

Towards multi-order hard x-ray imaging with multilayer zone plates



M. Osterhoff¹, R.N. Wilke¹, J. Wallentin¹, C. Eberl², F. Döring², H.U. Krebs², M. Sprung³, T. Salditt¹

¹ Institut für Röntgenphysik, Georg-August-Universität Göttingen

² Institut für Materialphysik, Georg-August-Universität Göttingen

³ DESY Photon Science, Hamburg



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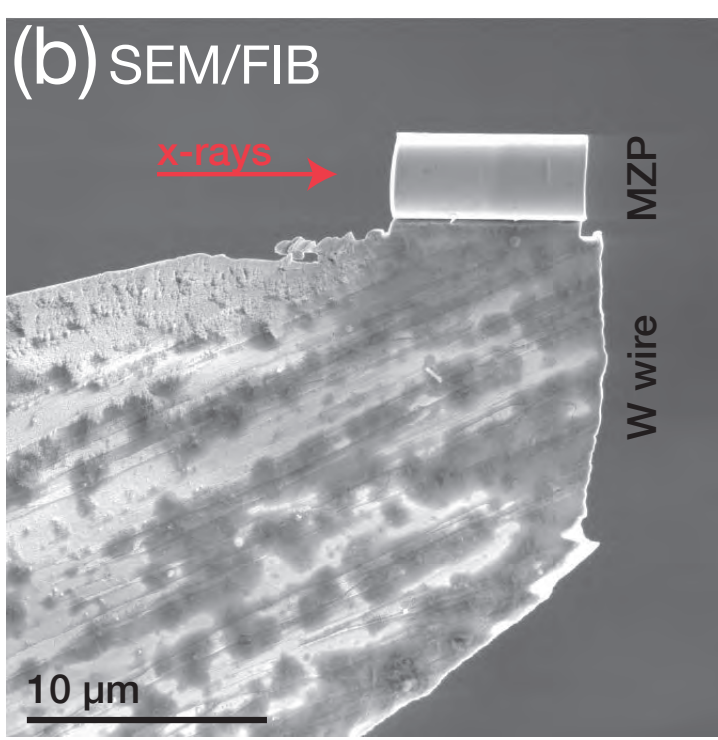
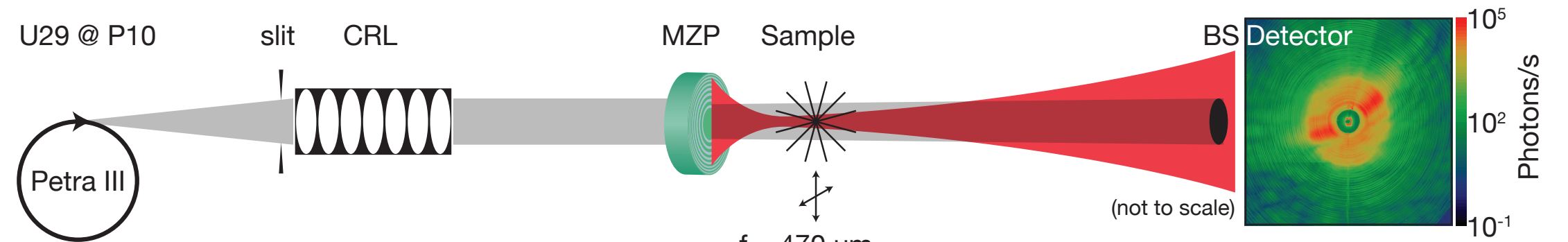
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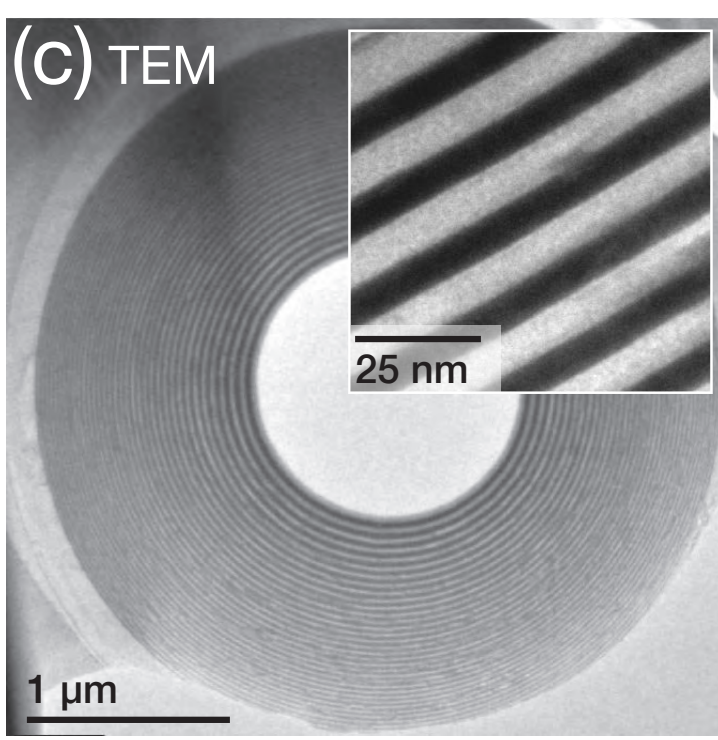
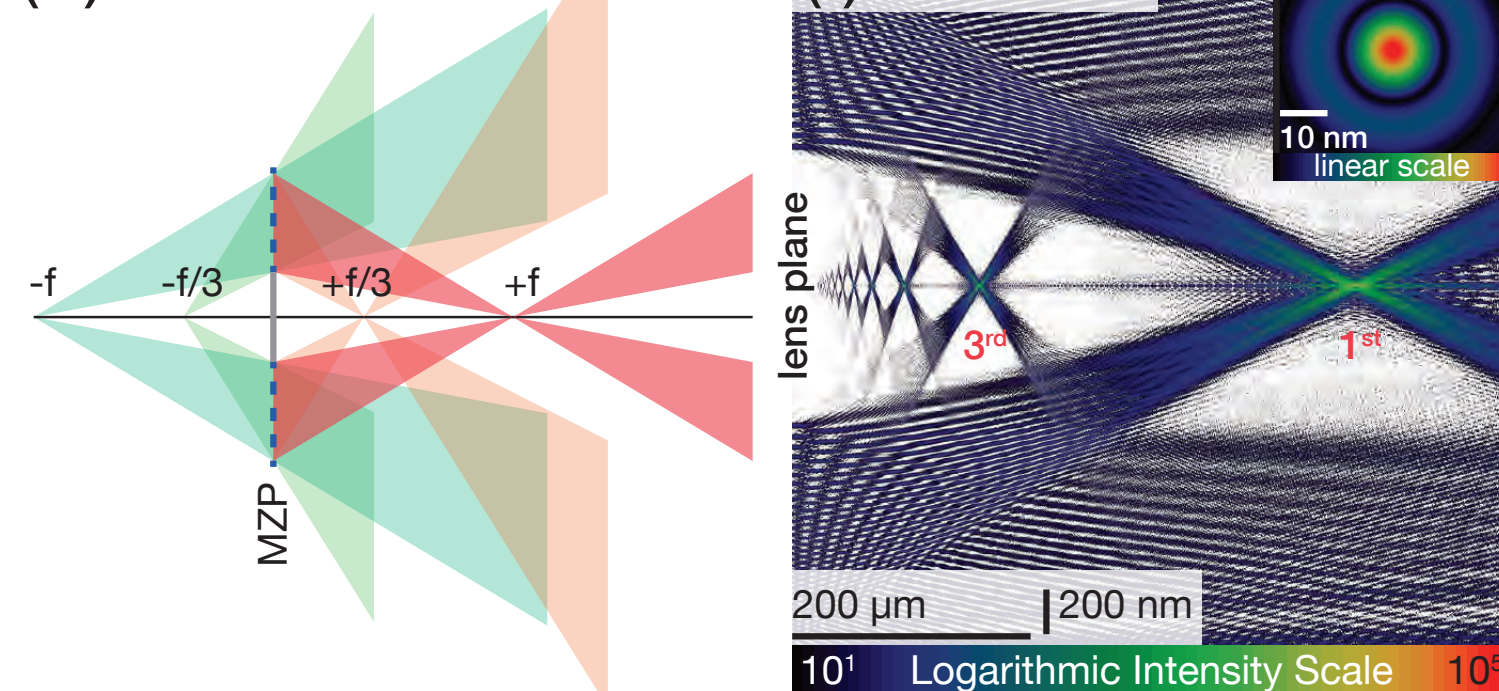
Multi-Order Zone Plates

- high-resolution Multilayer Zone Plate (MZIP) focusing down to 5 ... 10 nm (2D, $7.9 < E < 18$ keV); note that pre-focusing adapts the coherence length of $> 50 \mu\text{m}$ to the lens size of $< 5 \mu\text{m}$
- lens fabrication with Focused Ion Beam
- overview and detail of MZIP zone quality with TEM
- diffraction optics yield a multitude of foci:
can we image without an Order Sorting Aperture?
- idealised detector image to show than one can disentangle 1st from 3rd order (but +/- still remains)
- simulation of intensity along optical axis, showing +1st and +3rd order focus and higher orders; compare to d
- typical detector image; compare to e

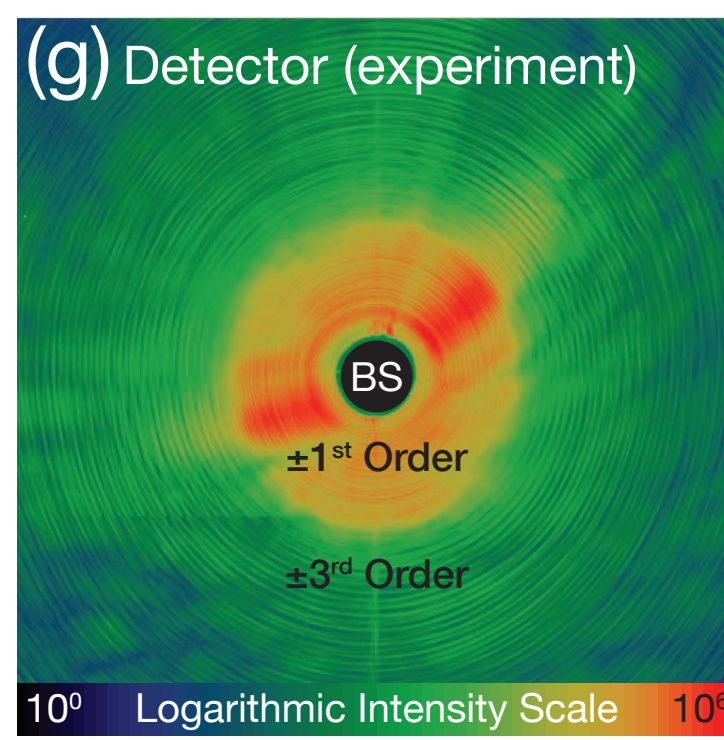
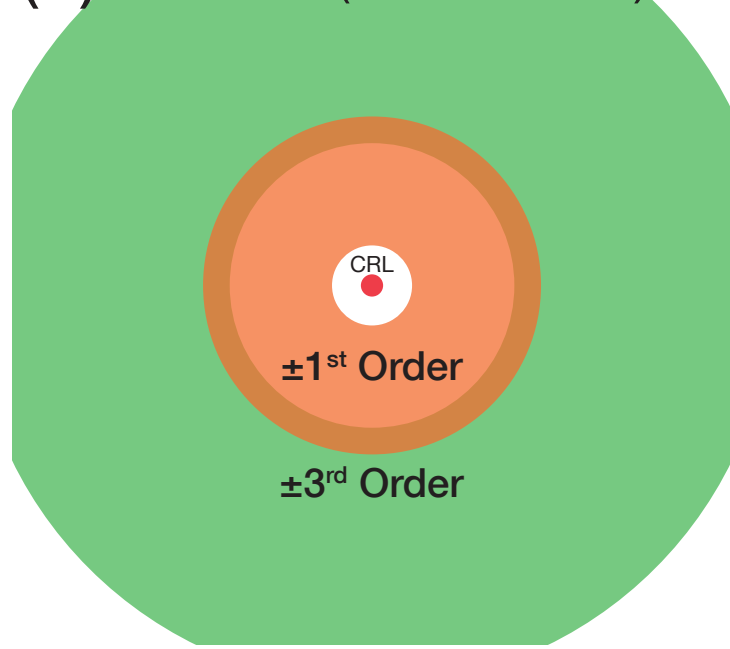
(a) Experimental Setup



(d) Orders



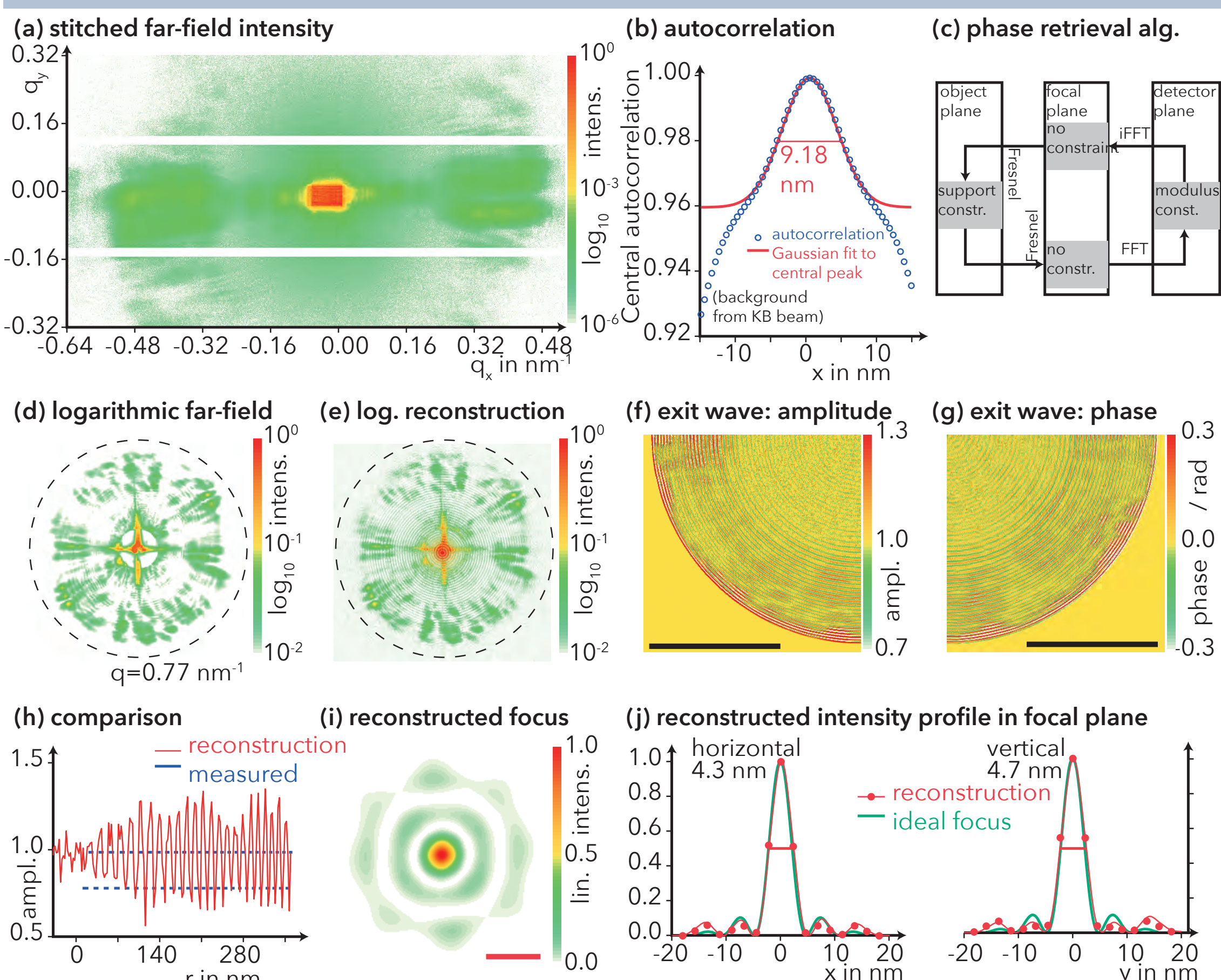
(e) Detector (ideal sketch)



Determining the focal spot size

- far-field intensities (Pilatus detector, horizontally stitched); its auto-correlation yields ...
- a "typical length scale" of the diffraction strength; full width at half maximum of 9.18 nm corresponds to focus structures of 4.6 nm
- three-planes phase reconstruction scheme [H.M. Quiney et al., Nature Physics 2, 2006]
- log-scaled colour coded measured intensity (sCMOS detector)
- log-scaled colour coded reconstructed intensity
- reconstructed amplitude in lense plane,
- reconstructed phase in lense plane;
- due to beam stop (necessary to protect detector from 0th order), zones inside central core appear
- reconstructed zones agree with measured layers
- reconstructed intensity in focal plane (interpolated)
- horizontal and vertical line profiles of i yield FWHMs of 4.3 nm \times 4.7 nm, the so-far smallest 2D hard x-ray focus

experiment at 7.9 keV; focal length 50 μm ; W / Si
similar results at 13.8 keV; focal length 250 μm ; W / ZrO₂



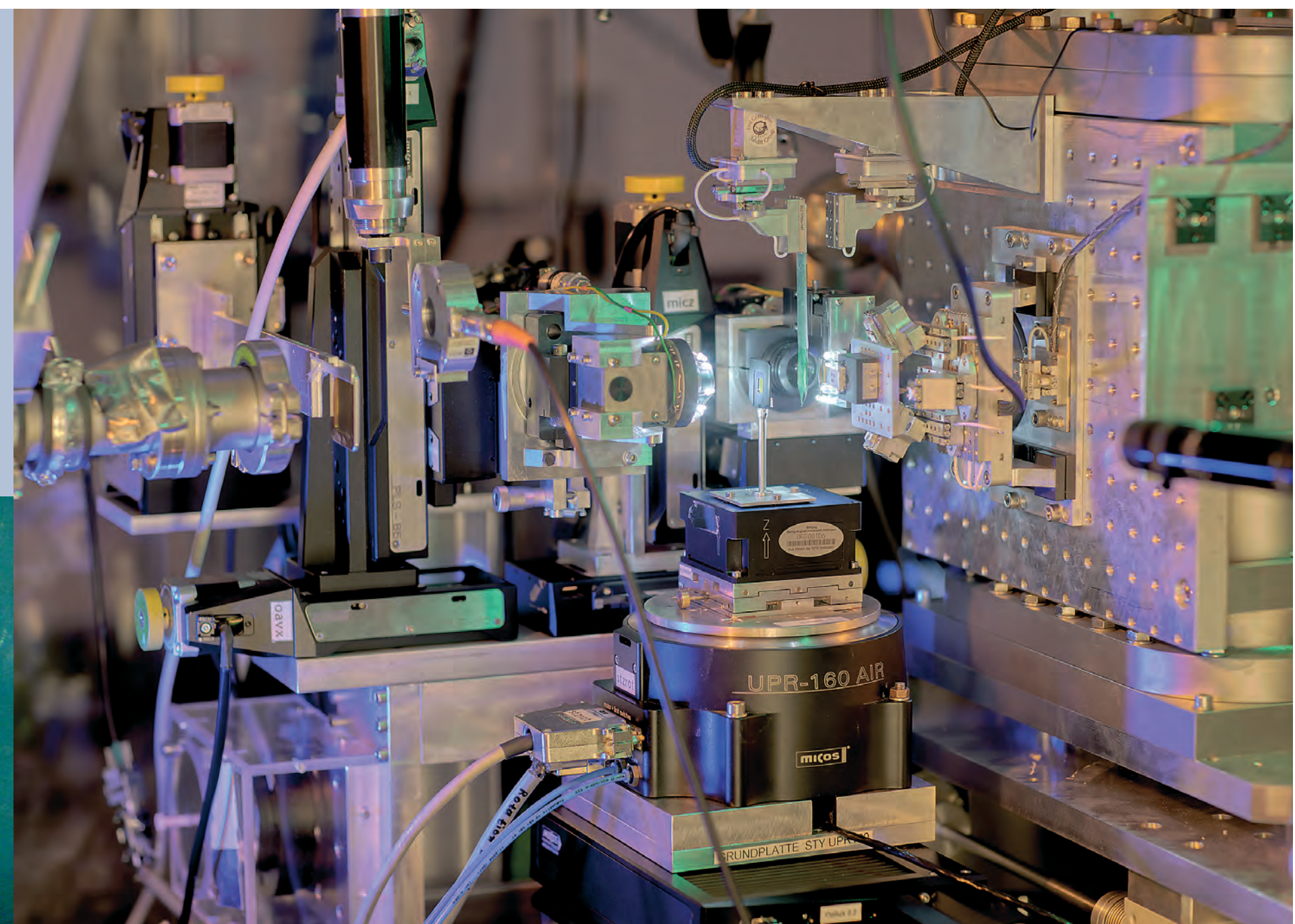
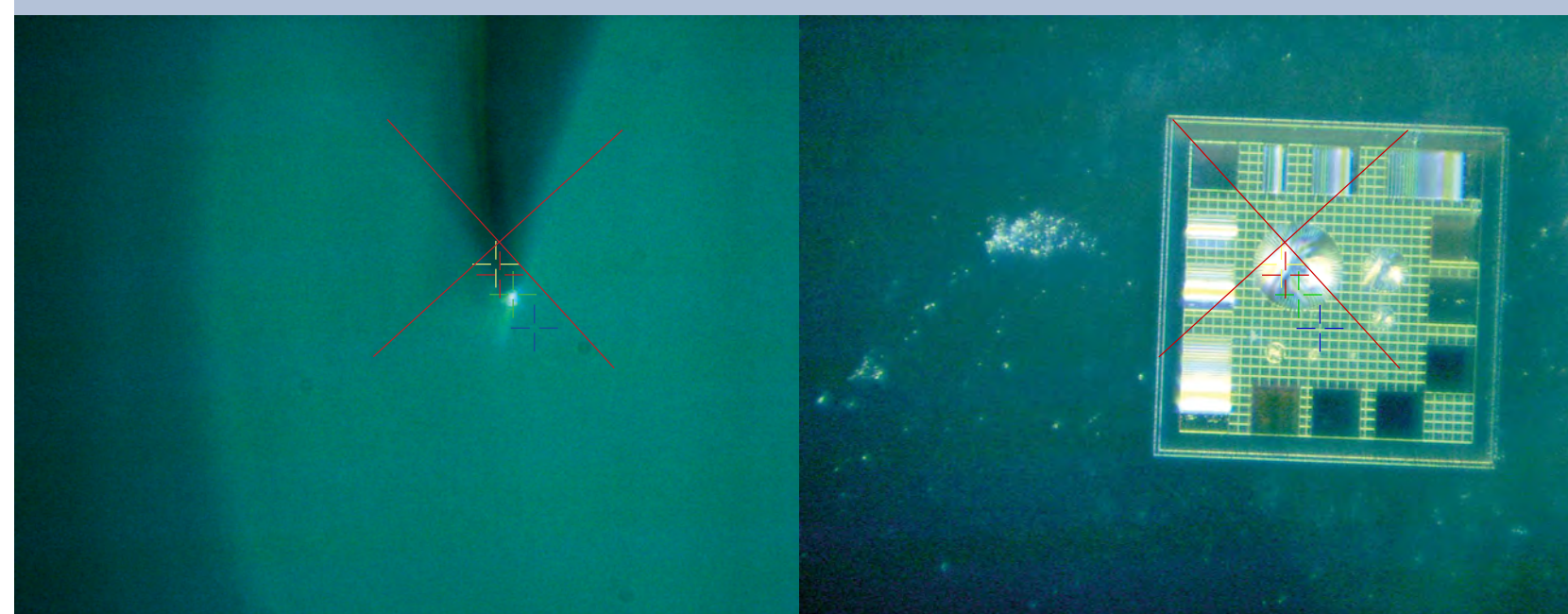
Imaging Experiments

right: GINIX set-up at P10 beamline, PETRA III

bottom: alignment of lens (left) and sample (right) is facilitated by optical in-line microscope

Experiments so far:

Energy:	7.9 keV	13.8 keV	18.0 keV
Focal length:	50 μm	250 μm	470 μm
Materials:	W / Si	W / ZrO ₂	Ta ₂ O ₅ / ZrO ₂
Pre-focussing:	KB mirrors, CRL		
Detectors:	Pilatus 300k, sCMOS		

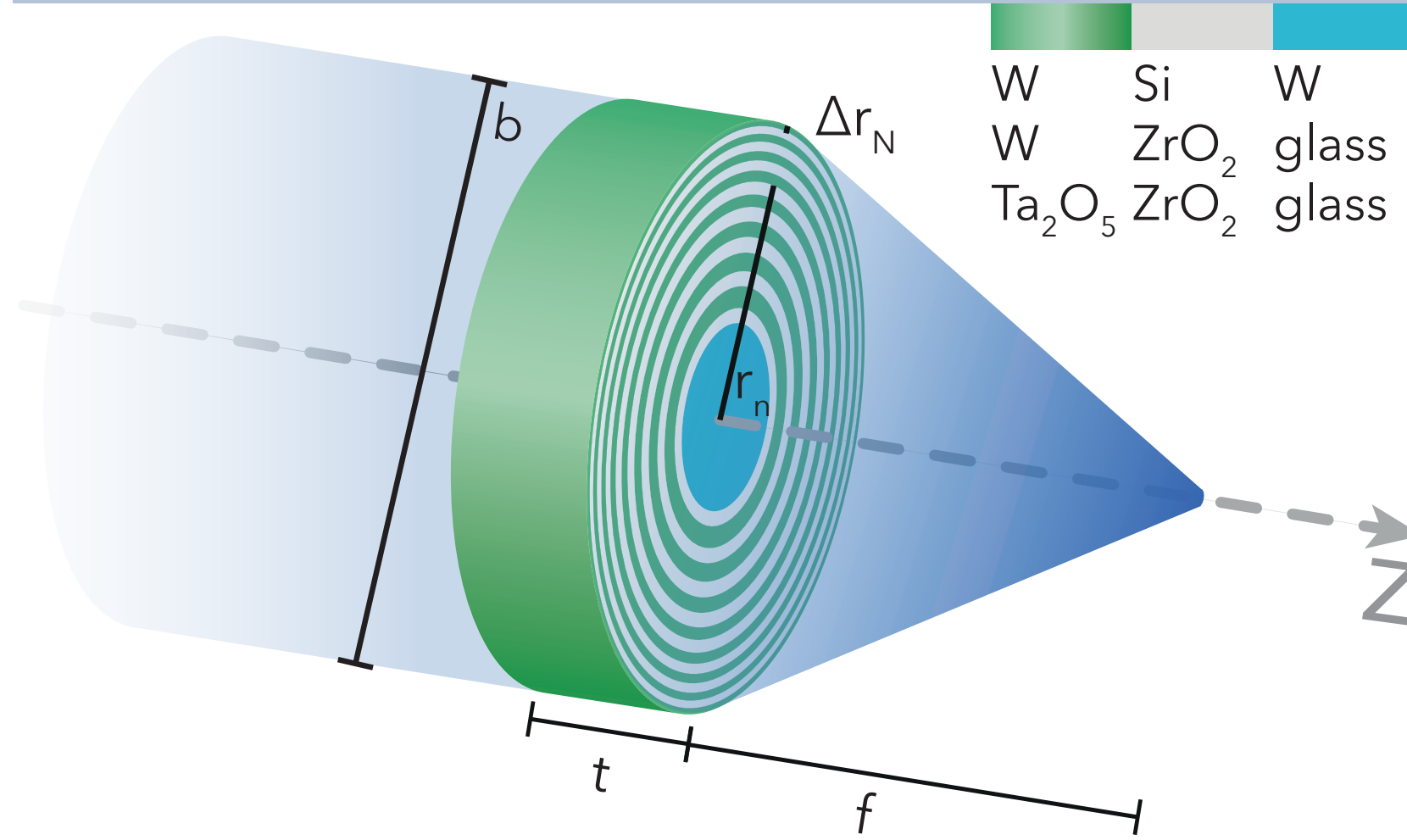


Göttingen MZIP

Central core: thin glass fibre of $800 \text{ nm} \leq \varnothing \leq 2 \mu\text{m}$; so far lenses with outer radius of $r_N \leq 7 \mu\text{m}$ fabricated by Pulsed Laser Deposition of alternating layers, optical thin and optical thick material, e.g. W / Si

Best results found for material combination Ta₂O₅ and ZrO₂ found:

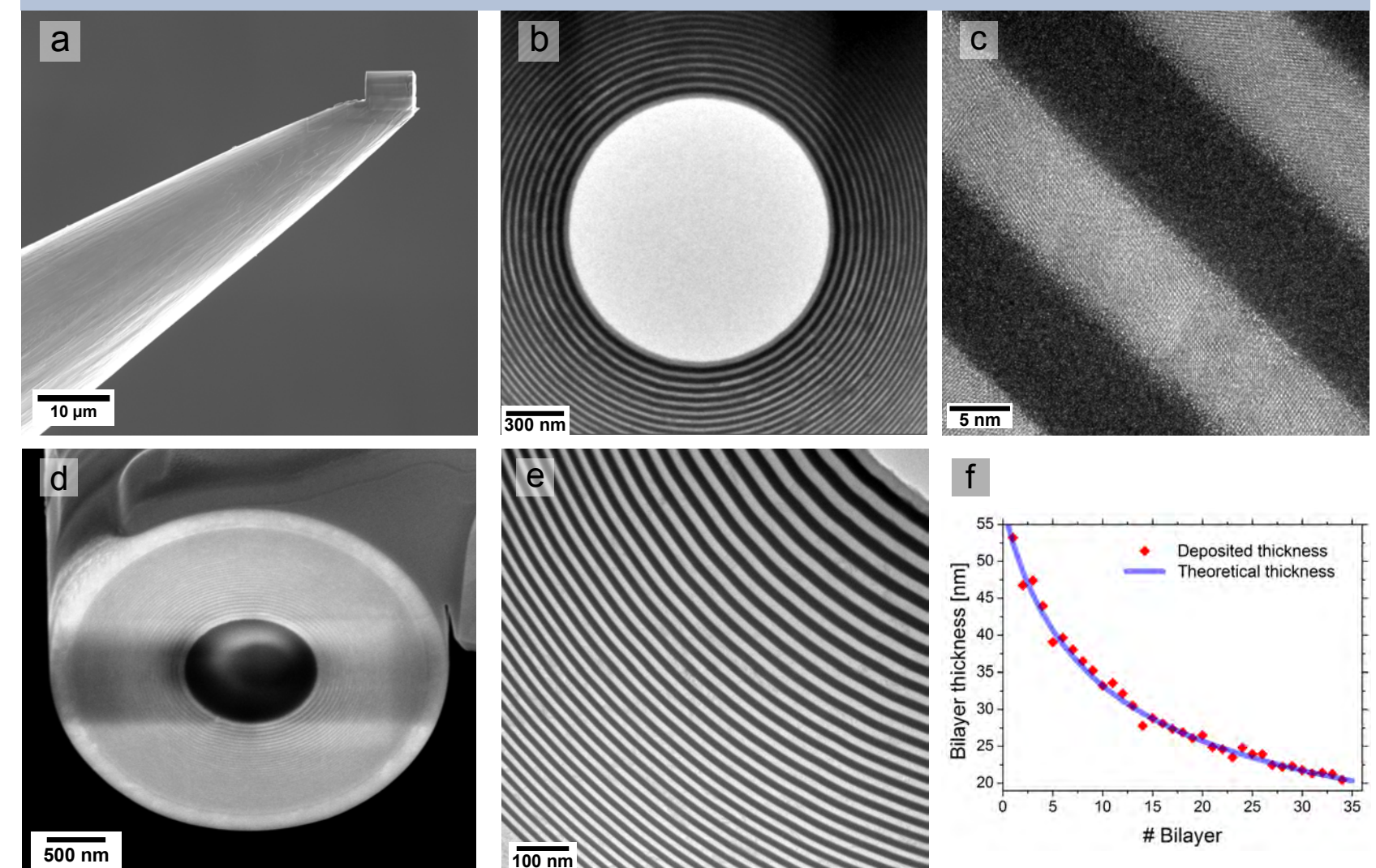
- very constant and high PLD rates during deposition,
- no droplet formation, opposed to Si,
- very clean and thin layers possible, up to 3 nm and below,
- *cumulative smoothing* residual roughness of core,
- focussing efficiency into +1st order: 6.9 % at 18 keV
- in principle, optical length $\gg 10 \mu\text{m}$ possible



MZIP fabrication

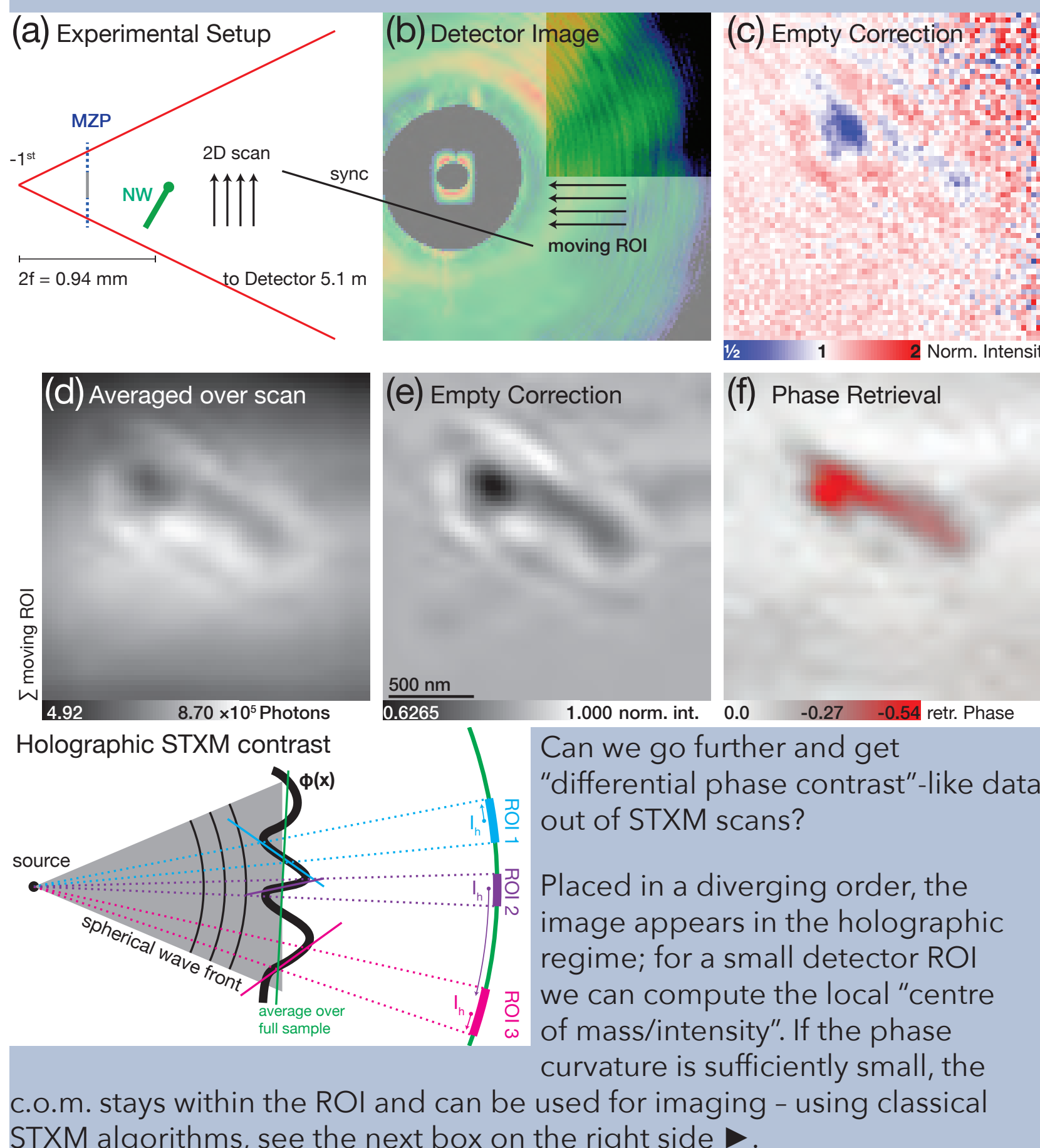
Pulsed Laser Deposition (PLD) is a convenient method to grow alternating layers on a rotating glass fibre

- using Focused Ion Beam (FIB) facility, the lens can be attached to a mount and cut to the desired optical length
- overview TEM image of a lens
- high-resolution view showing 5 nm layers of high surface quality
- final polishing of the MZIP with FIB
- detailed TEM image shows high quality layers, without cumulative roughness
- layer thicknesses closely match the zone plate law



Holography

- 2D scan of semiconductor nano wire ...
- b,c in diverging -1st MZIP order shows holographic images ...
- averaged over 1647 positions, with detector ROI co-moving; no encoder / interferometer positions used yet
- empty corrected image to cancel shadow from beamstop and inhomogeneous illumination
- phase retrieval: holographic reconstruction plus Gerchberg-Saxton



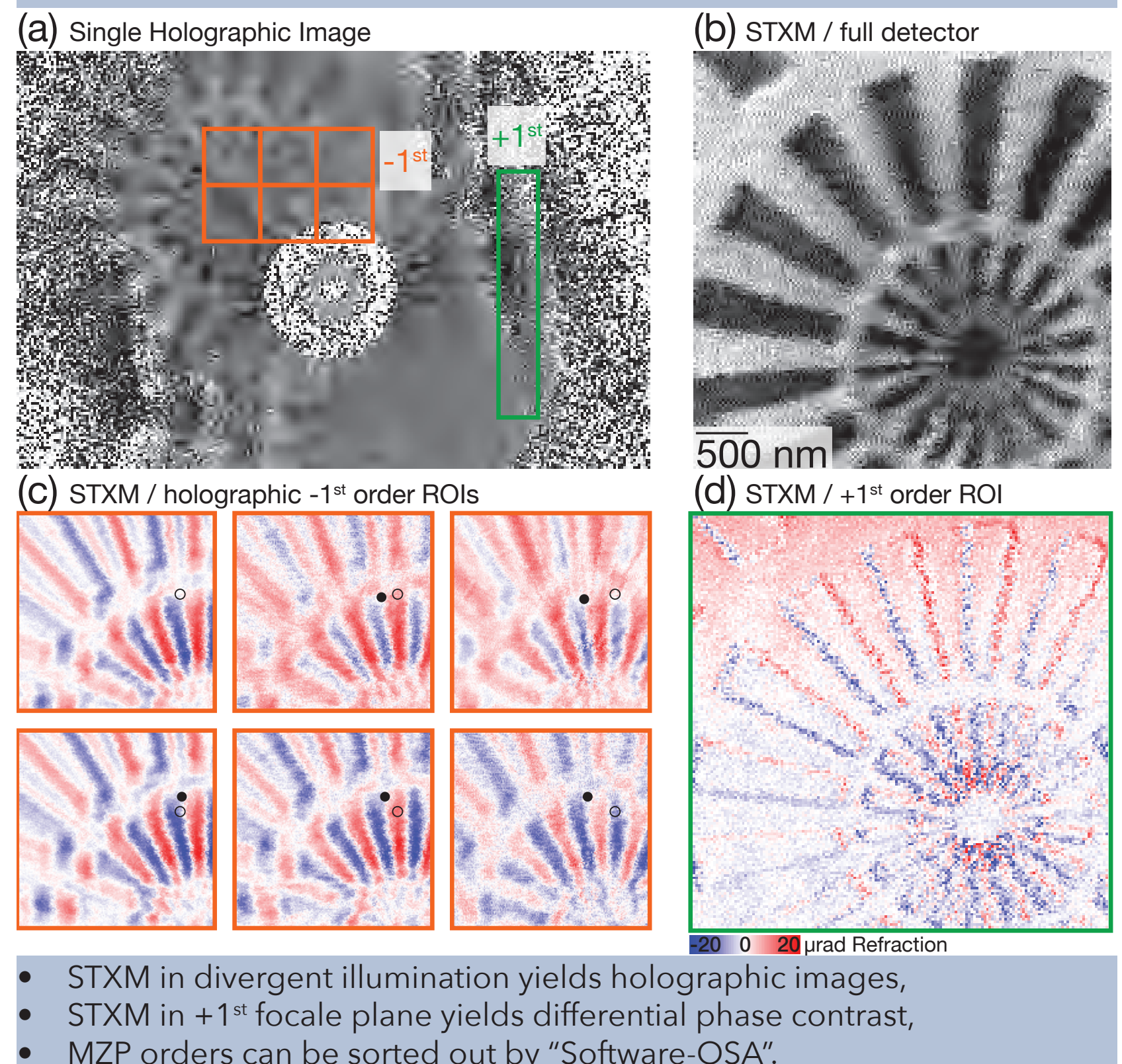
Imaging by scanning

A Siemens star test pattern was scanned laterally in the +1st focal plane. a far-field showing a holographic image of the star; this is attributed to the divergent illumination of the -1st order.

Intensities $I(X,Y)$ are recorded for every scan position (x,y) . Differential phase contrast (horizontal) $I_h(x,y)$:

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

- shows this signal, summed over full detector,
- shows I_h only for orange detector ROIs,
- shows the signal for the green ROI.



References

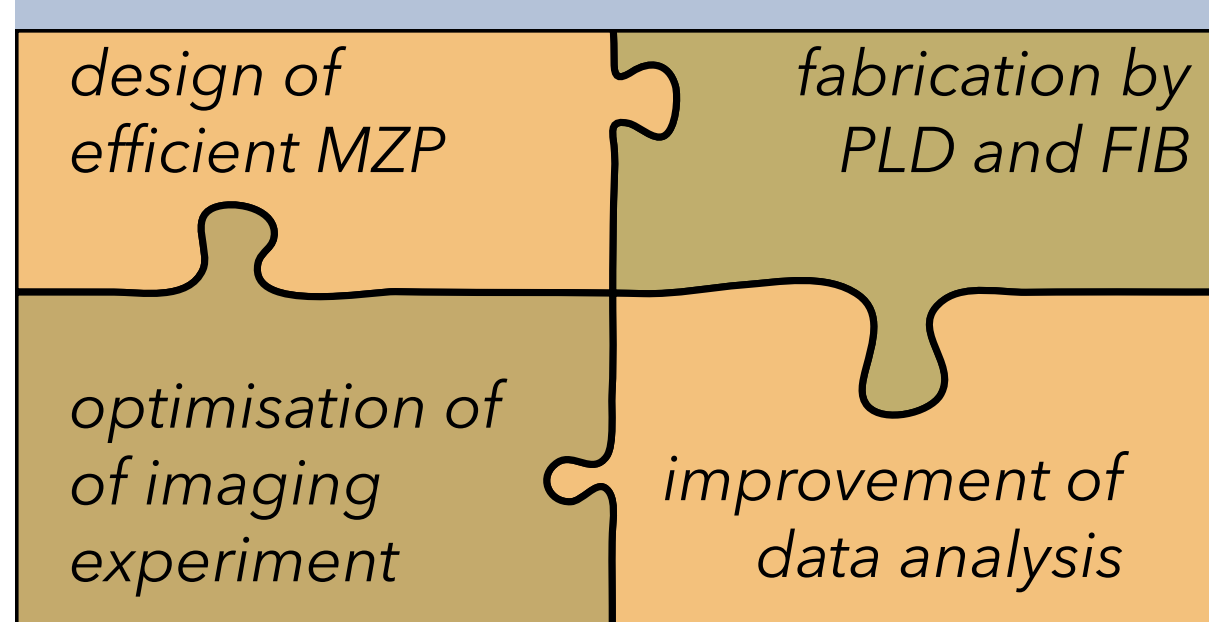
- [Osterhoff 2015] M. Osterhoff, C. Eberl, F. Döring, R.N. Wilke, J. Wallentin, H.U. Krebs, M. Sprung, T. Salditt
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Fabrication of laser deposited high-quality multilayer zone plates for hard x-ray nanofocusing
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Sub-5 nm hard x-ray point focusing by a combined Kirkpatrick-Baez mirror and multilayer zone plate
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A combined Kirkpatrick-Baez mirror and multilayer lens for sub-10 nm x-ray focusing
AIP Advances 2, 012175 (2012)

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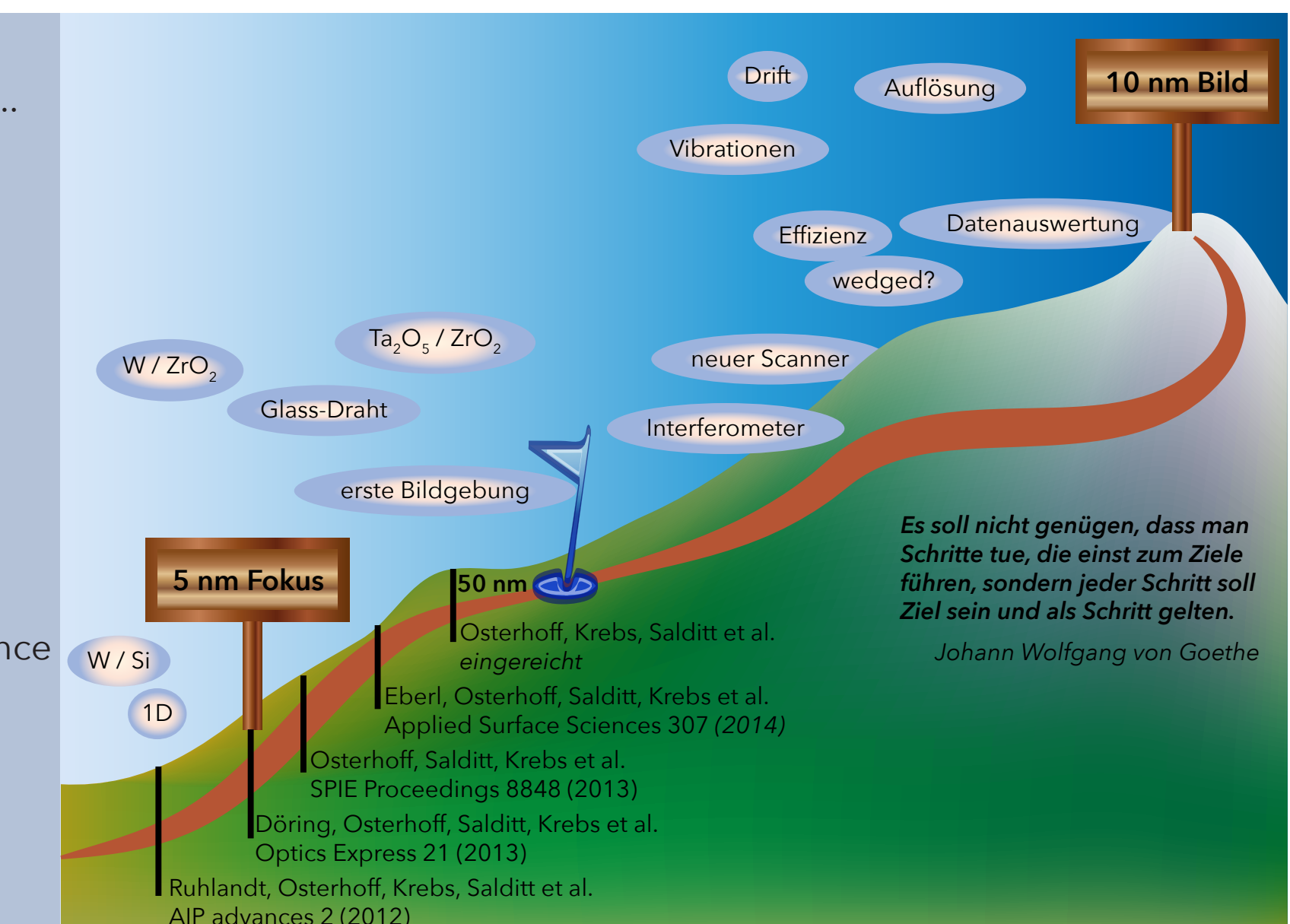
Future

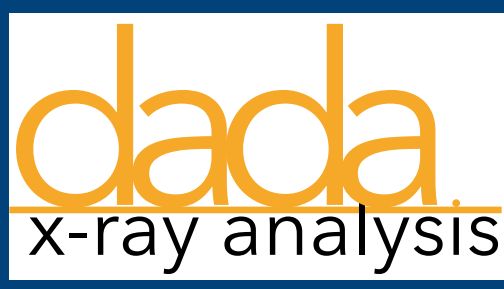
We are envisioning hard x-ray imaging using MZIPs with 10 nm resolution, addressing ...

- drift: interferometric control in commissioning
- vibrations: new sample tower / interferometry
- slow scans: faster piezo scanner in commissioning
- efficiency 6.9%: thicker lenses (already got to 7 μm); optimised layer shape along optical axis, e.g. wedged



- understand and control volume diffraction / waveguiding effects
- check stability and endurance in FEL beam
- use for nano-diffraction, nano-fluorescence, nano-stimulation (e.g. XBIC)



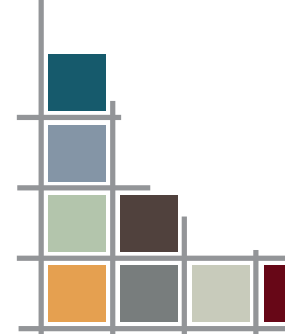


dada: a web-based collaborative x-ray images visualisation and analysis tool



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M. Osterhoff¹, T. Salditt

Institut für Röntgenphysik, Georg-August-Universität Göttingen

¹mosterh1@gwdg.de

Motivation

Challenges as university users of synchrotron / lab sources:

- different detectors / formats / compressions (changes even between beamtimes)
- different folder structure / file name formats (the famous %04d with varying 0's and 4's, **"the mess"**)
- Bachelor students come and go
- their need: "just show me that image!"
- often heard on the floor: "I got **this algorithm** from her, but the one from him is **newer**, but then I included **this piece of code** taken from someone else to open that image, but now I forgot the **value of alpha**, and **tomorrow I have to print my thesis.**"

Our idea

- centralised, unified **access** to (2D, x-ray detector) images
- standardised **algorithms**
- UI: **web-GUI**, Matlab **interface**, {C, C++, Python} libraries possible
- **no local copy** of data
- use **central** cluster
- **cached results** / **archive** by URL, ID
- **control** and **view** from *anywhere*, i.e., the internet
- **https** and **authentication**, fully transparent **proxy**



result:
data, plot

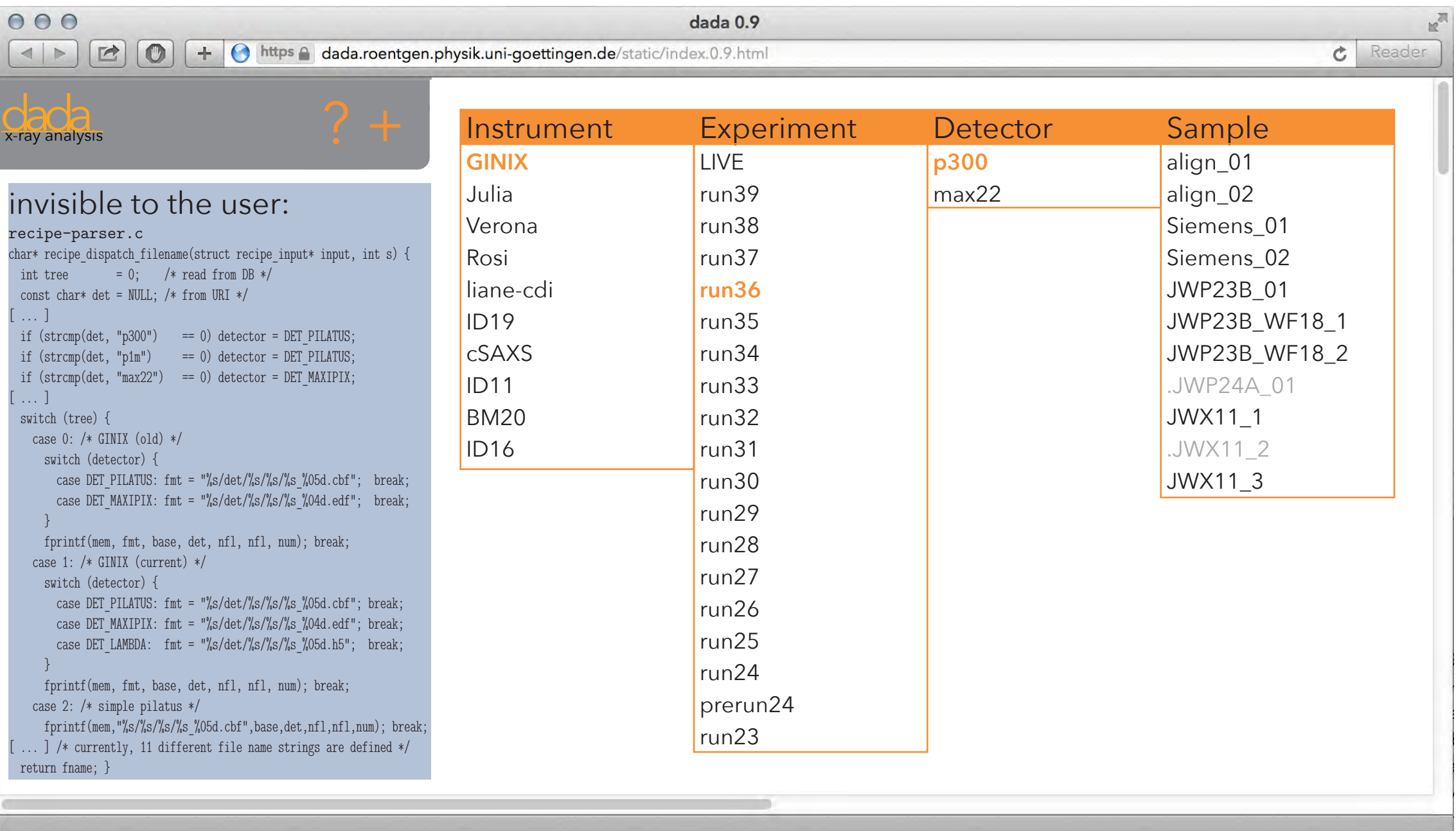


database:
- beamtime,
- detectors,
- formats

web GUI,
library



storage



dada

data daemon:

- database of instruments, experiments, used detectors
- one implementation of folder structure / file names
- web-based GUI, export to PDF, import from Matlab, other libraries planned
- basic tasks:
find that image!
show that image!
analyse those images!

Status

Detectors:

- Pilatus (cbf, edf, tiff)
- Maxipix (edf)
- FReLoN (edf)
- certain TIFFs
- certain HDF5s

SPEC scans (currently, 1D)

Single image:

- ROI, binning, summing, divide, subtract, movie, lin/log, colourbar, line profiles, export to png/pdf/Matlab

Composite and STXM

- darkfield, differential phase

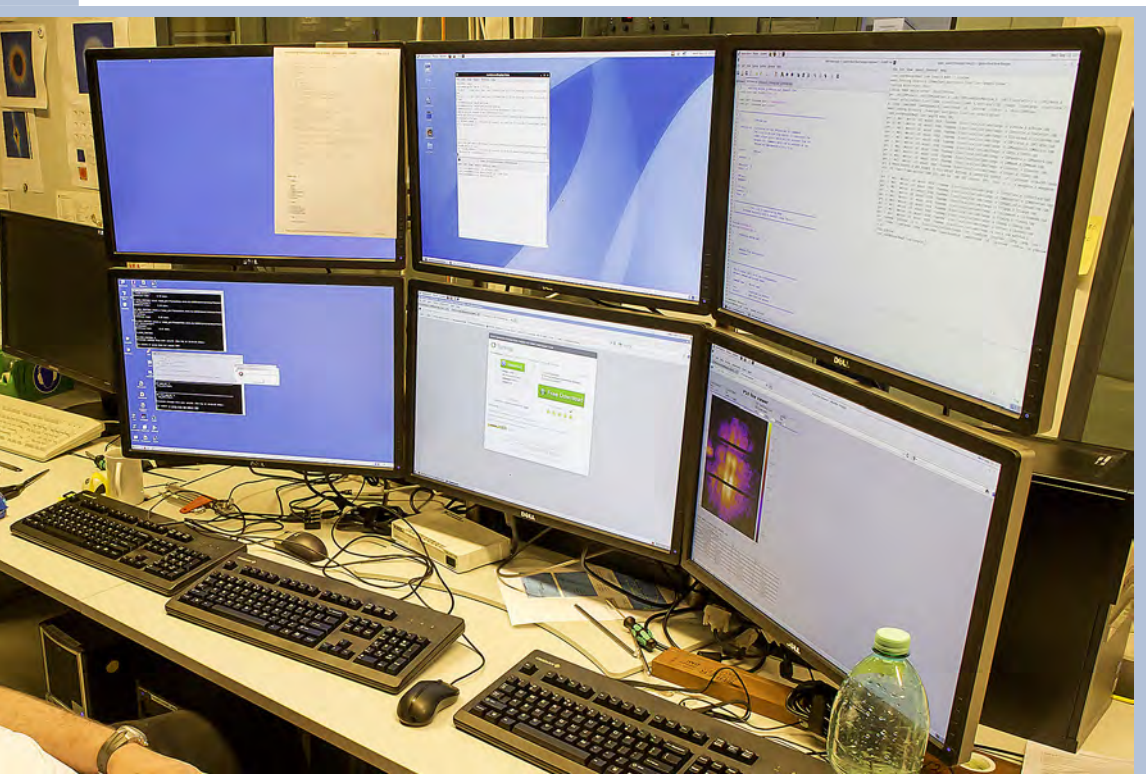
At the beamline

GINIX set-up @ P10 beamline

Control software: SPEC

Feedback from dada:

- counting on 2D detectors,
- centre-of-mass,
- peak width



Online **analysis**:

- print-outs including counting time and motor positions
- STXM measurements live from SPEC
- composite images (2D array of 2D images)
- clever combinations of ROI/binning/composite

Try it!

If you can see this text, the tablet is obviously absent.

Please visit



<http://dada.k-raum.org>
for a small demo.

Plans

Detectors:

- advanced HDF5 routines, fluorescence detectors

2D SPEC scans

Single image:

- radial / azimuthal profiles, fluorescence maps

Composite and STXM

- more contrasts, combine with in-line mic

Behind the curtains

C back-end, lighttpd for HTTPS, authentication and proxy web-GUI let's you browse images and adjust parameters

URI defines "module" (show, scan, stxm etc) and experiment / detector / sample / image number

query string defines modifiers:

lin/log scale, colour map, ROI, STXM parameters etc

URI is transformed into JSON-encoded "recipe":

<http://dada/stxm/ID11/proposal/maxipix/nanowire1/42?horz=161&vert=161&palette=bwr&scale=log...>

from short URI to JSON' recipe ...

```
{
  "type": "stm",
  "version": "v.01",
  "input": {
    "instrument": "ID11",
    "experiment": "hol182",
    "detector": "maxipix",
    "sample": "nanowire1",
    "number": 42,
    "dimensions": 2,
    "size": [161, 161]
  },
  "roi": {
    "x": 700,
    "y": 166,
    "w": 50,
    "h": 50
  },
  "colour": {
    "scale": "log",
    "palette": "bwr"
  },
  "output": {
    "format": "pdf"
  }
}
```

* JavaScript Object Notation

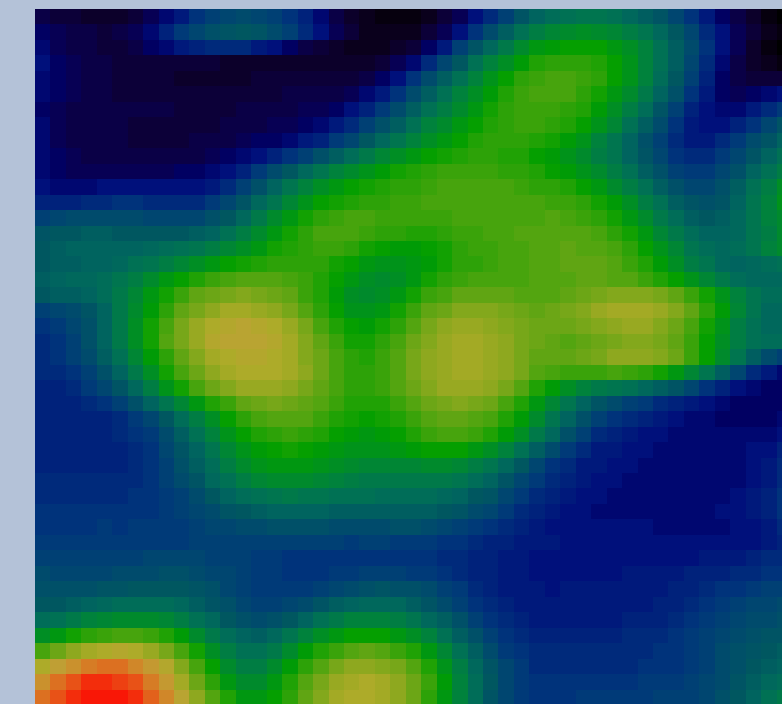
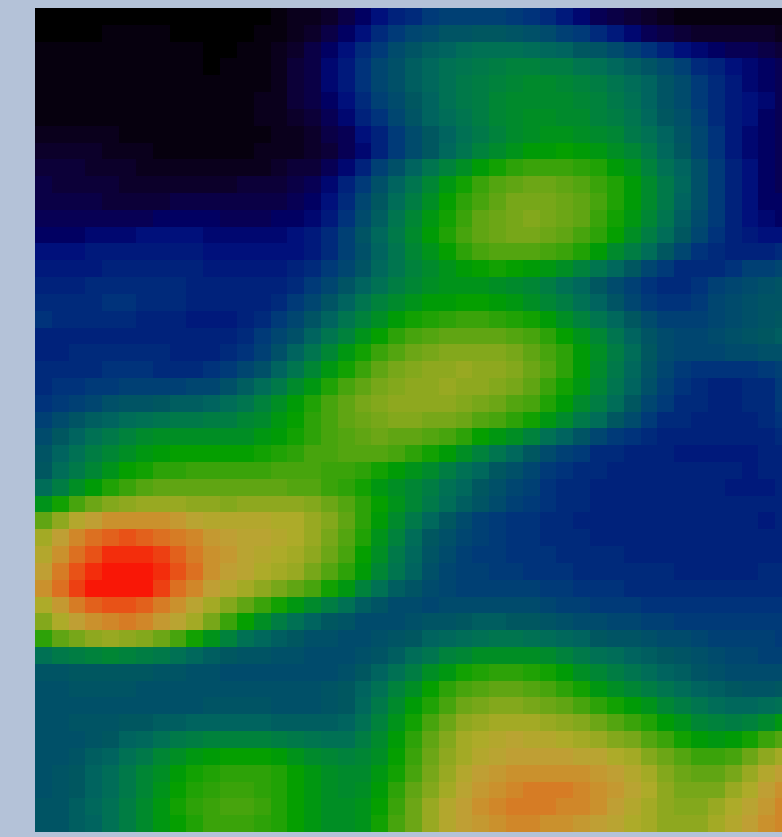
hash(JSON): look-up in cache database (sqlite3)

if cached: return { image, processed data, meta data etc. }

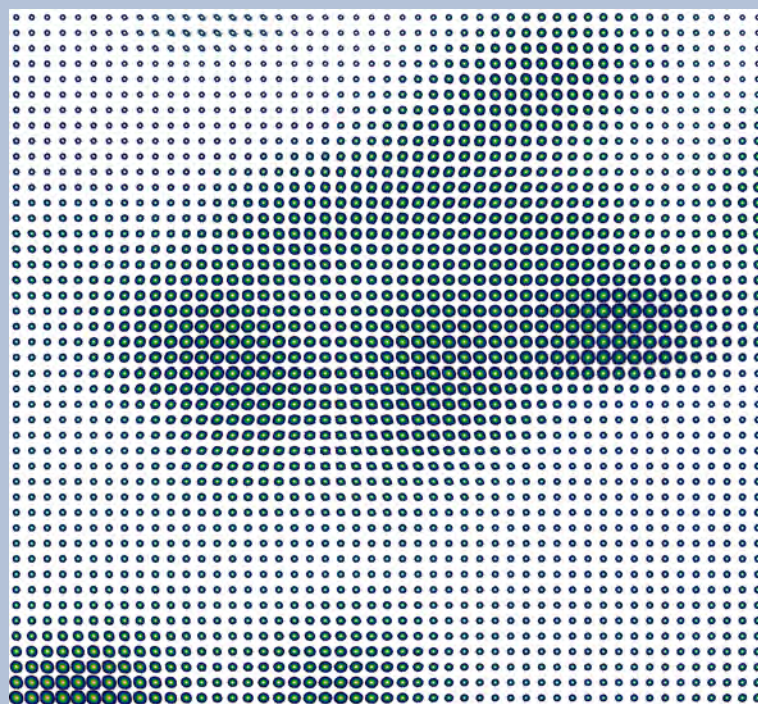
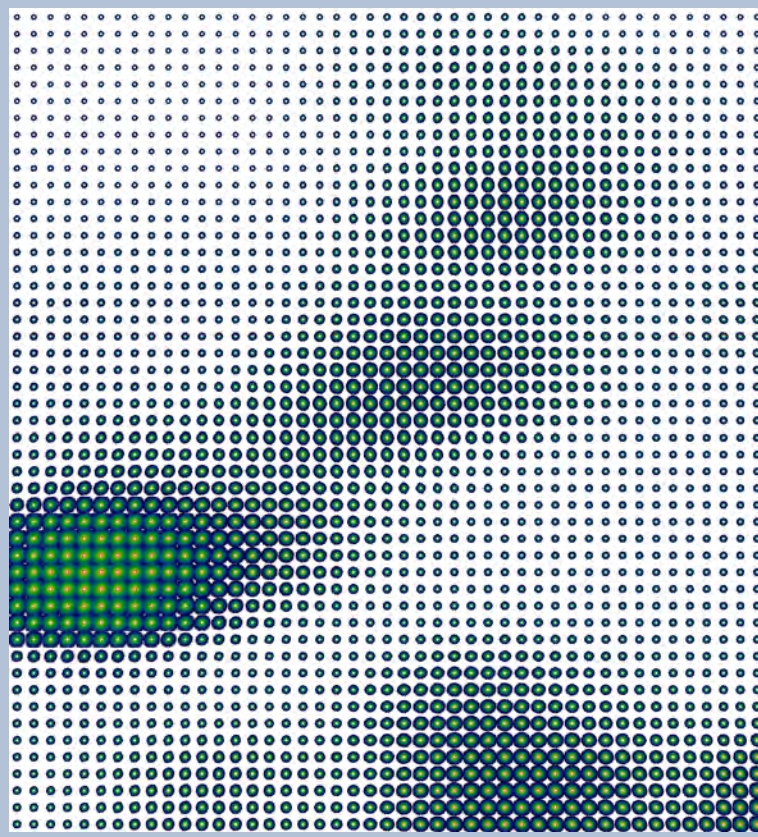
otherwise: process, calculate, render, cache, serve

Examples

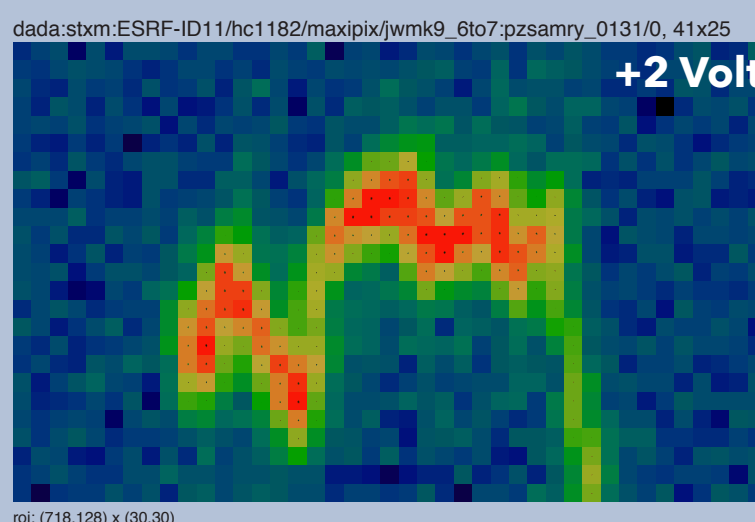
STXM darkfield



composite image

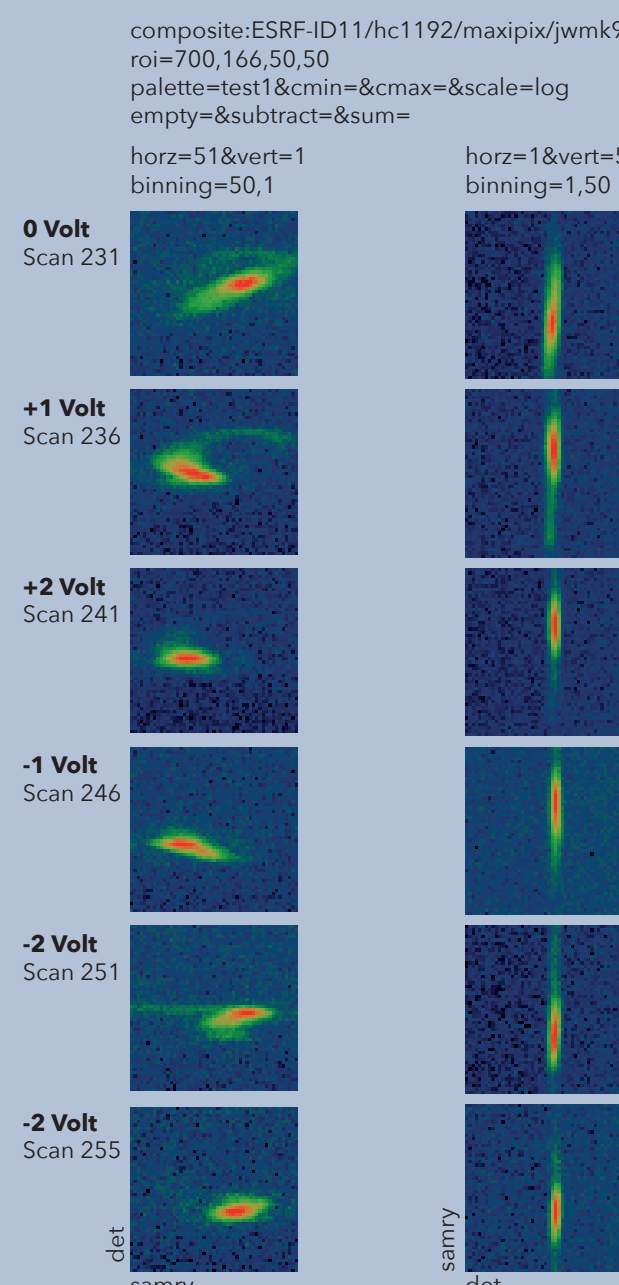


overlay STXM/image



combining STXM signal with detector images per position to measure strain in nanowire (J. Wallentin, unpublished)

1D comp + 1D binning



horizontal composite of vertically binned images (and vice versa) to visualise rocking scans of *in operando* contacted nanowire; here for different bias voltages (J. Wallentin, unpublished)