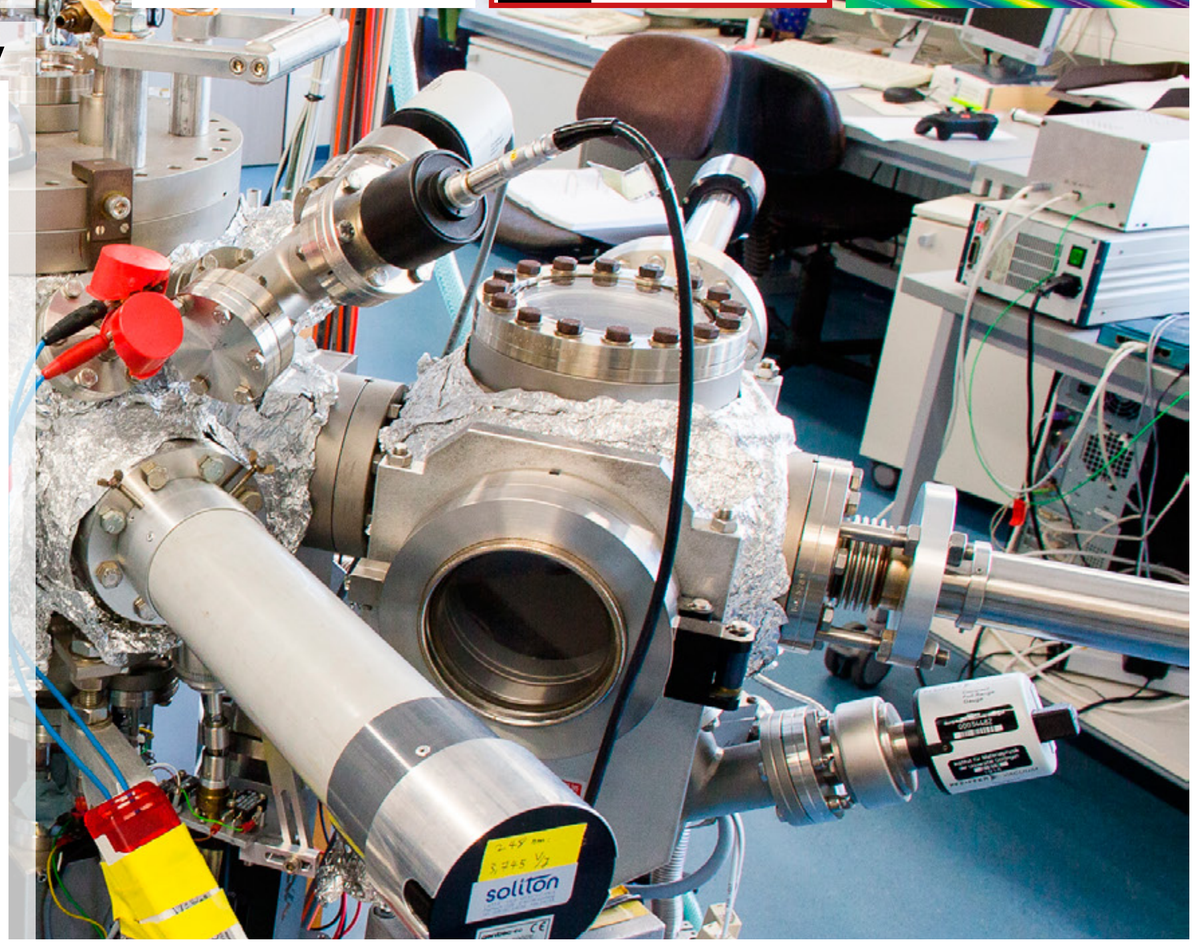
Focusing experiments @ GINIX set-up, P10 beamline, PETRA III

$$u_{\mathrm{zp}} = D_f\, u_{\mathrm{f}} = \mathcal{F}^{-1}\left[\mathcal{F}u_{\mathrm{f}} \cdot \mathcal{F}\frac{\exp\left(-ikf\rho_{\mathrm{f}}^2\right)}{i\lambda f}\right]$$

$$u'_{\mathrm{f}} = \begin{cases} \mathcal{F}^{-1}\left[\sqrt{I_{\mathrm{d}}(x_{\mathrm{d}}, y_{\mathrm{d}})} \cdot \arg \mathcal{F}u_{\mathrm{f}}\right] & \text{outside beamstop,} \\ \mathcal{F}^{-1}\mathcal{F}u_{\mathrm{f}} & \text{inside beamstop.} \end{cases}$$



Quiney et al., Nature Physics, 2006



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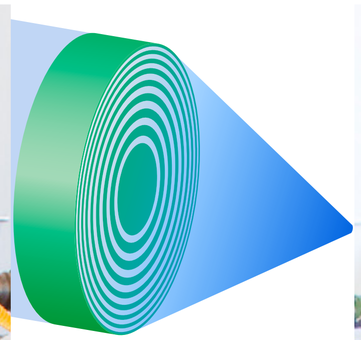

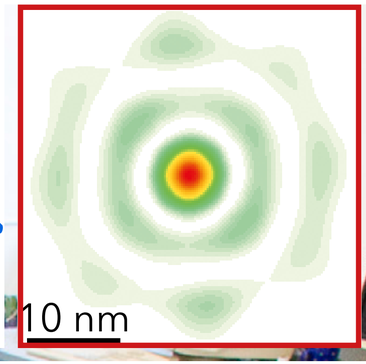

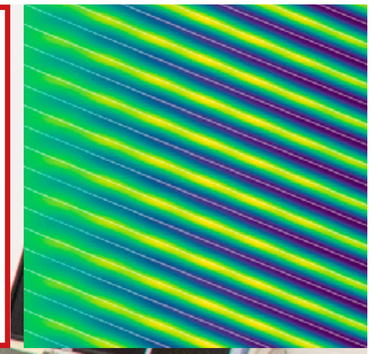

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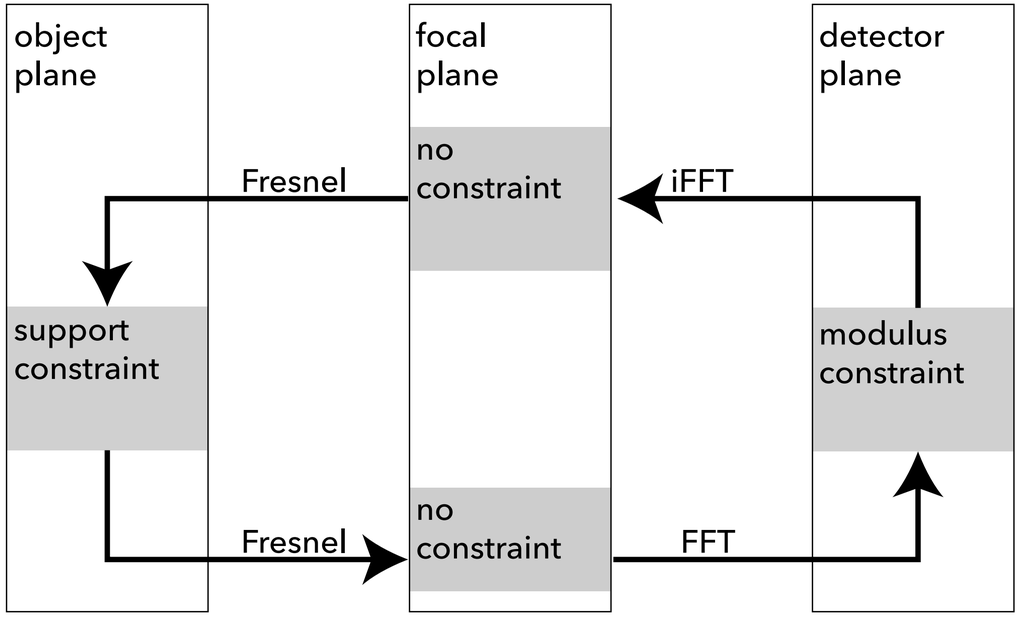
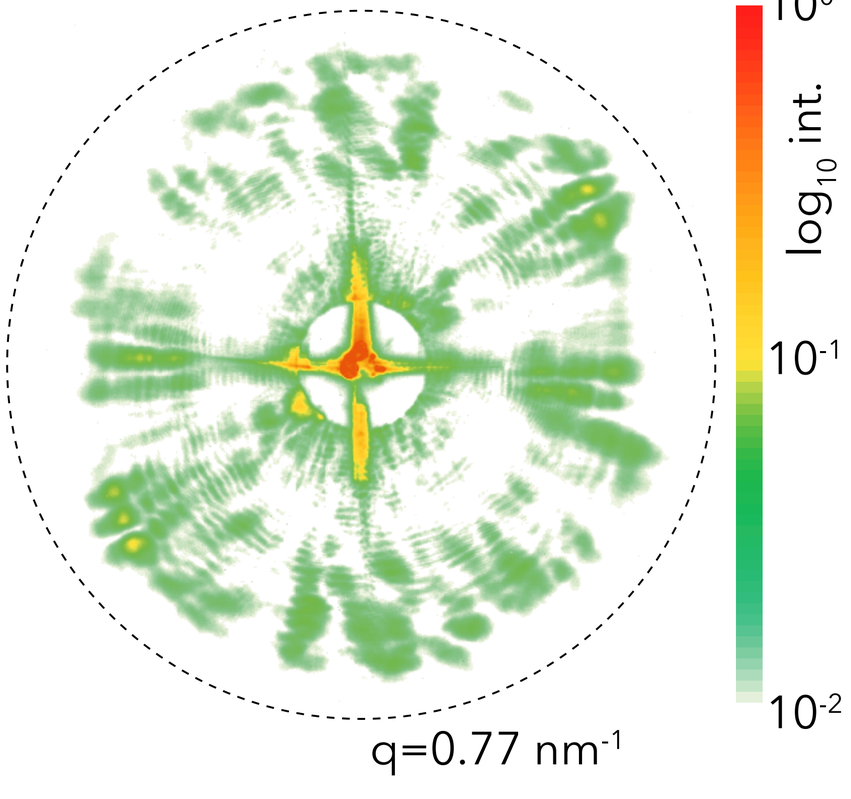


Quiney et al., Nature Physics, 2006

logarithmic far-field intensity



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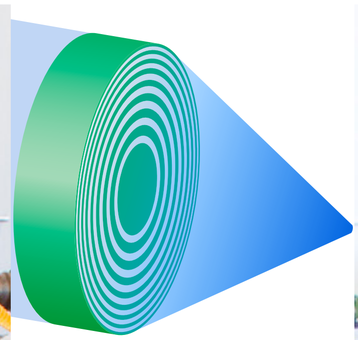

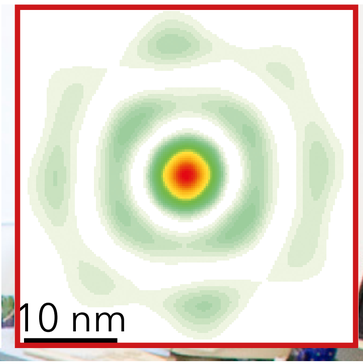

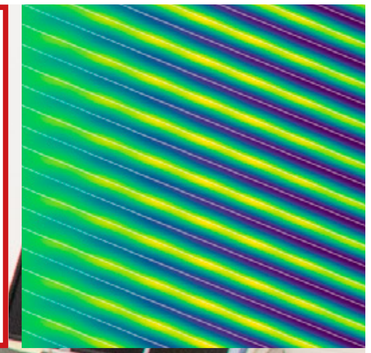

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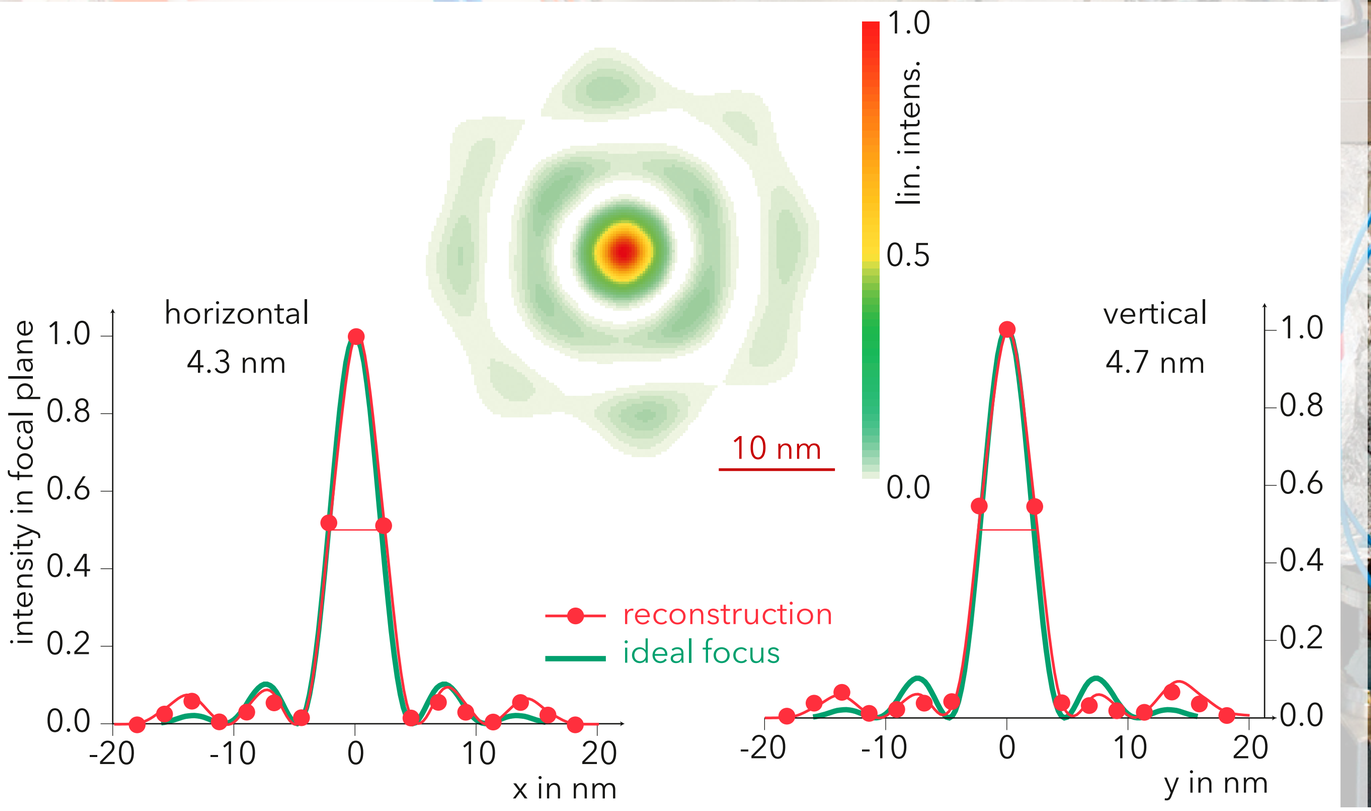
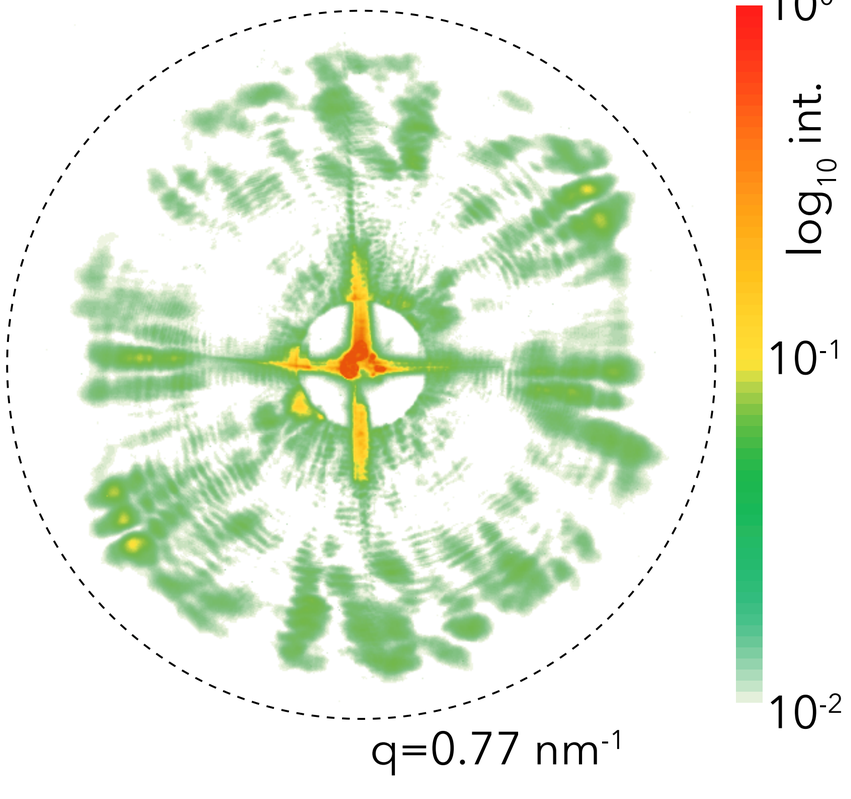


logarithmic far-field intensity



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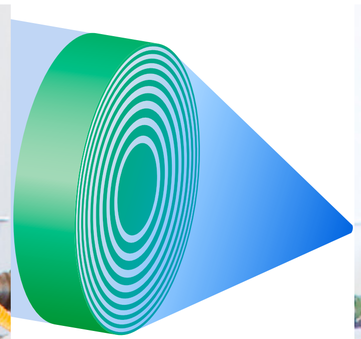

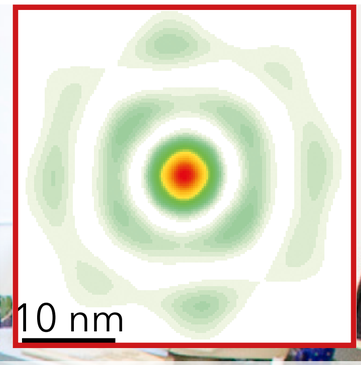

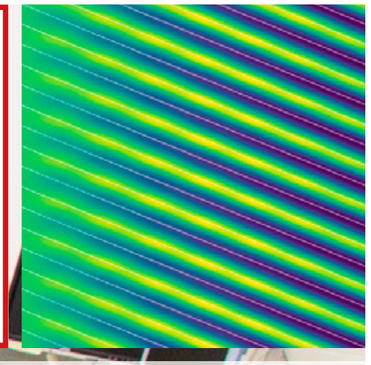

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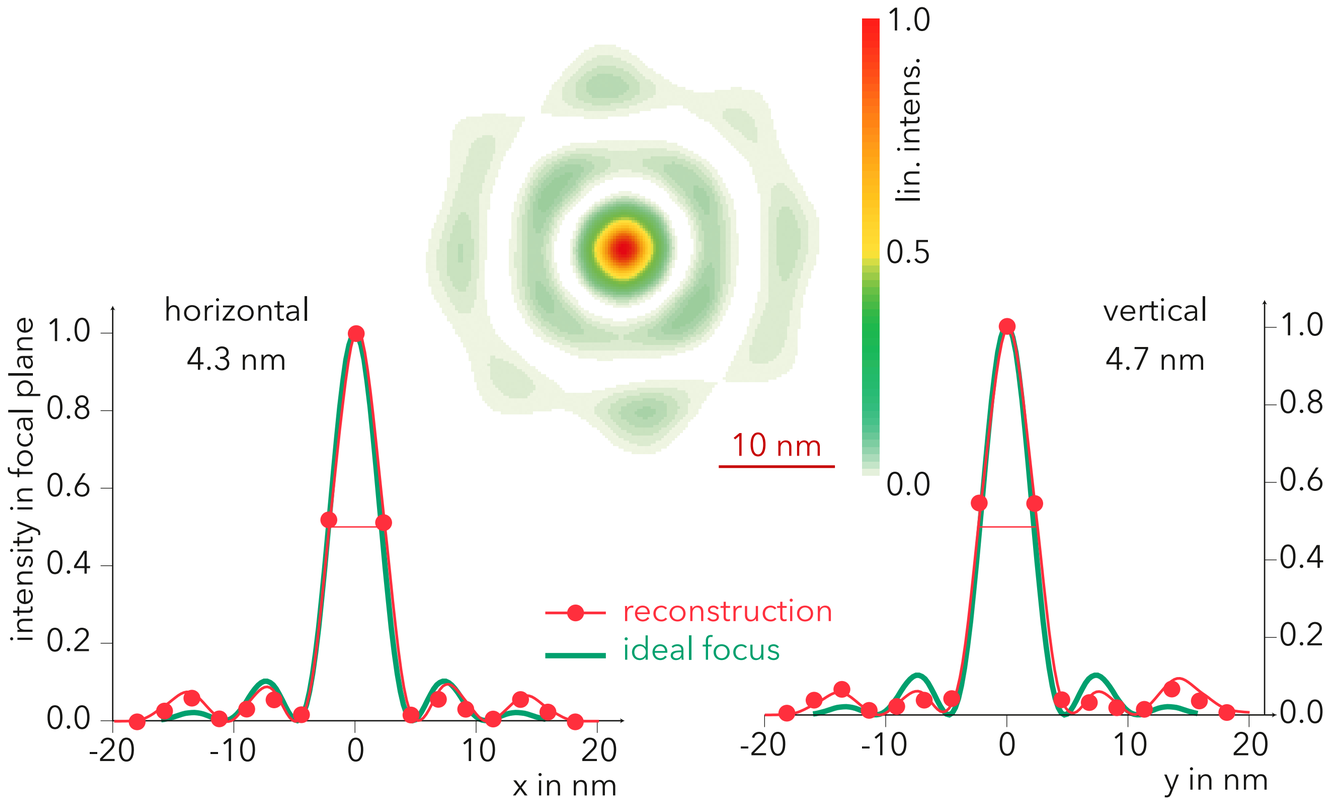
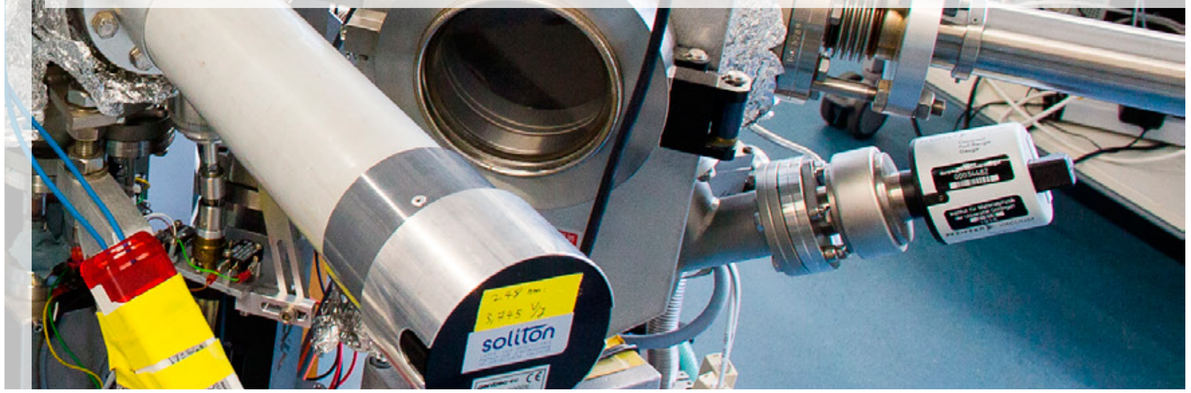


Year	Energy	Experiment	Design
2012	7.9 keV	≤ 4.7 nm	5 nm
2013	13.8 keV	≤ 5.9 nm	5 nm
2014	18.0 keV	10.0 nm	10 nm
2015	8.0 keV	≤ 5.3 nm	5 nm
2016	100 keV	?	10 nm

Efficiency:
Increase from ~1 % (2012) to ~7 % (2015)



D13 – largest diameter so far

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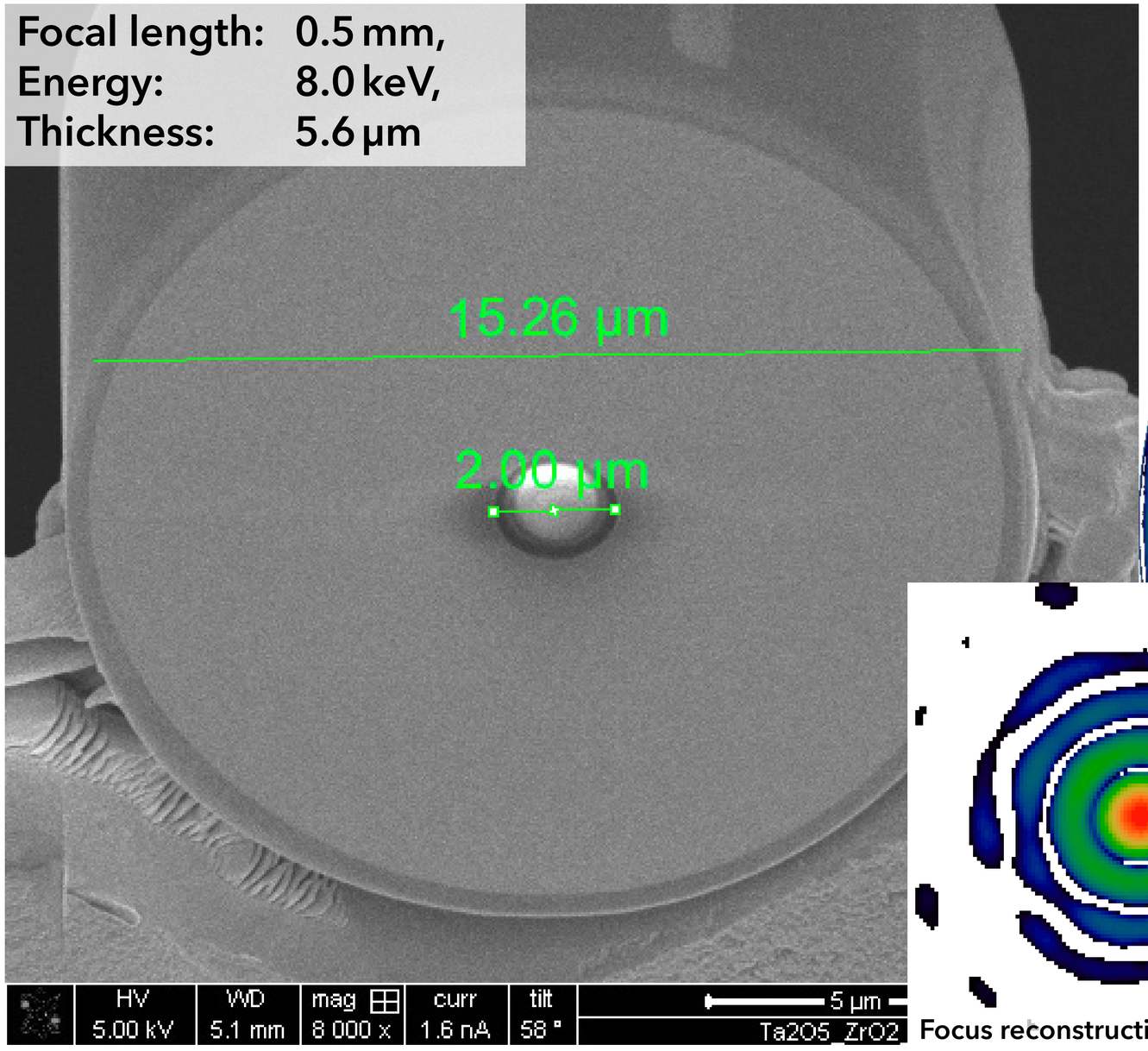
IRP / SFB 755

Focal length: 0.5 mm,
Energy: 8.0 keV,
Thickness: 5.6 µm



Focus reconstruction (log scale)

Far-field intensity pattern,
Pilatus 1M @ 5 m distance,
logarithmic colour palette

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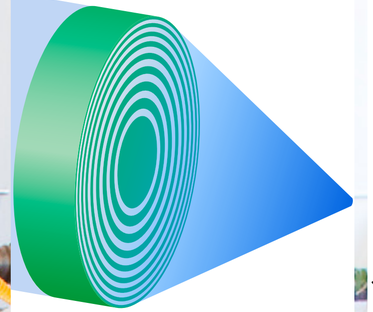
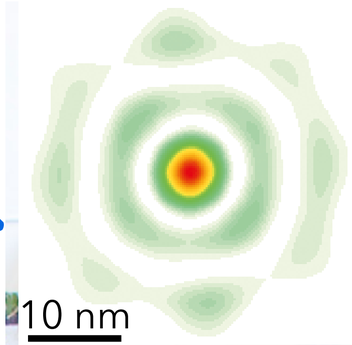
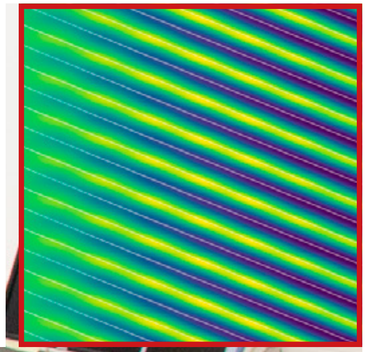
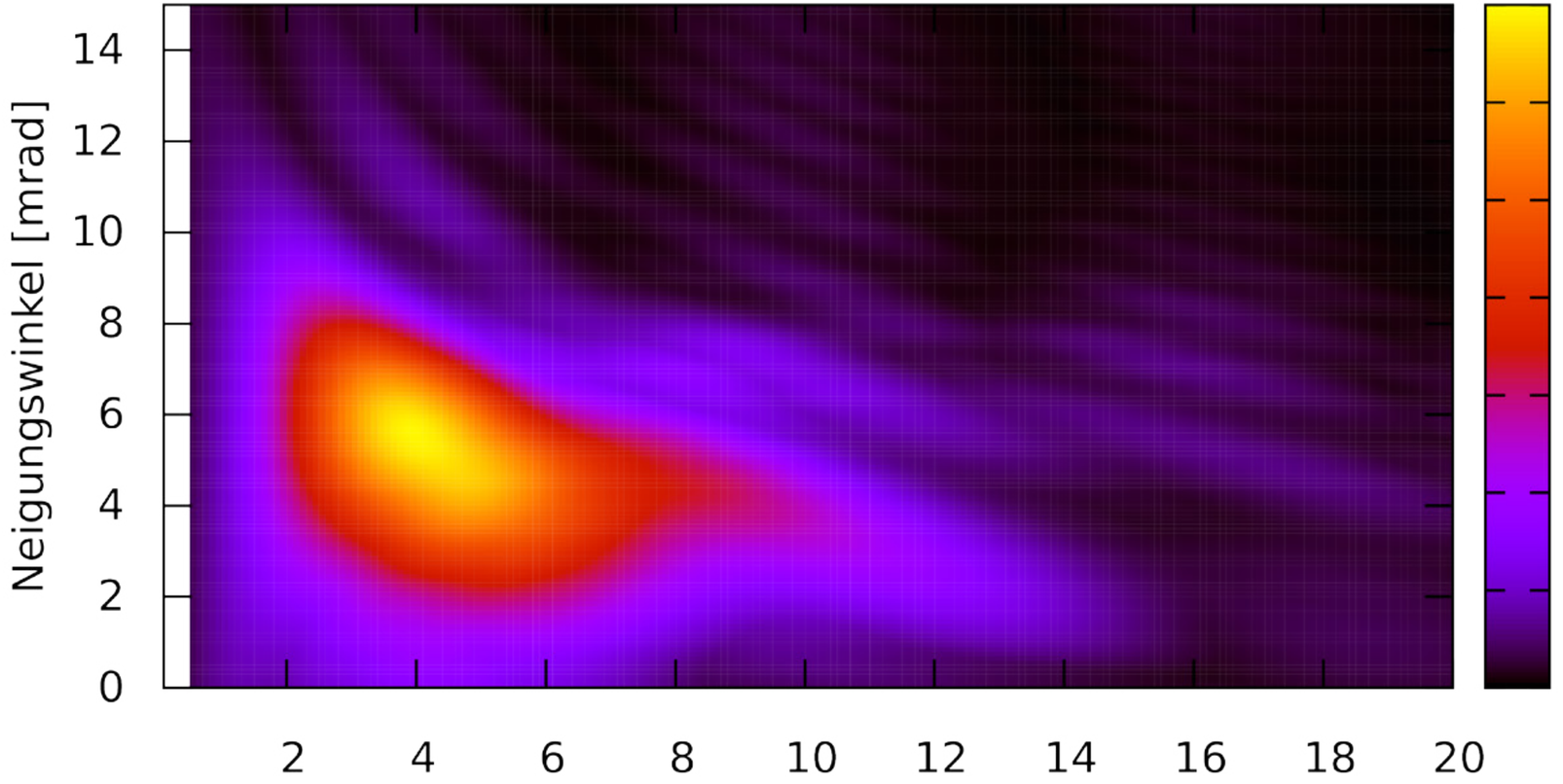
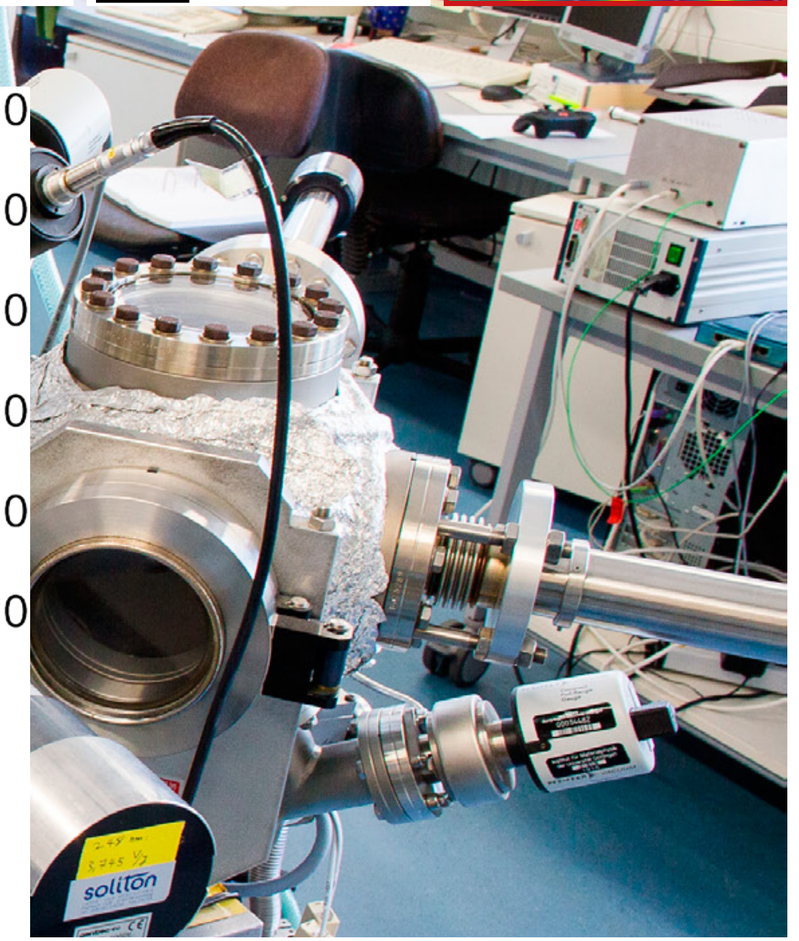






Focus intensity as function of taper angle / optical thickness





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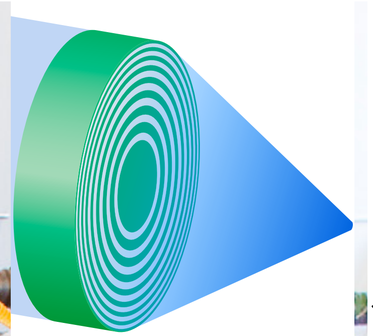
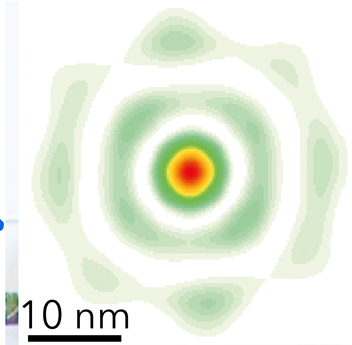
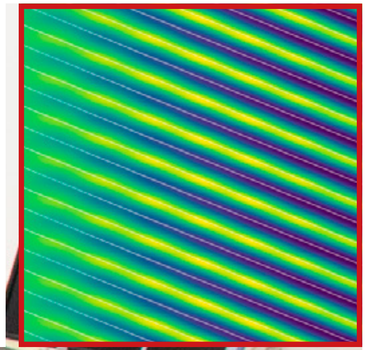
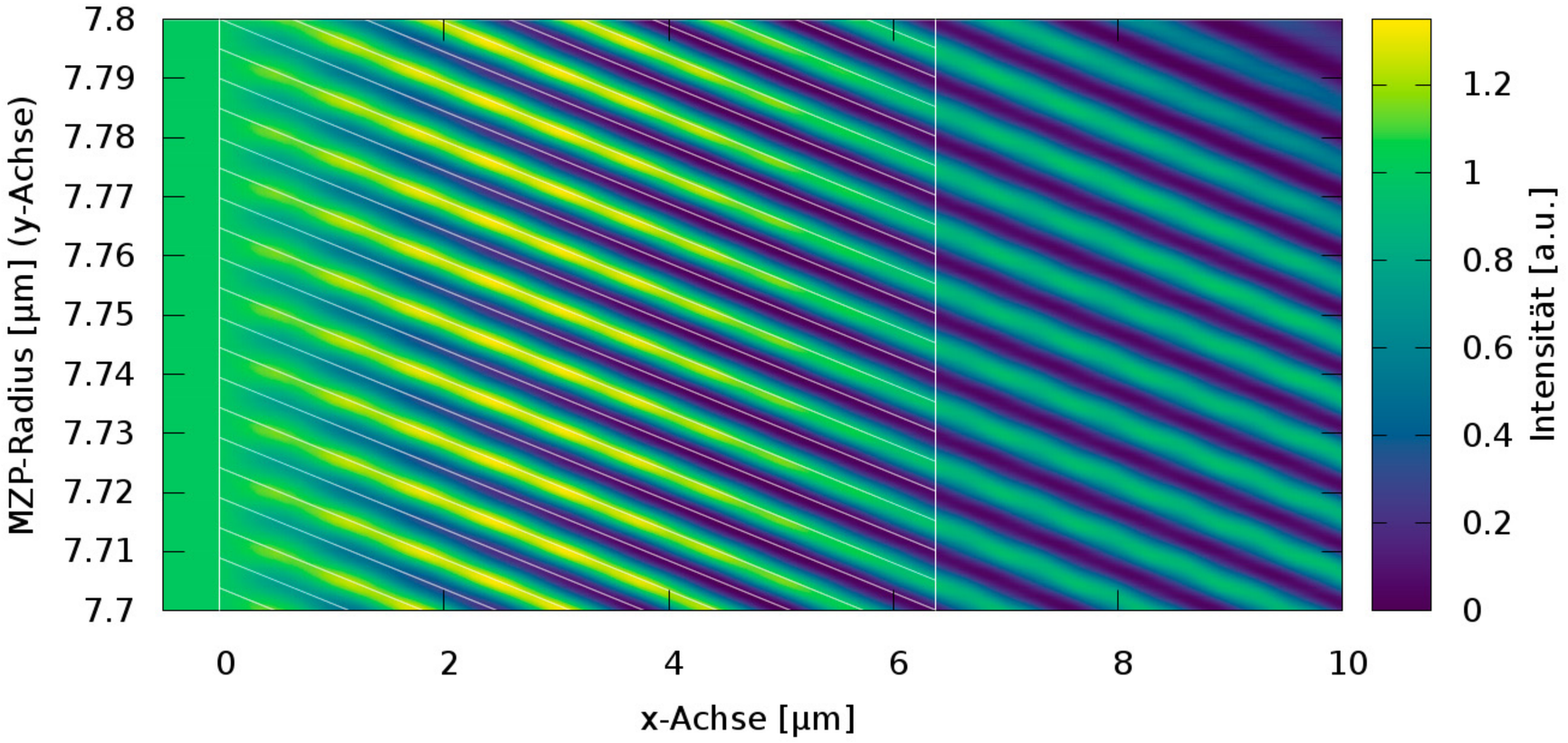
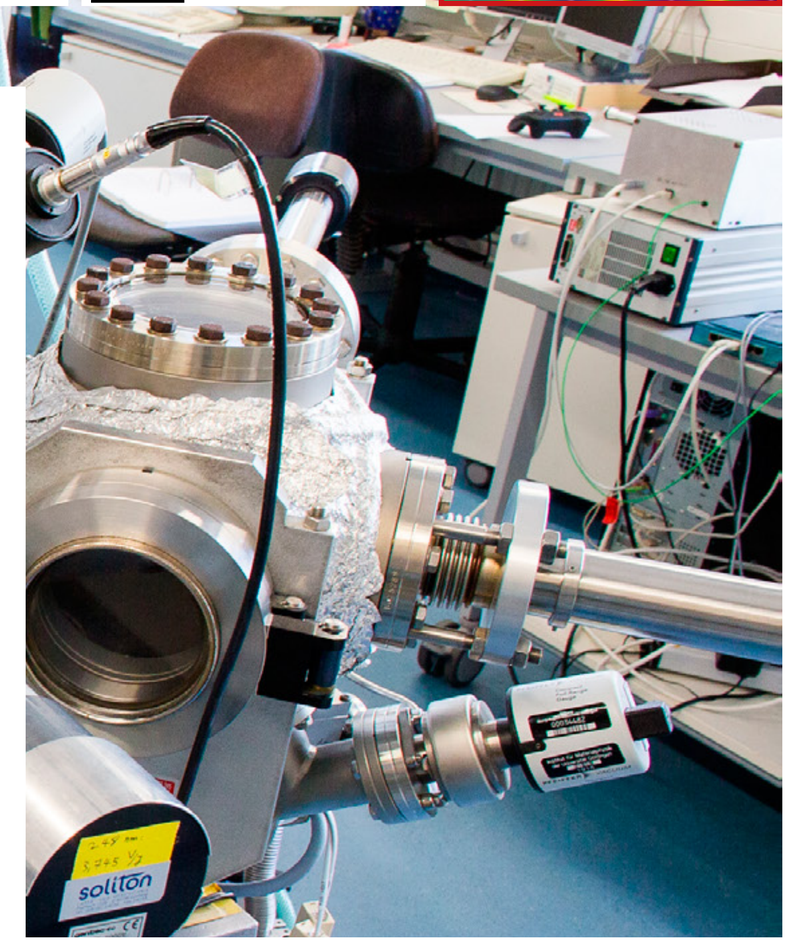






Propagation inside 5 nm-layers of wedged MZP





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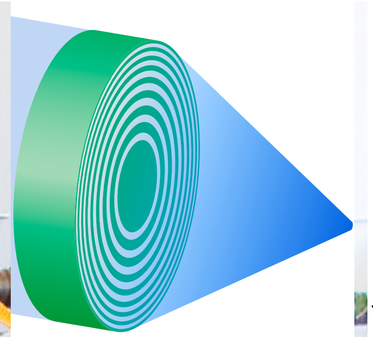
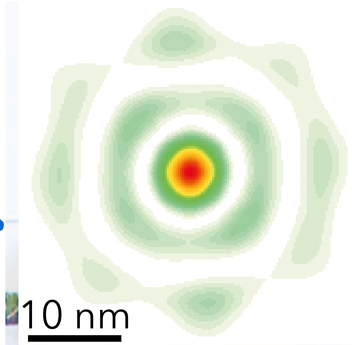
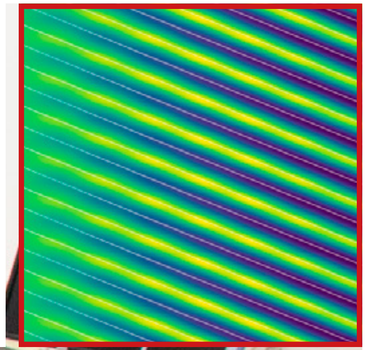
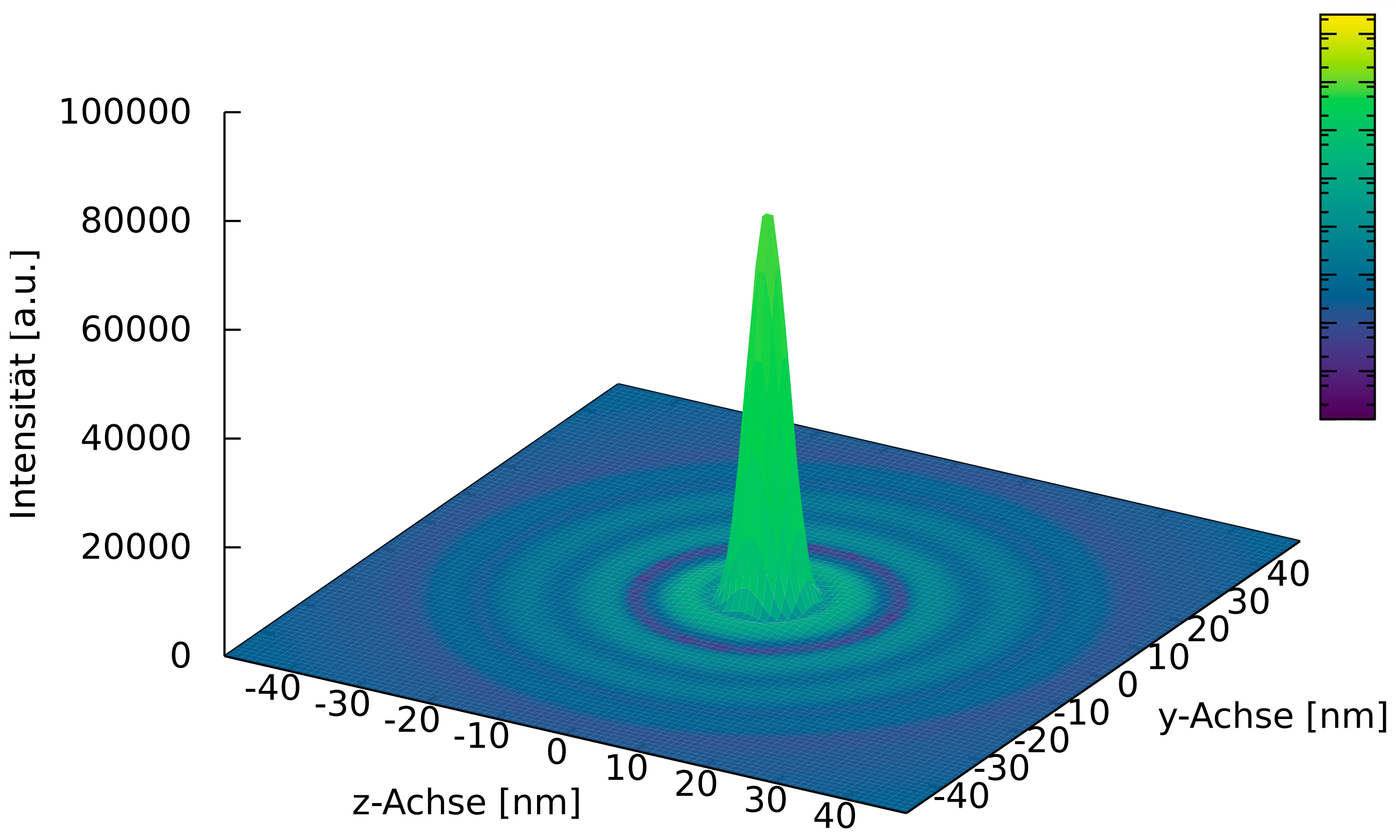
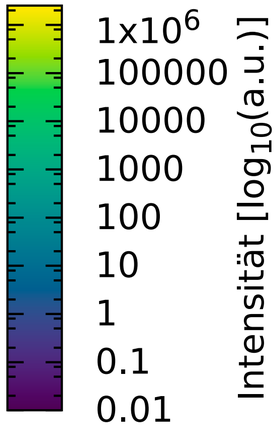


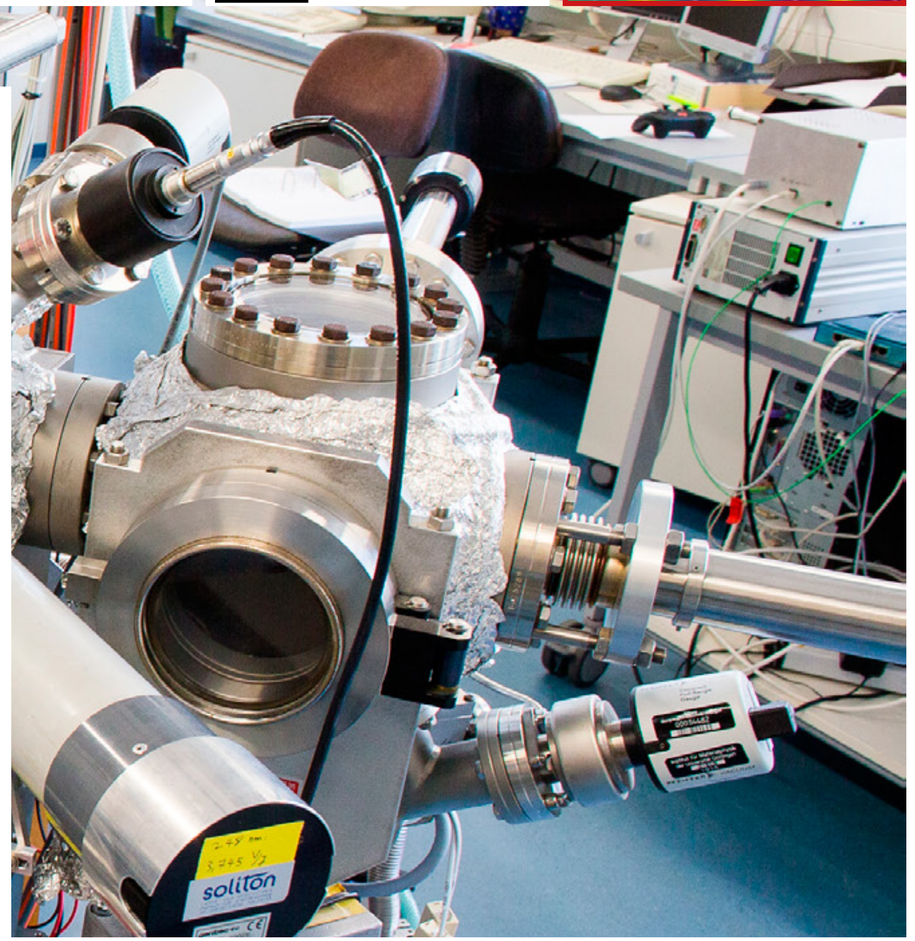




Focus simulation for flat MZP







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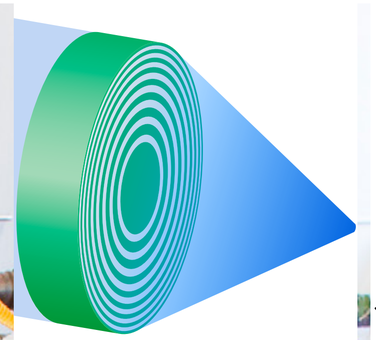
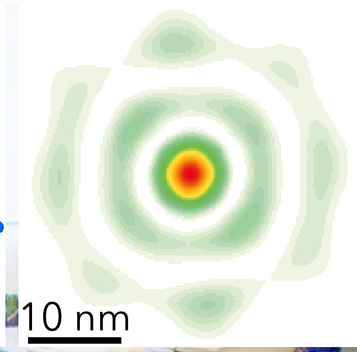
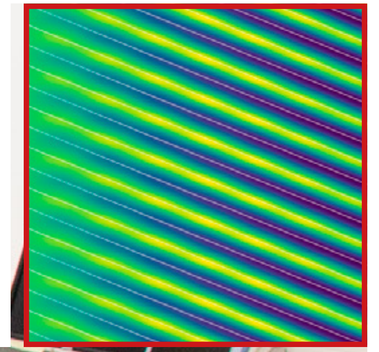
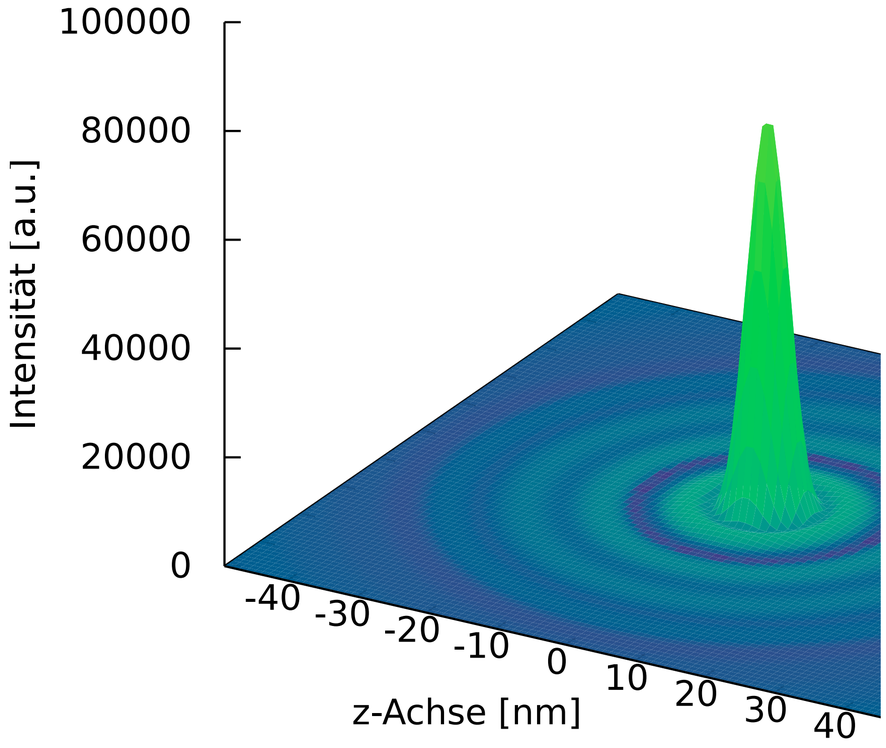
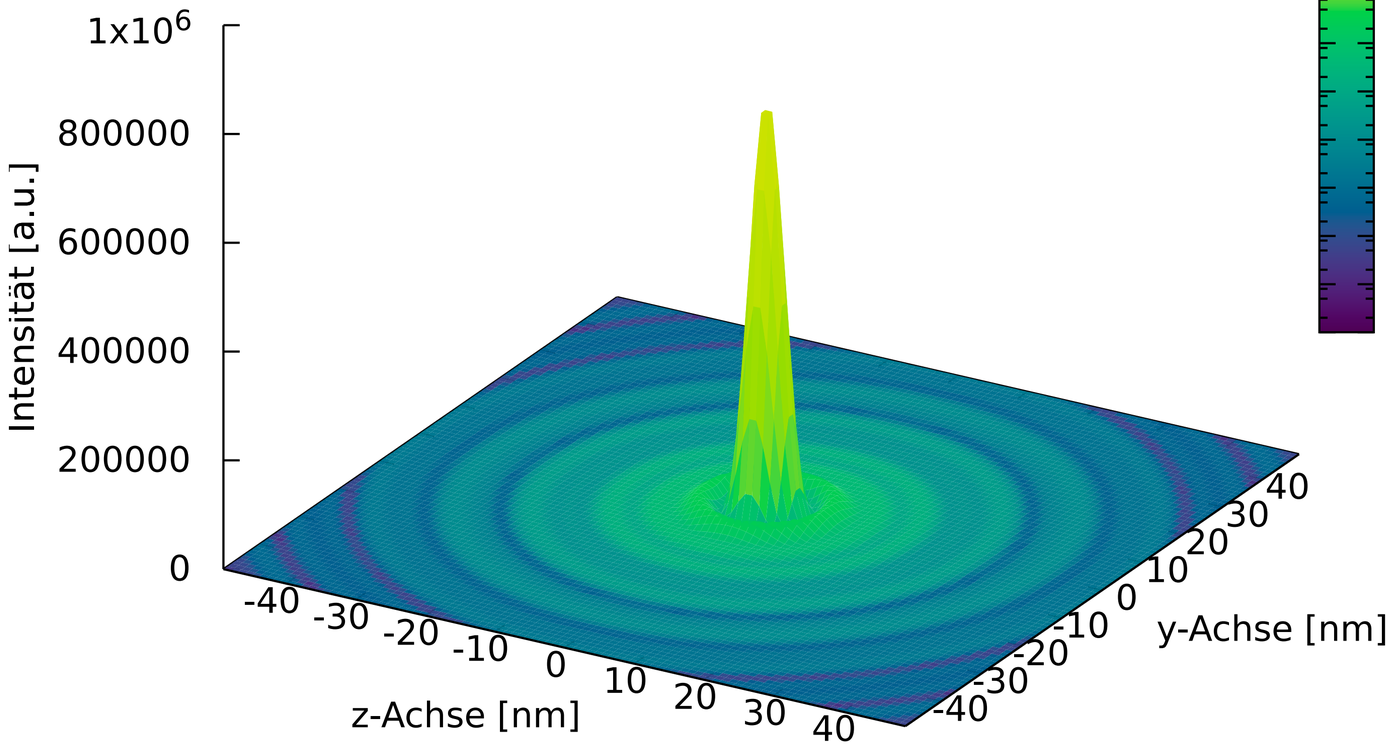


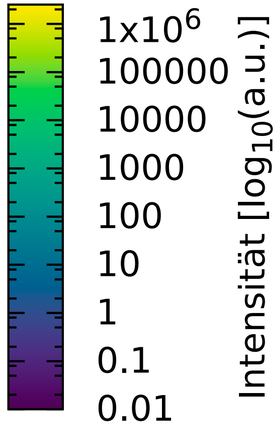




Focus simulation for flat and tilted MZP







MZP instrumentation

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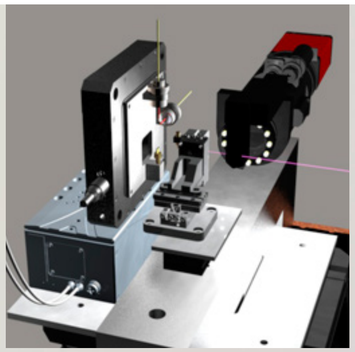
IRP / SFB 755

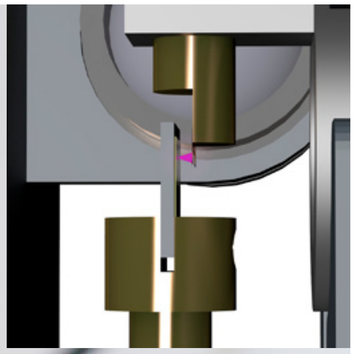
Mobile set-up:

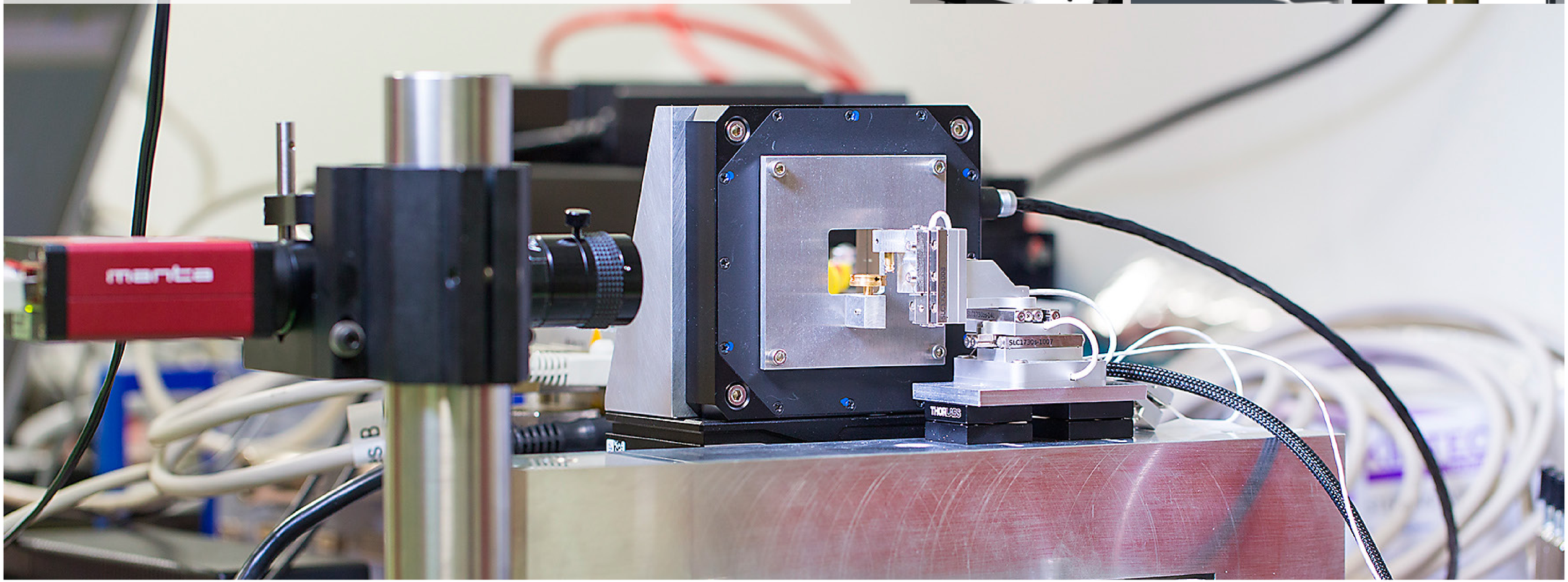
MZP alignment motorisation (in revision),

Sample alignment + fast Piezo scanner,

Interferometric position control (in progress)







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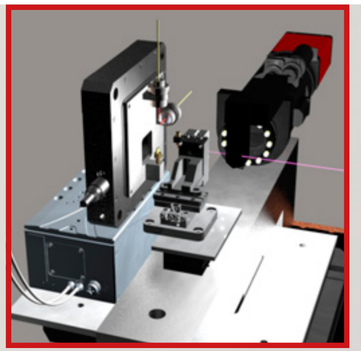
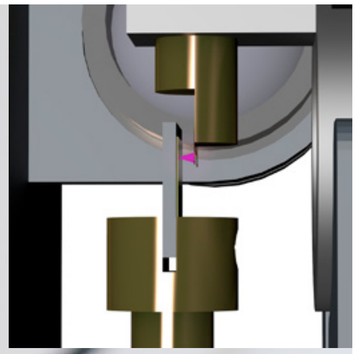
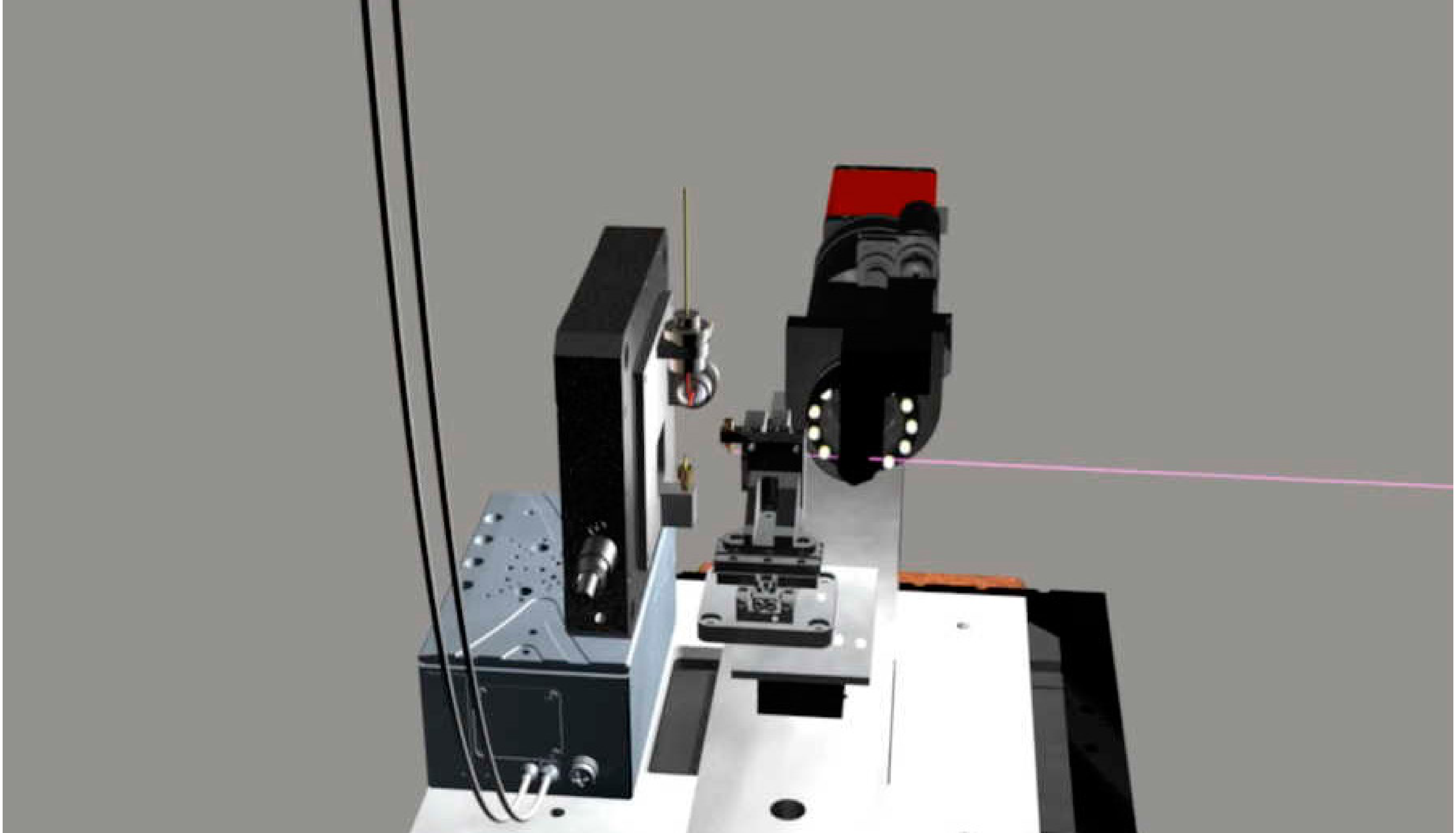




purpose	motor	axes	range	accuracy
lens, lateral	SLC–1730	x,y,z	21 mm	few nm
lens, rotation	STT–12.7	yrot,zrot	± 5°	≤ mrad
sample, align	M–686	x,y	25 mm	sub–µm
sample, align	N–765	z	6.5 mm	few nm
sample, scan	P–733	y,z	30 µm	sub–nm



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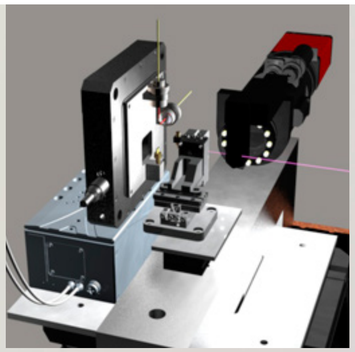
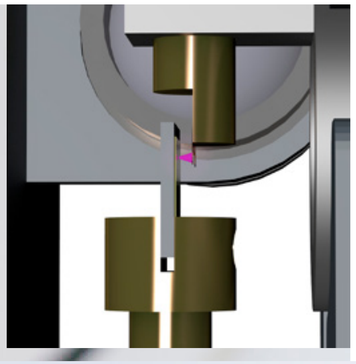
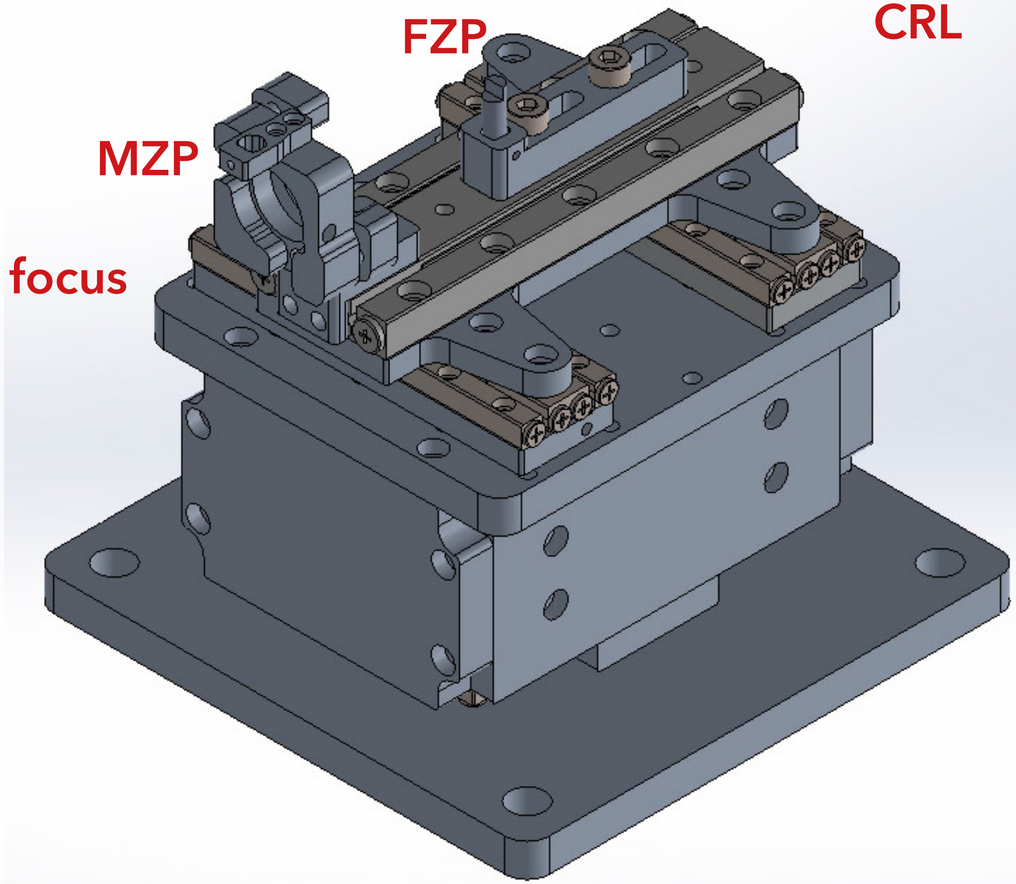




new FZP + MZP motorisation under construction

fast change of different optics:

CRL beam @ 1 µm (P10),
FZP beam @ 30 nm (C. David),
MZP beam @ 5 nm



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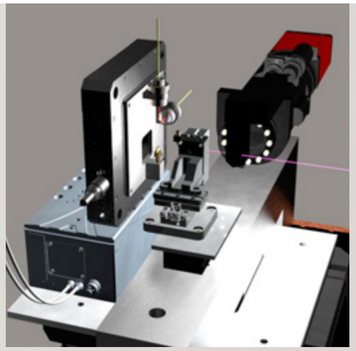
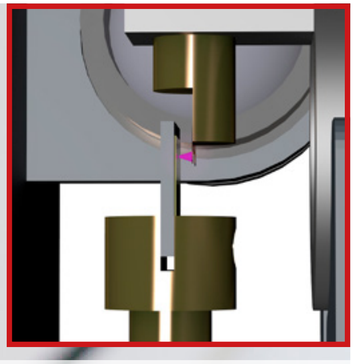
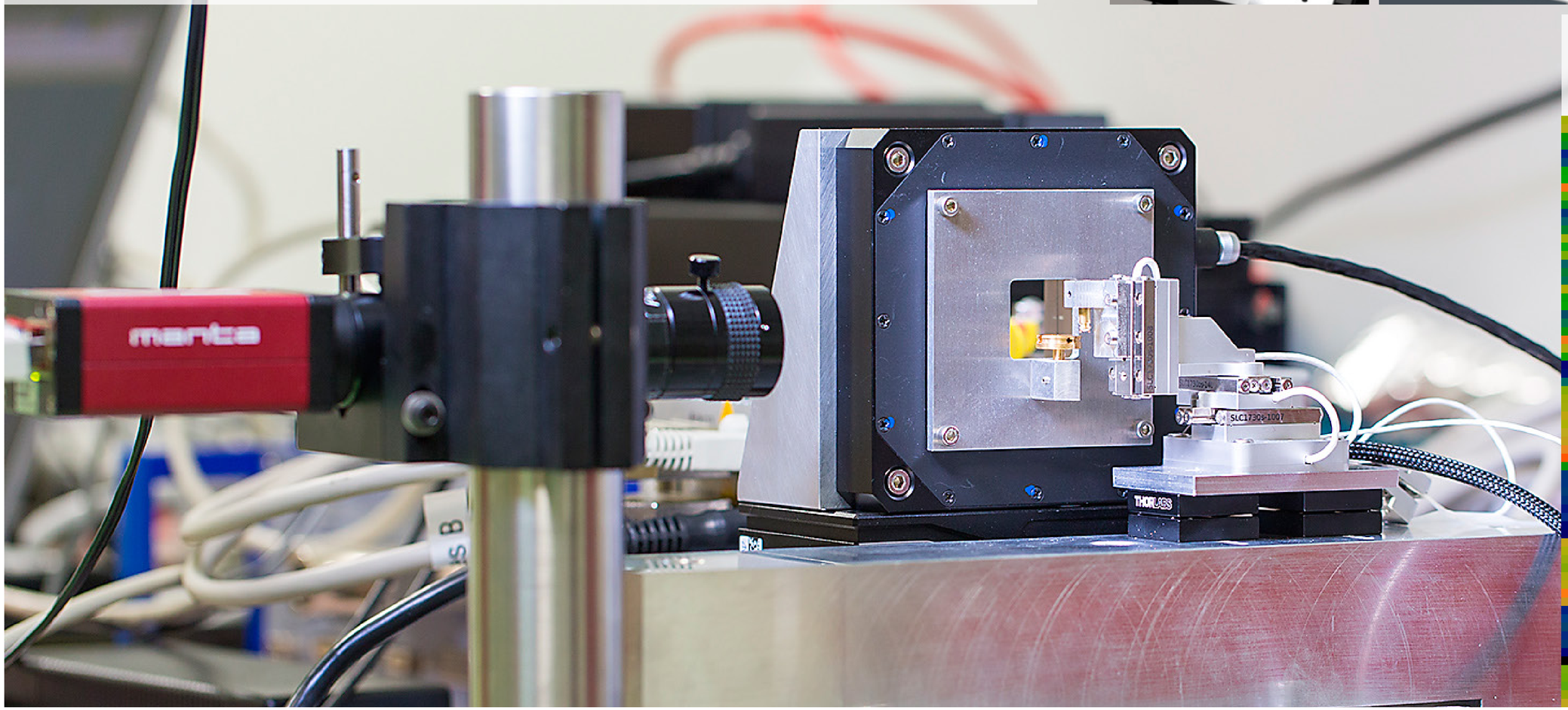
Continuous 2D scan

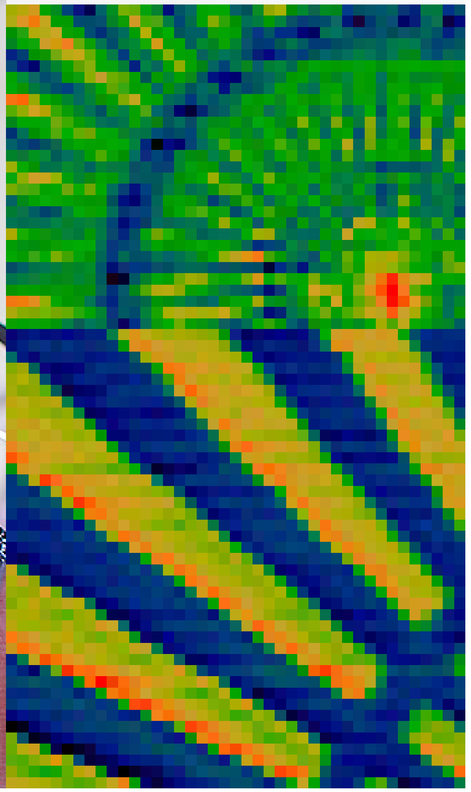
Eiger detector @ 750 Hz,
255 × 255 images, 1 µm² field of view
in less than 97 seconds





overivew step scans
during alignment



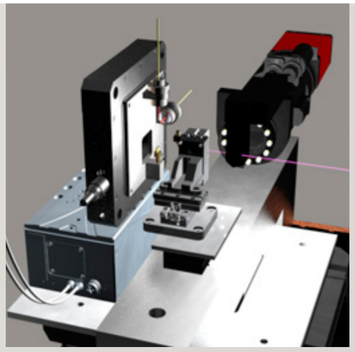


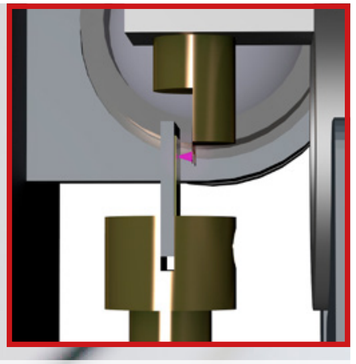
MZP instrumentation

Markus Osterhoff
IRP / SFB 755

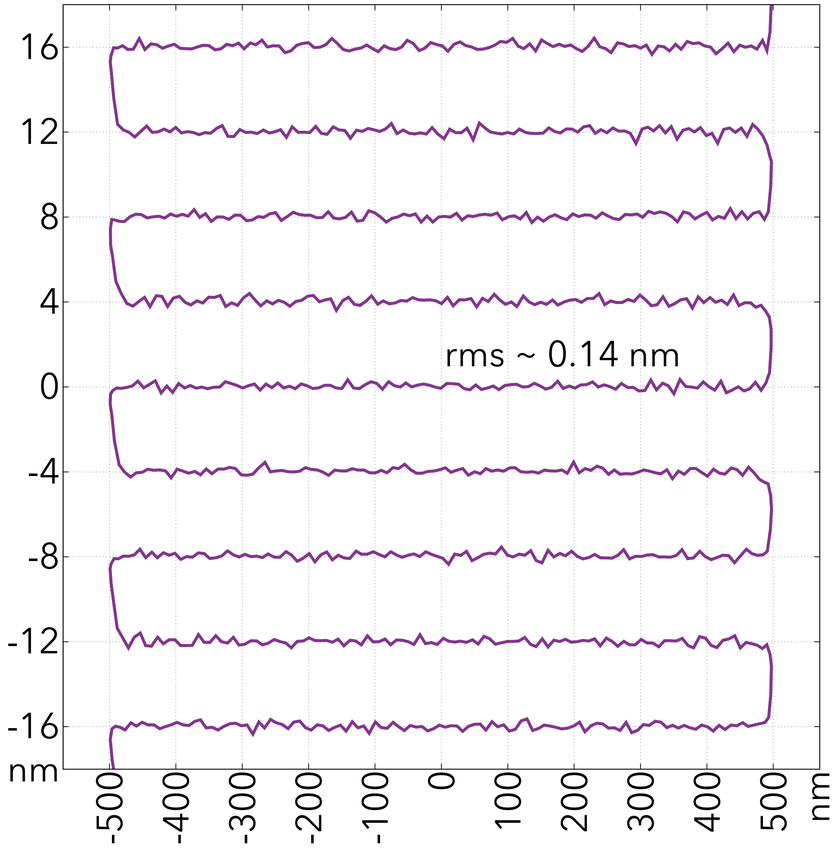
Continuous 2D scan

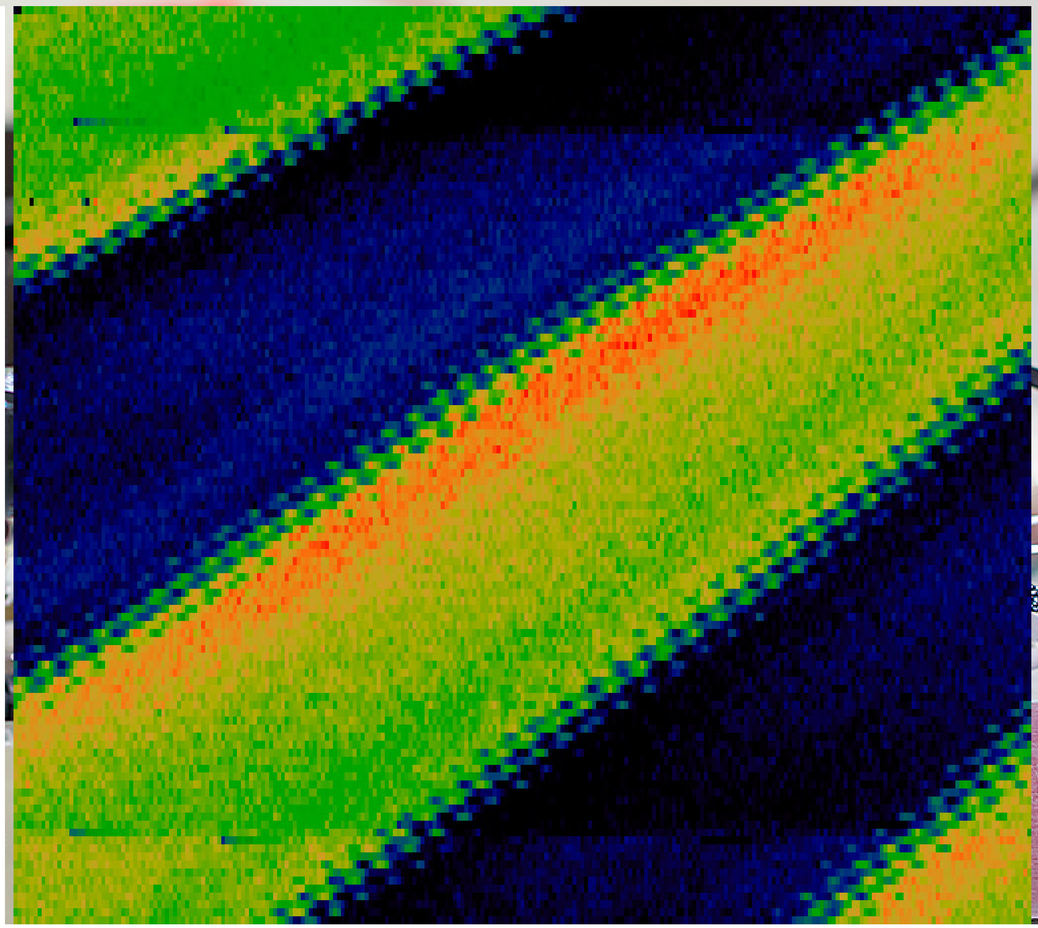
Eiger detector @ 750 Hz,
255 × 255 images, 1 µm² field of view
in less than 97 seconds



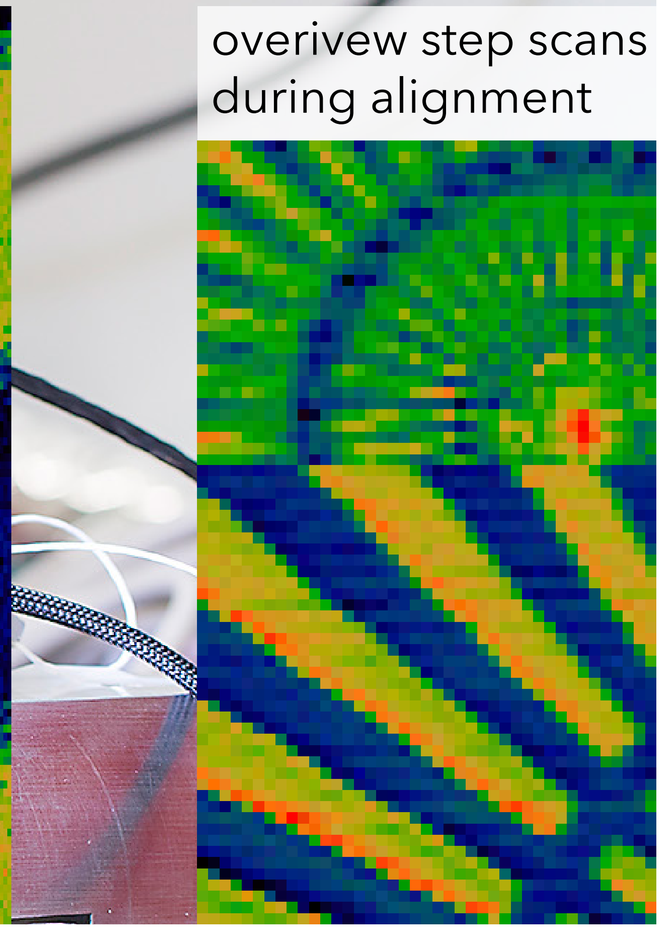


Piezo encoder during fast scan





overivew step scans during alignment



MZP imaging – our first baby steps

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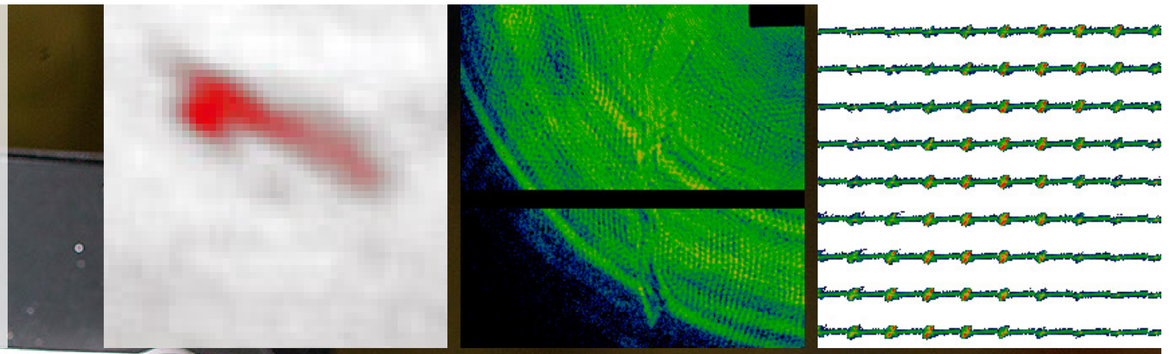
IRP / SFB 755

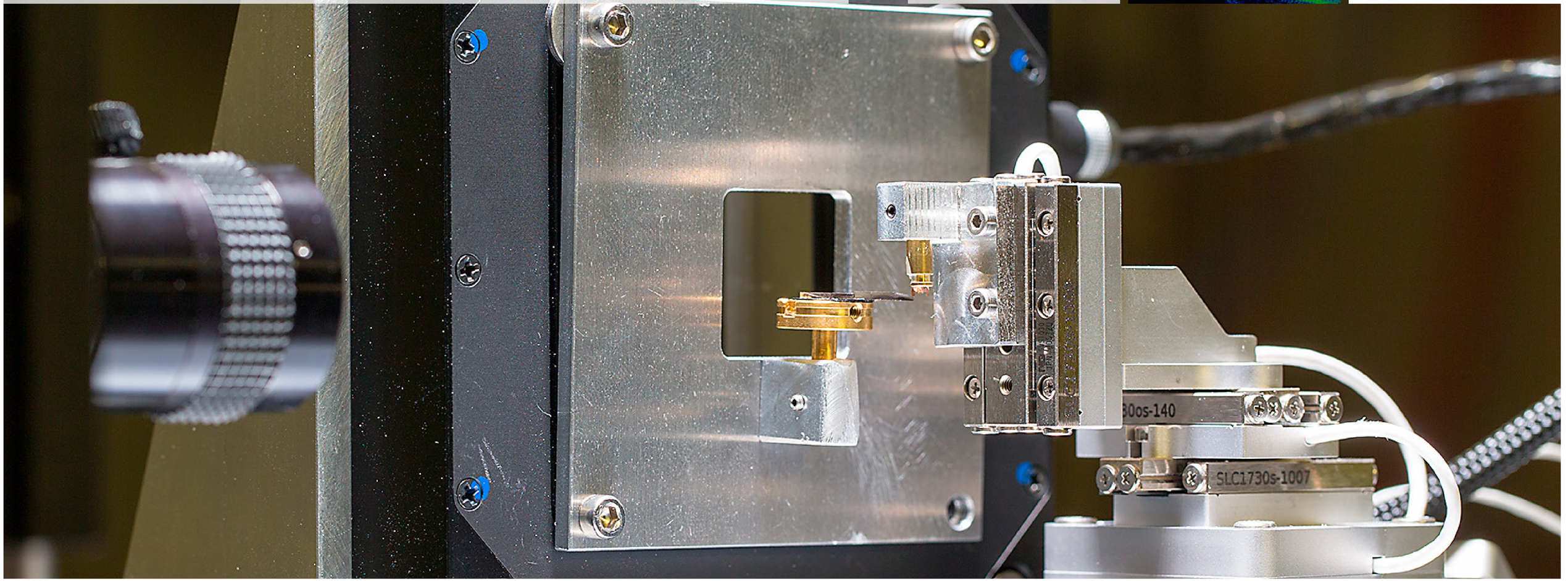
Proof-of-Principle Experiments, 2014 – ongoing:

Holography

Microscopy

Scanning WAXS





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Proof-of-Principle Experiments, 2014 – ongoing:

Holography

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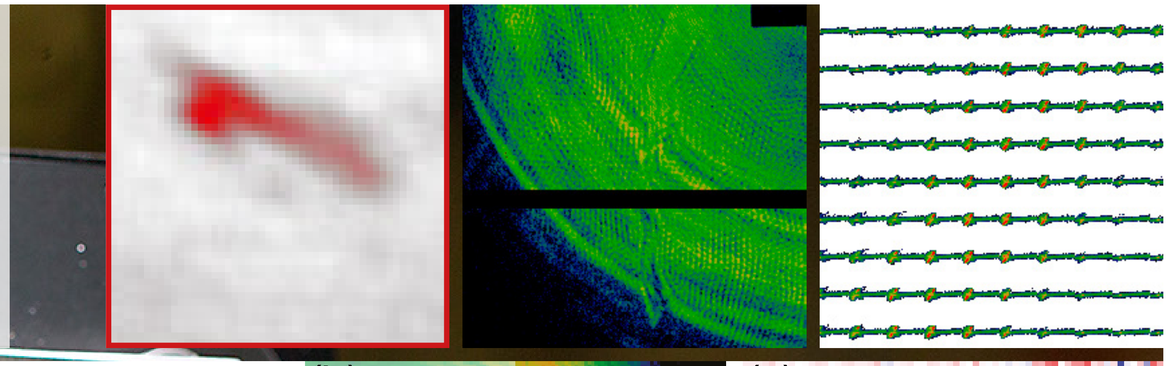


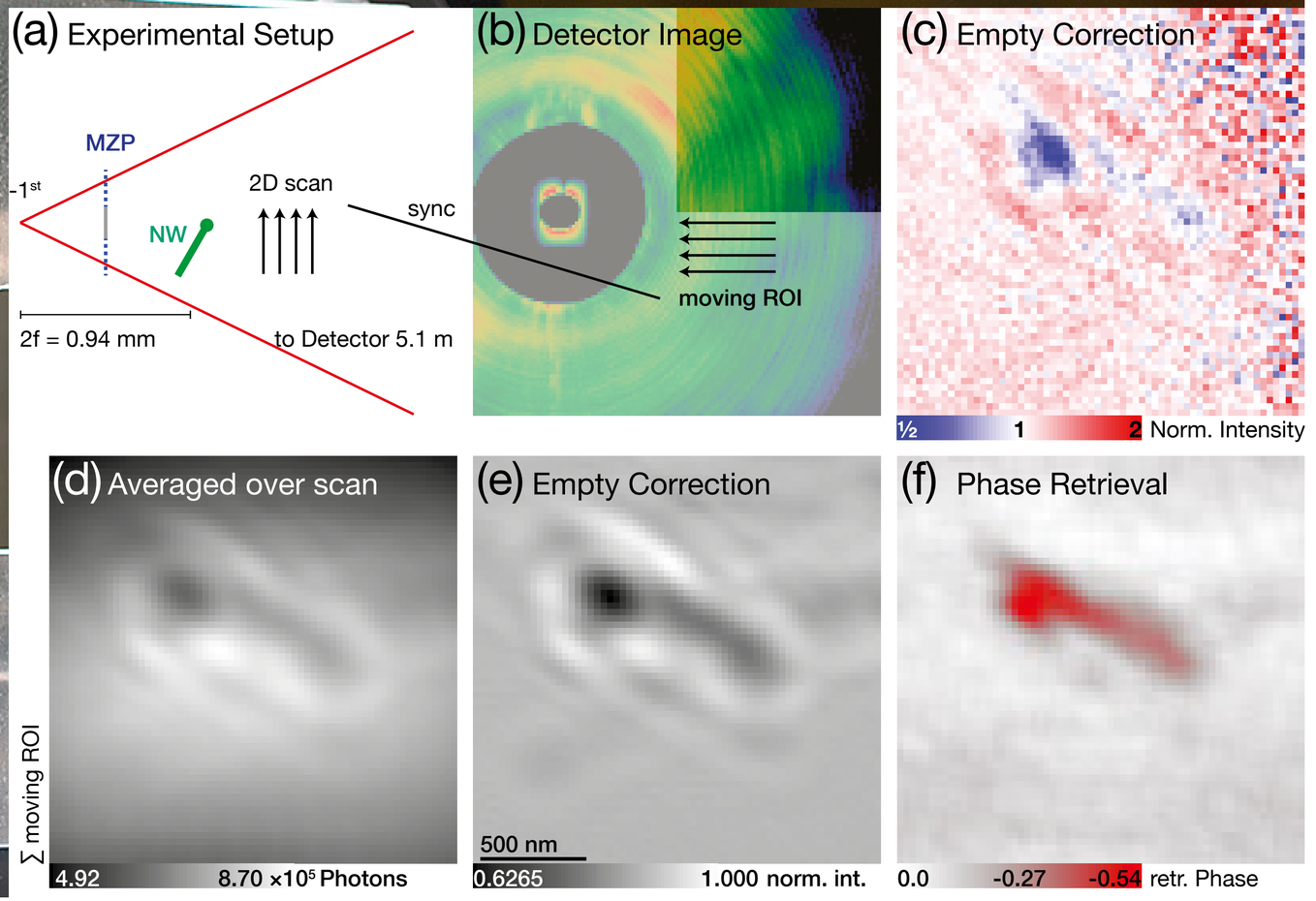
MZP for focusing, no objective lens

Holographic imaging in divergent -1st order

18 keV photon energy

GINIX @ P10

Sample:
GaAs nanowire, thickness ~ 300 nm, length ~ 2 µm



MZP imaging – our first baby steps

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Proof-of-Principle Experiments, 2014 – ongoing:

Holography

Microscopy

Scanning WAXS

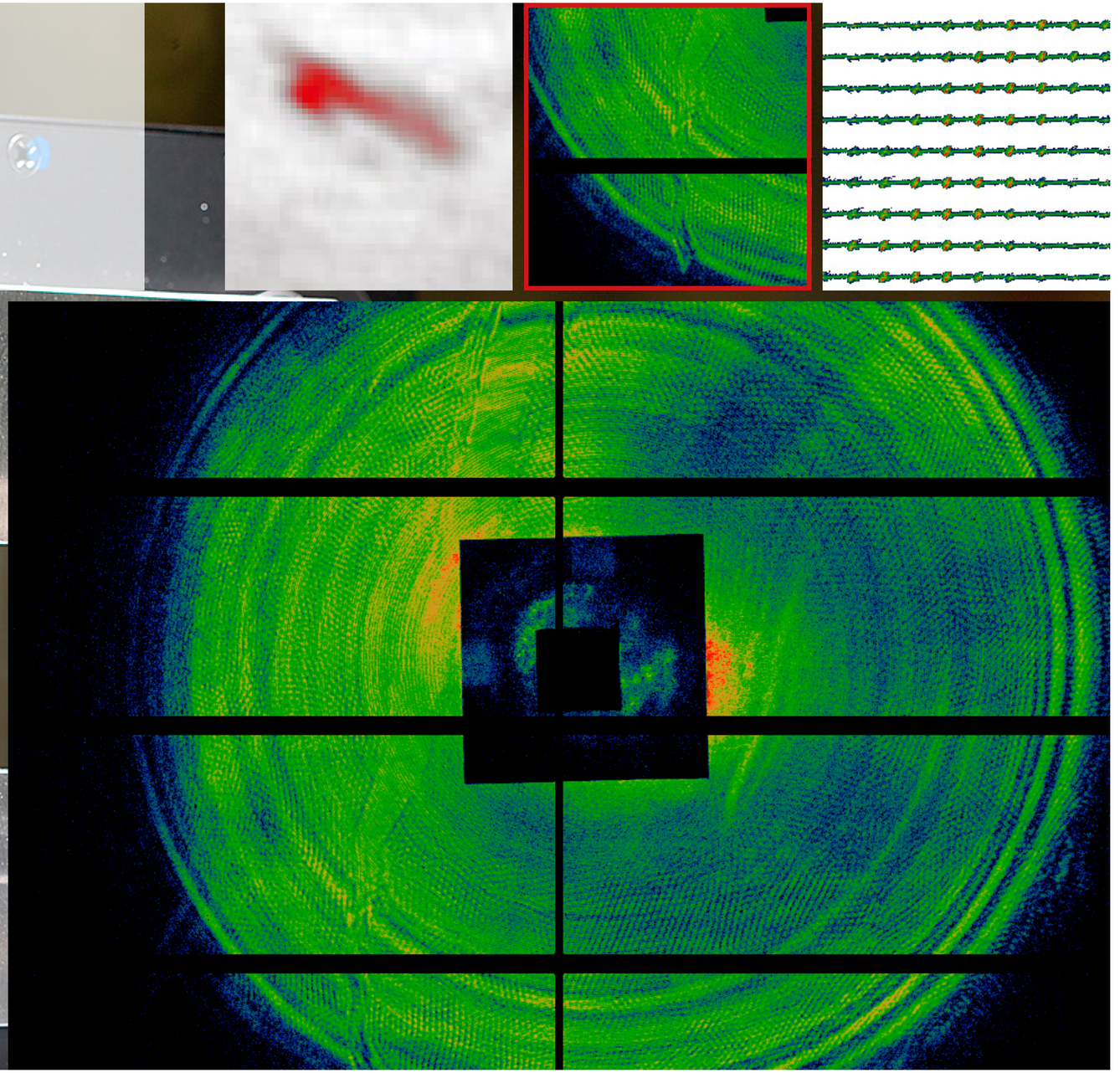
MZP as objective lens behind the sample

direct image on detector

magnification 10 200×
pixel size 16.9 nm

Pilatus 1M detector @ 5 m distance
pixel size 172 µm

Sample:
cracked nano-porous silicon



MZP imaging – our first baby steps

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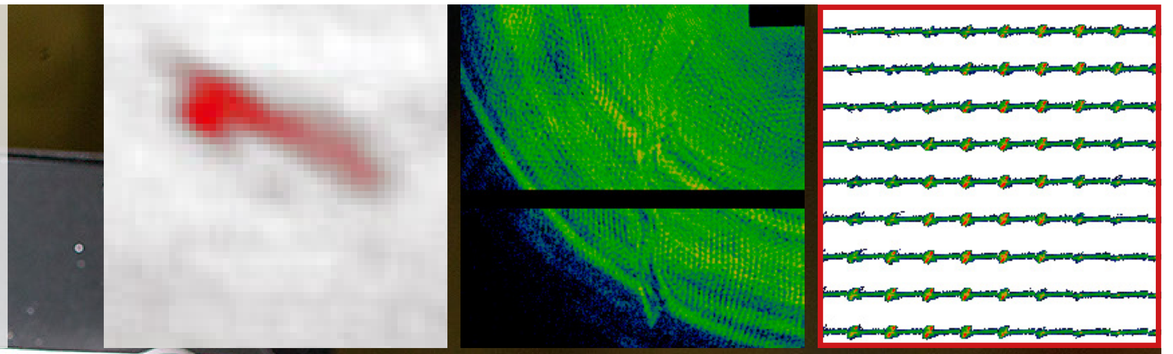
Proof-of-Principle Experiments, 2014 – ongoing:

Holography

Microscopy

Scanning WAXS

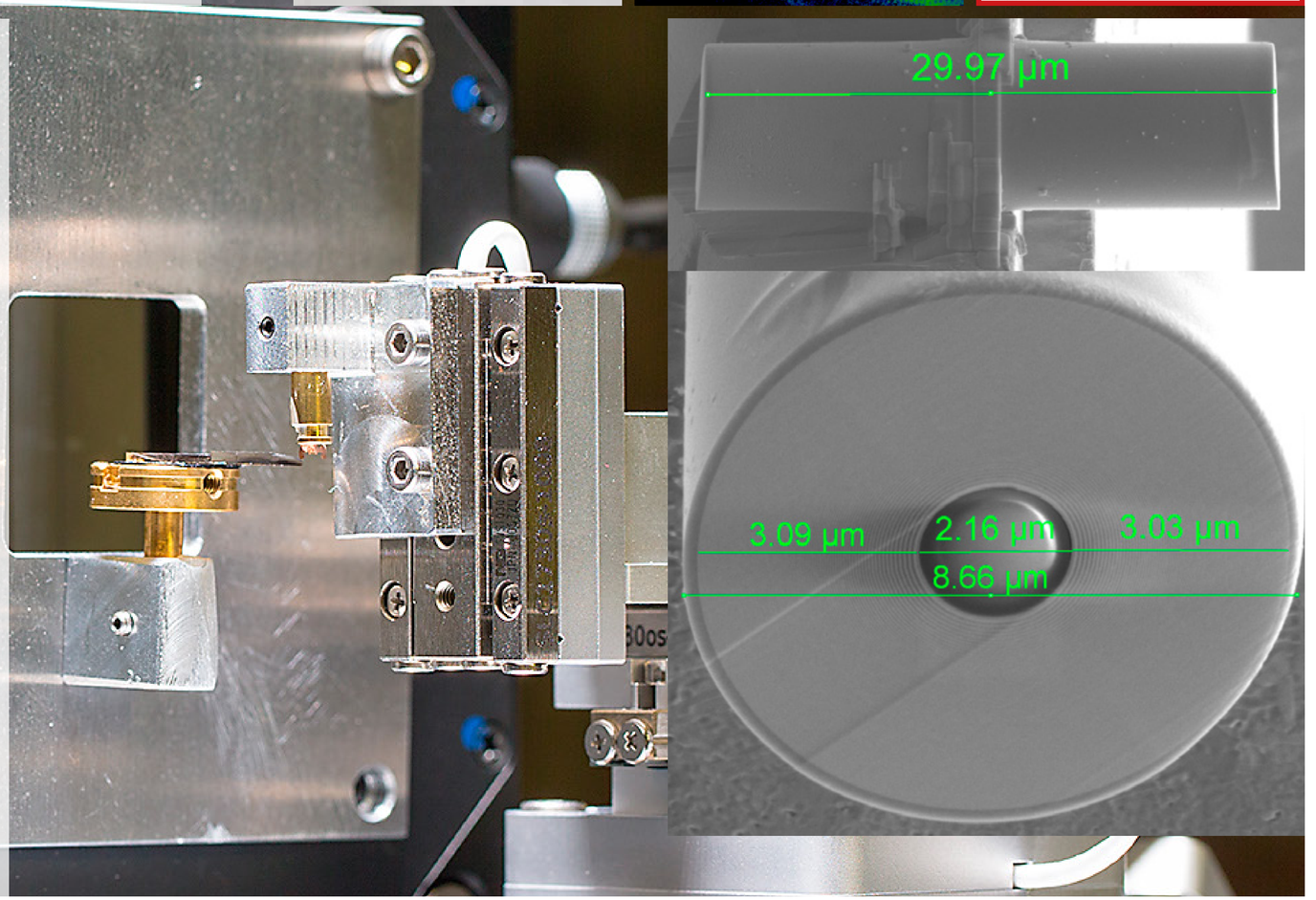


MZP as scanning probe @ ID-31
ESRF's high energy beamline

60 keV

MZP: diameter 8 µm, optical thickness: 30 µm
outermost zones: 10 nm

Sample:
Silicon droplets (crystalls) buried in ZrO_2



MZP imaging – our first baby steps

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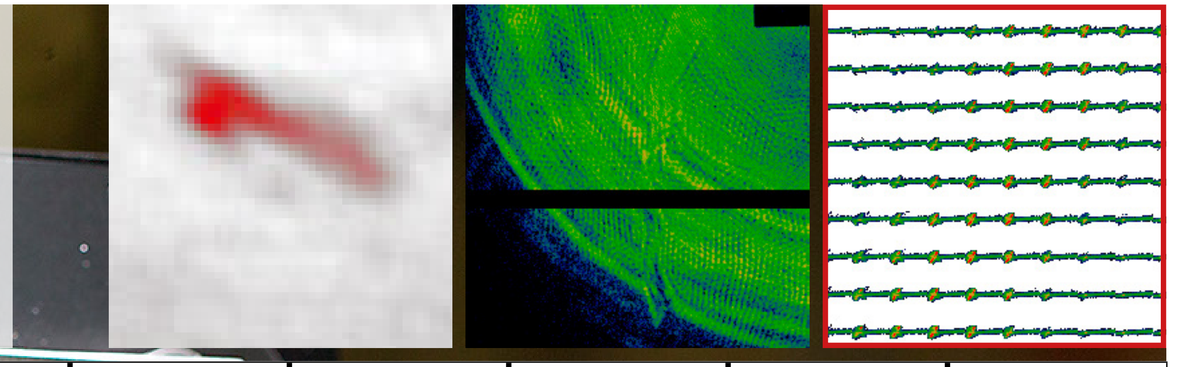
IRP / SFB 755

Proof-of-Principle Experiments, 2014 – ongoing:

Holography

Microscopy

Scanning WAXS



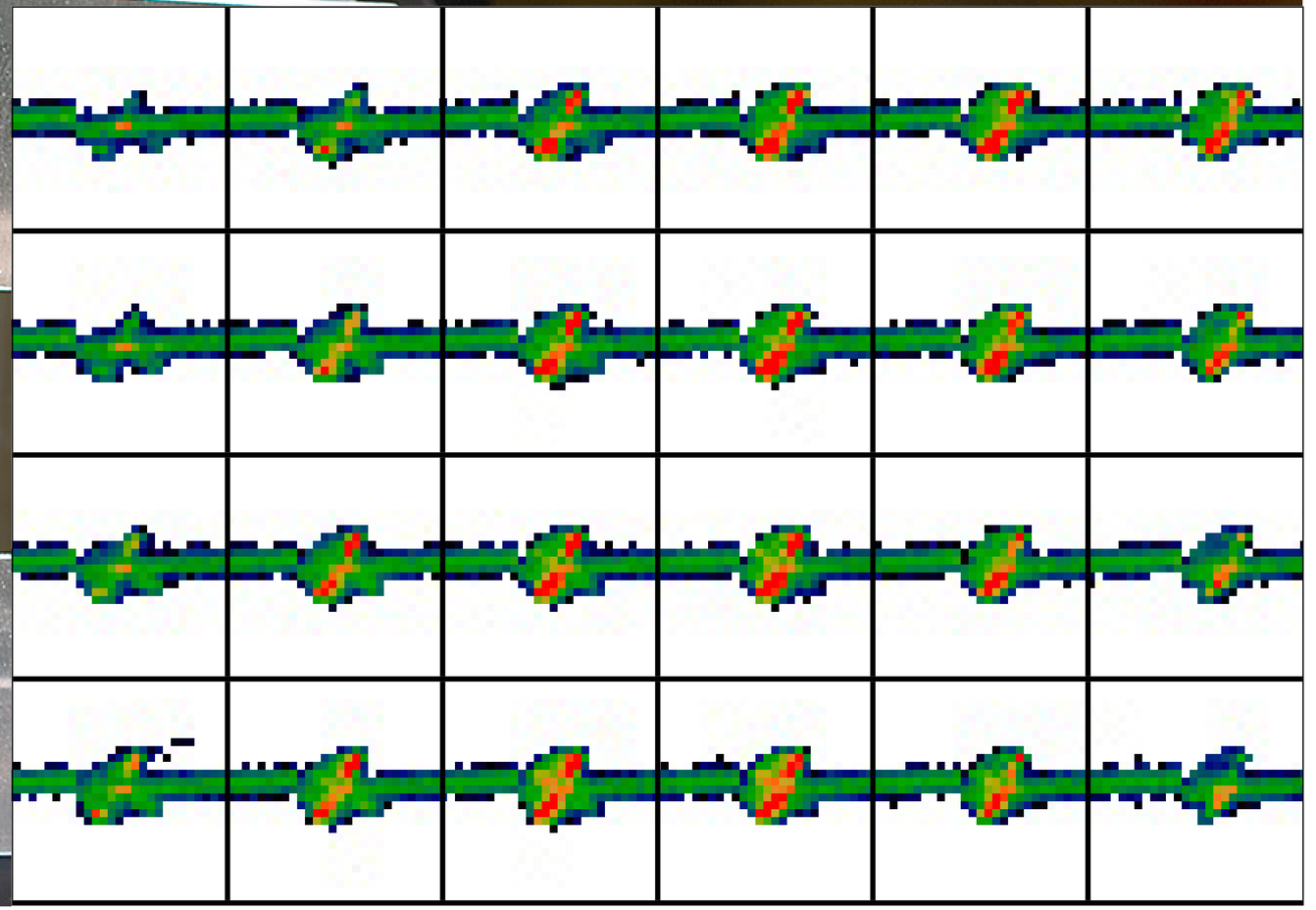
MZP as scanning probe @ ID–31
ESRF's high energy beamline

60 keV

MZP: diameter 8 µm, optical thickness: 30 µm
outermost zones: 10 nm

Sample:
Silicon droplets (crystalls) buried in ZrO_2

shown: Bragg peak at scan positions,
scan step size: 50 nm



MZP imaging – our first baby steps

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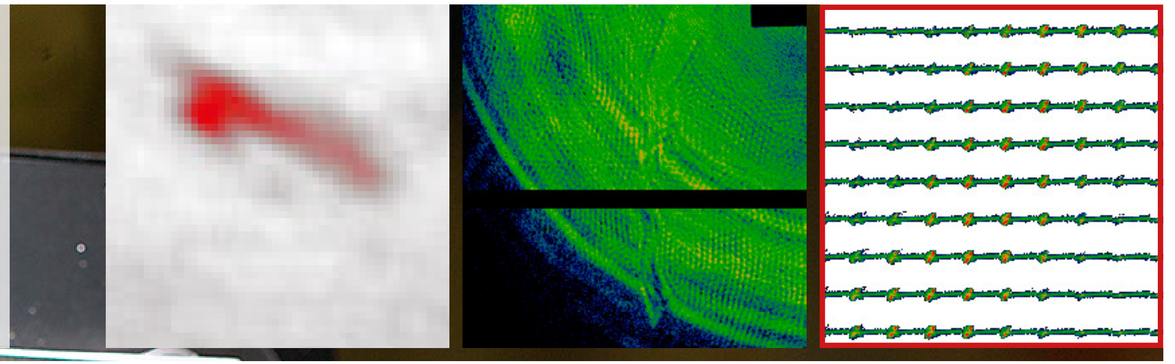
Proof-of-Principle Experiments, 2014 – ongoing:

Holography

Microscopy

Scanning WAXS



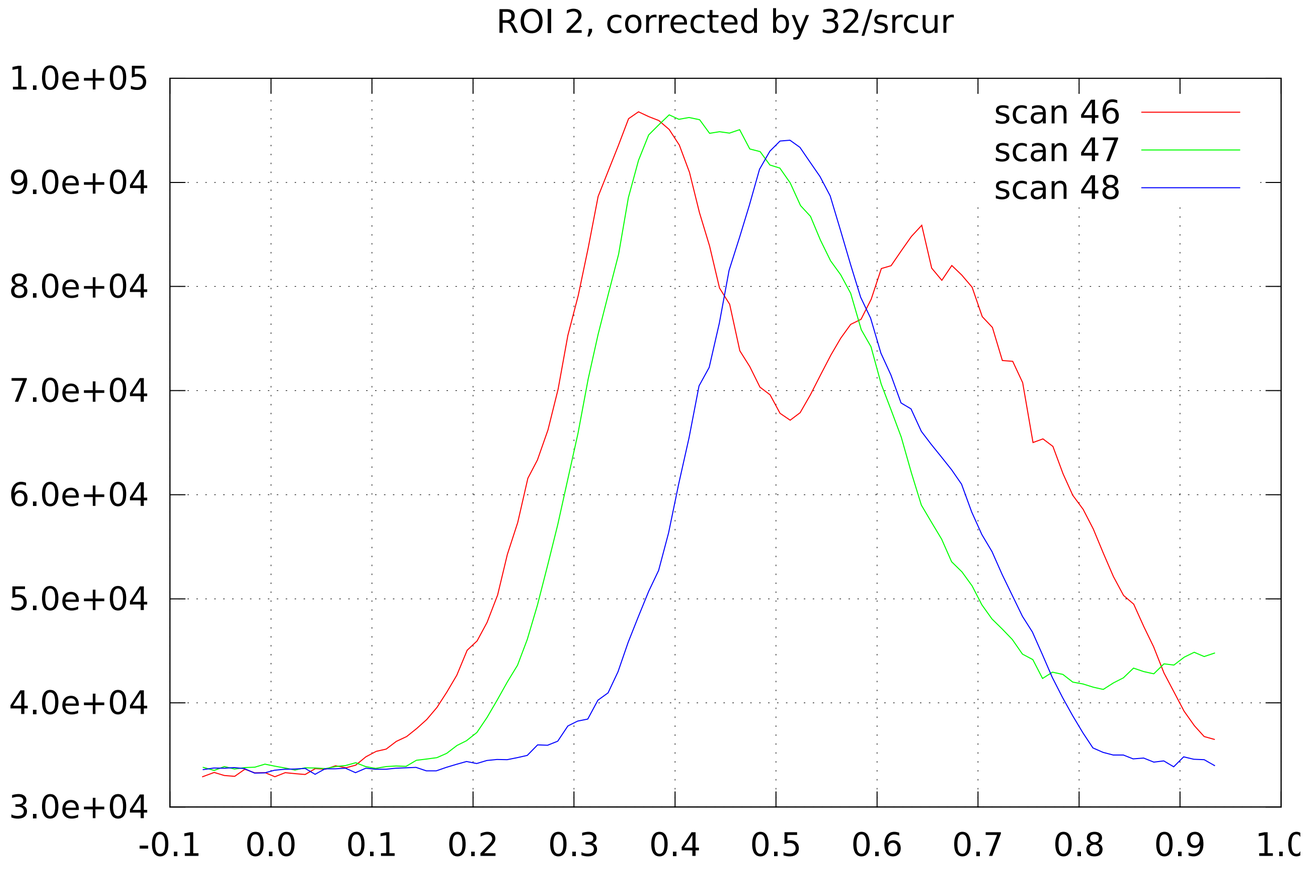
MZP as scanning probe @ ID–31
ESRF's high energy beamline

60 keV

MZP: diameter 8 µm, optical thickness: 30 µm
outermost zones: 10 nm

Sample:
Silicon droplets (crystalls) buried in ZrO_2

shown: Bragg peak intensity, horizontal line scans,
scan step size: 10 nm



MZP imaging – our first baby steps

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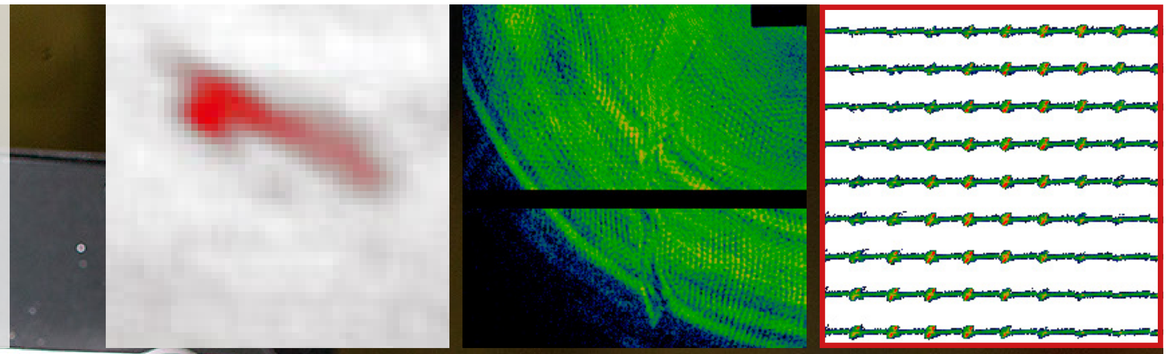
Proof-of-Principle Experiments, 2014 – ongoing:

Holography

Microscopy

Scanning WAXS



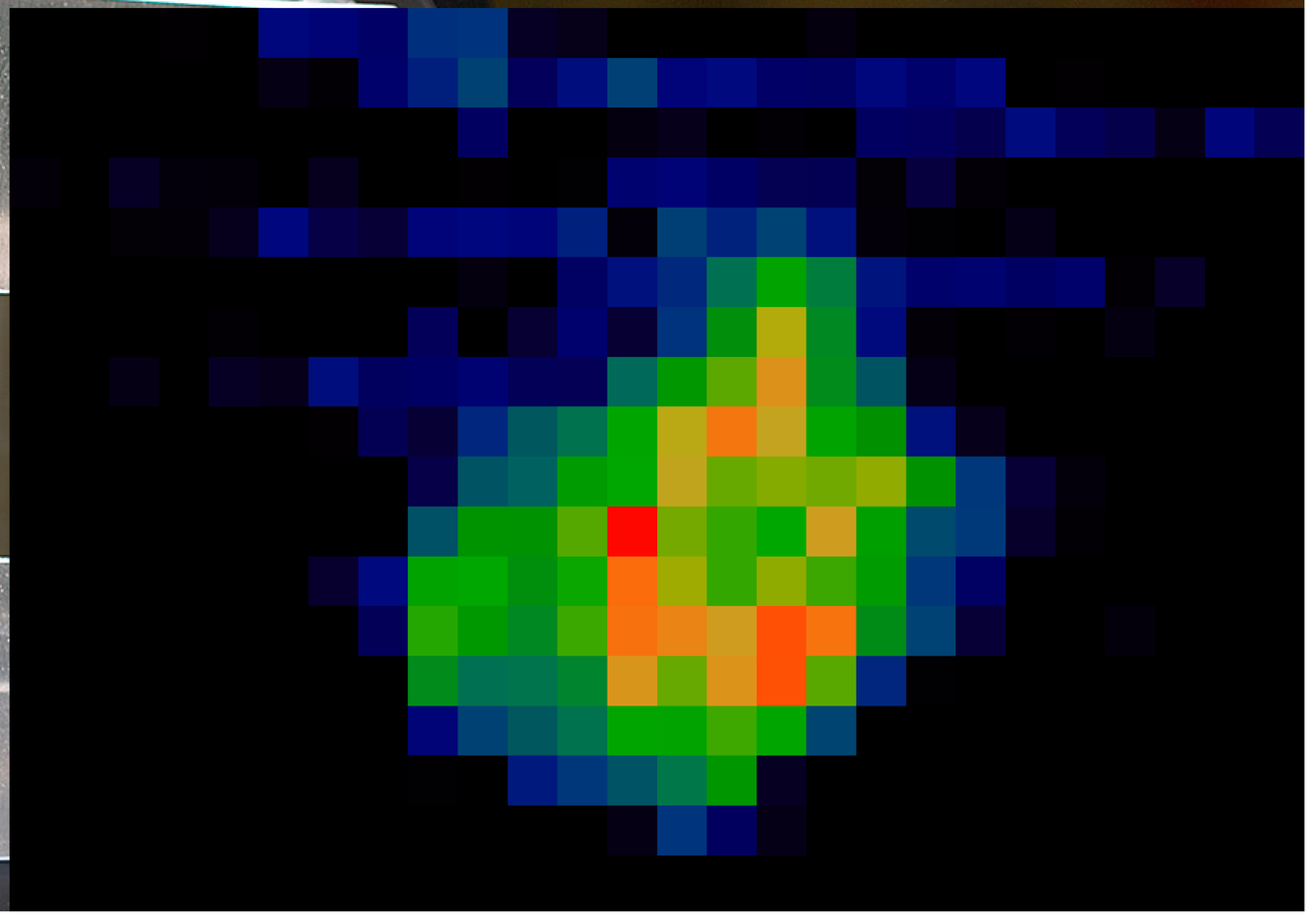
MZP as scanning probe @ ID–31
ESRF's high energy beamline

100 keV

MZP: diameter 8 µm, optical thickness: 30 µm
outermost zones: 10 nm

Sample:
Silicon droplets (crystalls) buried in ZrO_2

shown: Bragg peak at scan positions,
scan step size: 80 nm



Hard X-Ray MZP-Imaging at GINIX – Summary

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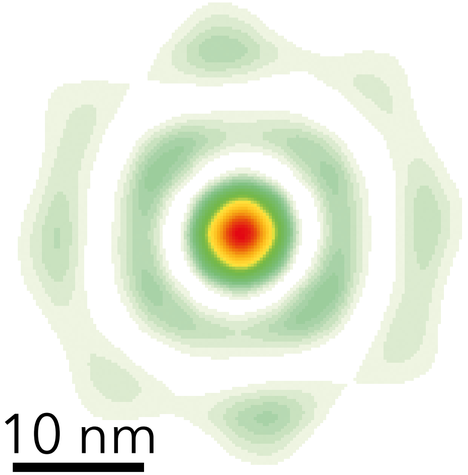
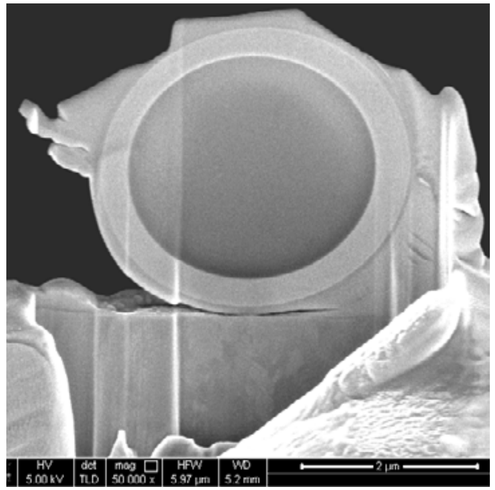
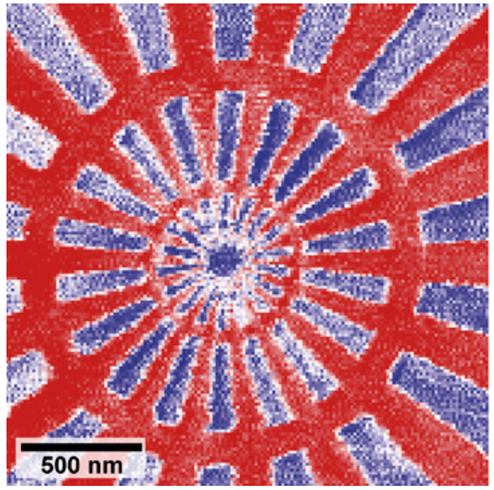
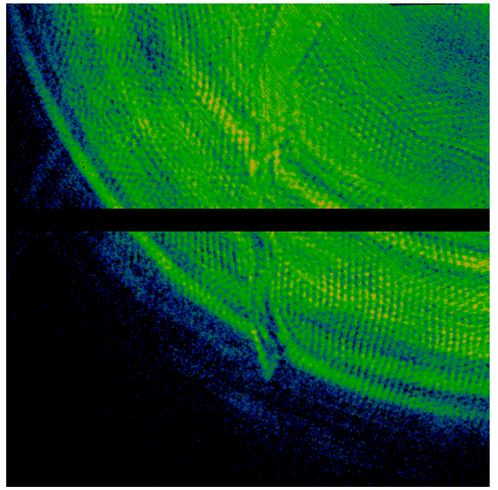
IRP / SFB 755

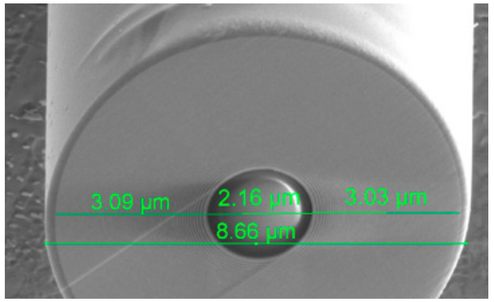
2012	2013	2014	2015	2016
W / Si	→ W / ZrO_2	→ Ta_2O_5 / ZrO_2	Ta_2O_5 / ZrO_2	Ta_2O_5 / ZrO_2
slow deposition		→ **fast deposition**	→ reproducible,	→ 30 µm long
droplets	→ **no droplets**		→ **15 µm aperture**	→ aspect ratio > 10^4
f = 50 µm E = 7.9 keV	→ 250 µm → 13.8 keV	→ 470 µm → 18.0 keV	→ 500 µm E = 8.0 keV	f = 4.0 … 6.6 mm → 60 … **100 keV**
bad W wire	→ glass fibre	→ first imaging	→ tapered glass fibre	

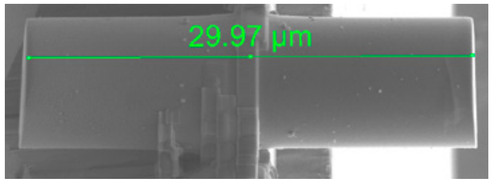












Hard X-Ray MZP-Imaging at GINIX – Summary

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PLD + FIB = Jelly Roll