In-Situ Hard X-ray Ptychography

Workshop on Coherence at ESRF-EBS

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DESY Photon Science
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HELMHOLTZ RESEARCH FOR GRAND CHALLENGES

PtyNAMi
Ptychographic Nano-Analytical Microscope

GEFÖRDERT VOM
Bundesministerium für Bildung und Forschung

DESY.

X-Ray Nanoscience and X-Ray Optics

Prof. Dr. Christian G. Schroer (DESY and Universität Hamburg)
Dr. Gerald Falkenberg (DESY - P06 beamline responsible)

S. Alizadehfanaloo, T. Böse, S. Botta, D. Brückner, R. Döhrmann, J. Garrevoet, J. Hagemann, M. Kahnt, M. Lyubomirskiy, C. Ossig, M. Scholz, A. Schropp, W. Schröder, F. Seiboth, M. Seyrich, K. Spiers, M. Stückelberger, P. Wiljes, F. Wittwer, and X. Yang

Scanning coherent X-ray micoscopy, using fluorescence (XRF), diffraction (SAXS, WAXS), absorption (XAS) and ptychographic (CXDI) contrast.

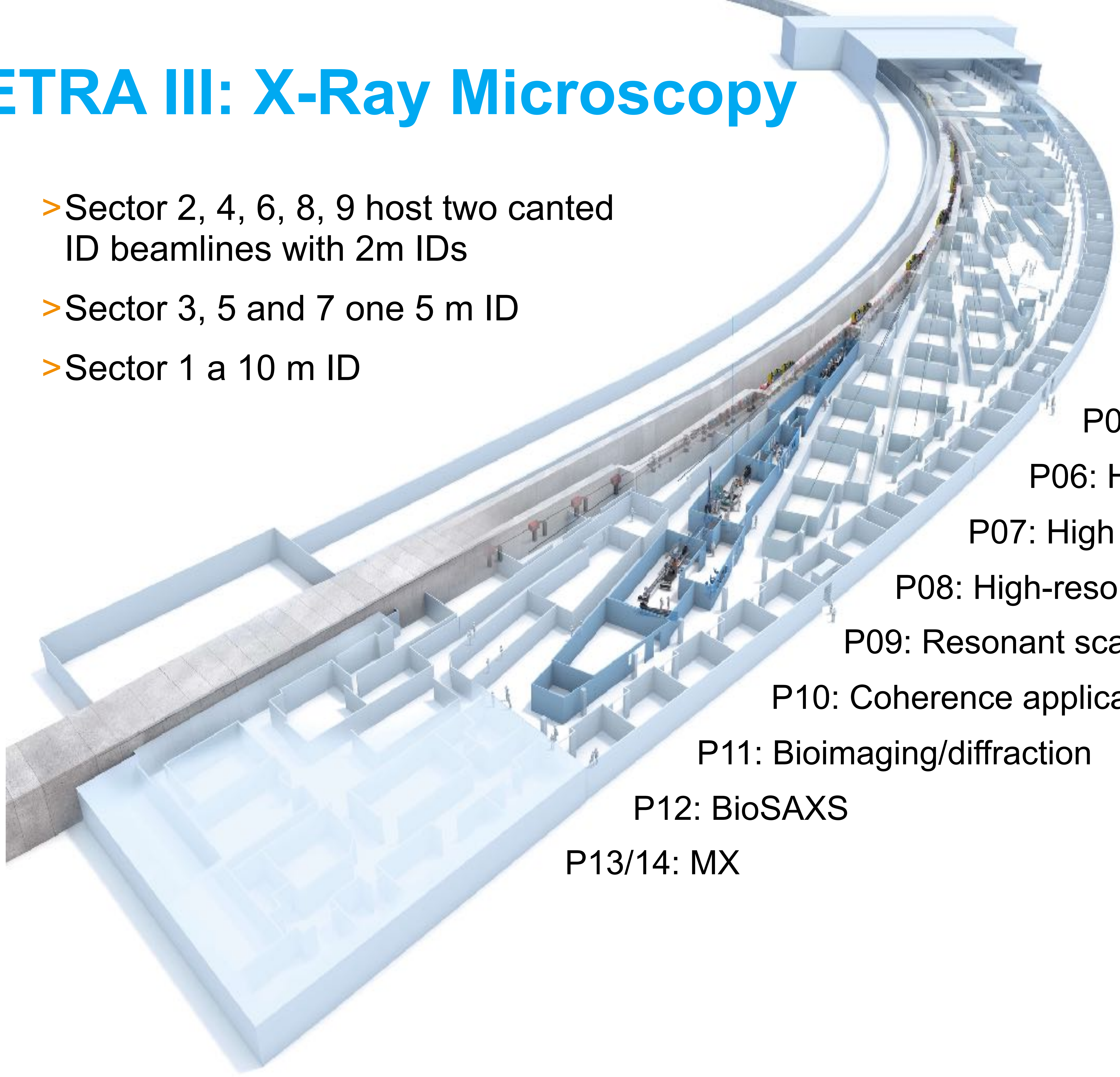
PETRA III: DESY's Brilliant Hard X-ray Source

- particle energy: 6 GeV
- stored current: 100 mA (top-up)
- emittance: 1.2 nm rad
- circumference: 2304 m
- # of undulators: 25 (incl. canted)
- # of experiments: 50
- X-ray wavelength: 10 – 0.05 Å
- annual operation: 5000 h (for users)

- **built in 1978 for high-energy physics**
- **rebuilt as a synchrotron radiation source starting in 2007**
- **user operation since 2010**
- **extension added: March 2014 - April 2015**

PETRA III: X-Ray Microscopy

- >Sector 2, 4, 6, 8, 9 host two canted ID beamlines with 2m IDs
- >Sector 3, 5 and 7 one 5 m ID
- >Sector 1 a 10 m ID



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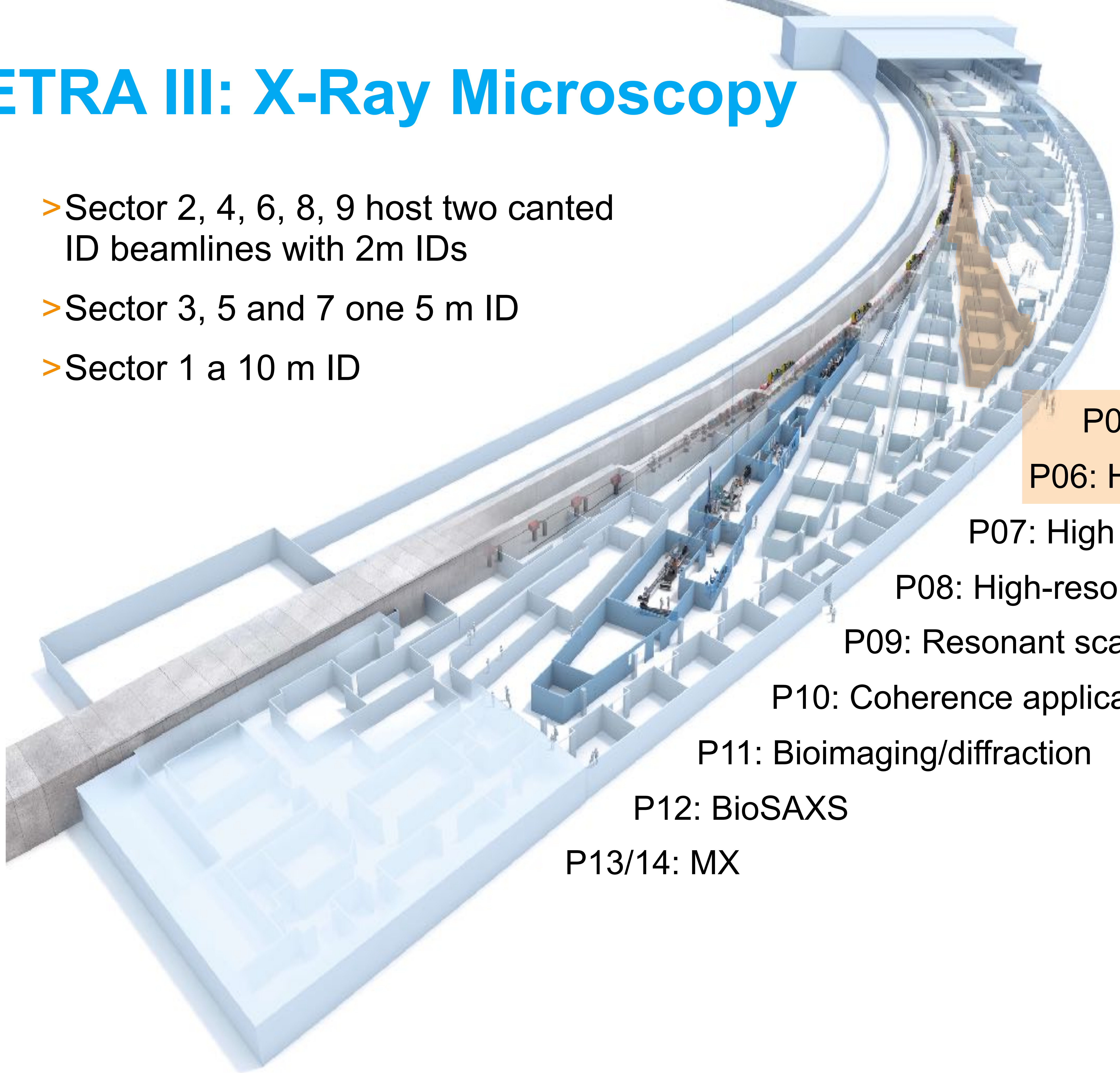


P01: Dynamics beamline, IXS, NRS

P02: Powder diffraction/Extreme Conditions

P03: Micro-, nano-SAXS, WAXS

P04: Variable polarization XUV

P05: Micro-, nano-tomography

P06: Hard x-ray micro-, nanoprobe

P07: High energy materials science

P08: High-resolution diffraction

P09: Resonant scattering/diffraction

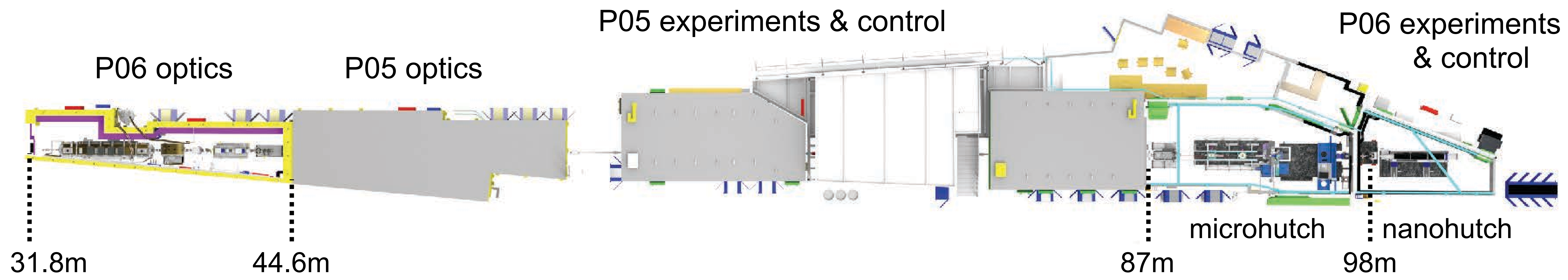
P10: Coherence applications

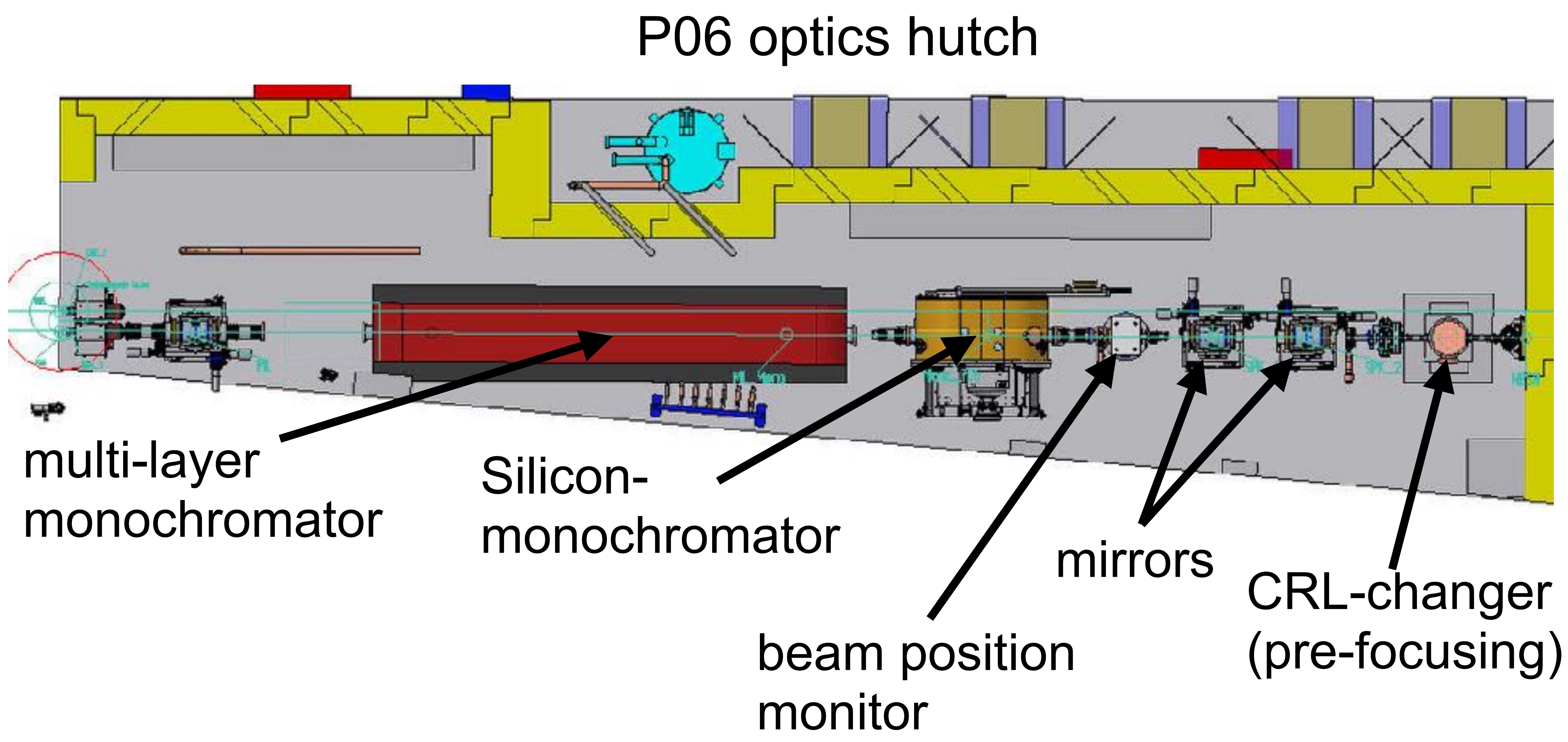
P11: Bioimaging/diffraction

P12: BioSAXS

P13/14: MX

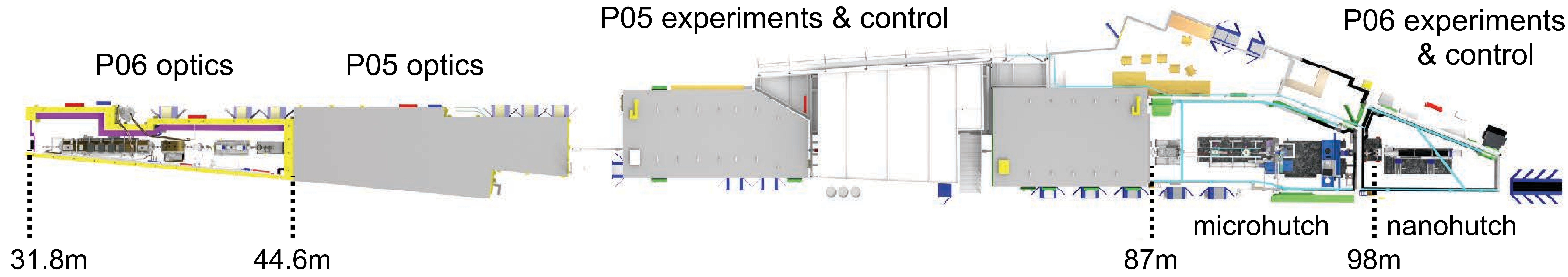
Beamline P06 at PETRA III

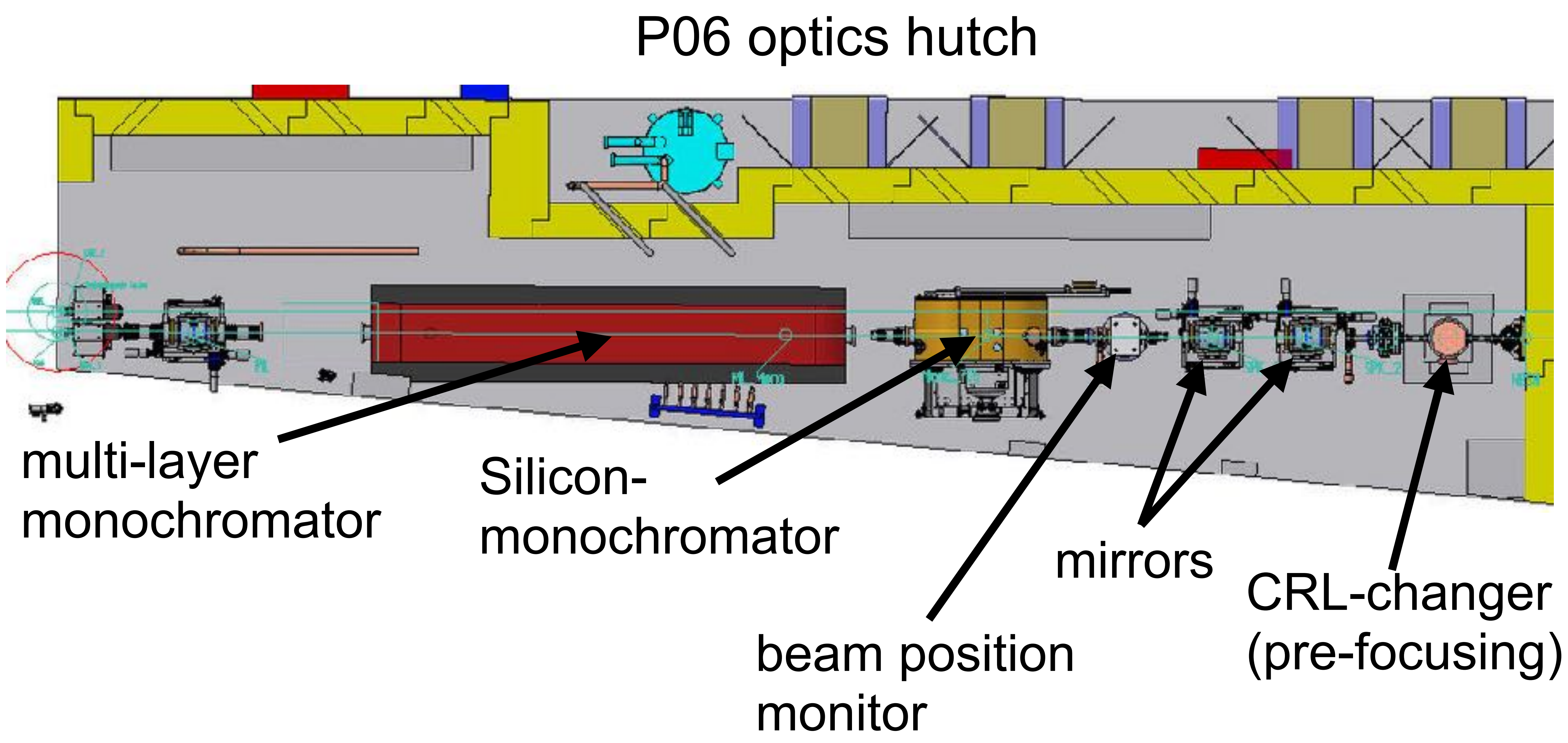


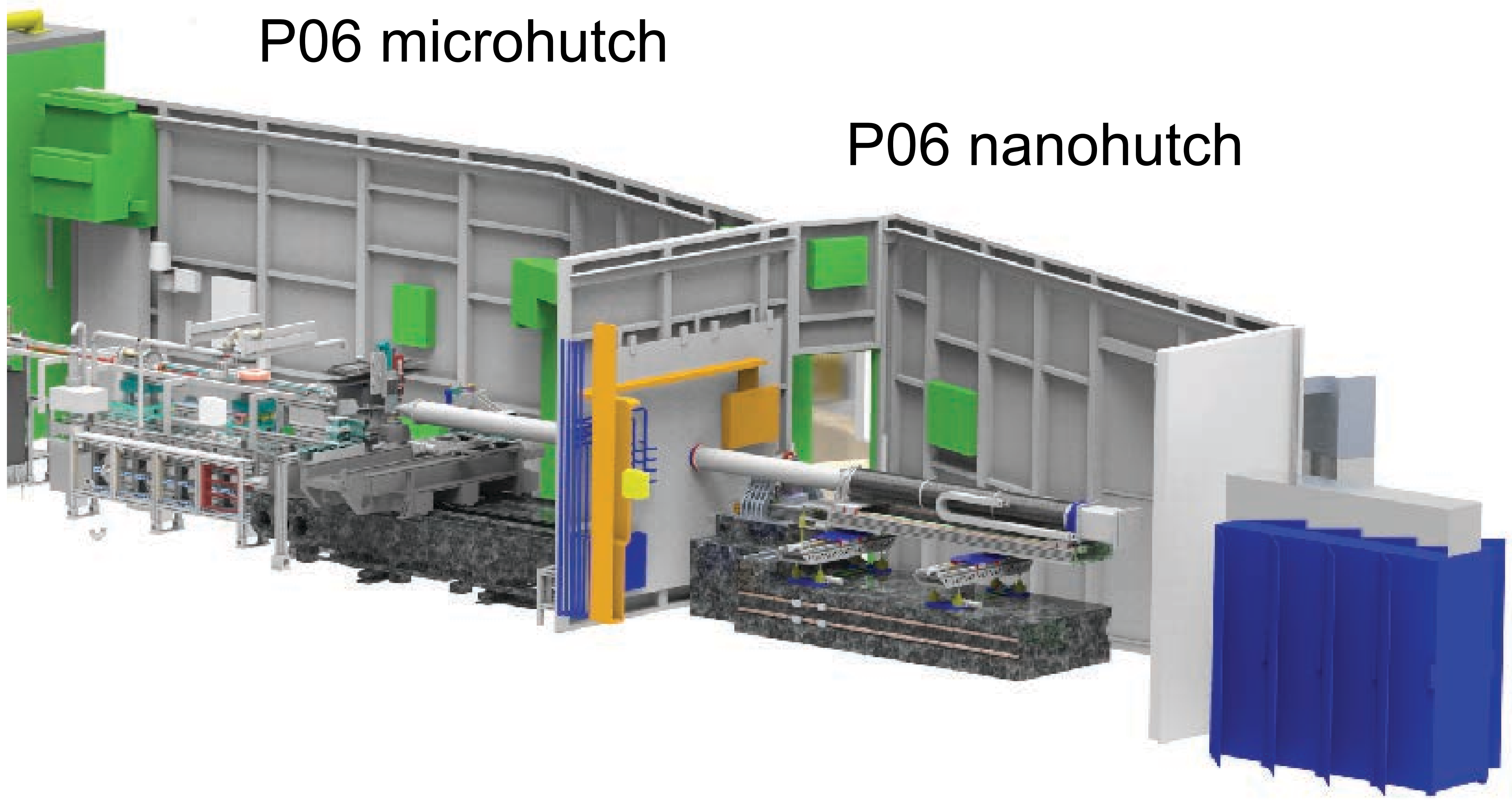


- monochromators:
  - Si(111)-channelcut (CC)
  - Si(111)-double crystal monochromator (DCM)
  - multilayer-monochromator (ML)
- horizontal offset mirrors (HO-mirrors)
- pre-focusing: CRL-changer

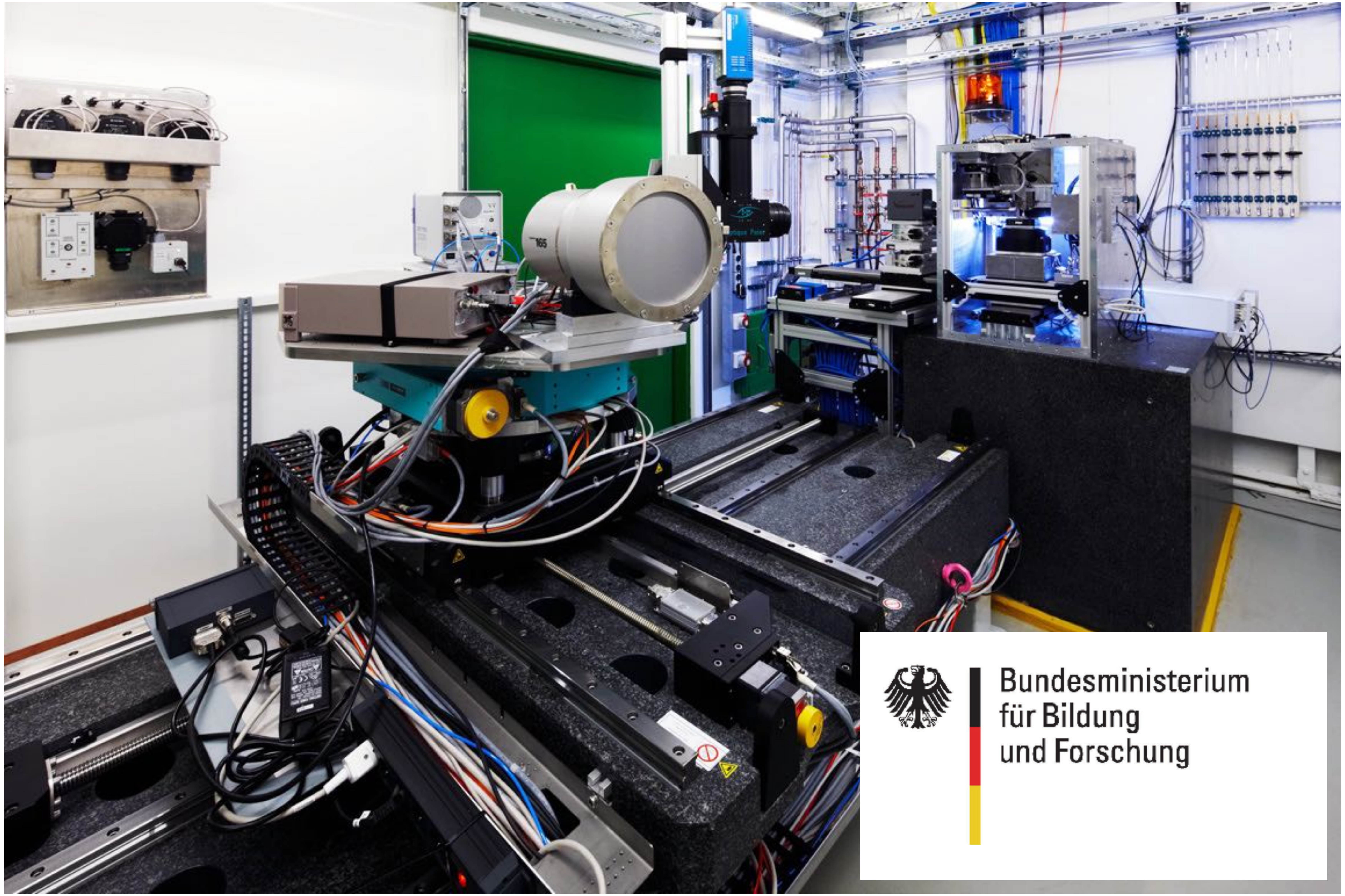
Beamline P06 at PETRA III







Nanoprobe Setup at Beamline P06 (2015)



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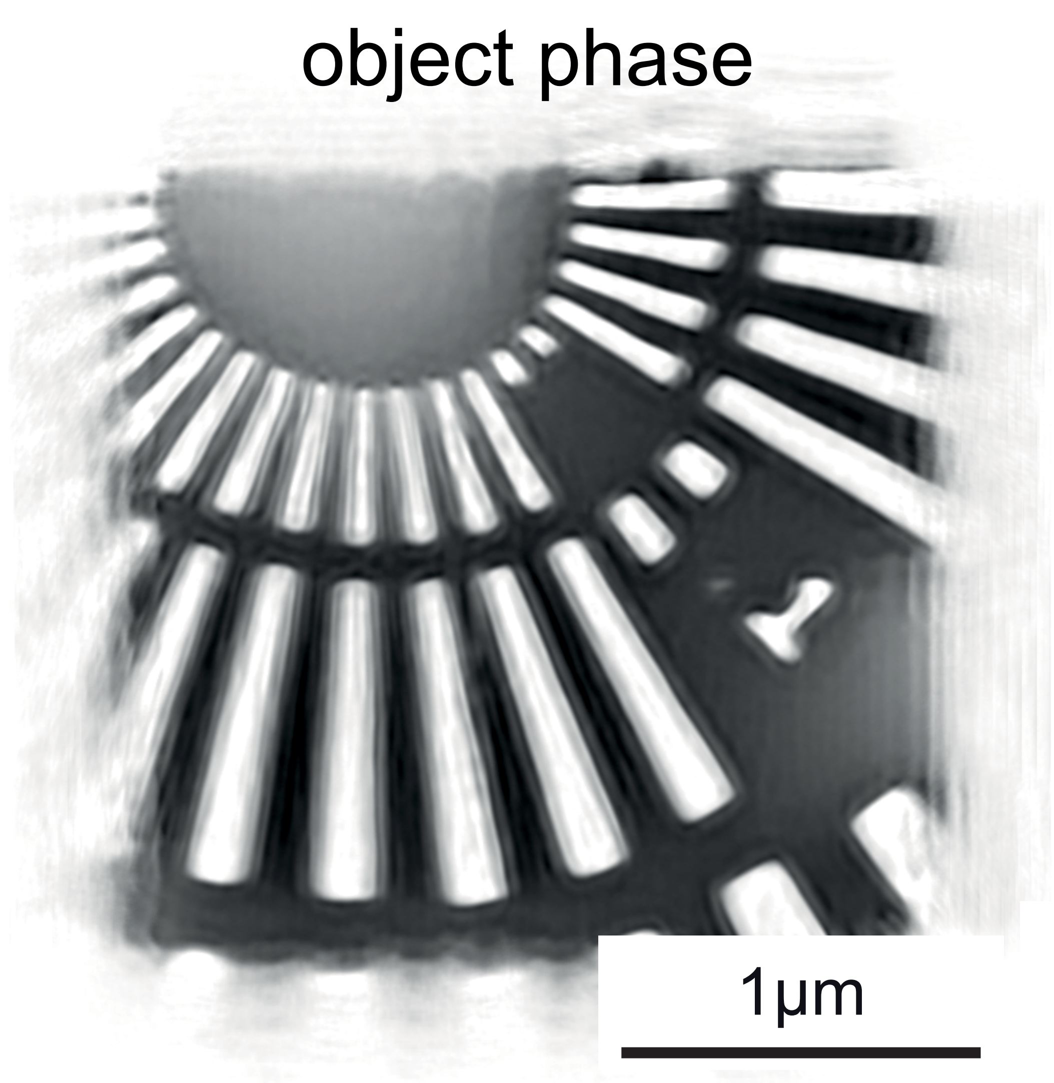
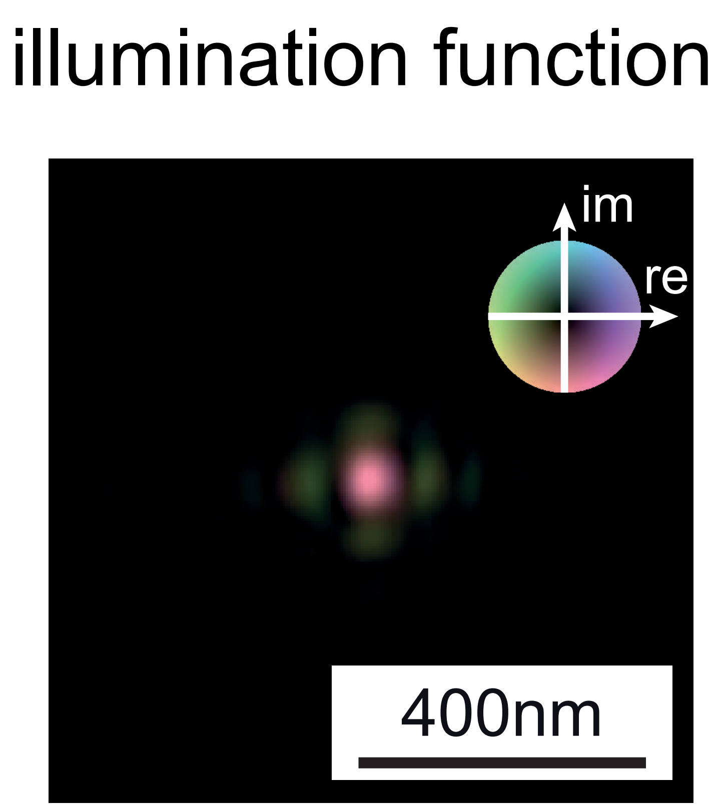
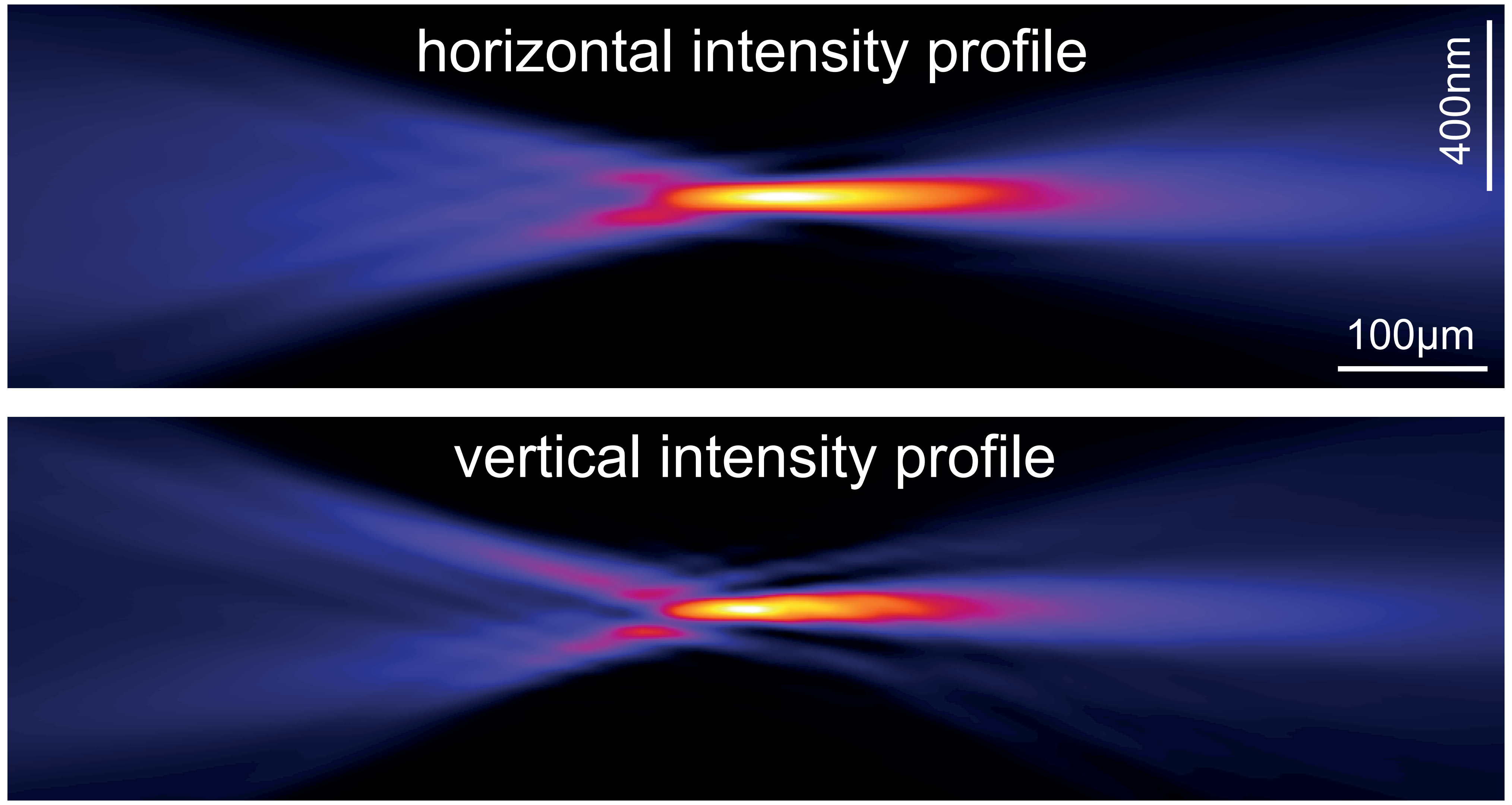
und Forschung

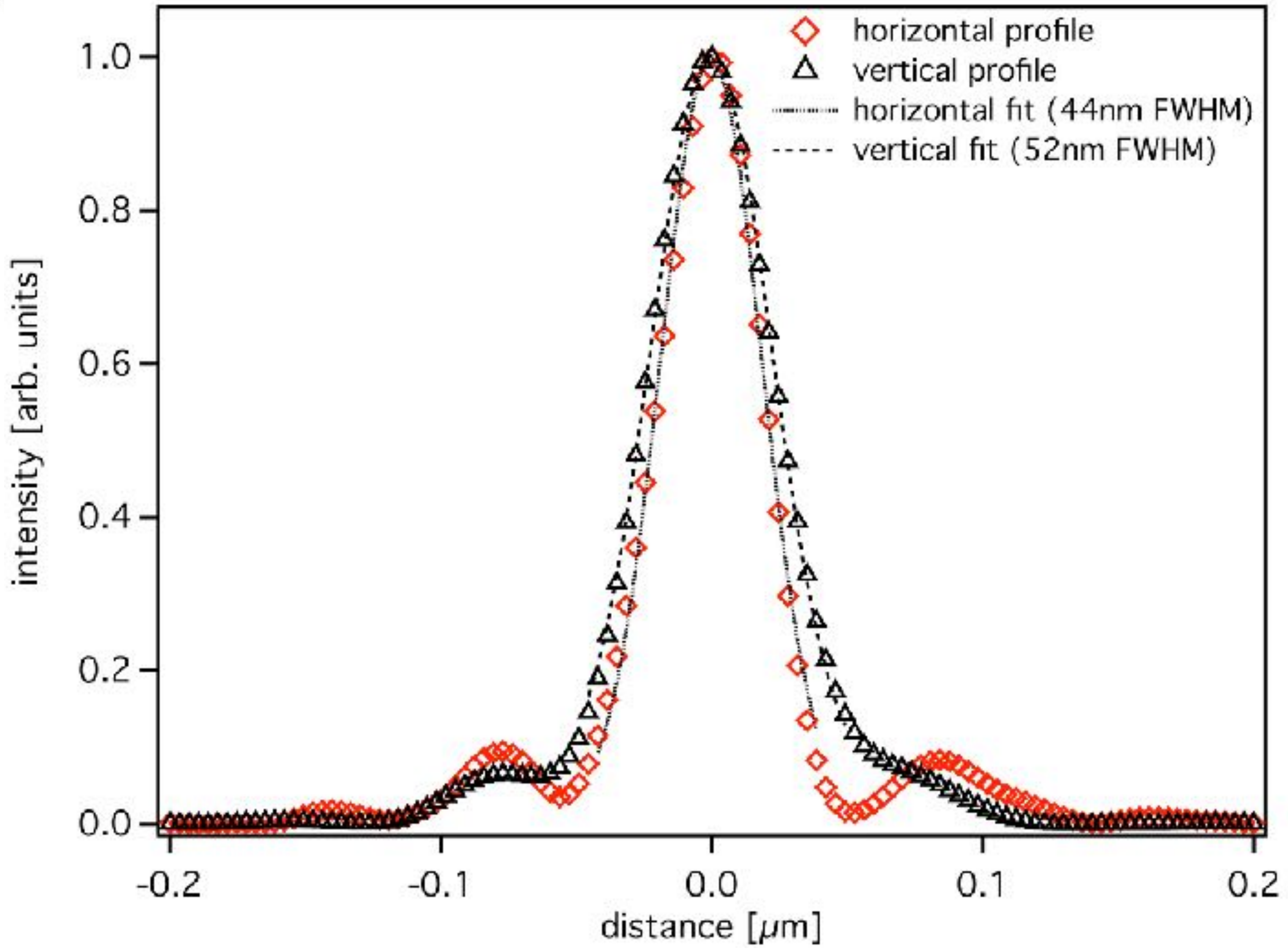
Nanobeam Characterization by Ptychography

- X-ray energy: 18 keV
- convergence after 200 iterations
- focus size 44 nm x 52 nm (FWHM)
- almost perfect nano-focused X-ray beam









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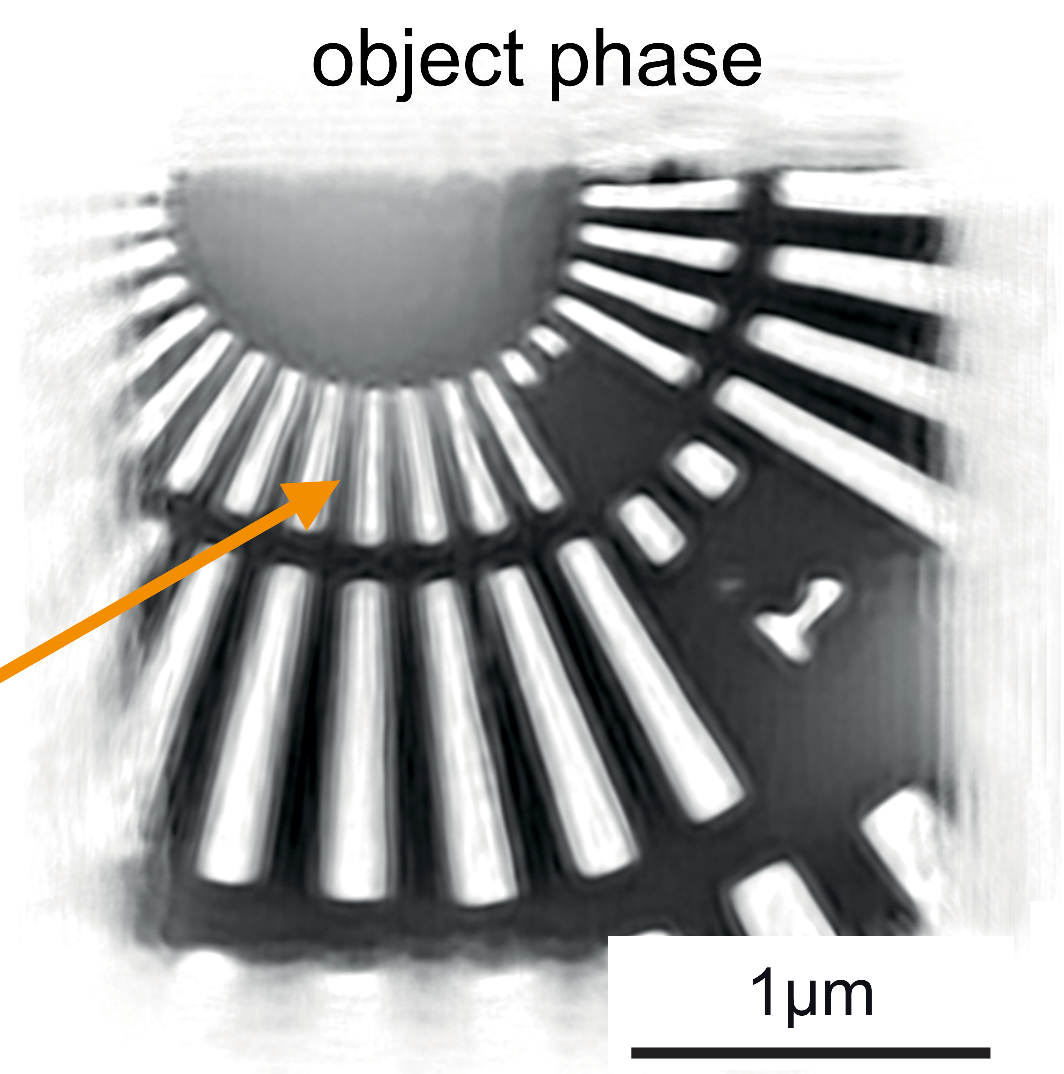
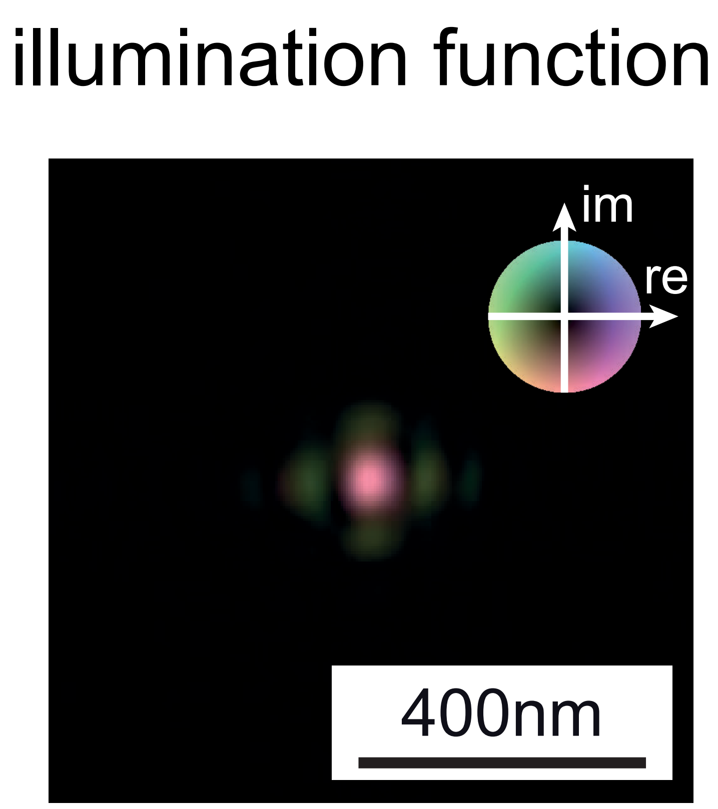
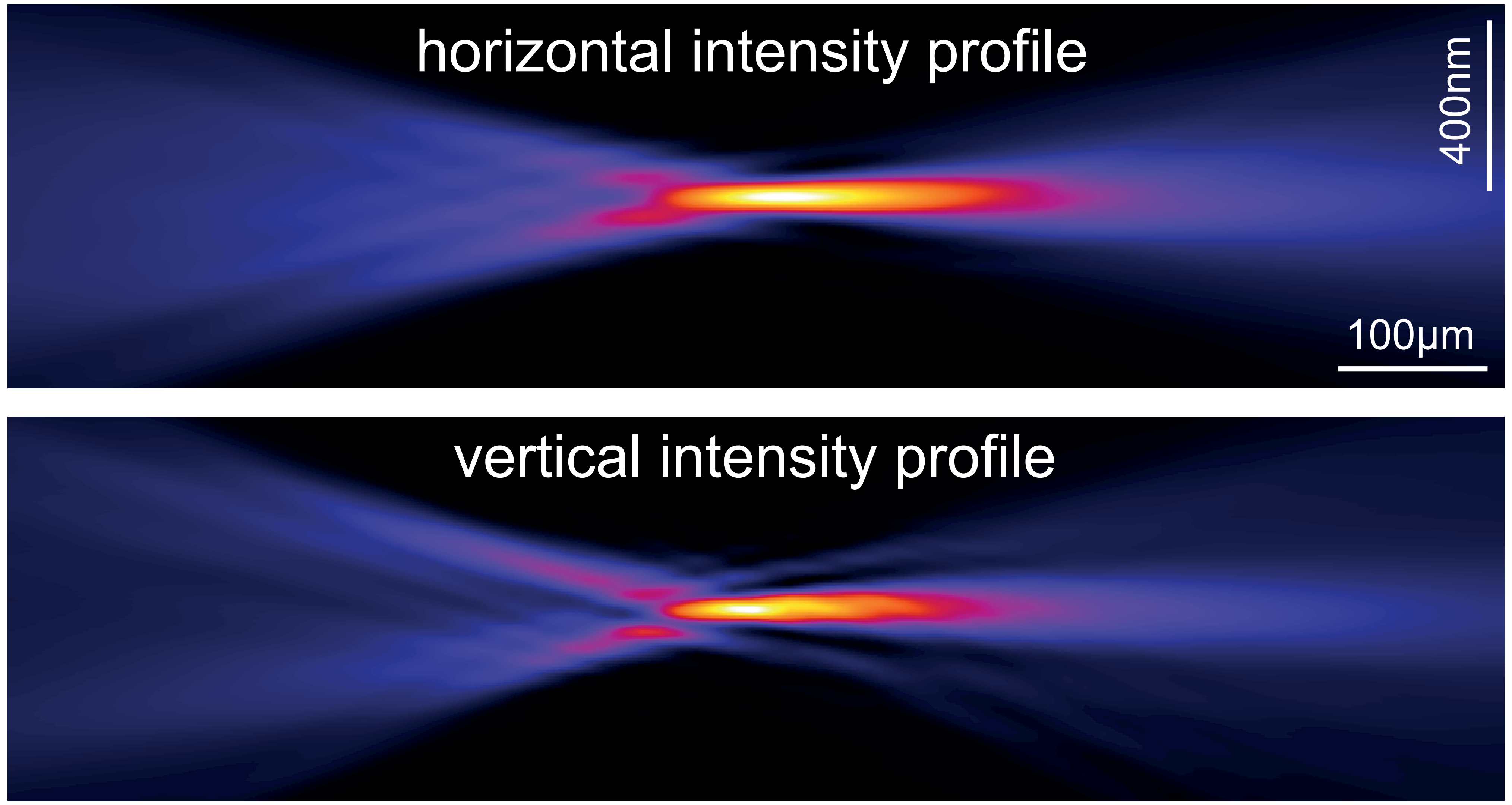
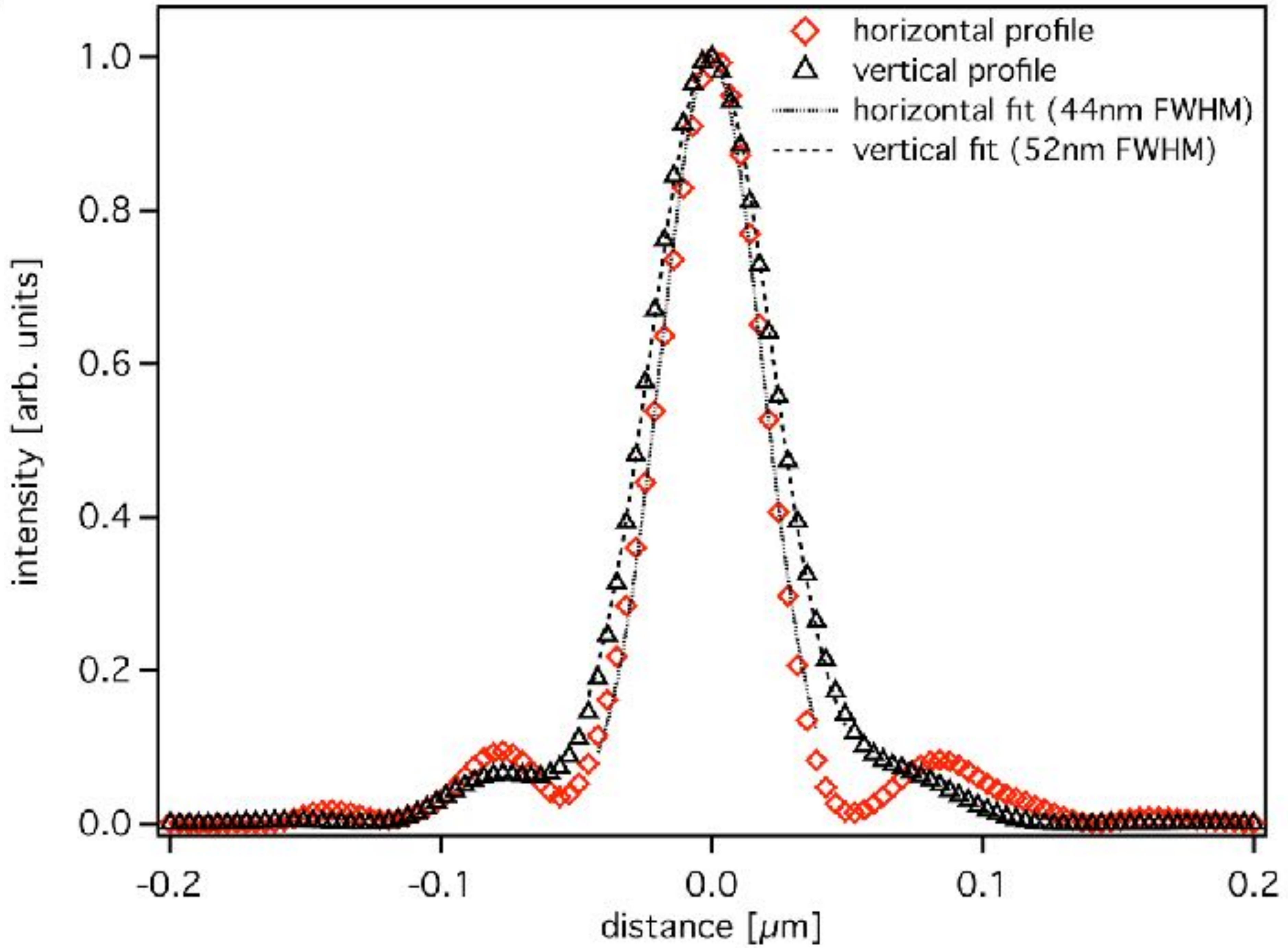
Stability of the setup crucial!





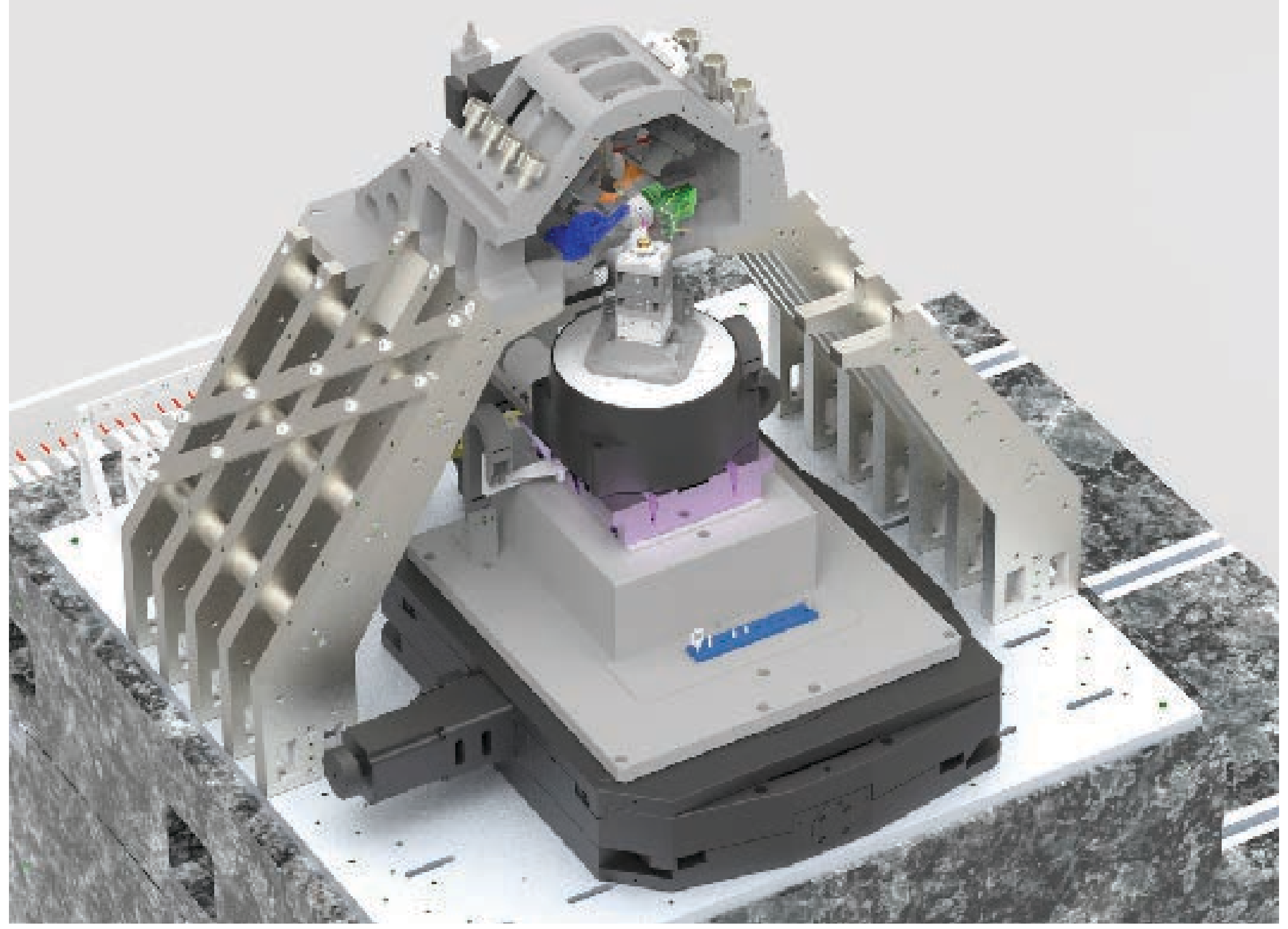
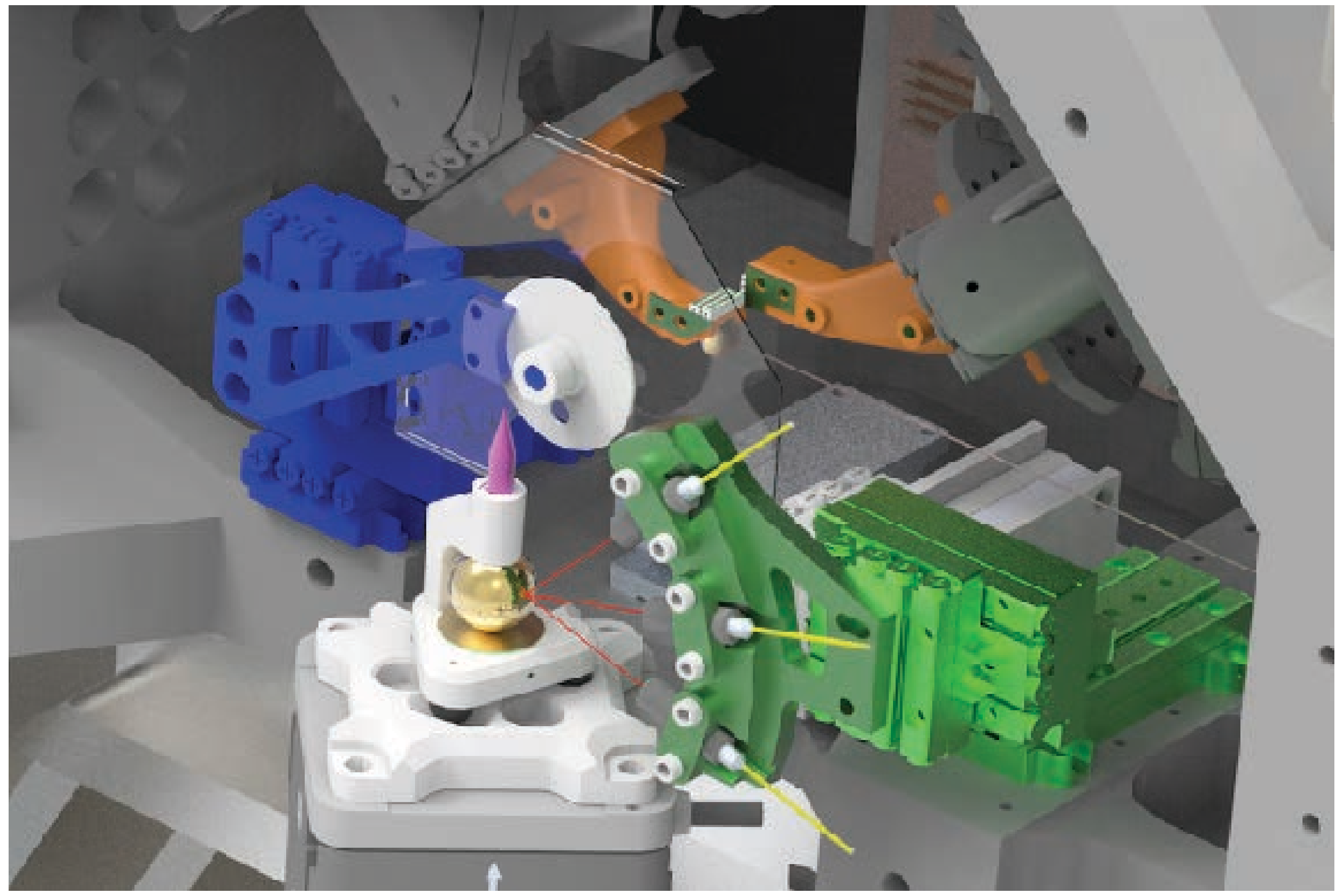
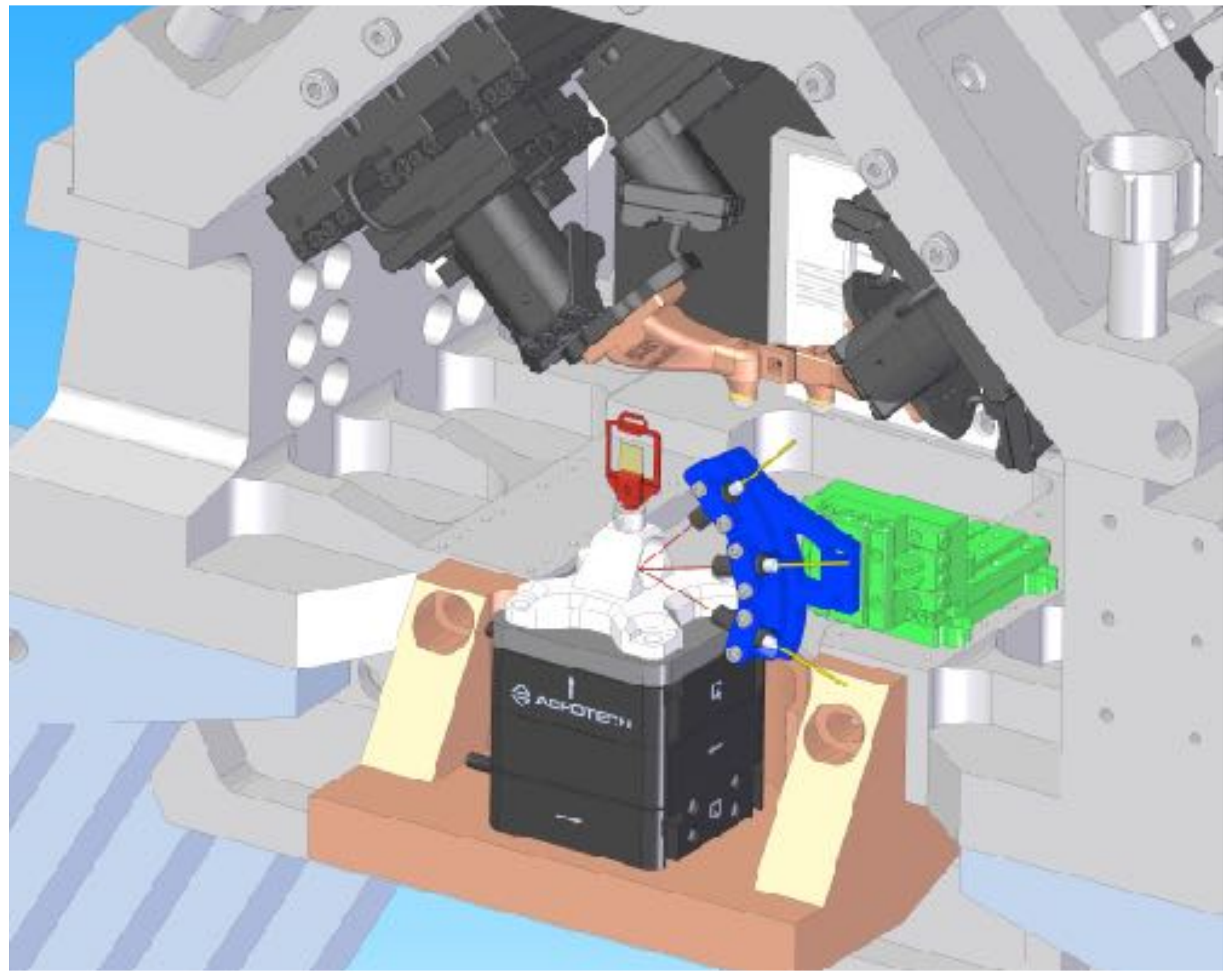
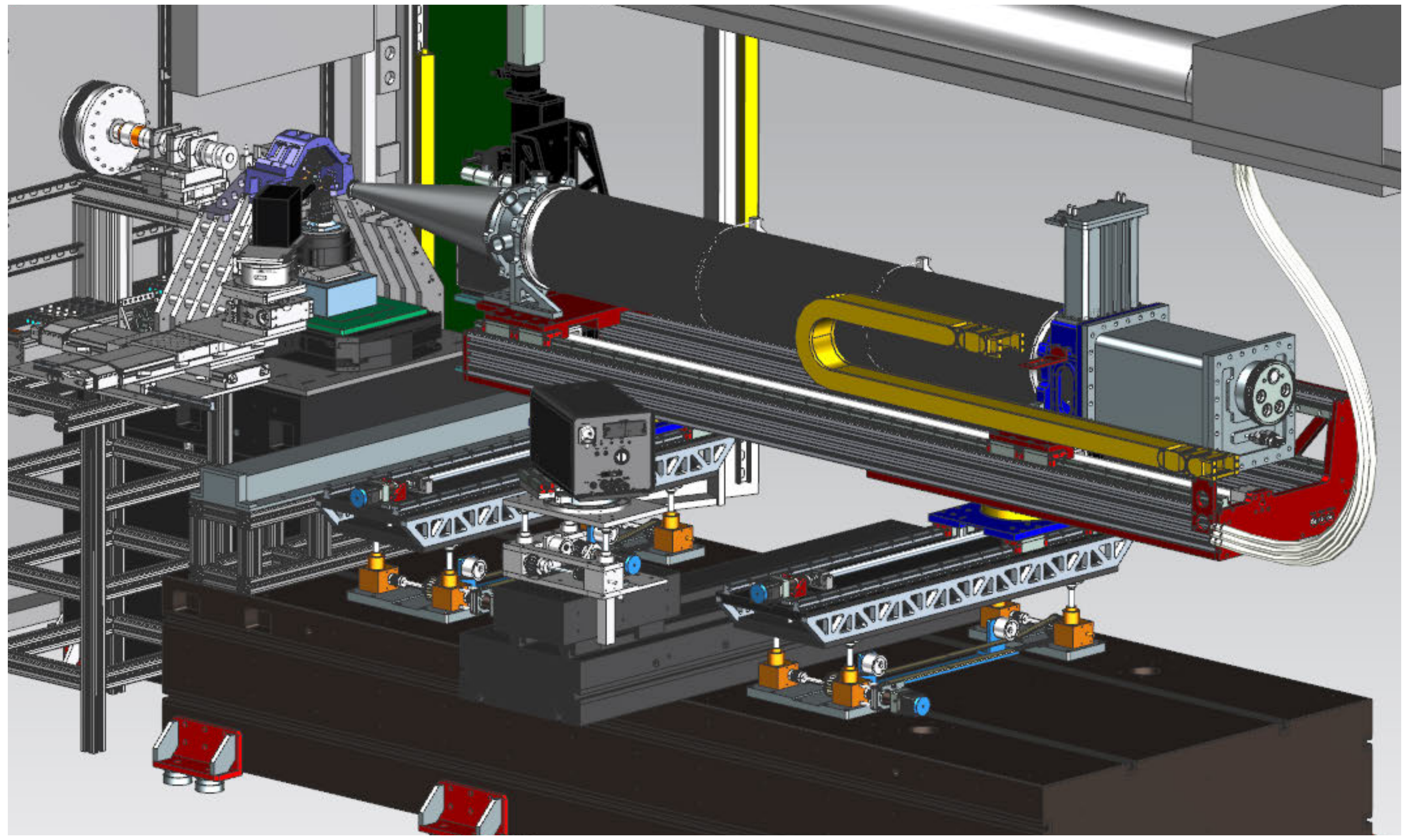




Ptychographic Nano-Analytical Microscope: PtyNAMi

Goals:
- high spatial resolution
- high sensitivity
- 2D and 3D imaging
- *in situ* & *operando*

Experimental requirements:
- optimized coherent flux
- high performance X-ray optics
- high mechanical stability and control
- low background

R. Döhrmann, S. Botta, P. Wiljes, H. Lindemann, *et al.*

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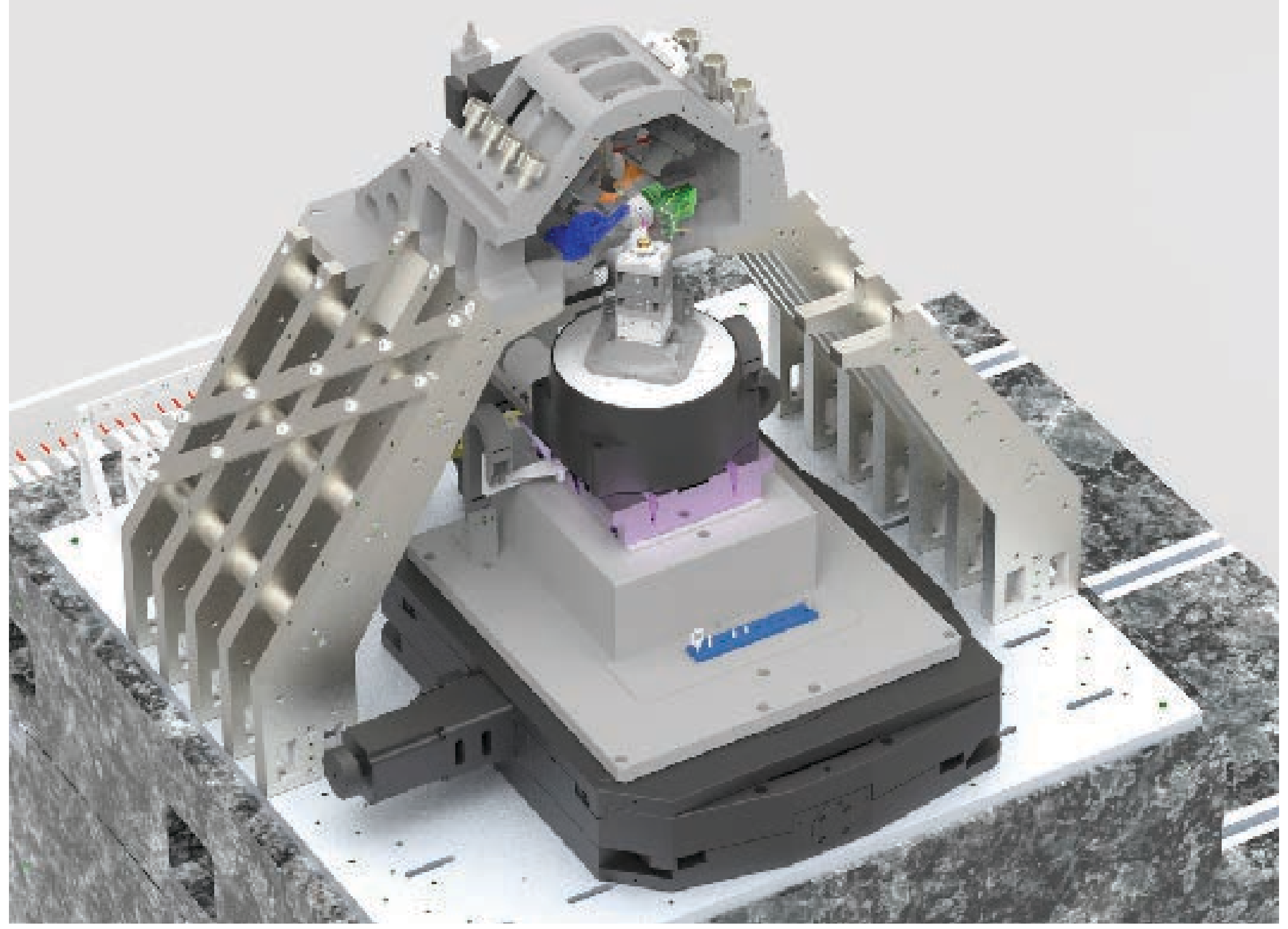

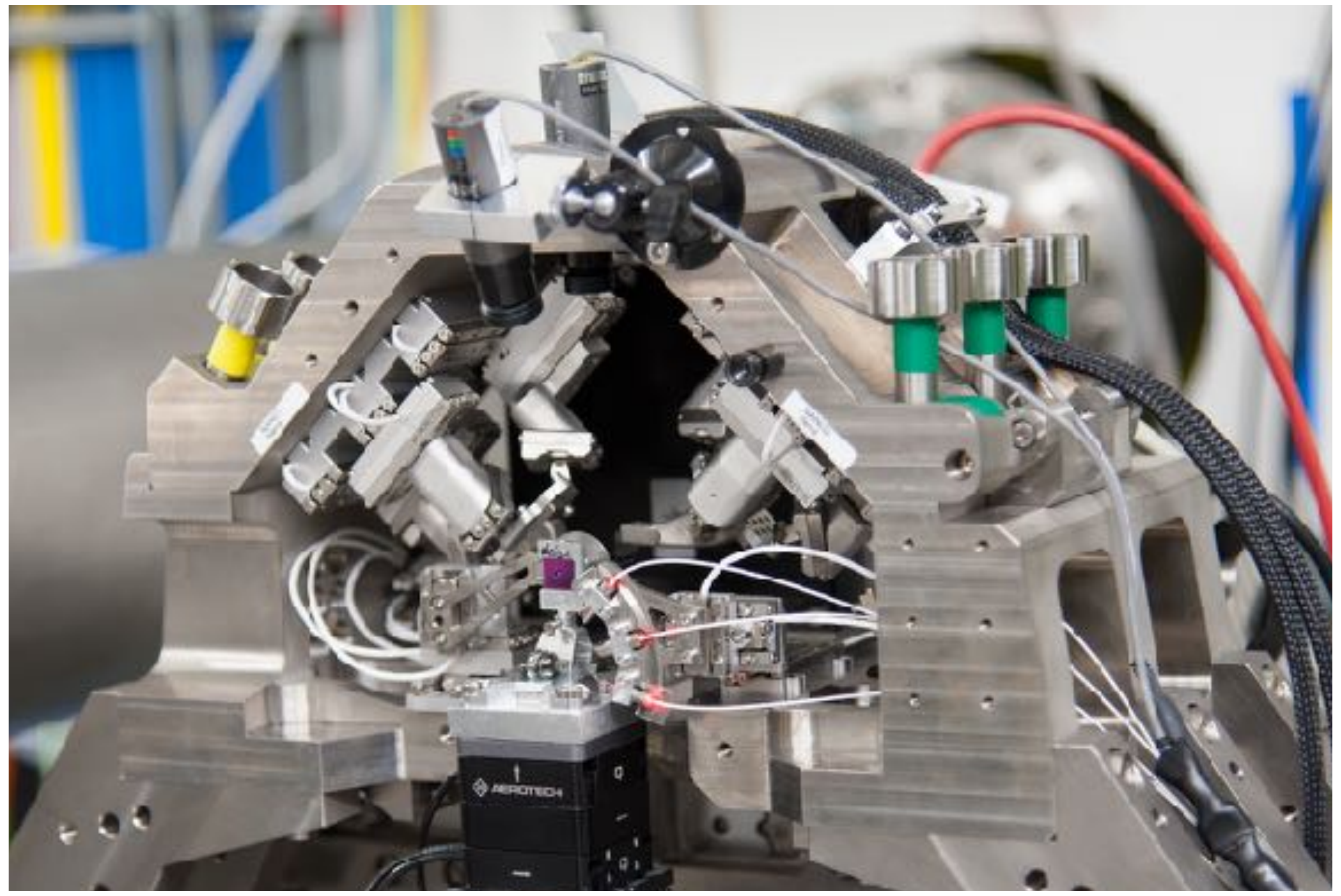

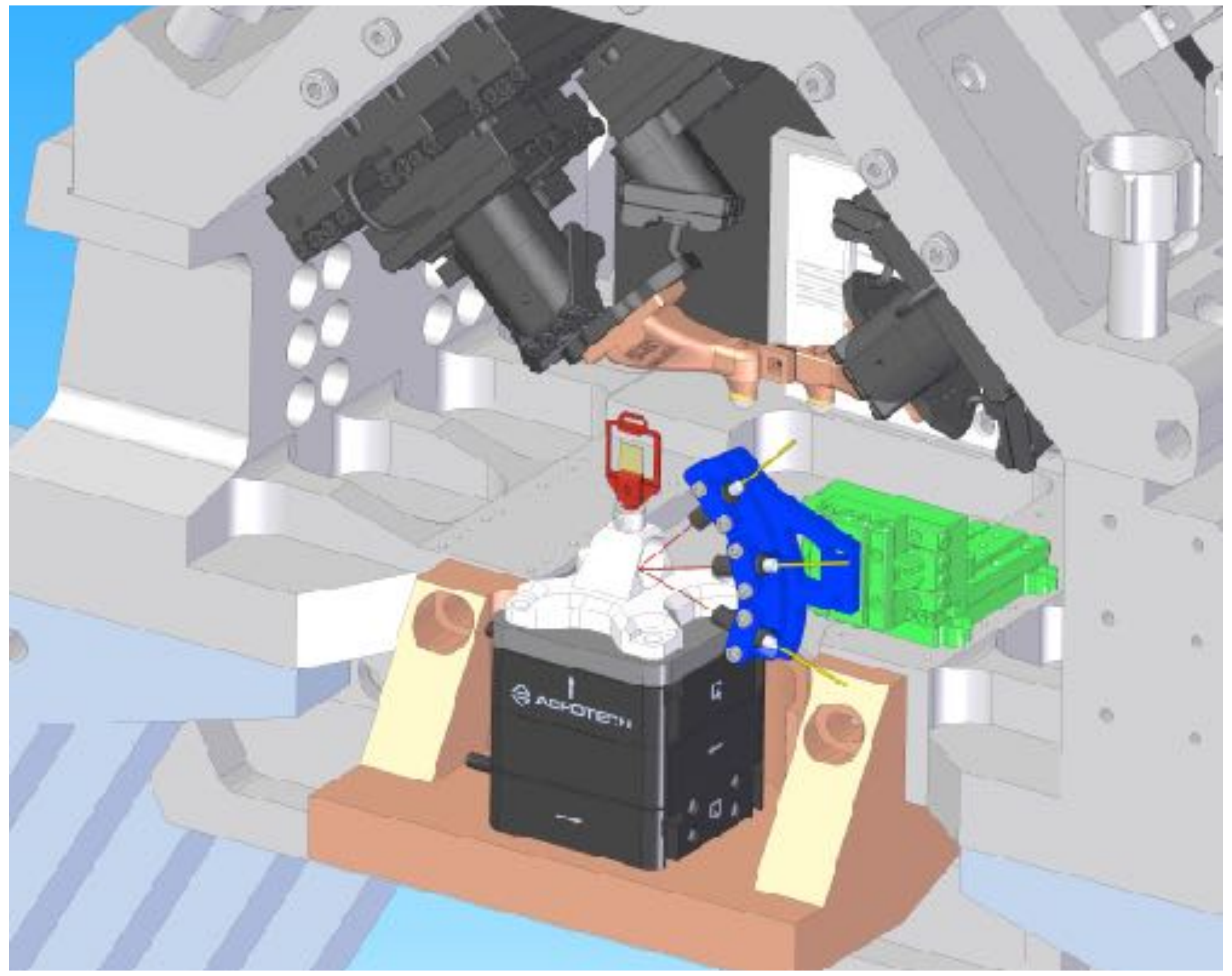

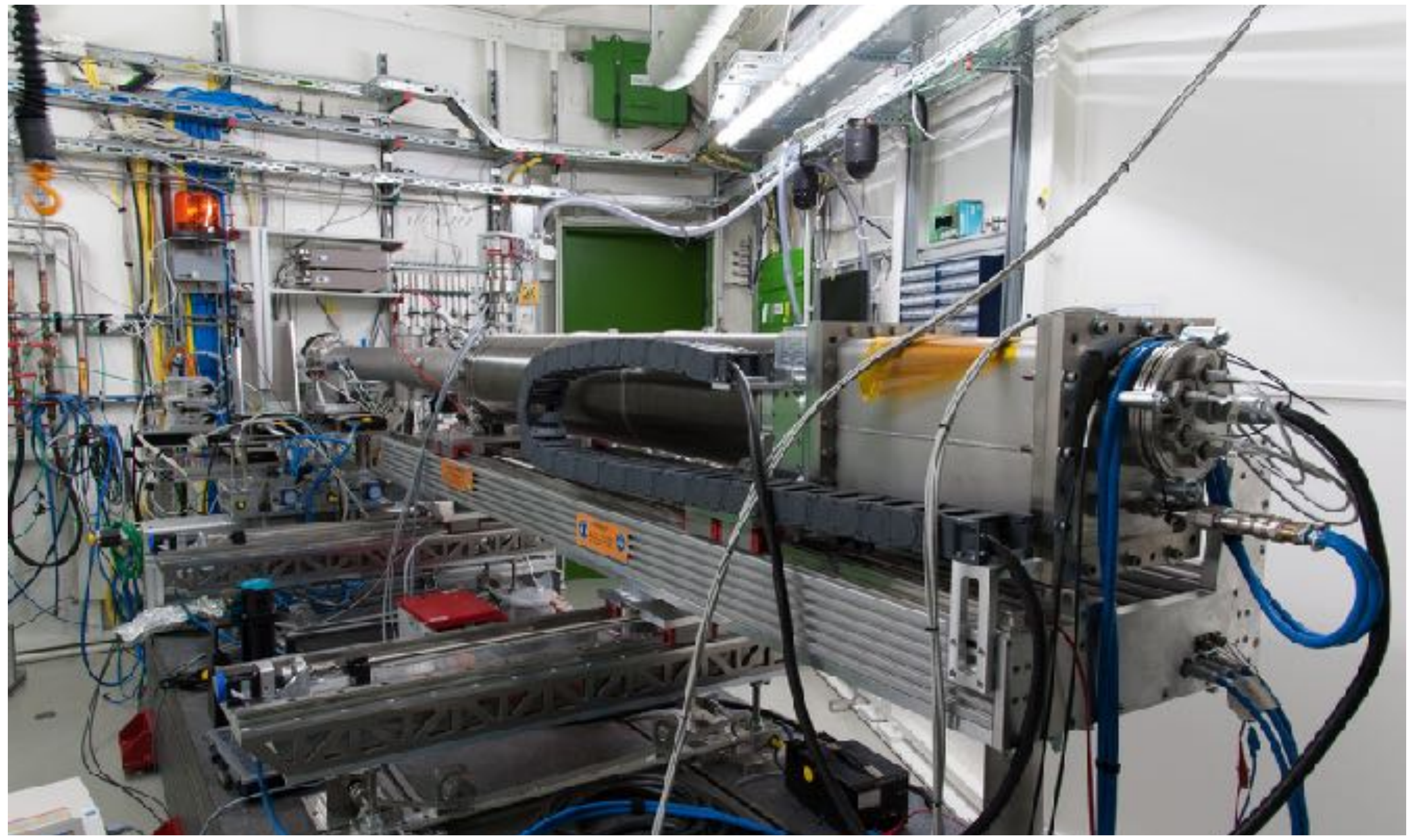

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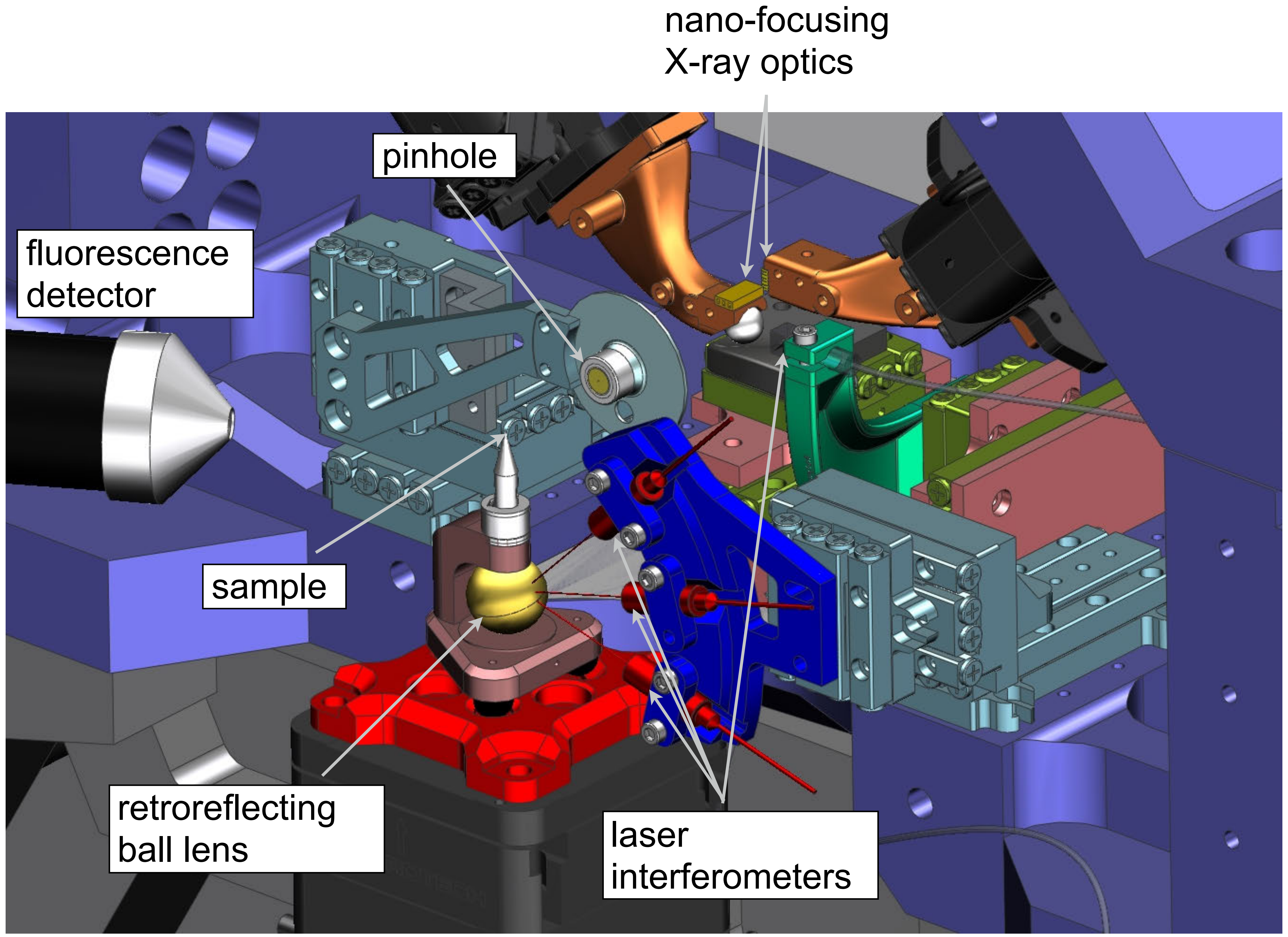
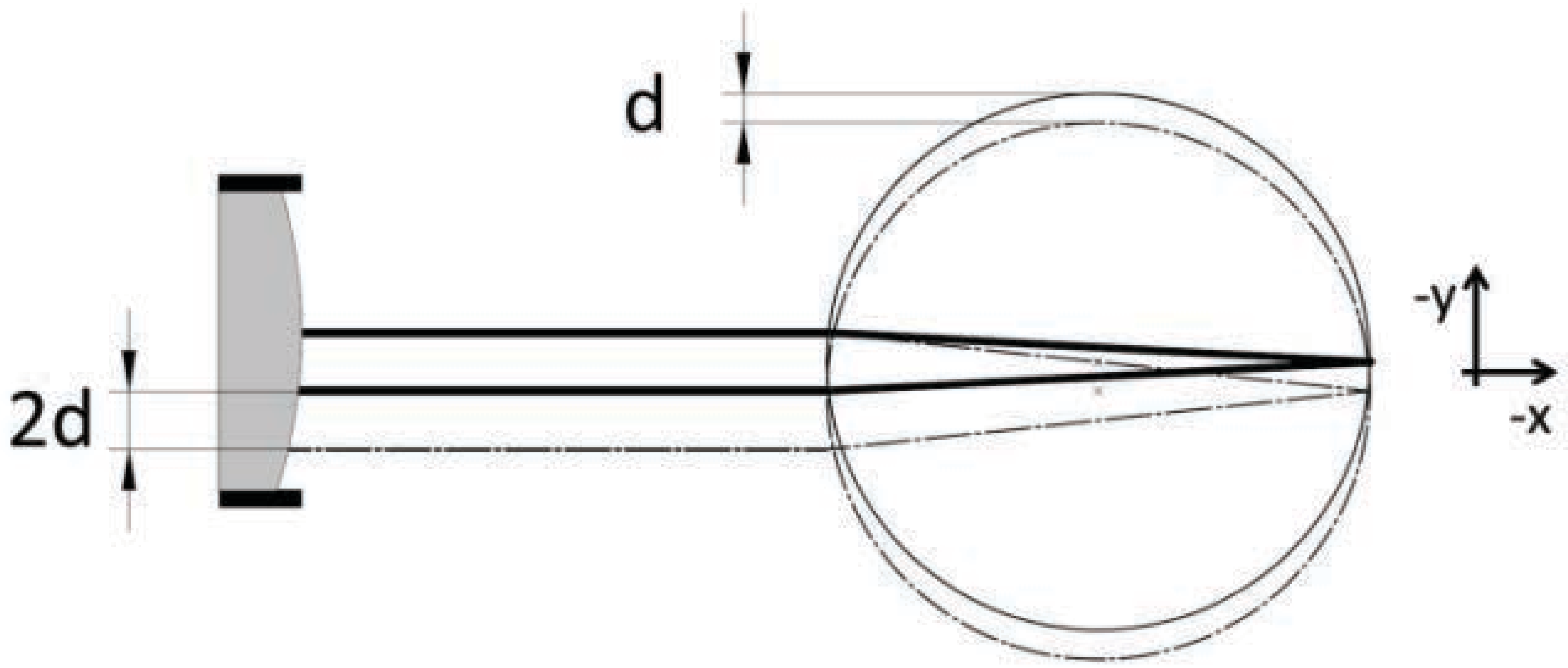
PtyNAMi — Optical Interferometers

Sample Environment and Positioning Control





focal length of a ball lens

$$f = \frac{nr}{2(n-1)}$$

$$n = 2 \Rightarrow r = f$$

→ retroreflection

our ball lenses:

n = 1.955 @ λ = 1550 nm and r = 5 mm

M Seyrich, *et al.*

PtyNAMi — Optical Interferometers

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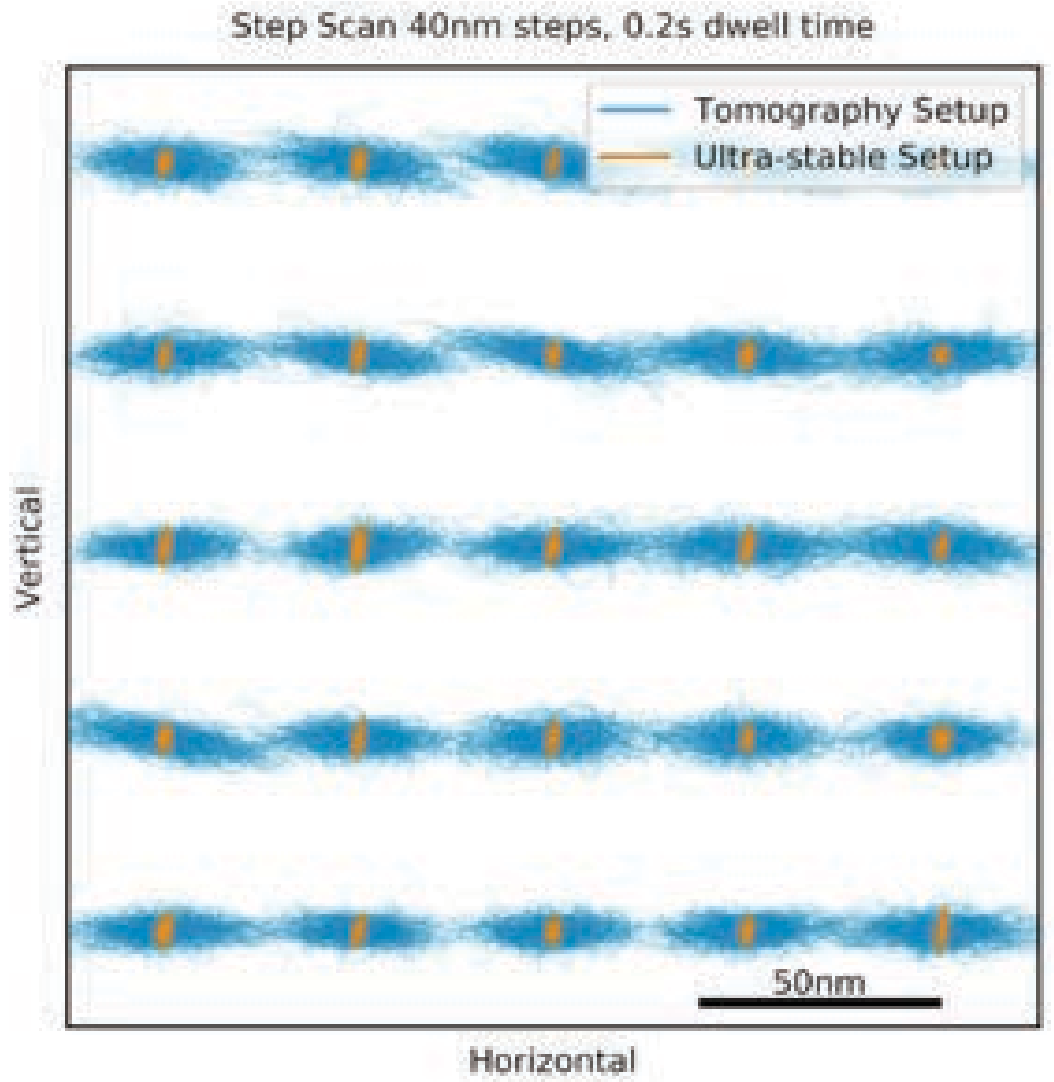

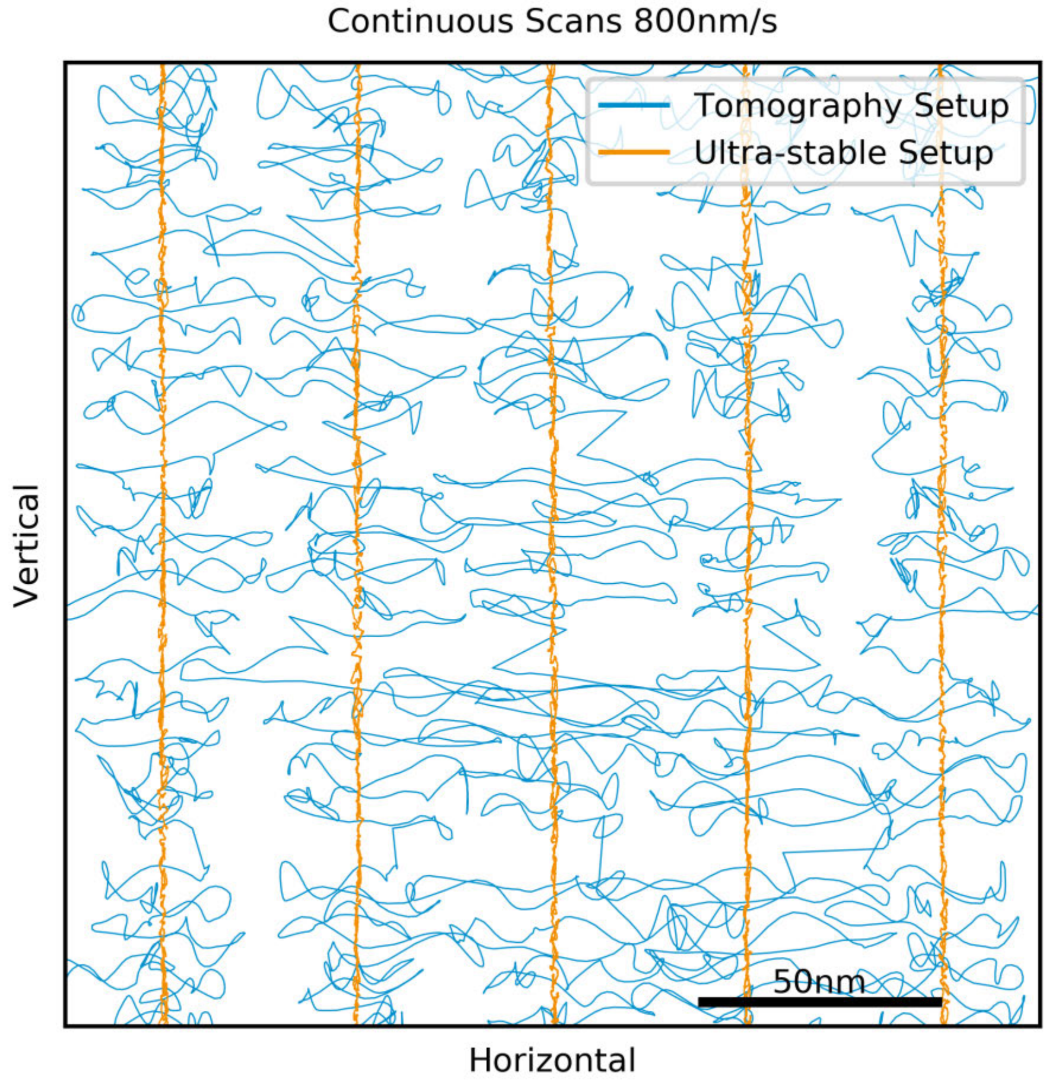

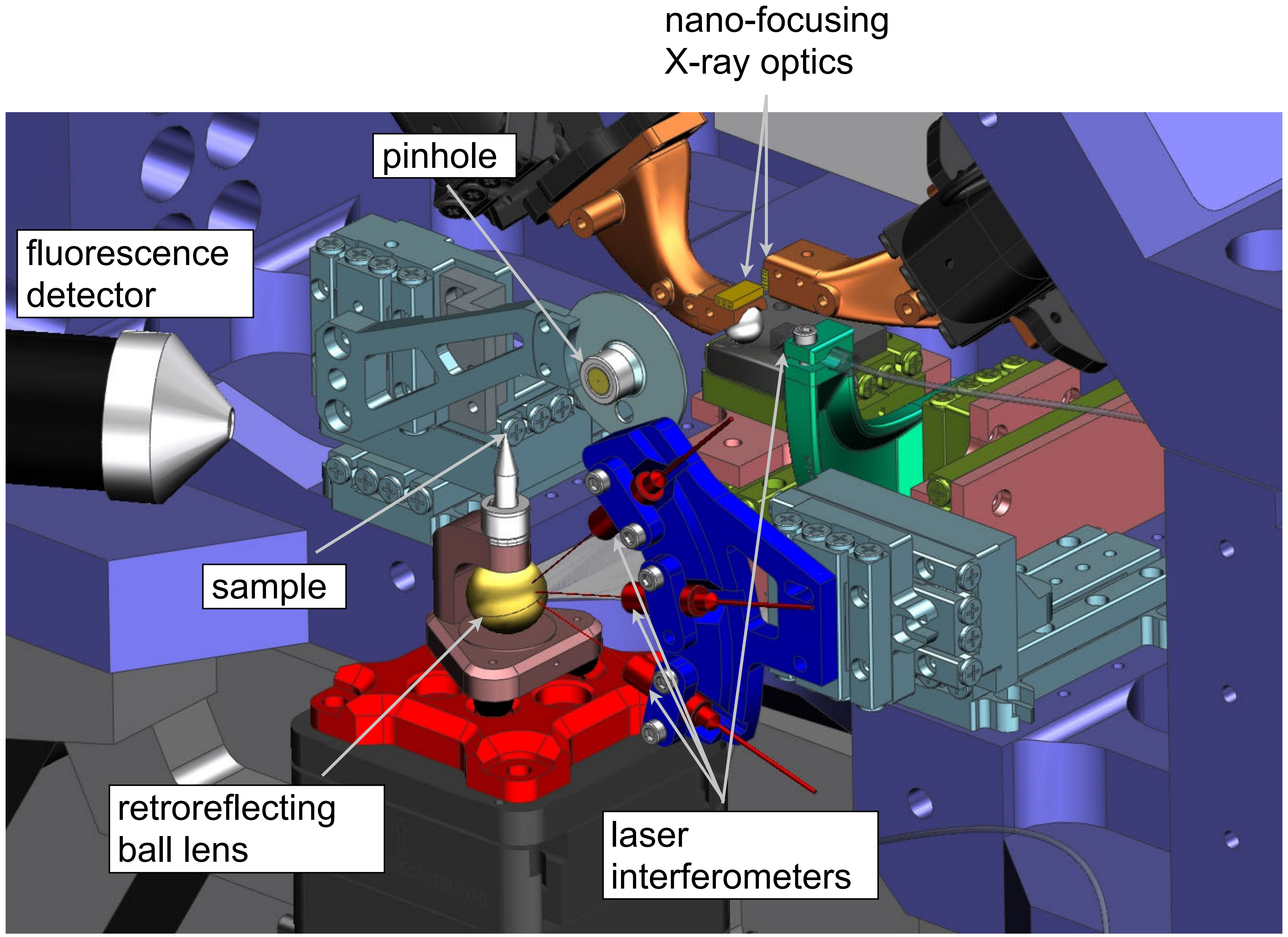

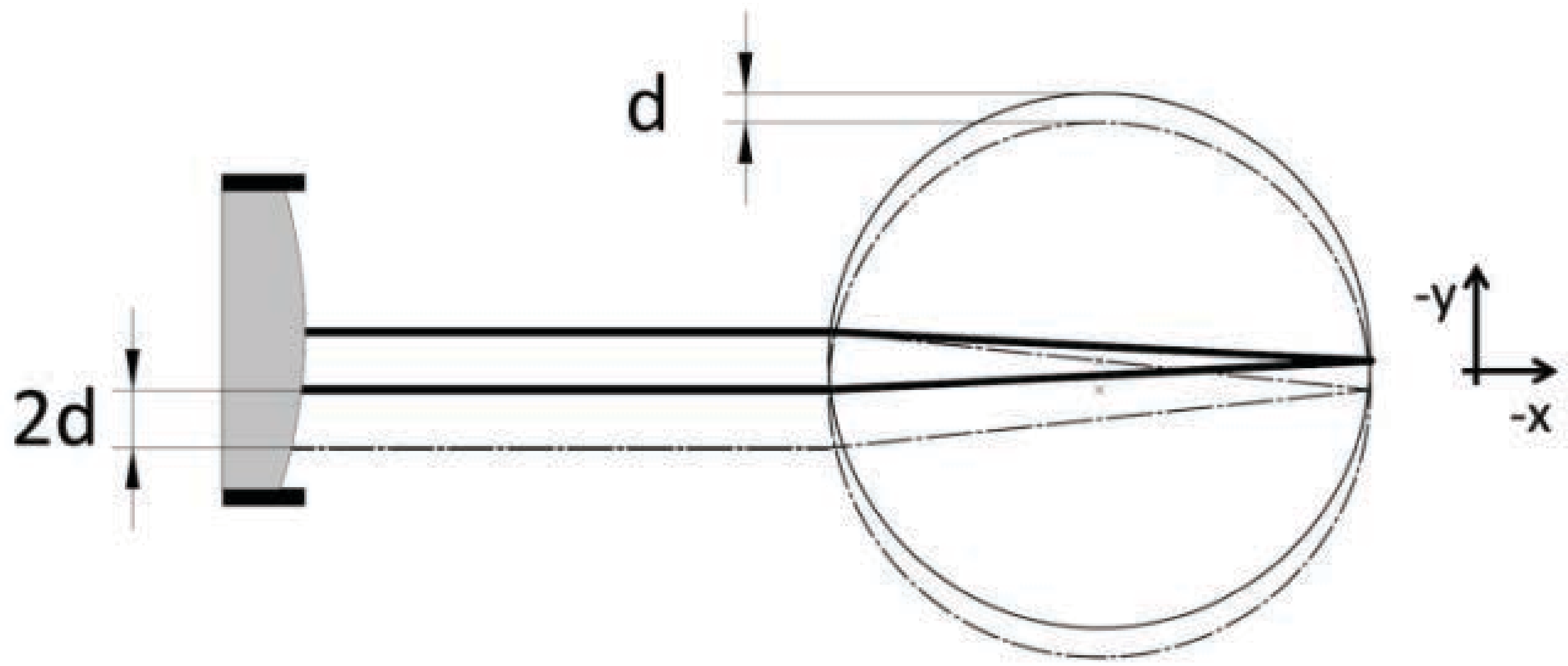

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PtyNAMi

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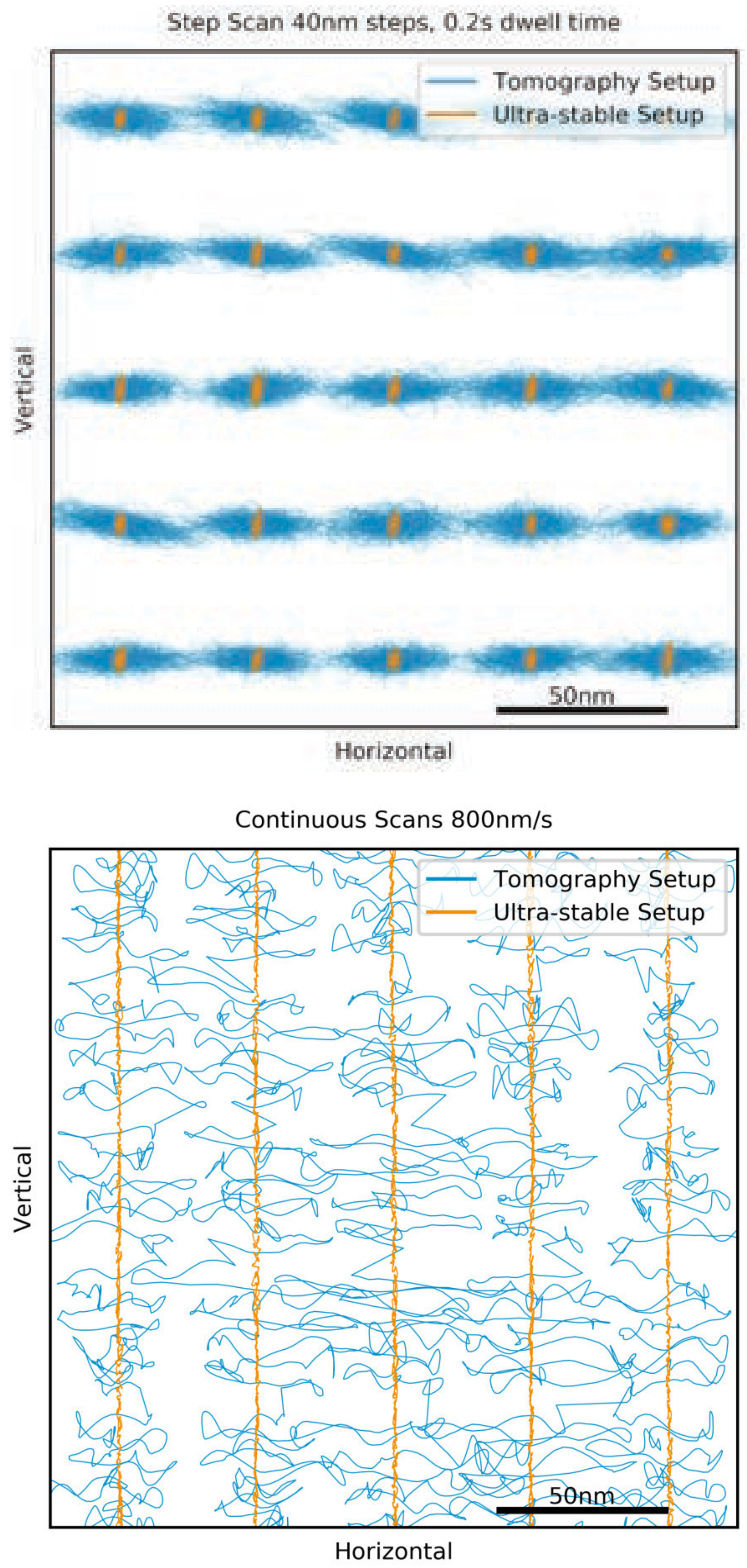
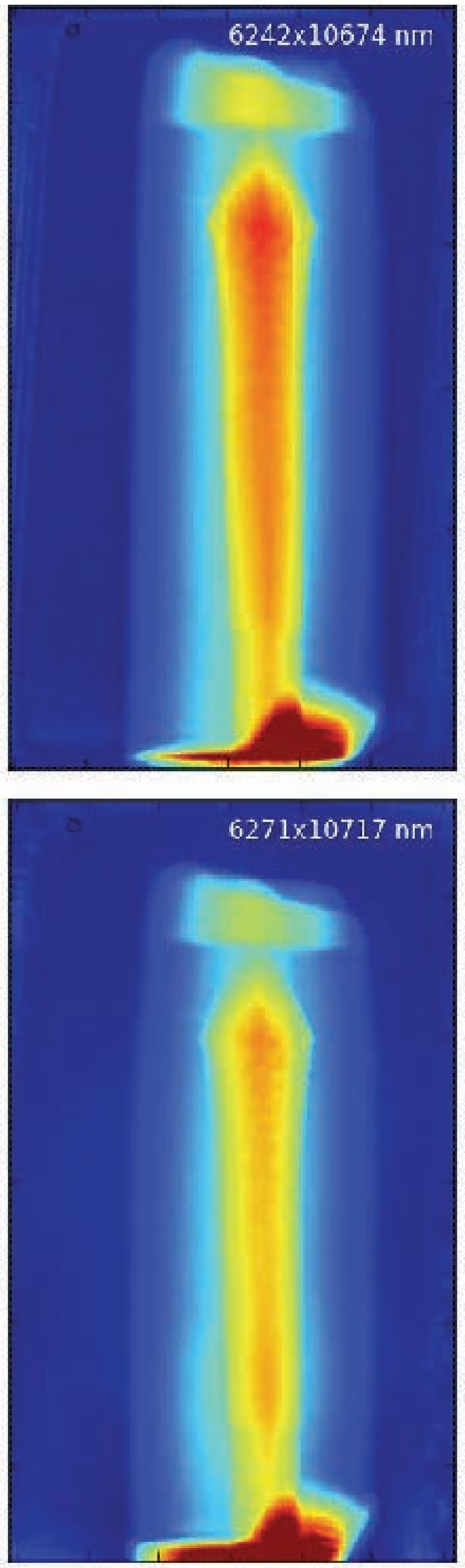
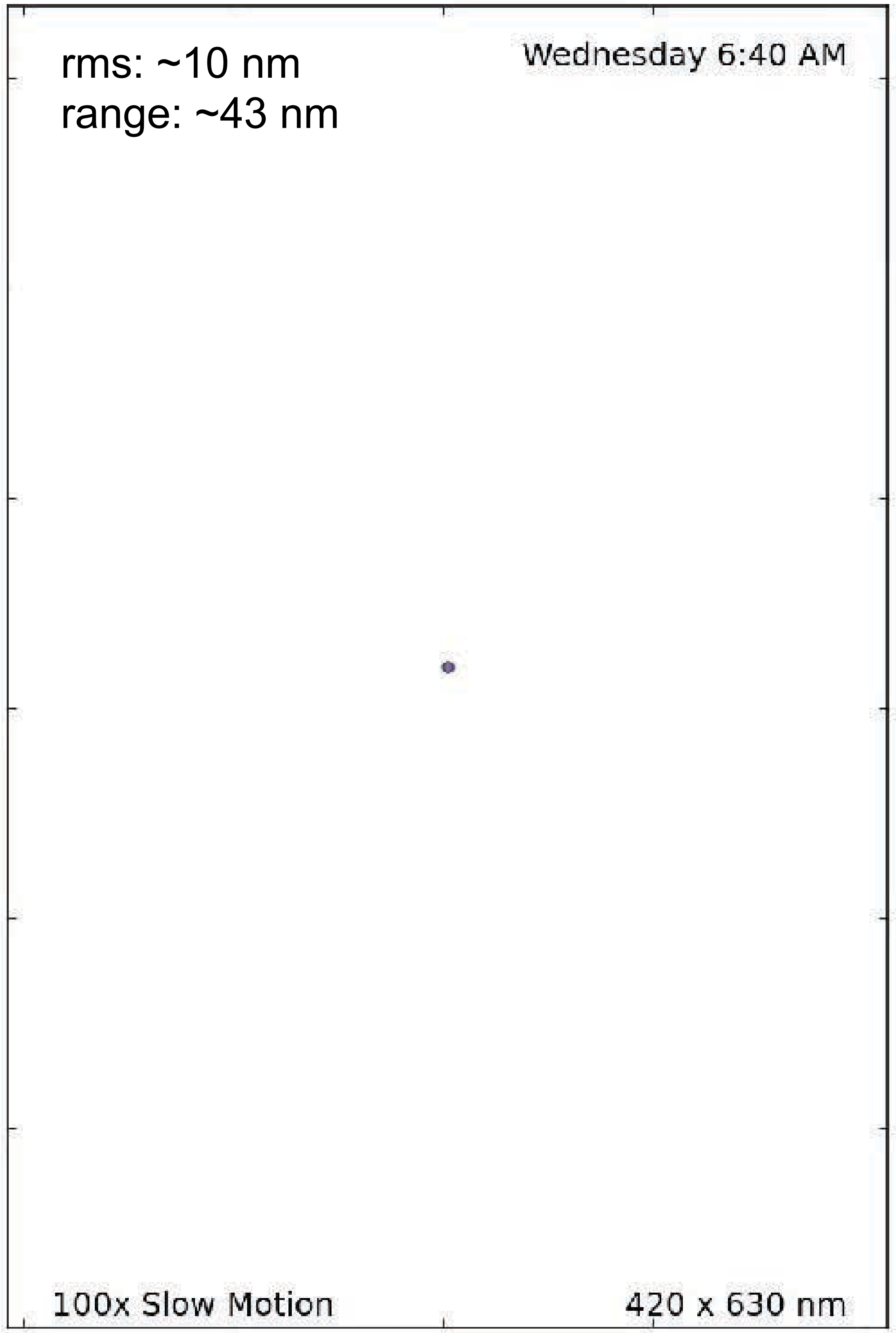
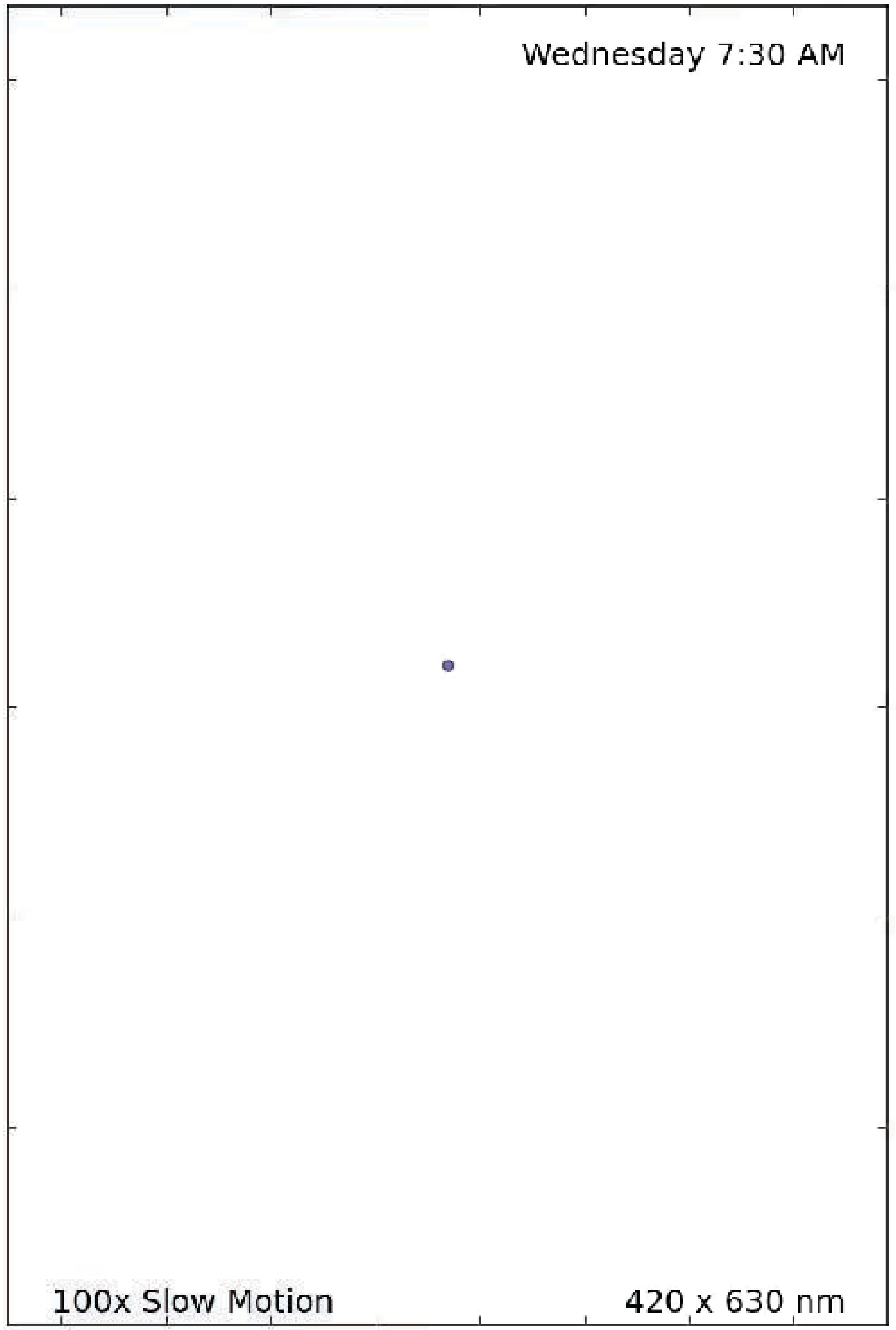




during construction work outside PETRA III



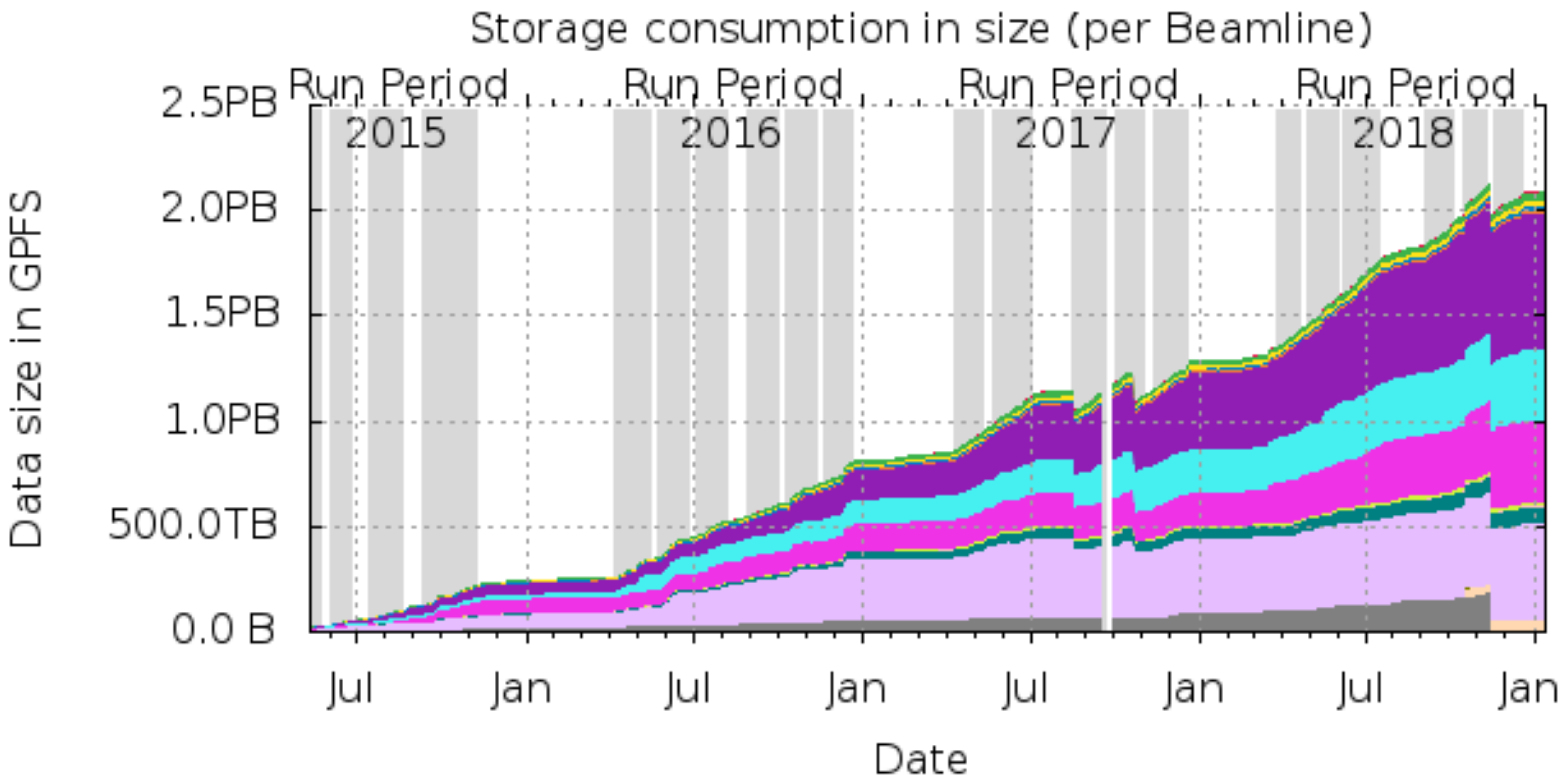


M Seyrich, *et al.*

GPFS: Total Use per Beamline

Data rate increases with development of faster detectors

4 Beamlines generate 80 % of the data on GPFS!



tomography (HZG)

micro-/nanoprobe

tomo & eng. mat. sci. (HZG)

macromolecular cryst.

Diffraction detectors running at high full-frame frequencies: “outrunning vibrations”.

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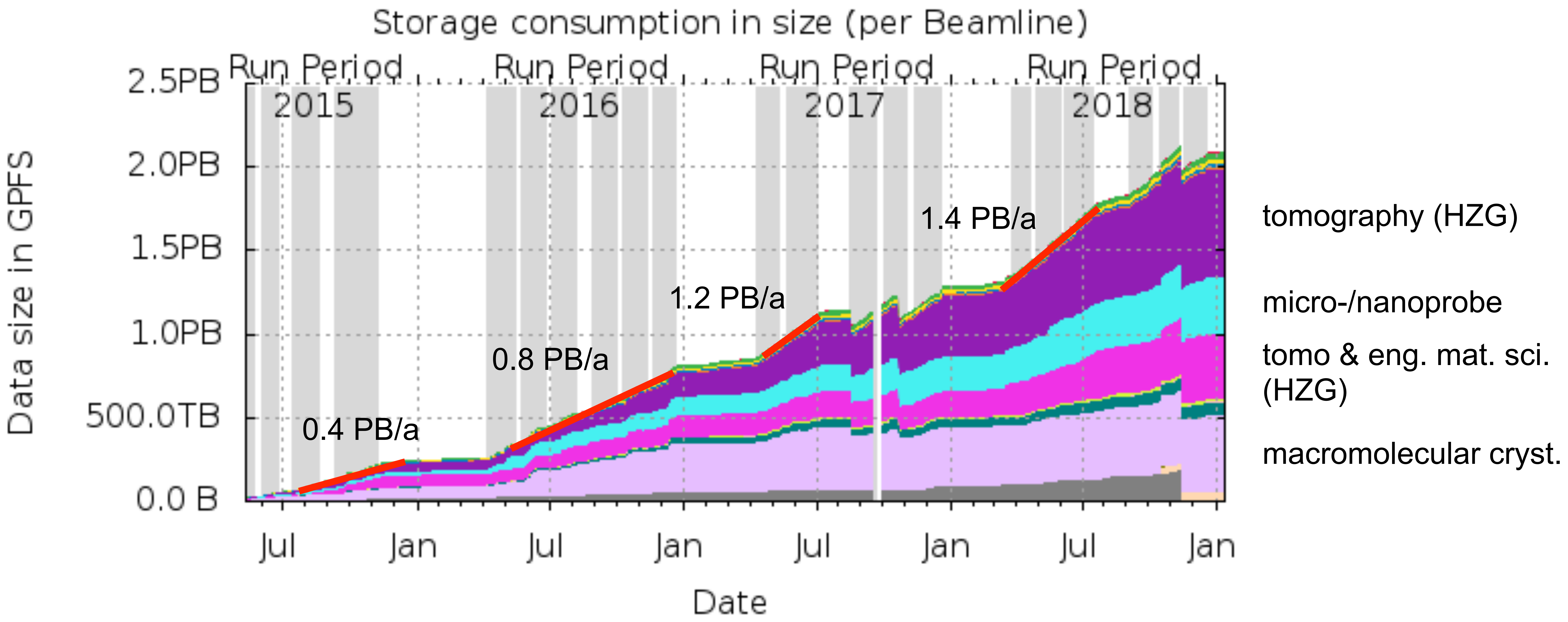
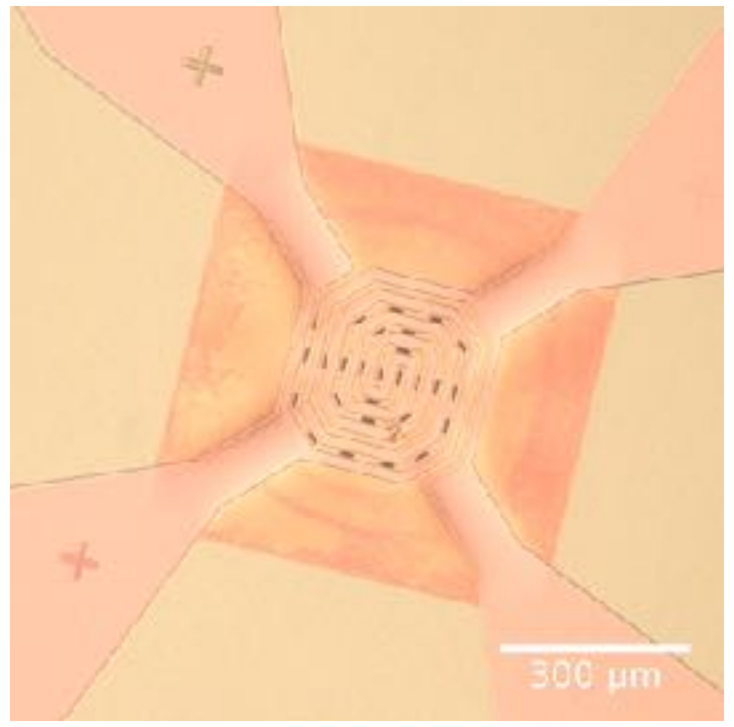


Diffraction detectors running at high full-frame frequencies: “outrunning vibrations”.

In-situ Ptychography

Example: In-Situ 2D Imaging of Gold Nanoparticles during Thermal Annealing

MEMS-chip



Why *in situ*?

- Most materials operate under specific environmental conditions.
- Uncover chemistry and structure of materials only present under reaction conditions.
- Reveal relationship between surface structure, materials composition and chemical properties.
- Imaging: retrieve local structural information in 3D.
- Design new and better materials.

In-situ Cell

In collaboration with KIT: Thomas L. Sheppard, Yakub Fam, Jan-Dierk Grunwaldt, *et al.*

Design requirements:

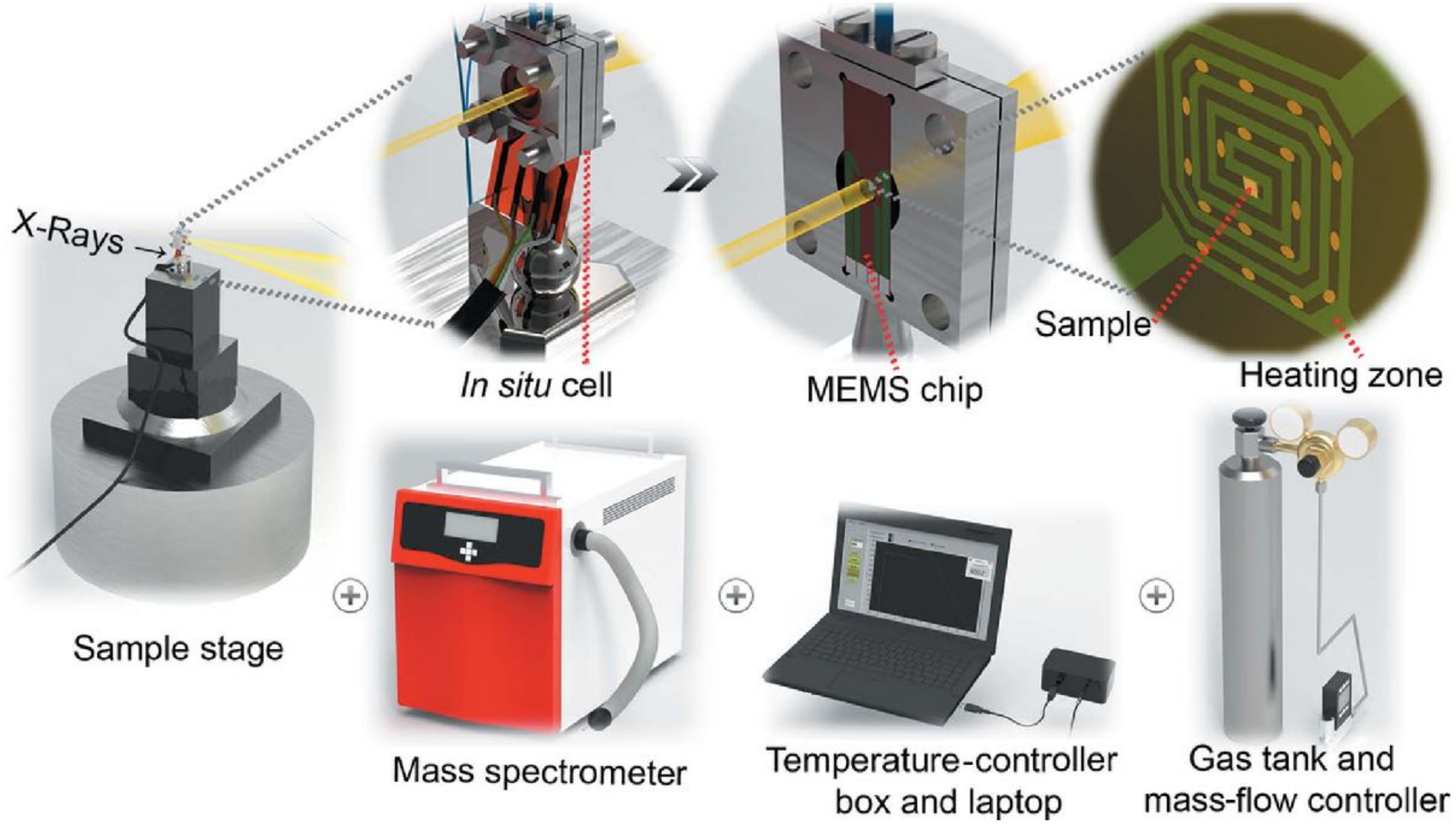
> small device to minimize the load on the piezoscanner (positioning accuracy)

> remote temperature control

> remote mass-flow control

> mass spectrometer for gas analysis

> compatible to limited-angle tomographic applications



Y. Fam, *et al.*, J. Synchrotron Rad. **26**, 1769-1781 (2019).

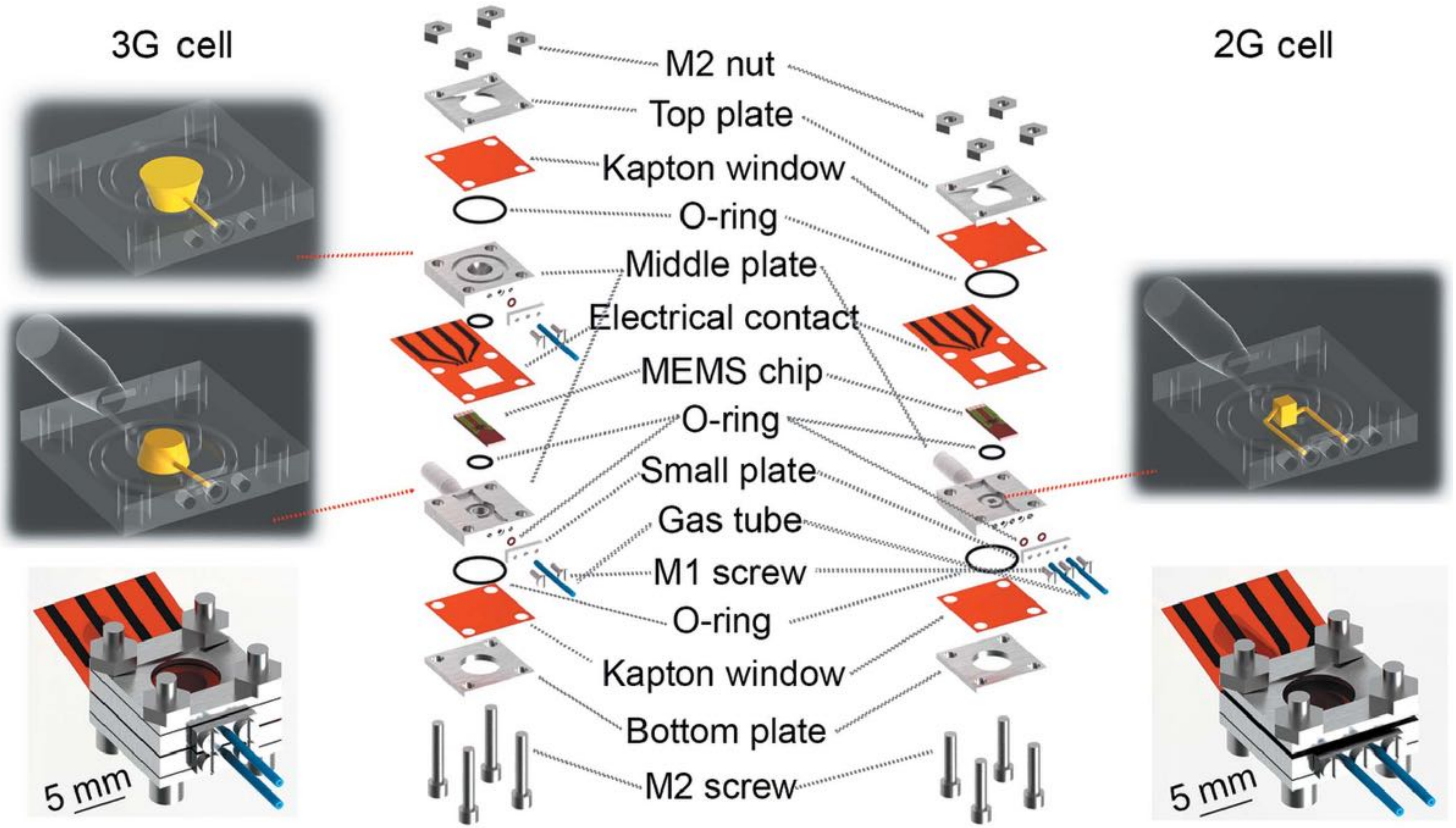
Different Generations of *In-situ* Cells

In collaboration with KIT: Thomas L. Sheppard, Yakub Fam, Jan-Dierk Grunwaldt, *et al.*

Cell design:

- based on commercial MEMS-chips
- temperature control up to 1573K
- pressure up to 100kPa
- compatible with hard X-ray ptychography and transmission electron microscopy

Multiple generations of cells have been designed and tested!



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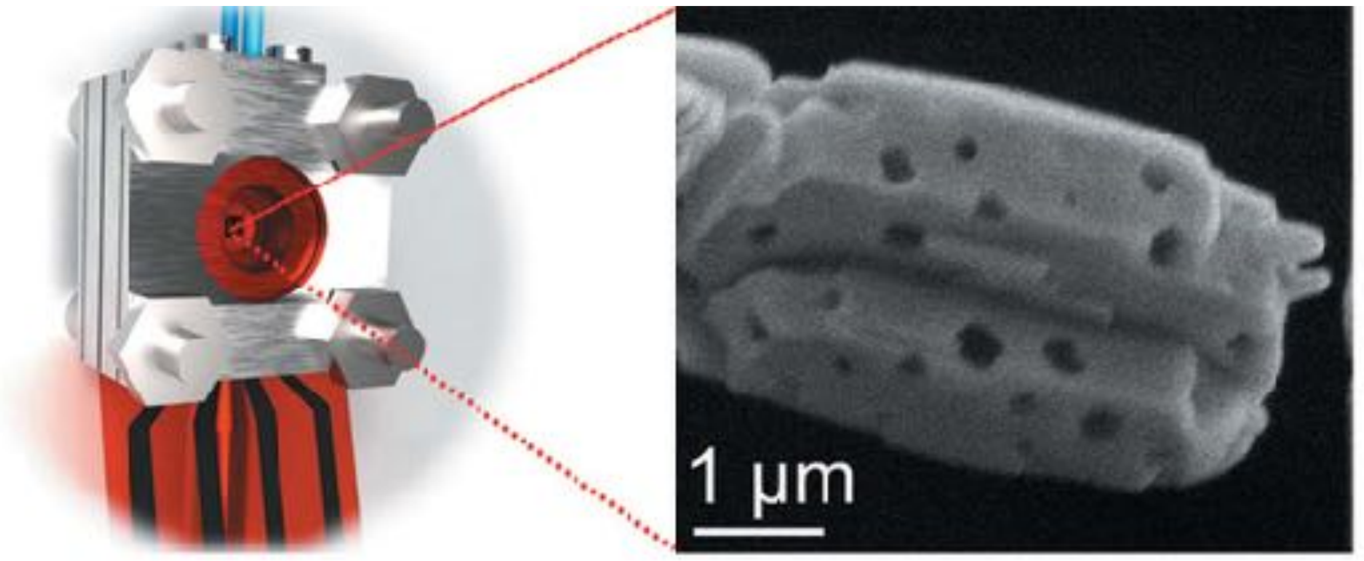
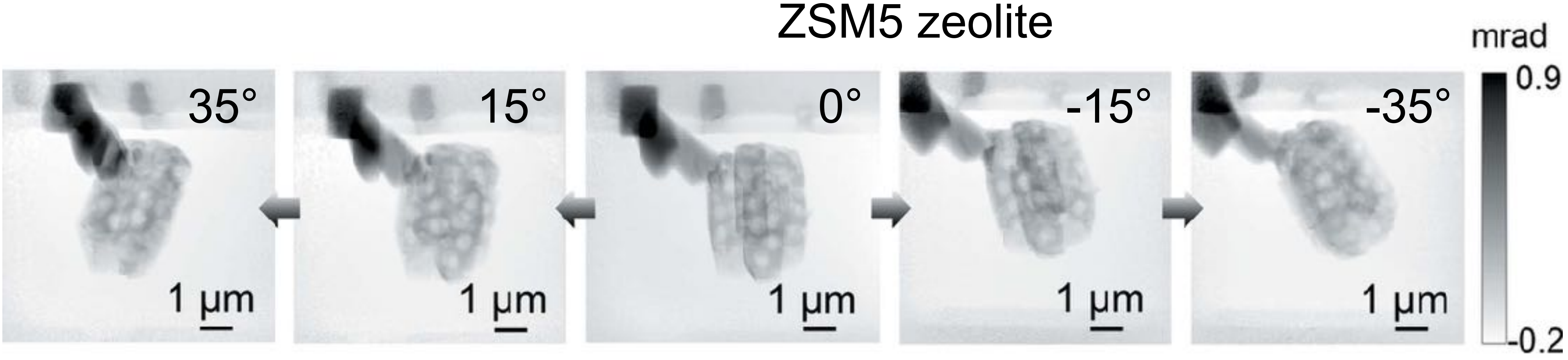
Limited-Angle Ptychography *In Situ*



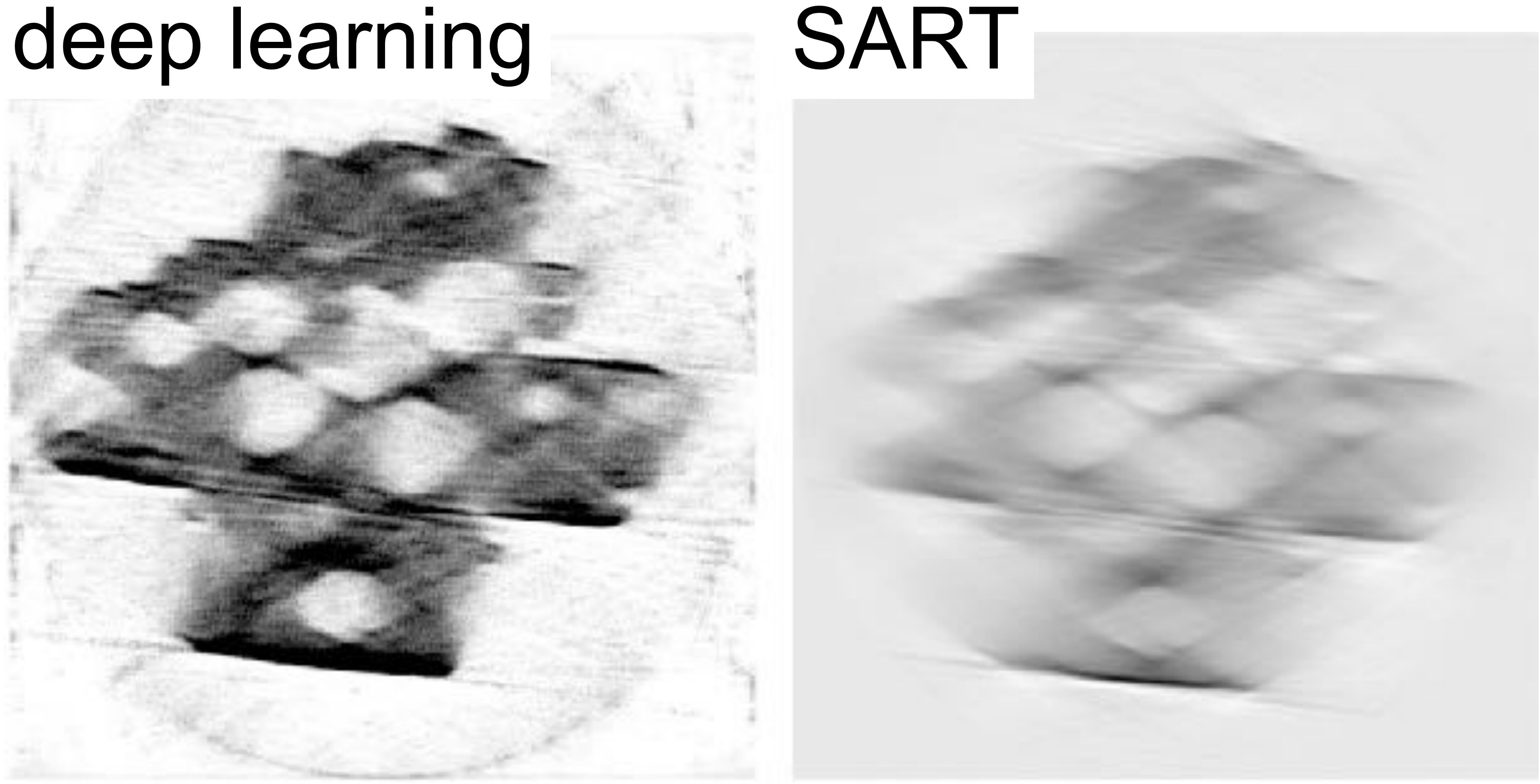
ZSM5 zeolite

Accessible angular range limited to about +/- 35°



deep learning

SART



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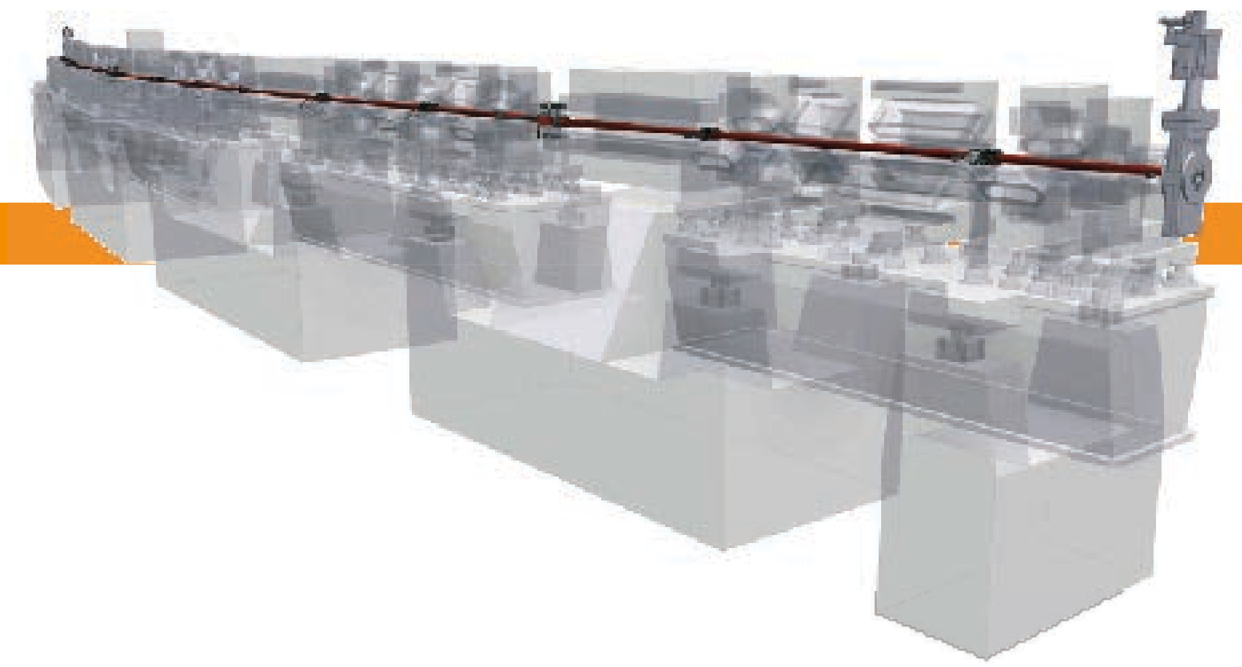
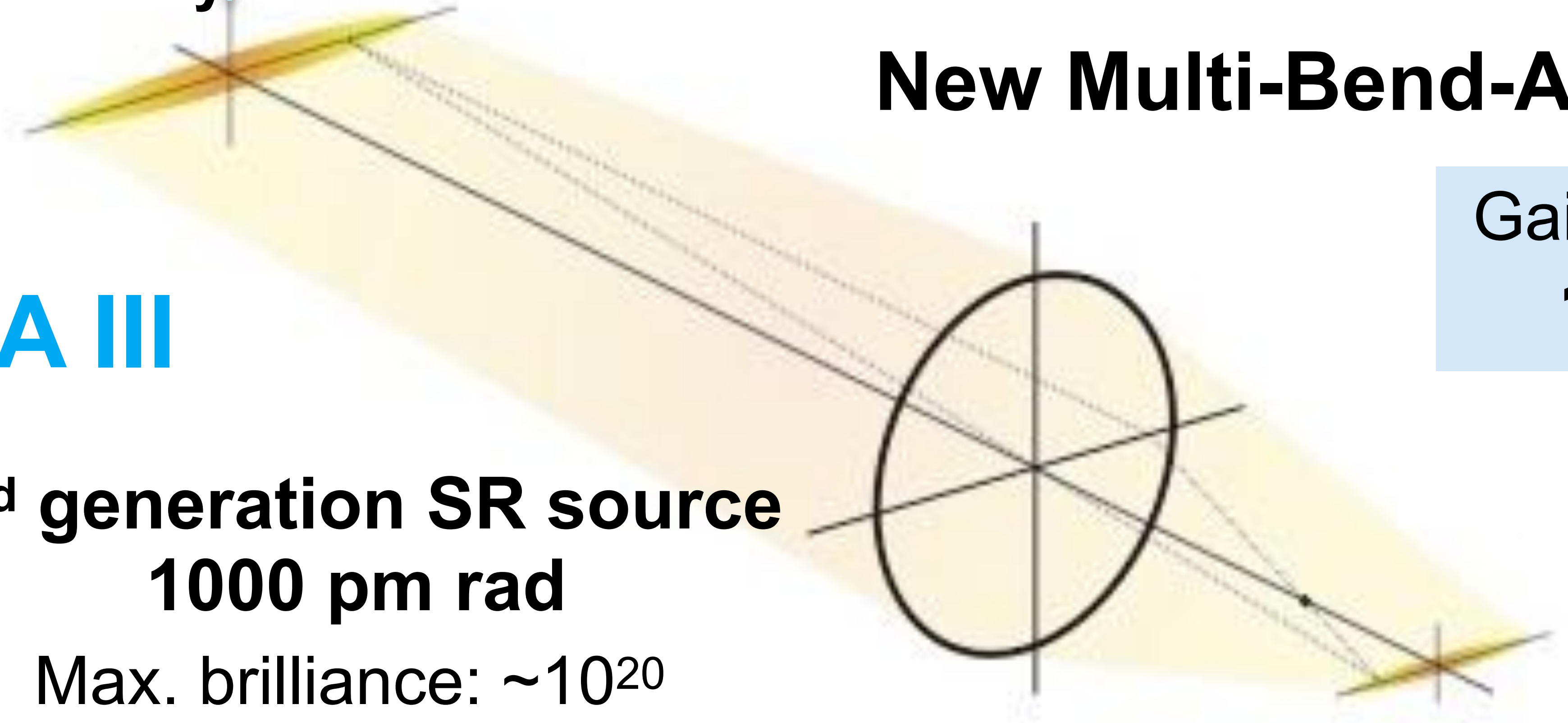
Perspectives: PETRA IV



source size:
140 µm x 6 µm

asymmetrical
x-ray beam

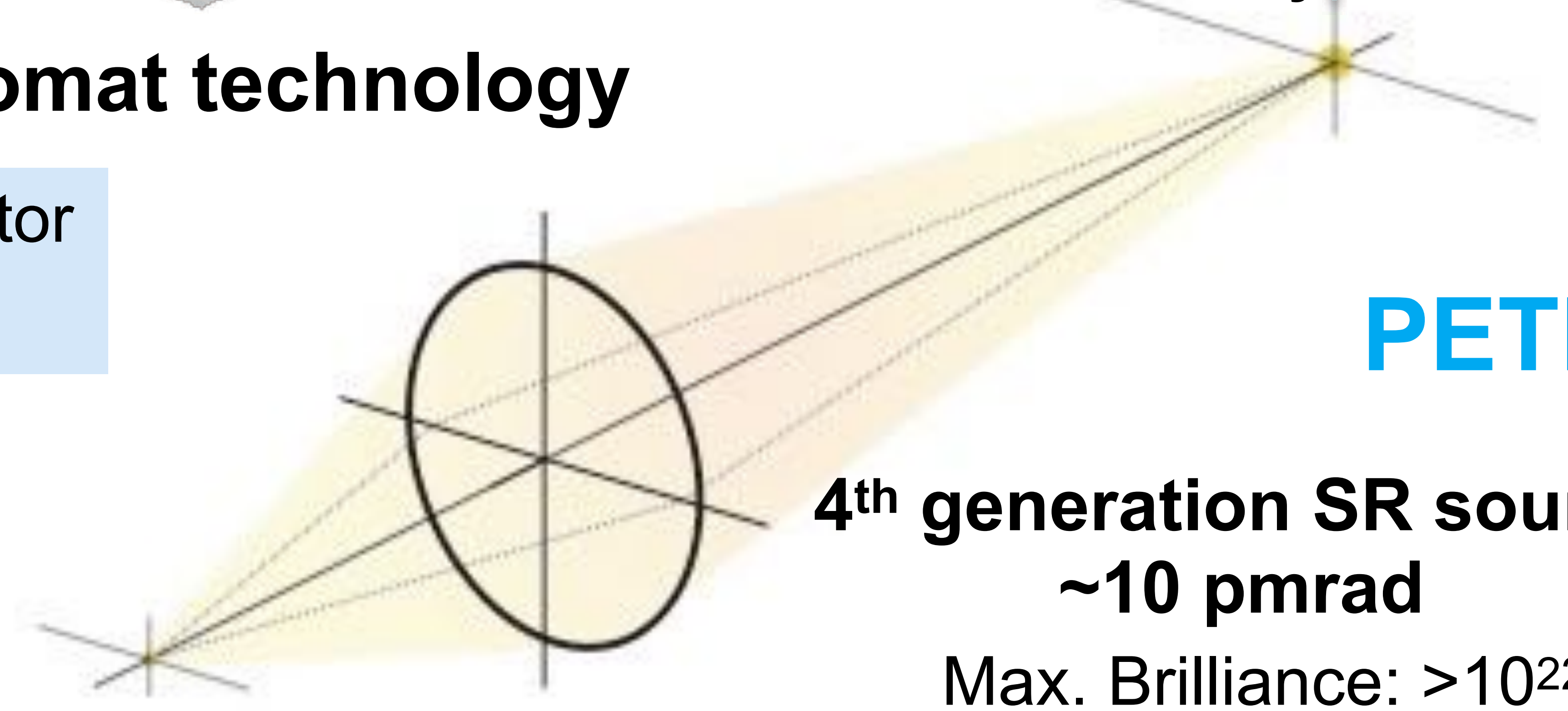


New Multi-Bend-Achromat technology

Gain factor
100

source size:
6 µm x 6 µm

round
x-ray beam



PETRA III

3rd generation SR source
1000 pm rad

Max. brilliance: ~10^{20}
coherence: ~0.1-1%

nanometer focusing

PETRA IV

4th generation SR source
~10 pmrad

Max. Brilliance: >10^{22}
Coherence: > 25%

PETRA IV

> new multi-bend-achromat (MBA) technology +
> 2.3 km circumference (largest SR source)
emittance scales as 1/(circumference)^3

→ diffraction limited down to a wavelength of 1 Å (ultimate storage ring)

Qualitative step in synchrotron analytics

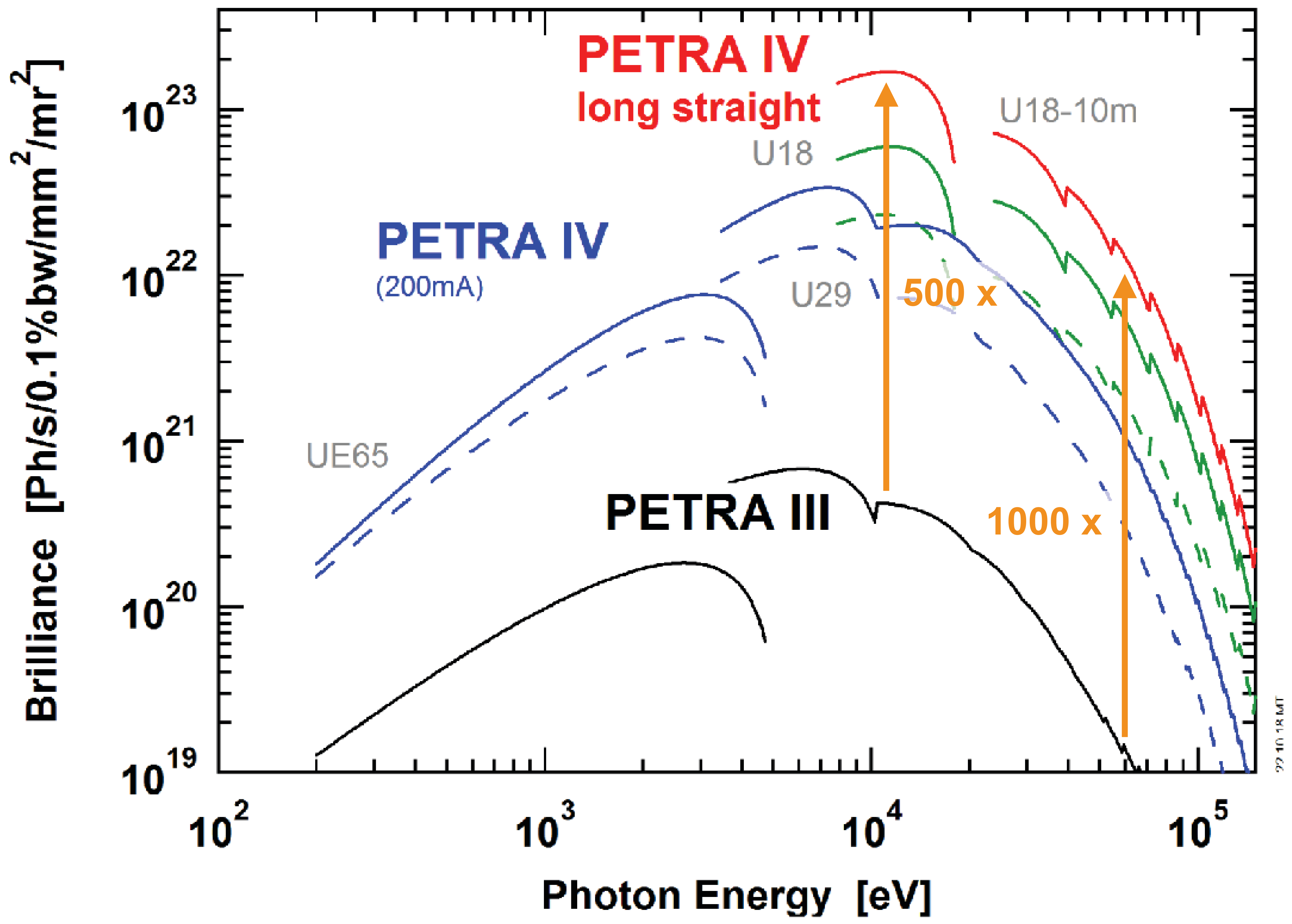
In-situ 3D-microscopy on nanometer scale

Operando nanoimaging of

> structure, chemistry
> electronic and magnetic properties
> dynamics on the sub-nanosecond scale

PETRA IV

Expected Brightness



Based on current design:

- > emittance:
  - ➔ coherence mode: < 20 x 4 pmrad²
  - ➔ timing mode: < 50 x 10 pmrad²
- > undulators: 5 m, 10 m
- > optimized beta (in 10 m section): 2 x 2 m²
- > ring current: 200 mA

Brightness increase by

- ➔ 500 x (hard X-rays)
- ➔ 1000 x (high-energy X-rays)

PETRA IV brightness at 100 keV same as for 10 keV at PETRA III today!!

C. G. Schroer, et al., JSR **25**, 1277 (2018).

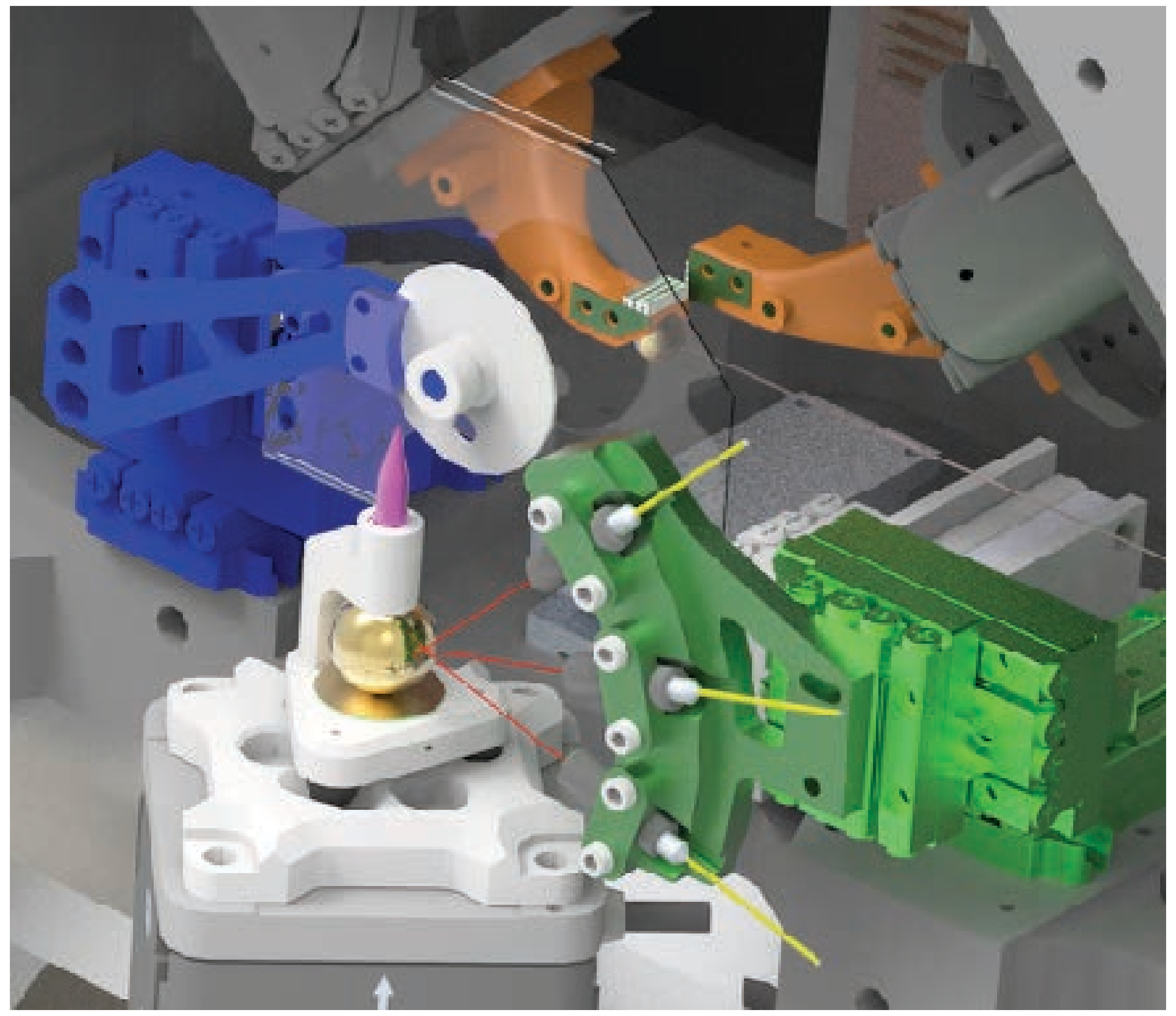
Summary and Outlook

PtyNAMi

- optimize coherent flux
- high-performance X-ray optics
- high mechanical stability and control
- low scattering background

In-situ cell developments at beamline P06

- based on commercial MEMS-chips
- temperature and pressure control, gas analysis
- limited-angle ptychographic tomography
- compatible with hard X-ray ptychography and transmission electron microscopy



Diffraction-Limited Storage Rings

- high coherent photon flux:
  fast *in-situ* hard X-ray microscopy with
  high spatial resolution (towards 1nm)

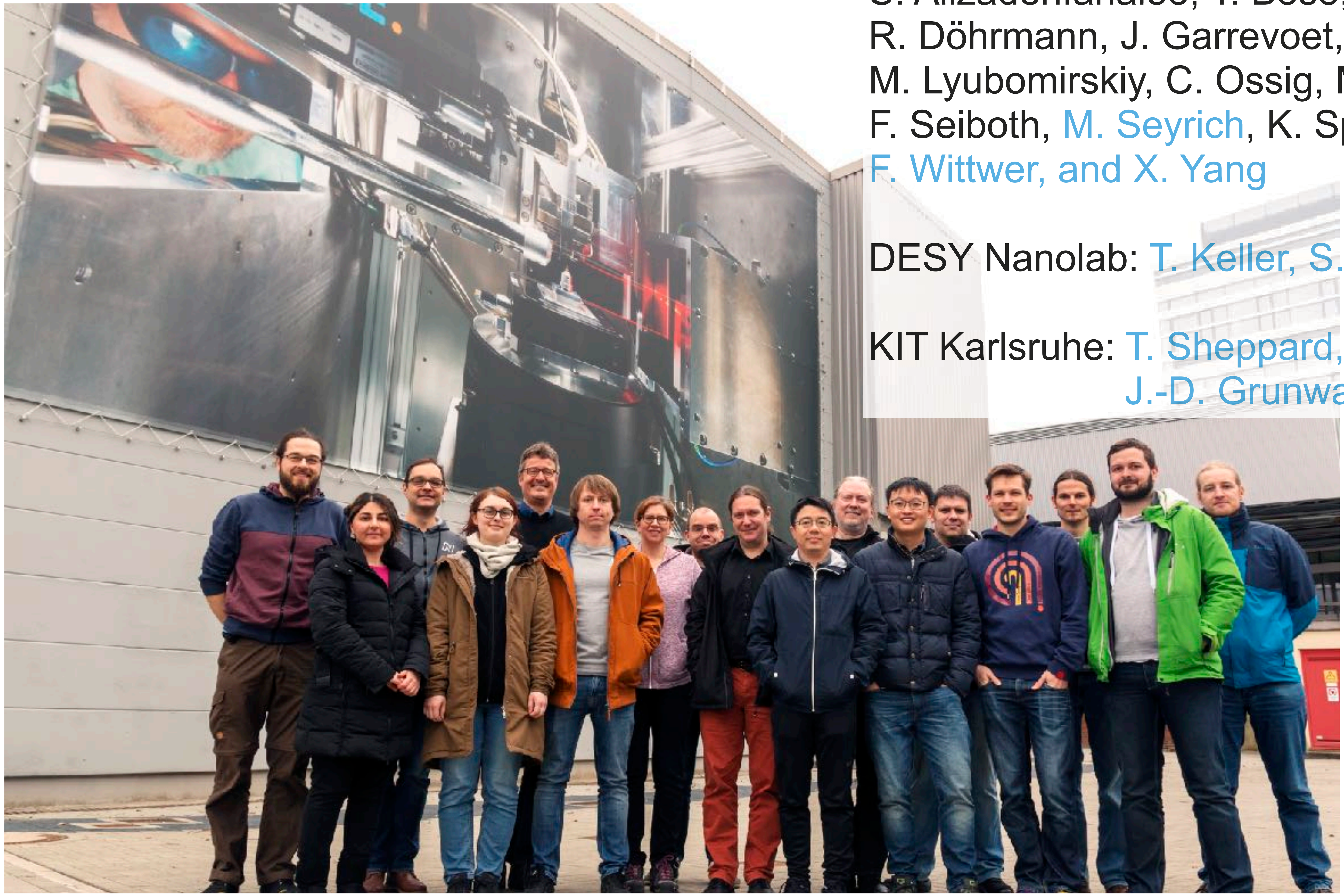
Acknowledgements

Prof. Dr. Christian G. Schroer (DESY and Universität Hamburg)
Dr. Gerald Falkenberg (DESY - P06 beamline responsible)

S. Alizadehfanaloo, T. Böse, S. Botta, D. Brückner, R. Döhrmann, J. Garrevoet, J. Hagemann, M. Kahnt, M. Lyubomirskiy, C. Ossig, M. Scholz, A. Schropp, W. Schröder, F. Seiboth, M. Seyrich, K. Spiers, M. Stückelberger, P. Wiljes, F. Wittwer, and X. Yang

DESY Nanolab: T. Keller, S. Kulkarni, A. Stierle

KIT Karlsruhe: T. Sheppard, Y. Fam, J. Becher, S. Weber, and J.-D. Grunwaldt



Thank you very much for your attention!