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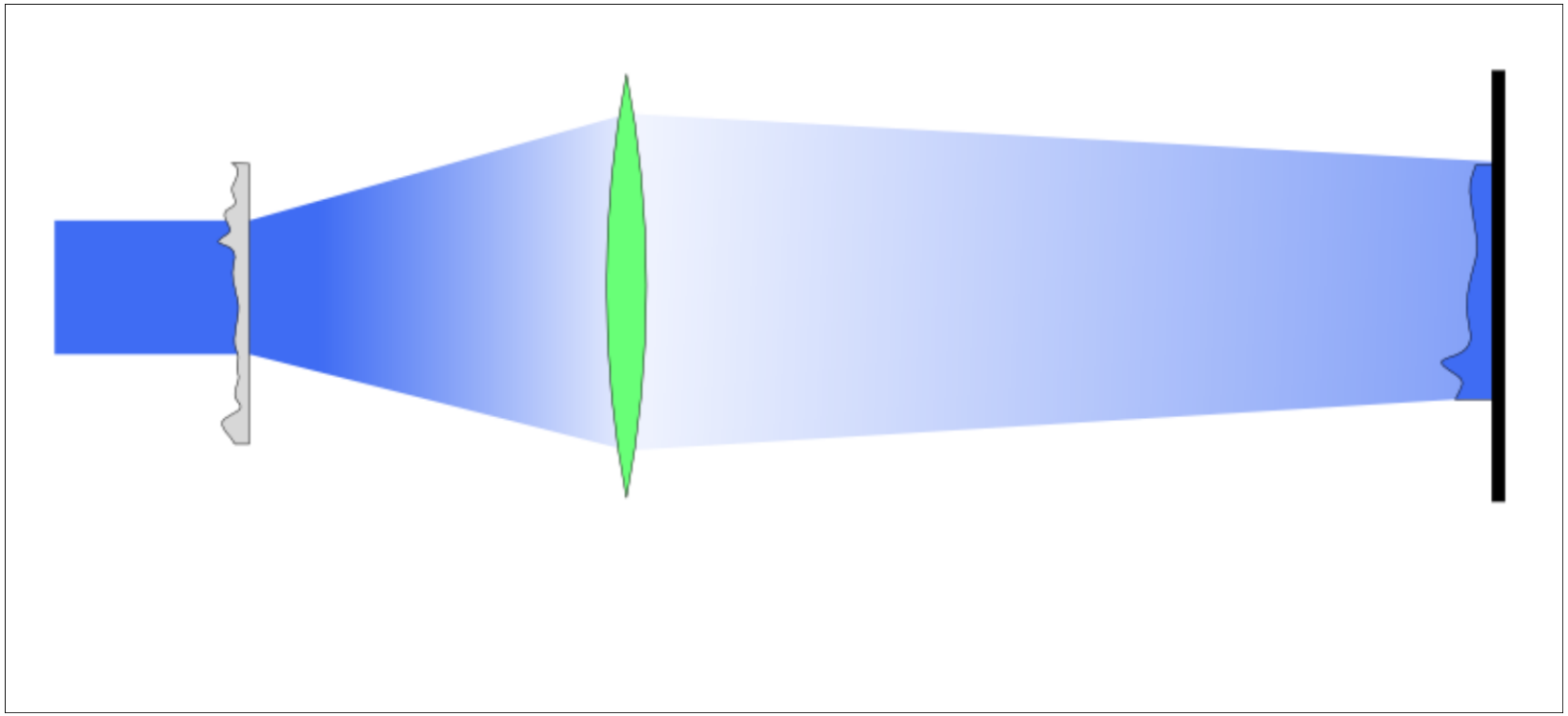
Mirko Holler :: Paul Scherrer Institut

OMNY – an instrument for high-resolution multi-keV X-ray tomography

Full field imaging

Traditional (x-ray, bright field) microscopy

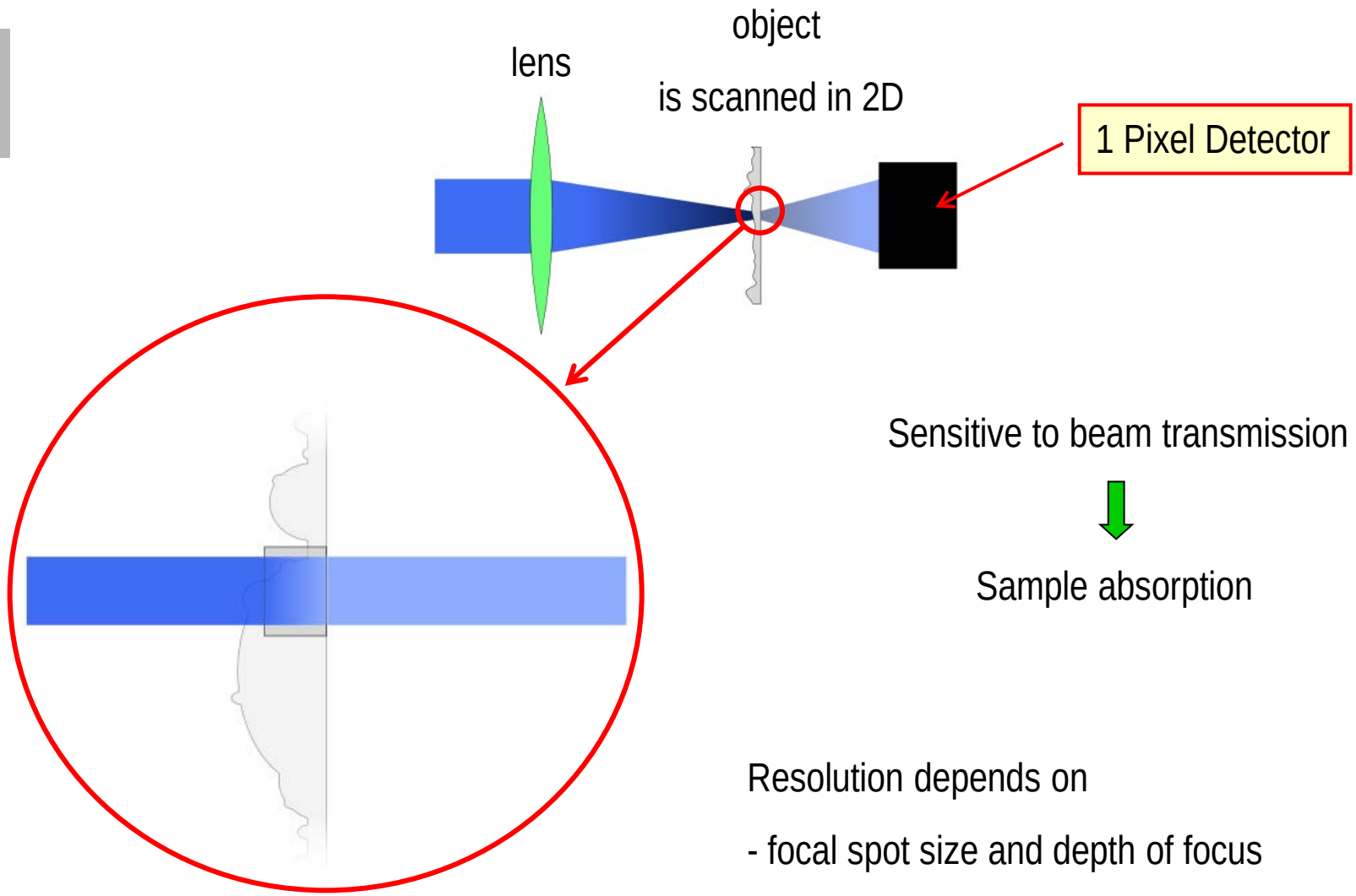


Object is directly imaged to a 2D detector.

Resolution depends on imaging optics, limiting factor especially at hard X-rays

Low efficiency of X-ray optics (Fresnel zone plate approx. 10%) → increased dose

Scanning Transmission X-ray Microscopy (STXM)



Sensitive to beam transmission

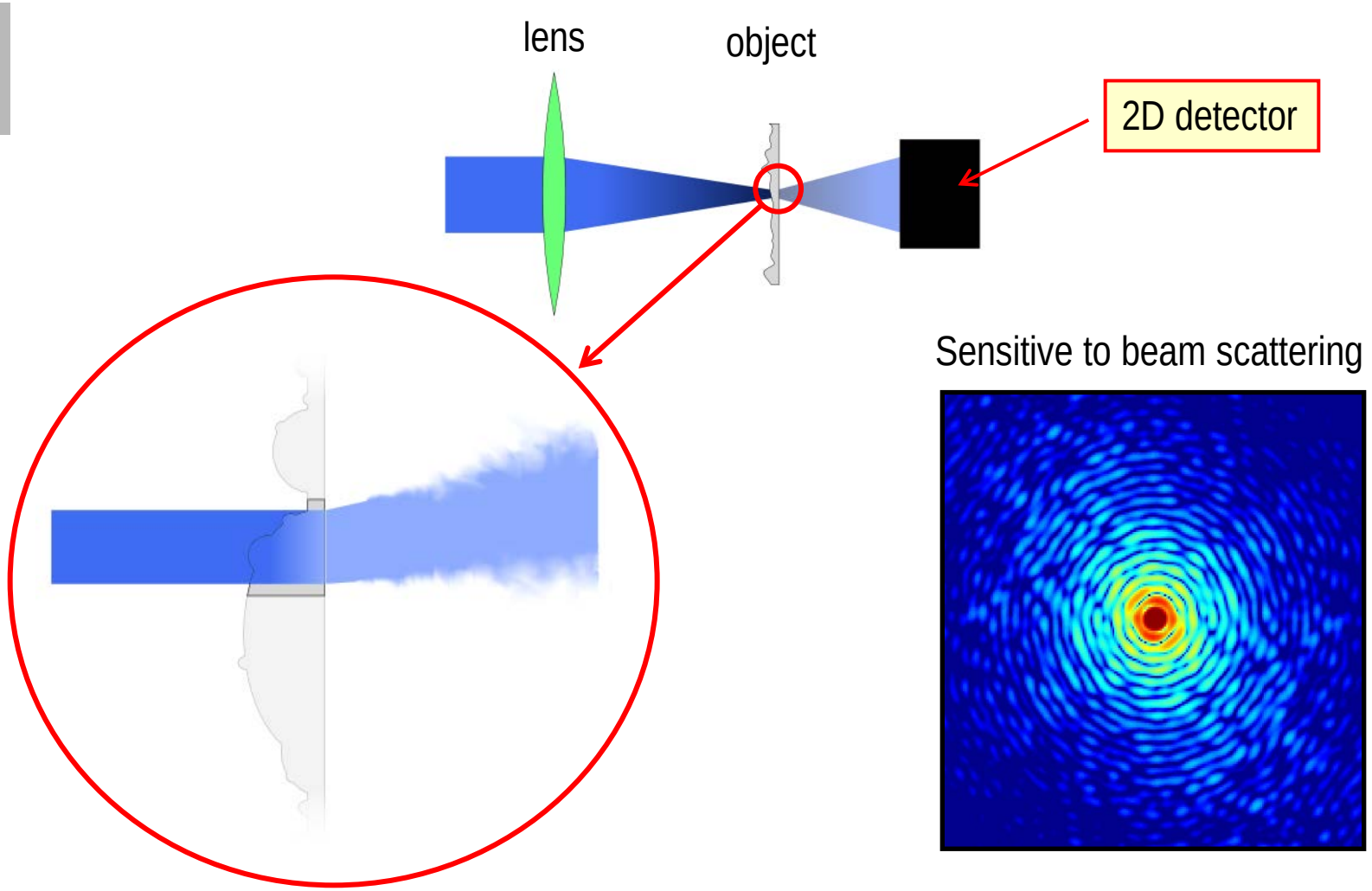
Sample absorption

Resolution depends on

- focal spot size and depth of focus

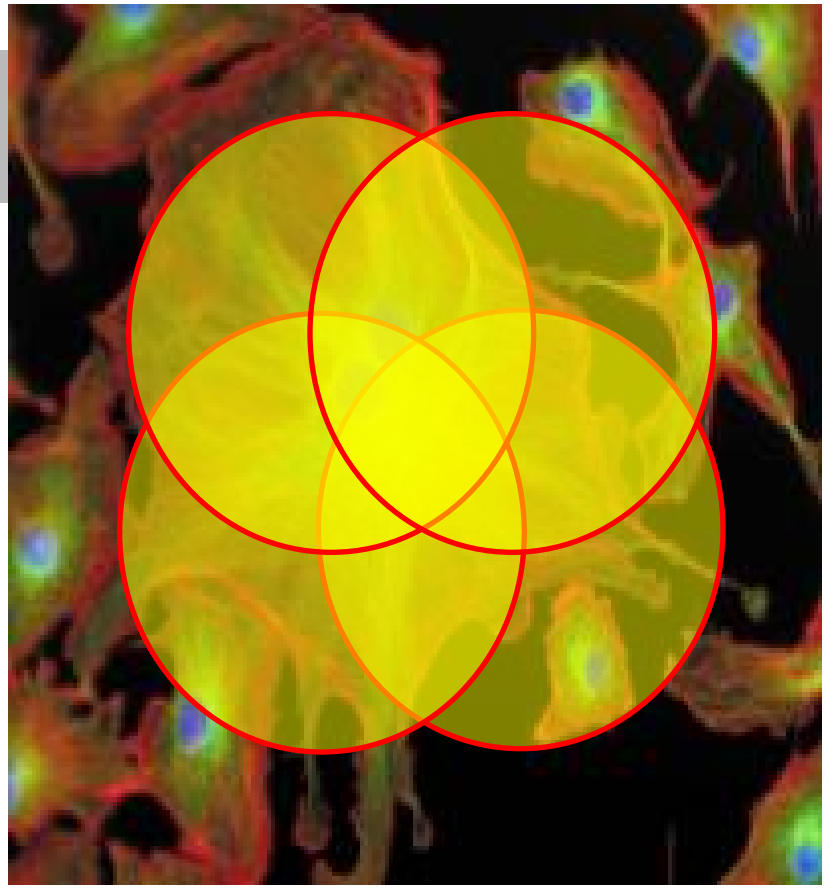
- positioning accuracy of sample in the beam

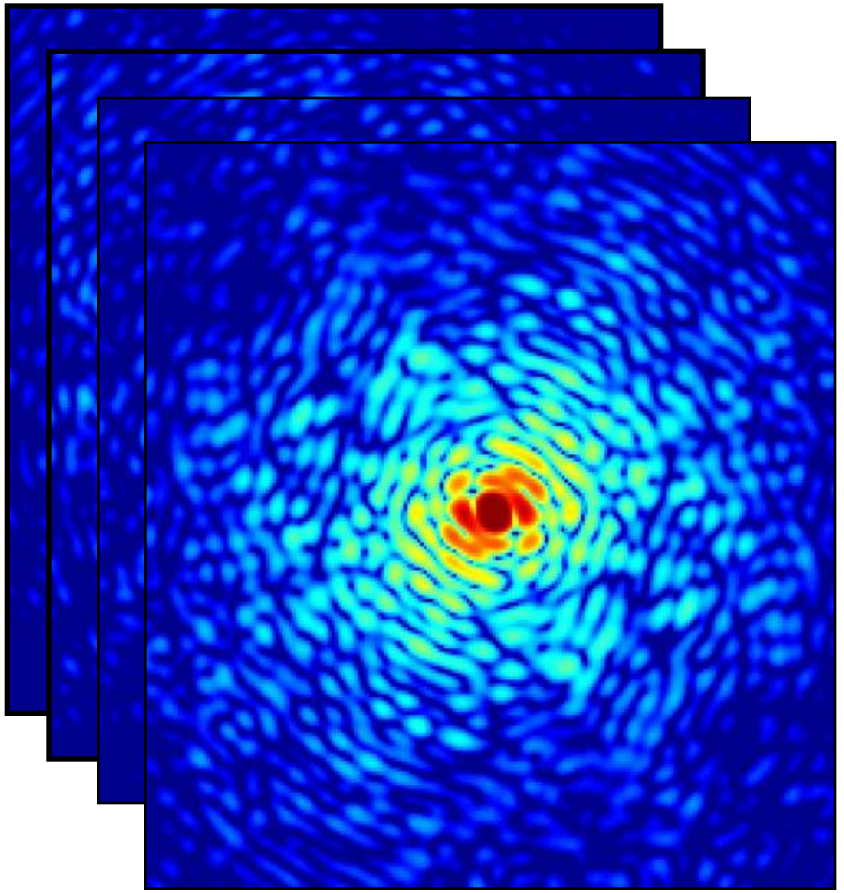
Scanning X-Ray Diffraction Microscopy
Ptychography



Combining coherent diffractive imaging with STXM

Scanning X-Ray Diffraction Microscopy
Ptychography

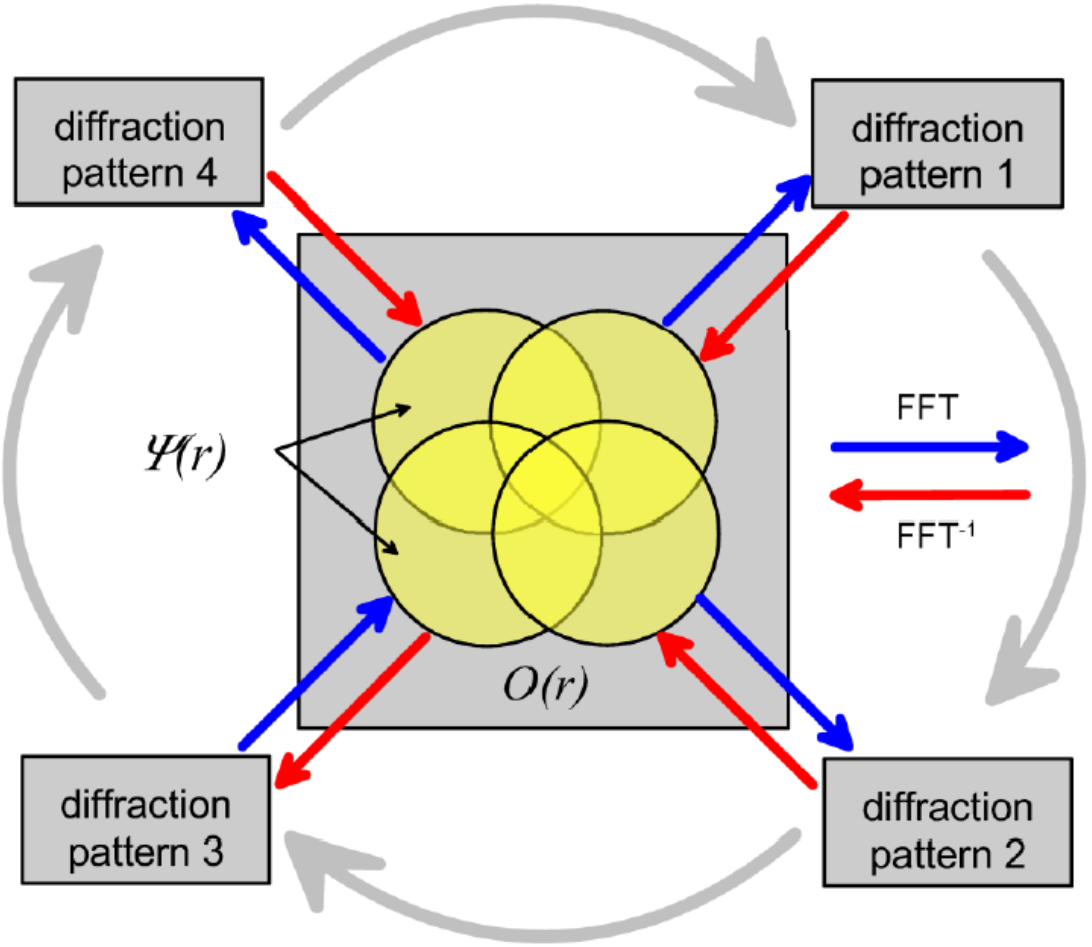




overlapping illumination → shared information → can be analyzed by iterative phase retrieval algorithms

Thibault P., et al., SCIENCE 321, 379 (2008).

Scanning X-Ray Diffraction Microscopy
Ptychography



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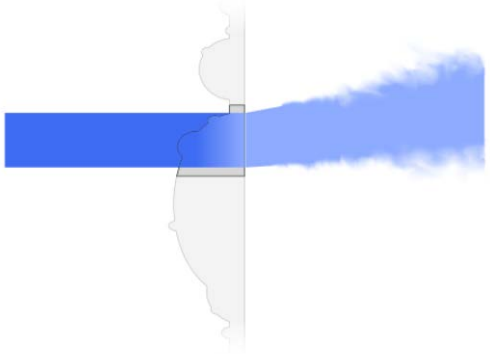
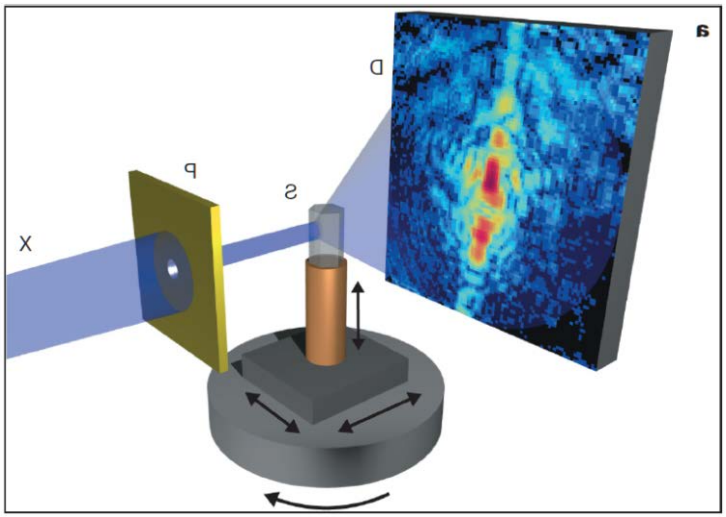
Scanning X-Ray Diffraction Microscopy
Ptychography



+ Resolution not determined by spot size of the X-rays
+ Suitable to hard X-rays (multi keV) → thick samples
+ Lens-less - no imaging optics between sample and detector
  → dose efficient, resolution not limited by optics
+ Phase and absorption contrast
+ Quantitative: imaging the electron density

- Data analysis is involved
- Resolution depends on sample positioning accuracy → involved instrumentation
- Coherent illumination of the sample needed!

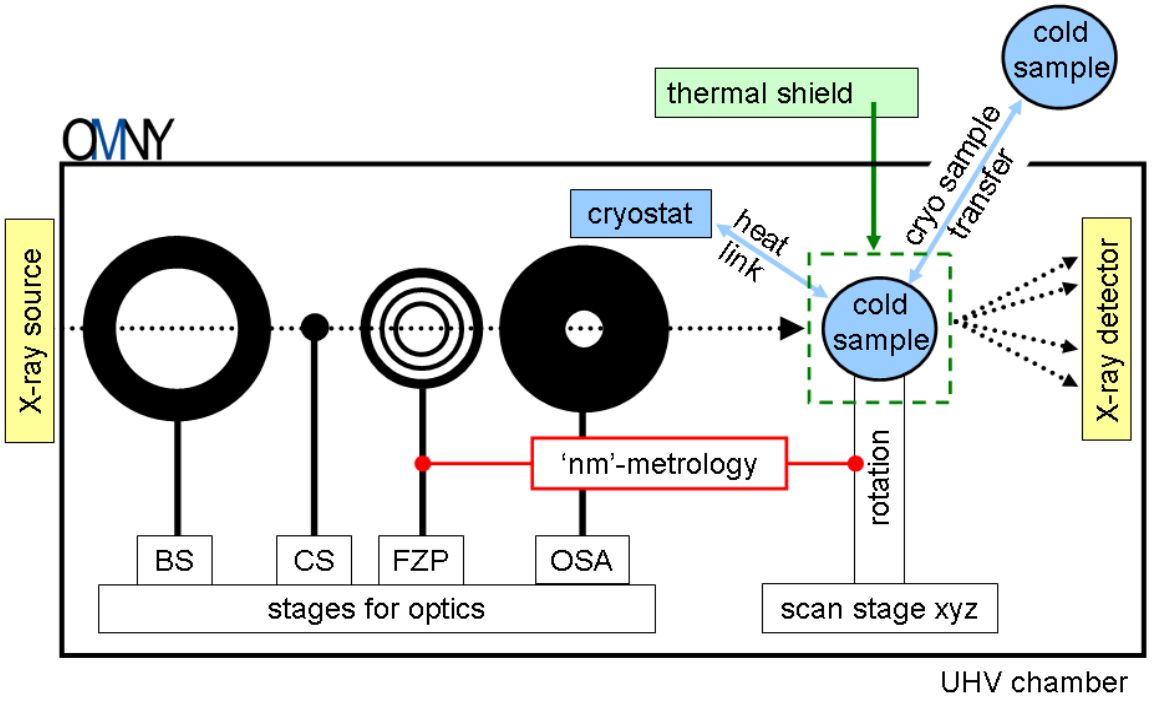


Access 3D information via
computed tomography
→ obtain many projections of the sample
at different angle of incidence.

OMNY – tOMography Nano crYo endstation

Goal: Development of a **dedicated instrument** to perform tomography at the **nano-scale** on **biological samples** and condensed matter physics samples using Ptychography in a **controlled sample environment**.

- Nano positioning of the sample
- Rotation of the sample for tomography (vertical axis)
- Beam conditioning: Fresnel Zone-plate illumination
- Cryogenic temperatures
- UHV environment
- Sample transfer of frozen bio-samples (contamination free)



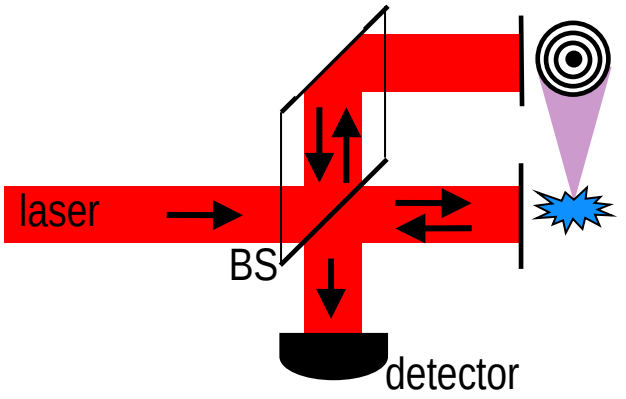
Resolution goal: 10 nm in 3D

→ aiming at positioning accuracy of sub 10 nm
(sample versus beam = sample versus X-ray lens)

Metrology for OMNY

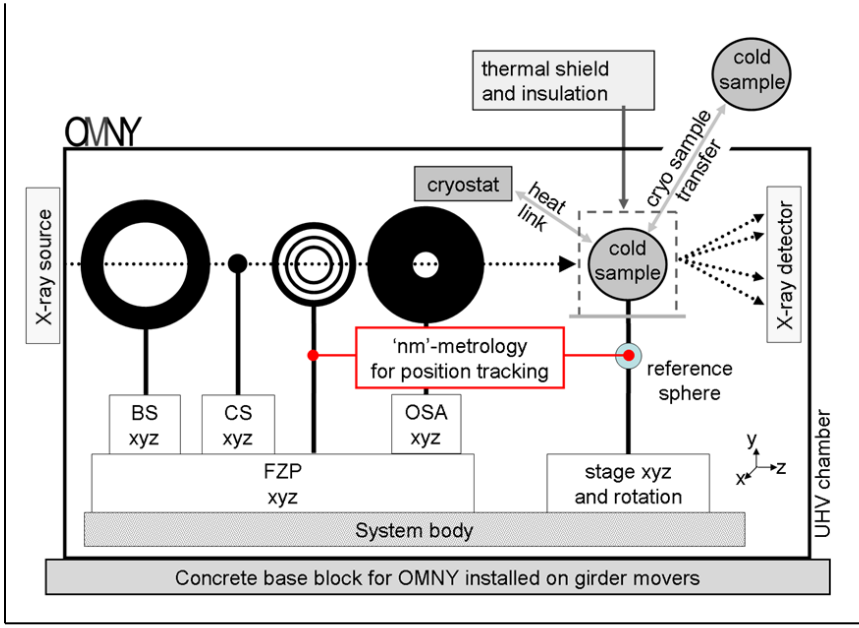
Accurate sample positioning
→ measure relative position FZP vs. sample

vertical rotation axis
→ for vertical measurement a plane mirror can be used



Fresnel zone plate

sample



Heterodyne laser interferometry

- Resolution: 0.3 nm
- non-contact, long range
- exteroceptive:
  include thermal drifts in the measurement
- linear, accurate and stable scale

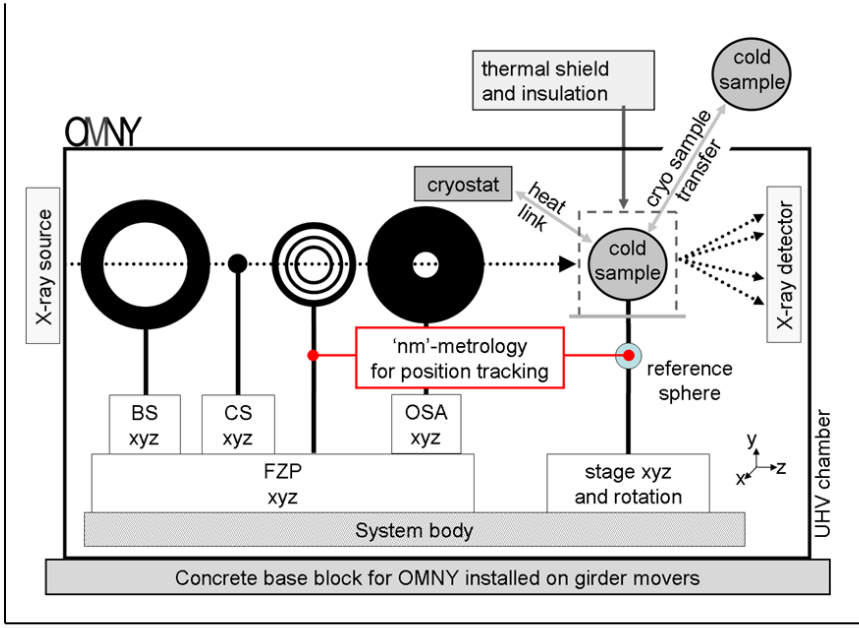
Metrology for OMNY

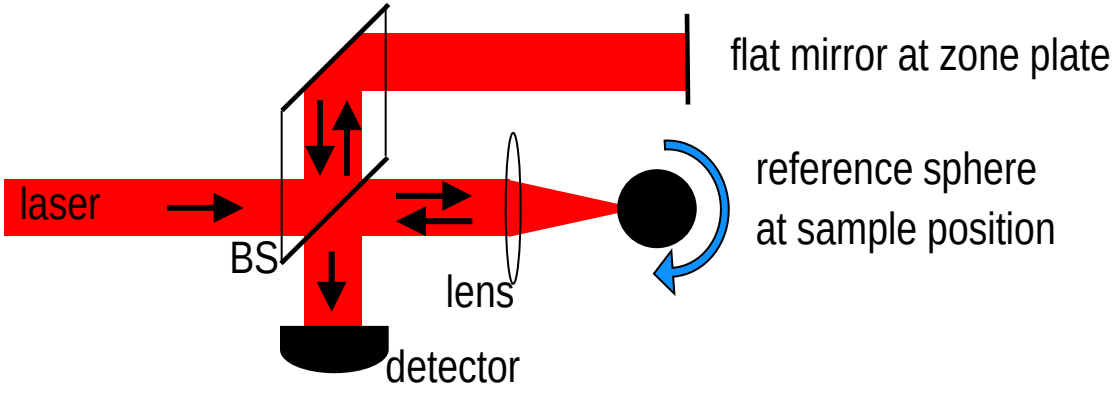
vertical rotation axis

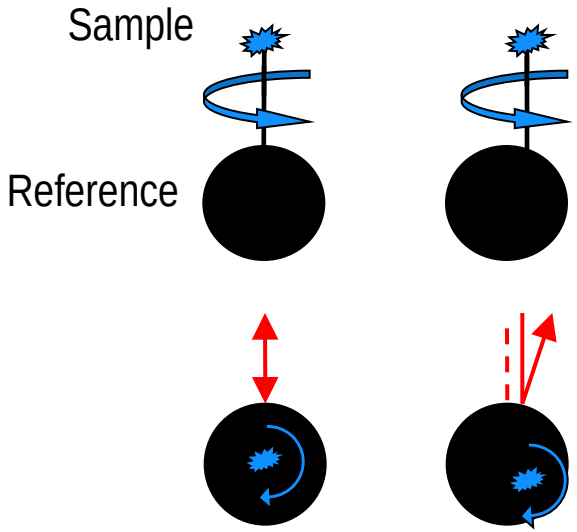
→ for horizontal measurement a spherical mirror is used (equator of a sphere)

BUT

→ no centering mechanics between mirror and sample → wobble of sphere → beam loss



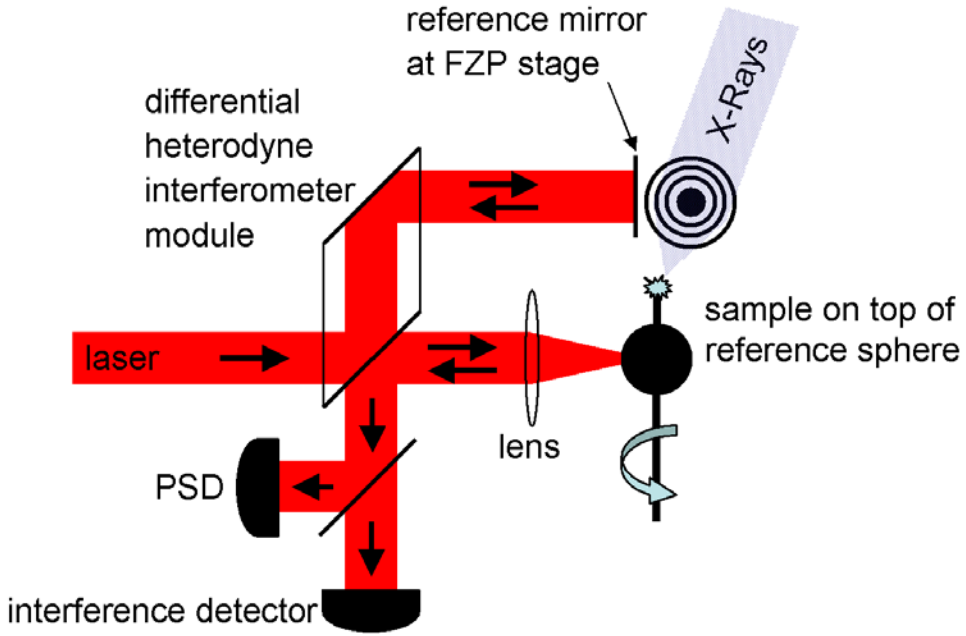


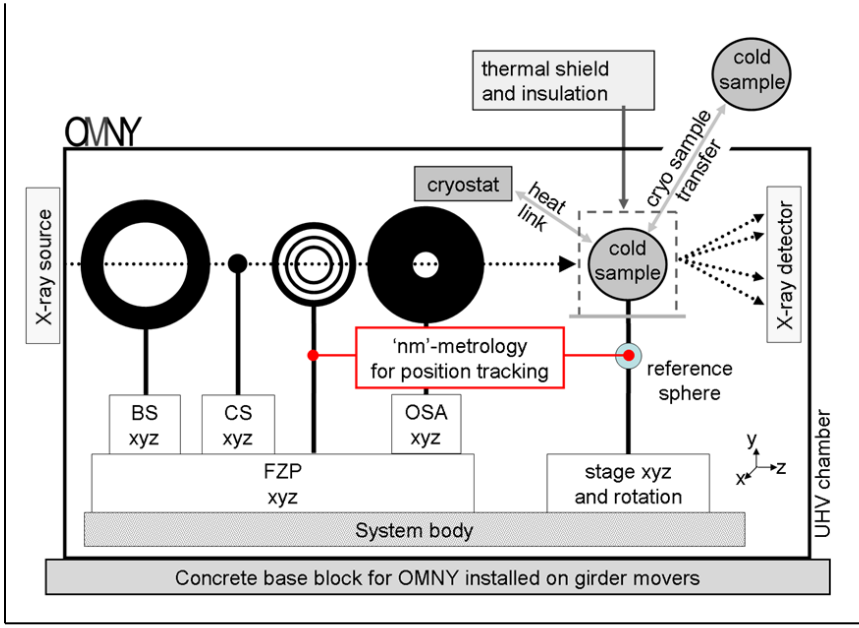


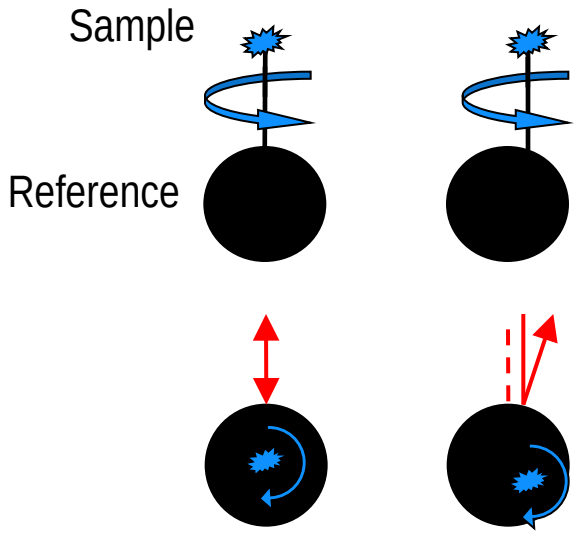
Metrology for OMNY: Tracking interferometer

Position sensitive detector measures sphere motion perpendicular to laser beam propagation.

Closed loop: interferometer tracks the reference sphere and keeps pointing a its center







Compensation of mechanical tracking error motion needed – details in M. Holler and J. Raabe, Opt. Eng. 54(5) 054101 (2015)

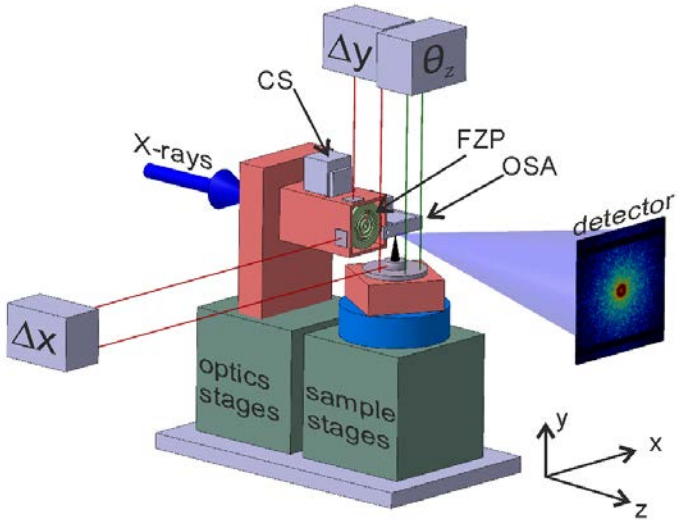
Pat. publication no. WO 2012079875 A1

Two instruments with different sample environements

metrology system ☑

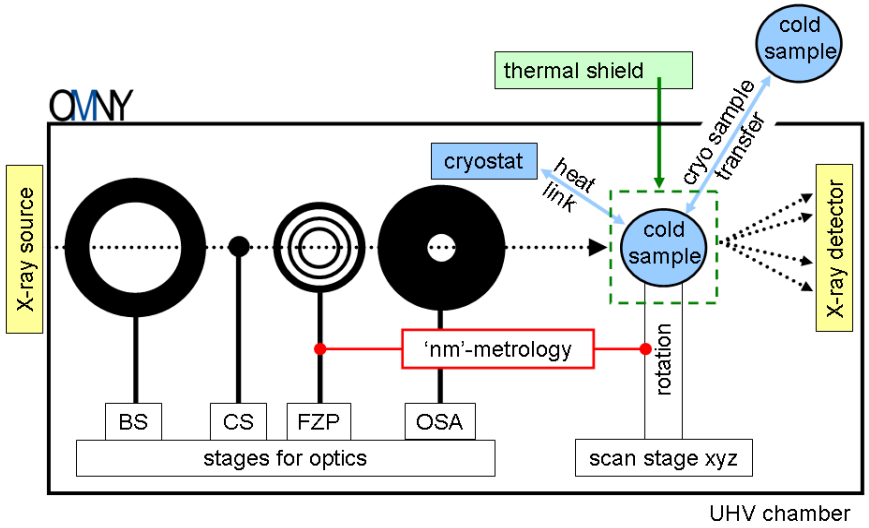
flOMNI (flexible tOMography Nano Imaging)

+ nano-positioning, tomography with interferometer
+ no cryogenic sample environment → limited to radiation hard samples
+ atmospheric pressure
+ breadboard style – flexible sample environments



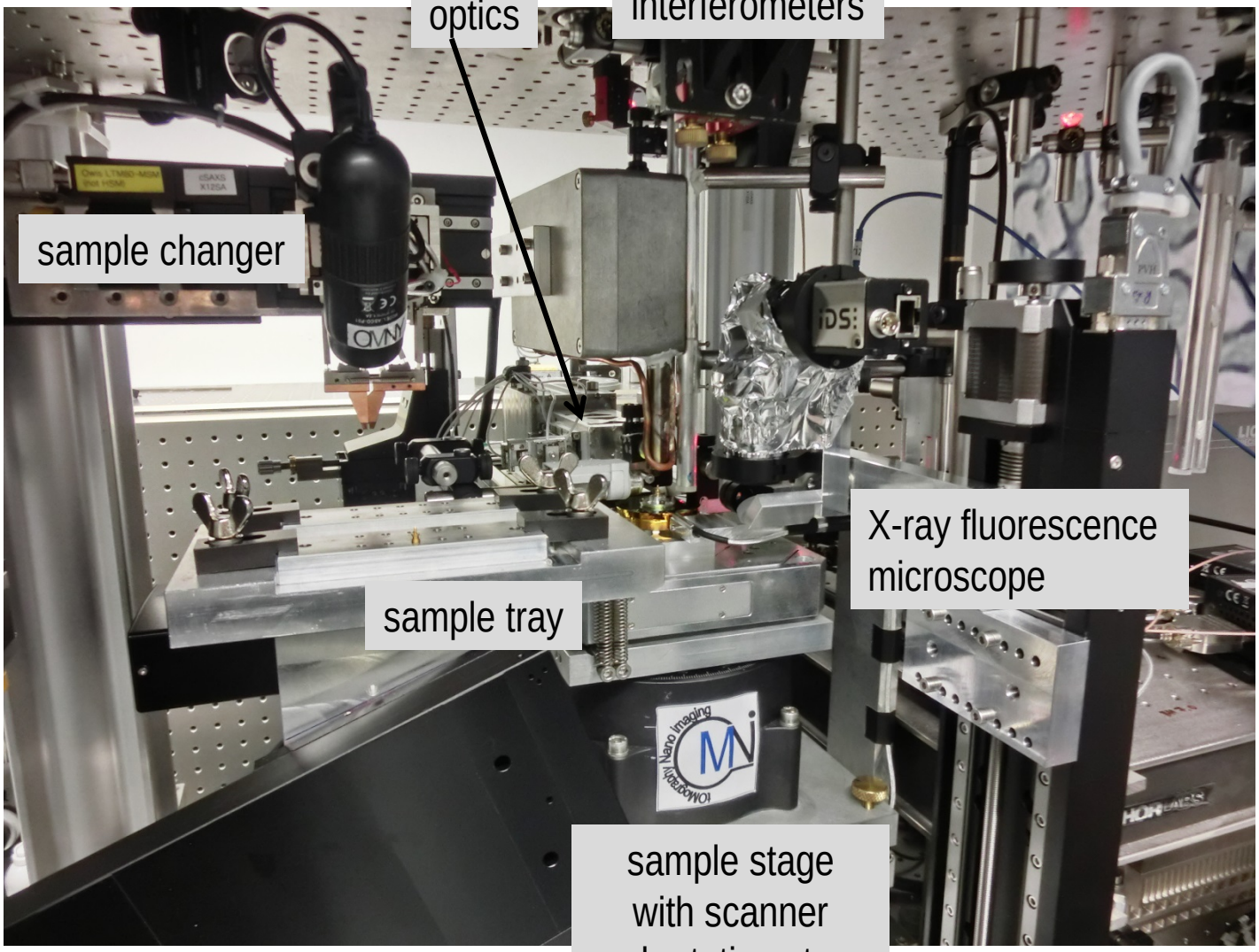
OMNY (tOMography Nano crYo)

+ optimized mechanical structures
+ cryogenic environment and UHV



M. Holler, et al., Rev. Sci. Instrum. 83, 073703 (2012)

flOMNI (flexible tOMography Nano Imaging)



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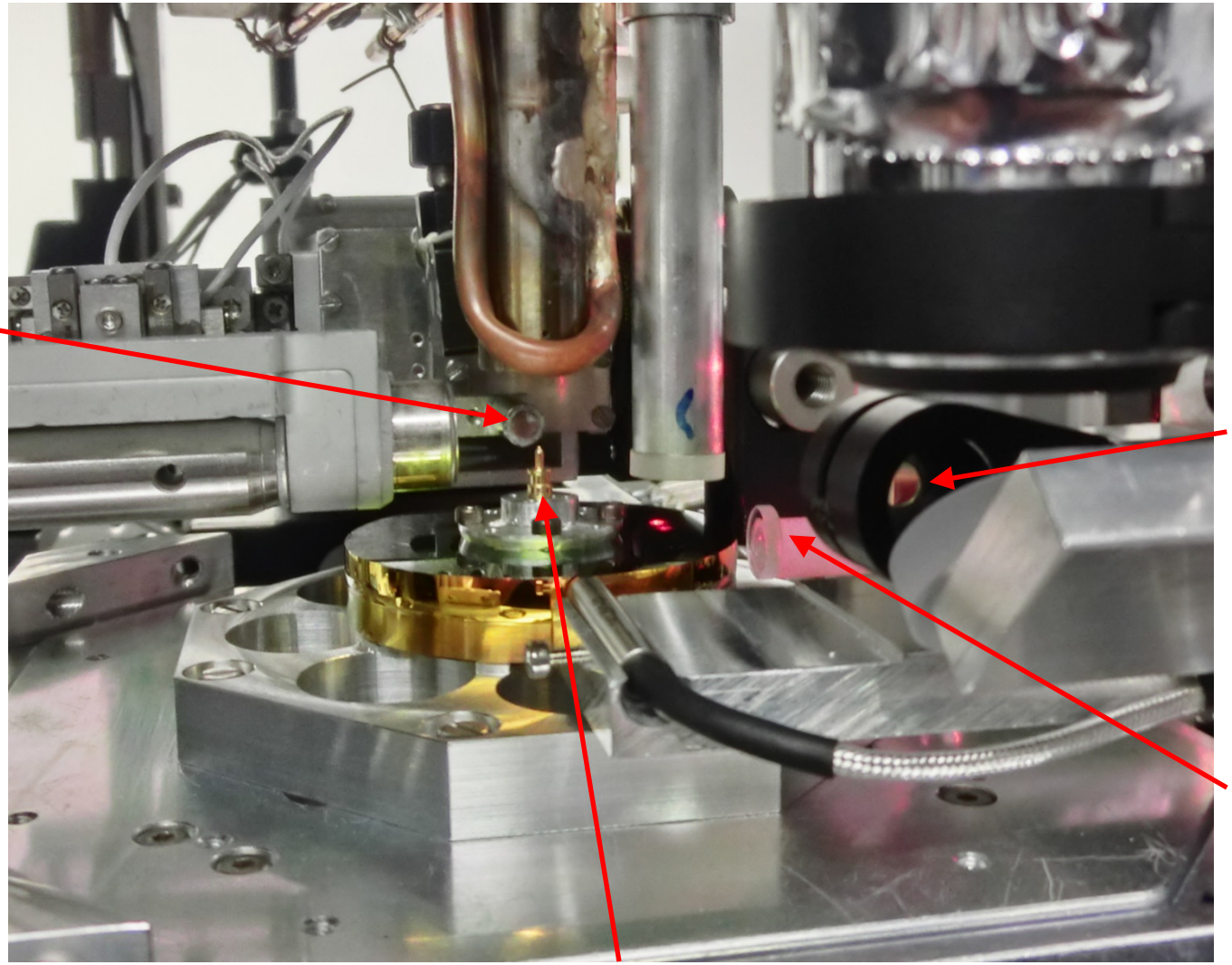

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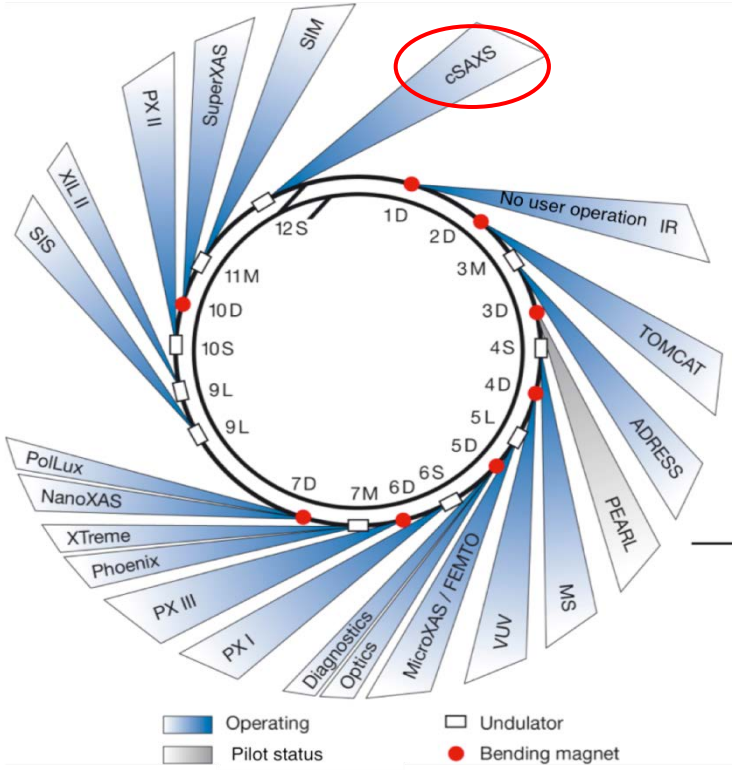
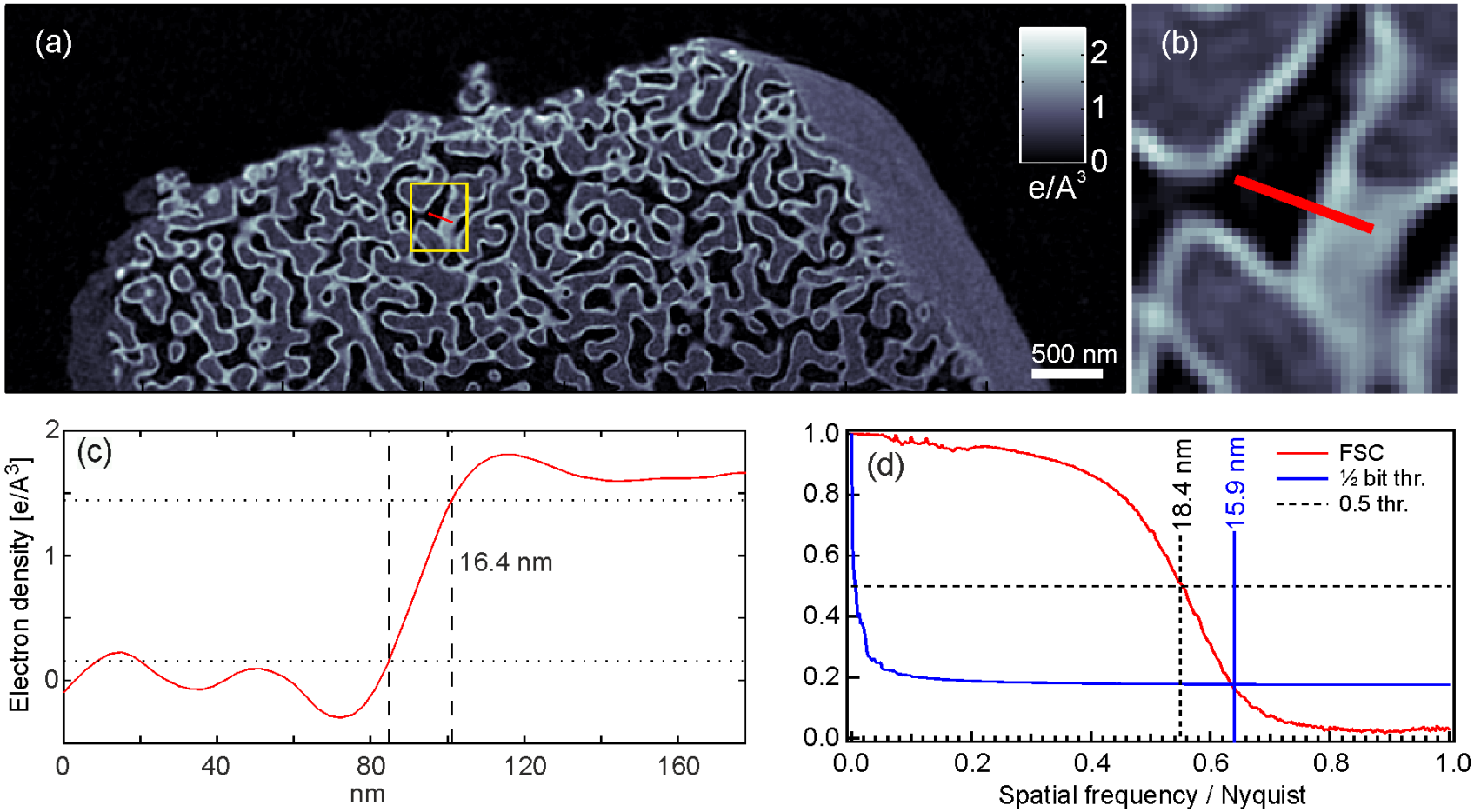
Swiss Light Source



flOMNI Resolution Test in 3D

3D Test sample: Nanoporous glass ERM®-FD122, Mean pore width 140 nm, ALD coated (Ta_2O_5) 37 nm

3D resolution: 16 nm (Fourier shell correlation), 720 projections, voxel size: 10 nm^3

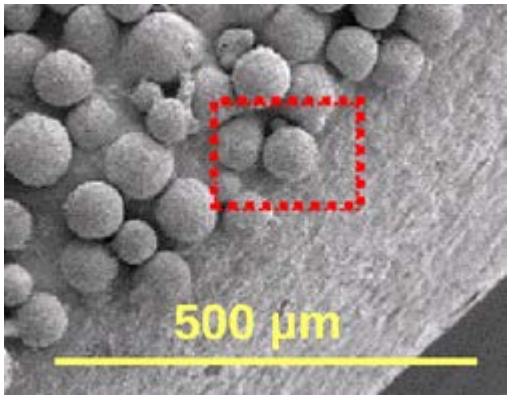


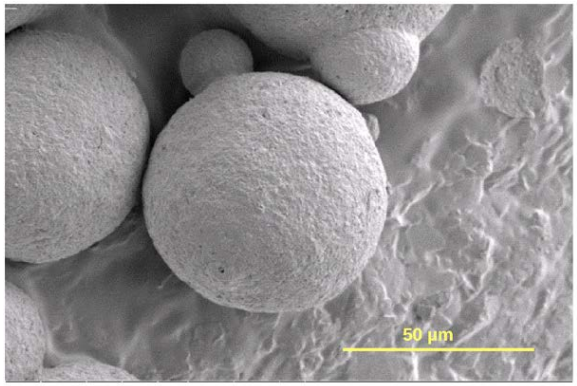
Scientific Reports 4, 3857 (2014)

Innovation Award on Synchrotron Radiation 2014
for high-resolution 3D hard X-ray microscopy

Example: Fluid Cracking Catalyst

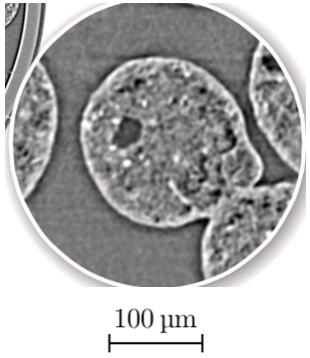
- characterization of interparticle pores at micro- and nanometer-length scales
- visualize and identify individual components in pre-shaped catalyst bodies used in fluid catalytic cracking.





SEM images

FIB sample prep

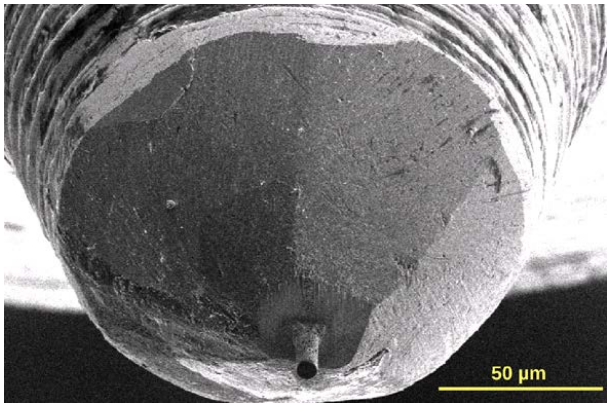


TOMCAT

Full-field, voxel size 0.3 microns

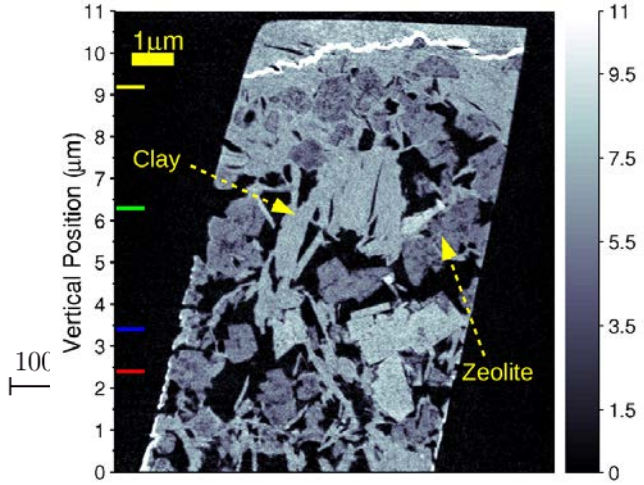
Resolution 0.7 microns

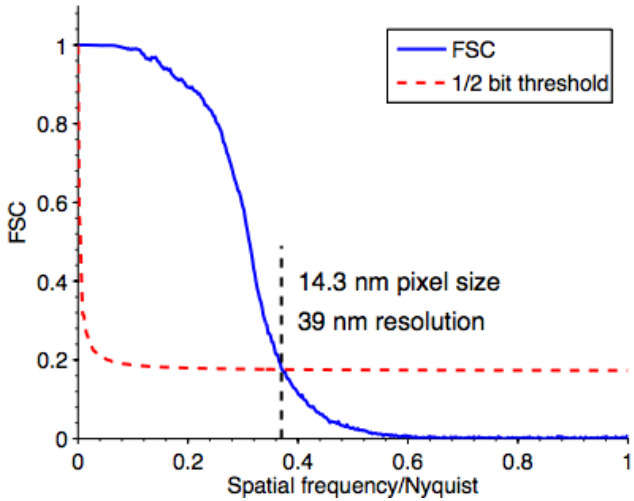
FOV 850x700 microns^2



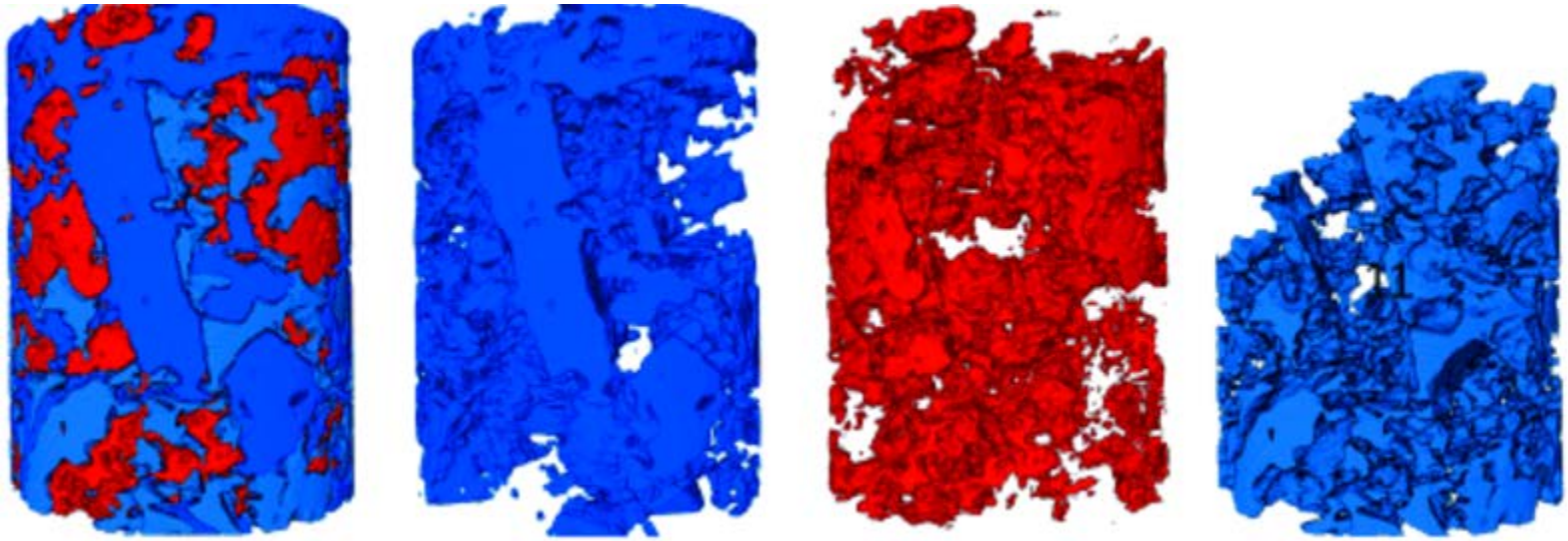
J. C. da Silva, *et al.*, ChemCatChem, 7, 413-416 (2015)

Example: Fluid Cracking Catalyst





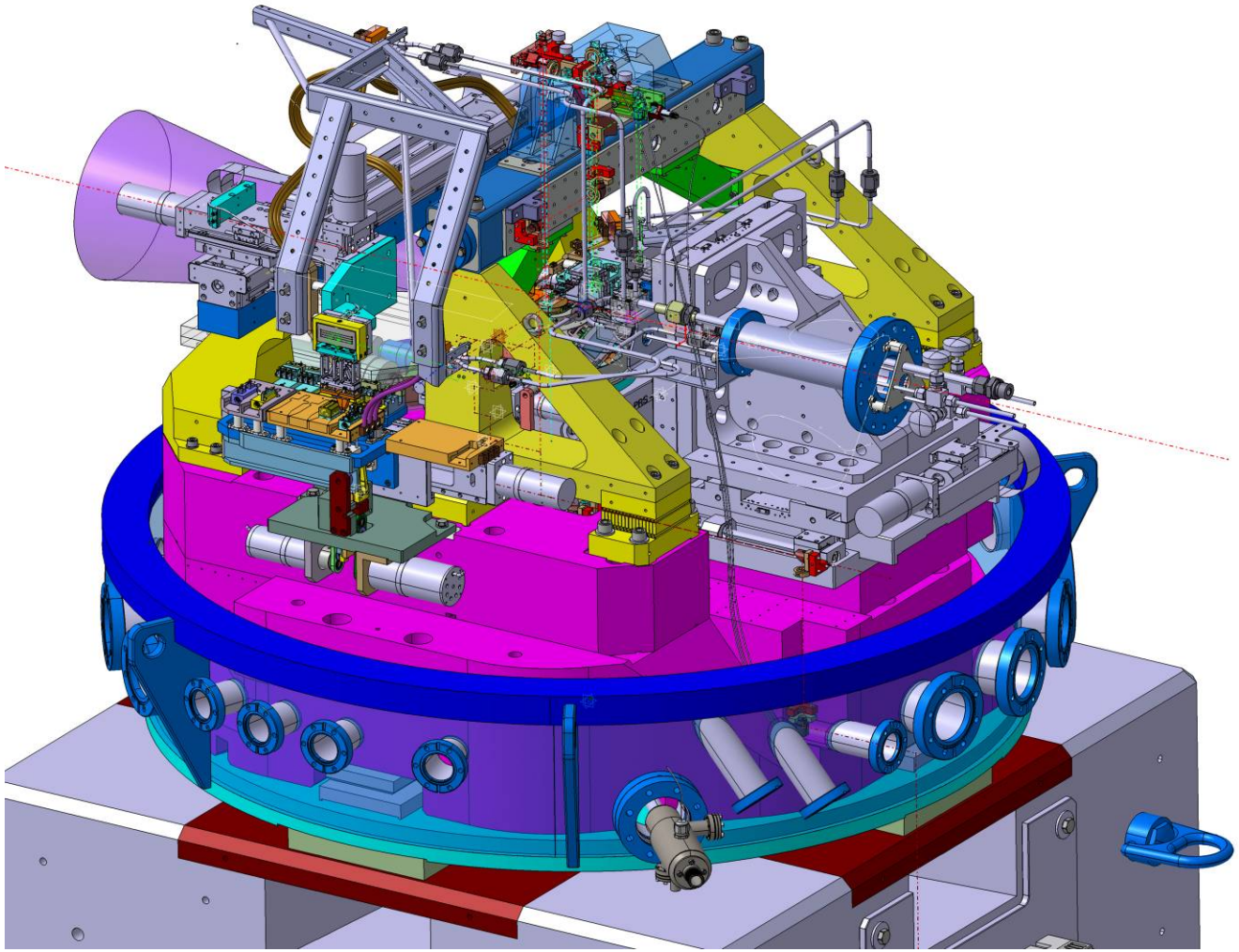
3 D rendering of the pores in light blue, the zeolite type Y in blue, and the metakaolin clay in red.



419 diffraction patterns per projection
450 projections

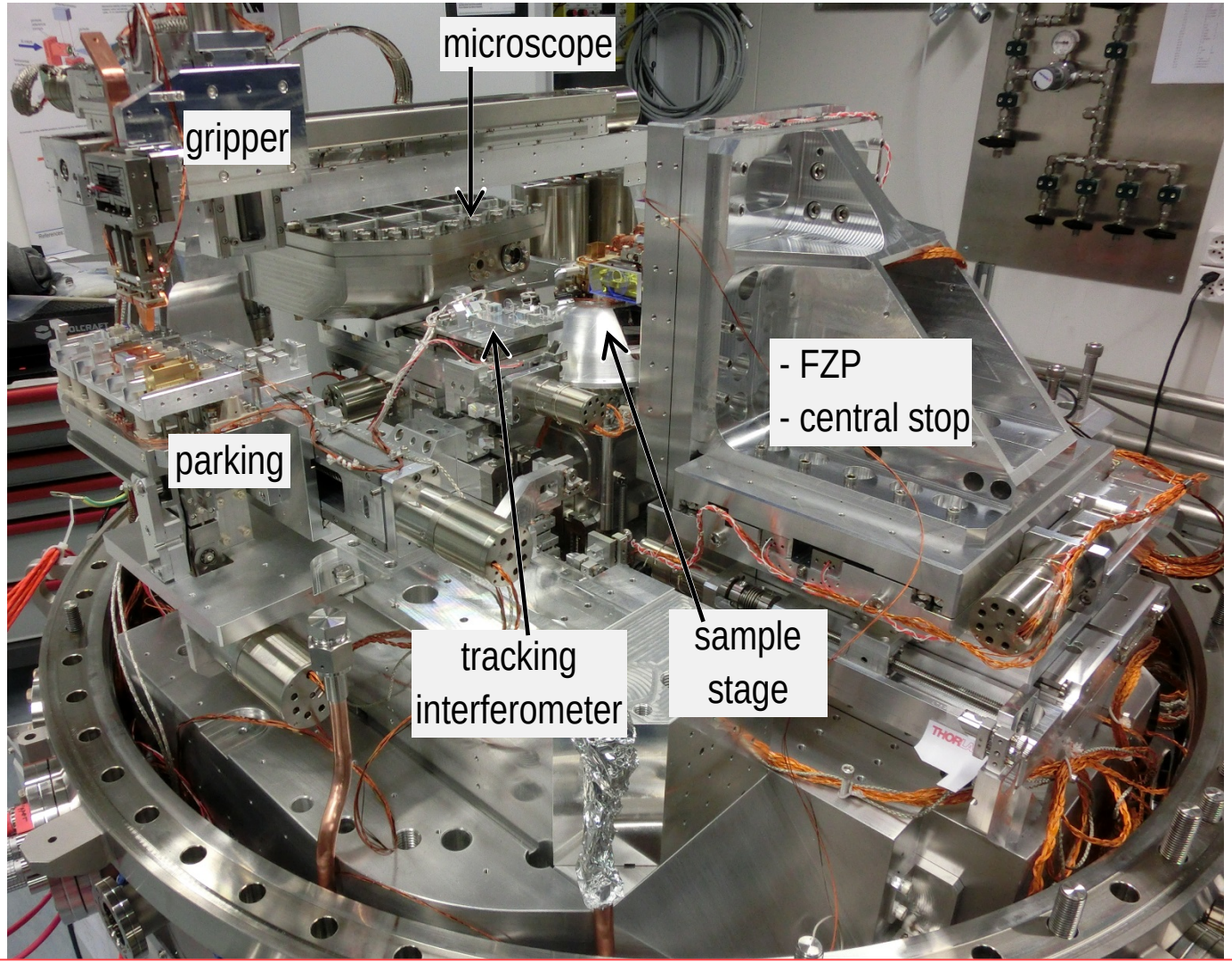
J. C. da Silva, *et al.*, ChemCatChem, 7, 413-416 (2015)

CAD rendering of cryogenic OMNY



Details of this instrument and new measurement results will be presented only and are not available in this reduced version of the talk!

View into the chamber



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