Sample Environments for X-Ray Photon Correlation Experiments (XPCS)

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DESY
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DESY

Acknowledgements

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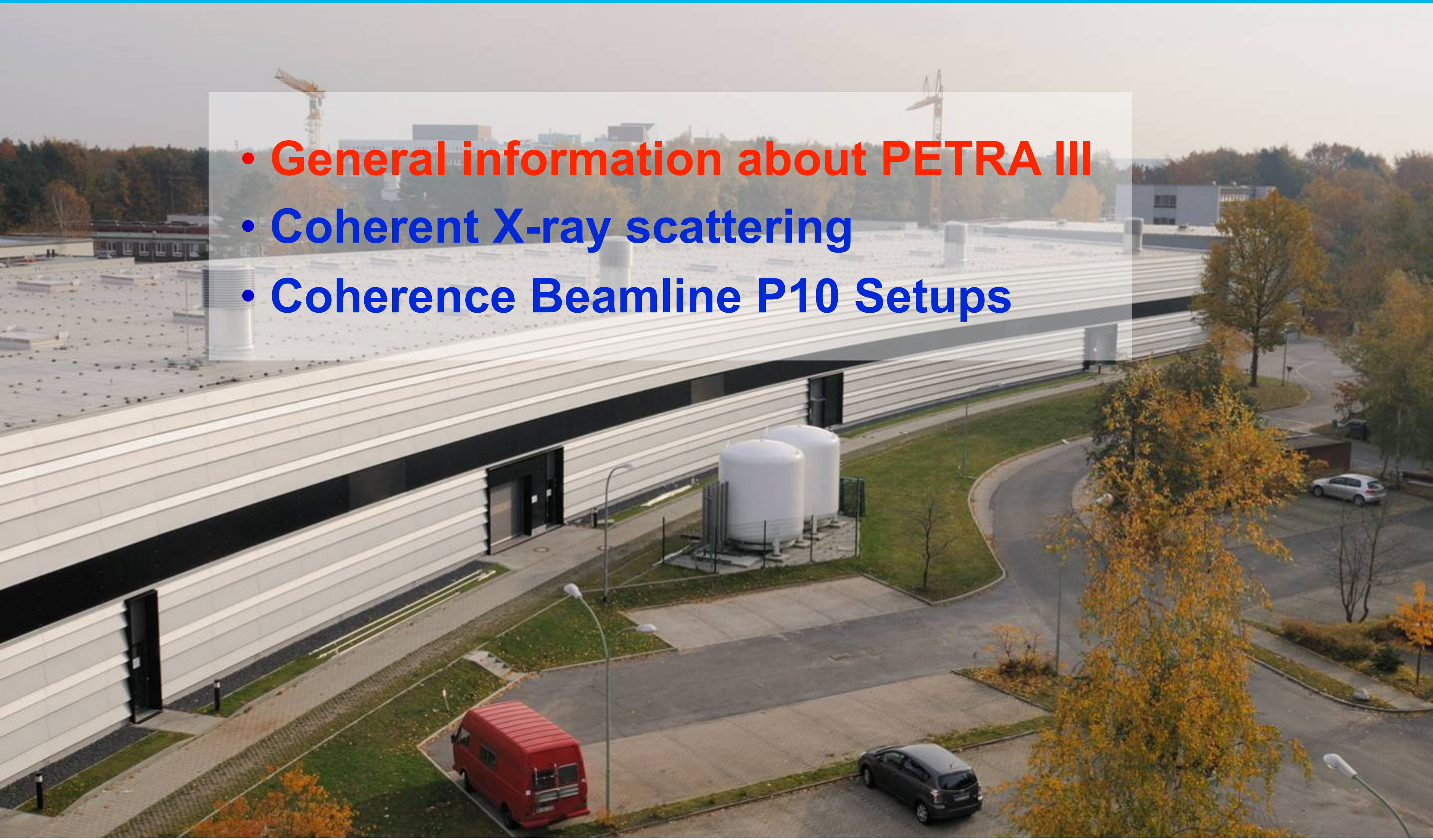
Overview

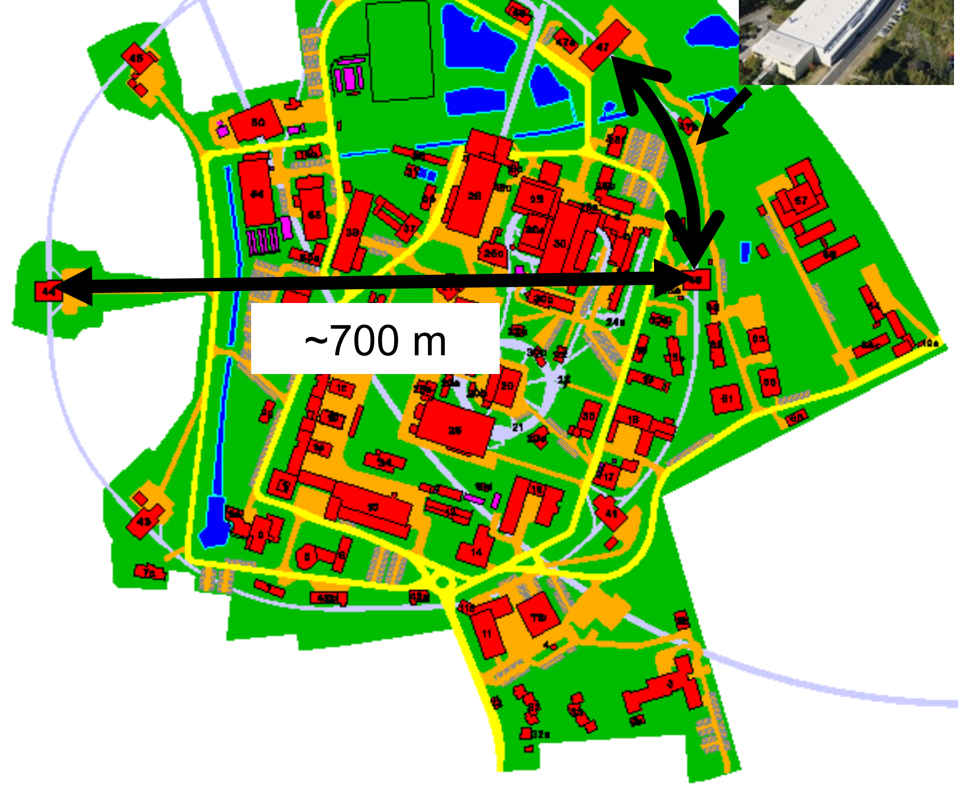


- General information about PETRA III
- Coherent X-ray scattering
- Coherence Beamline P10 Setups

Introduction to PETRA III: General Information

Original PETRA III project

- reconstruction of PETRA (2.3km long accelerator; Gluon) in 2007 to 2009
- construction of PETRA III (288 m hall)
- straight sections for 14 undulator beamlines in 1st experimental hall
- 80 m damping wiggler in the long straights
- renewal of the entire machine and injection system



- E = 6 GeV
- I = 100 mA (200 mA) – top-up
- 960 or 40 bunches
- $\varepsilon \approx 1.0$ -1.2 nm rad
- $B_u \approx 10^{21}$ 1/s mm^{2}mrad^2 0.1%BW

Introduction to PETRA III: Highlights and Current status

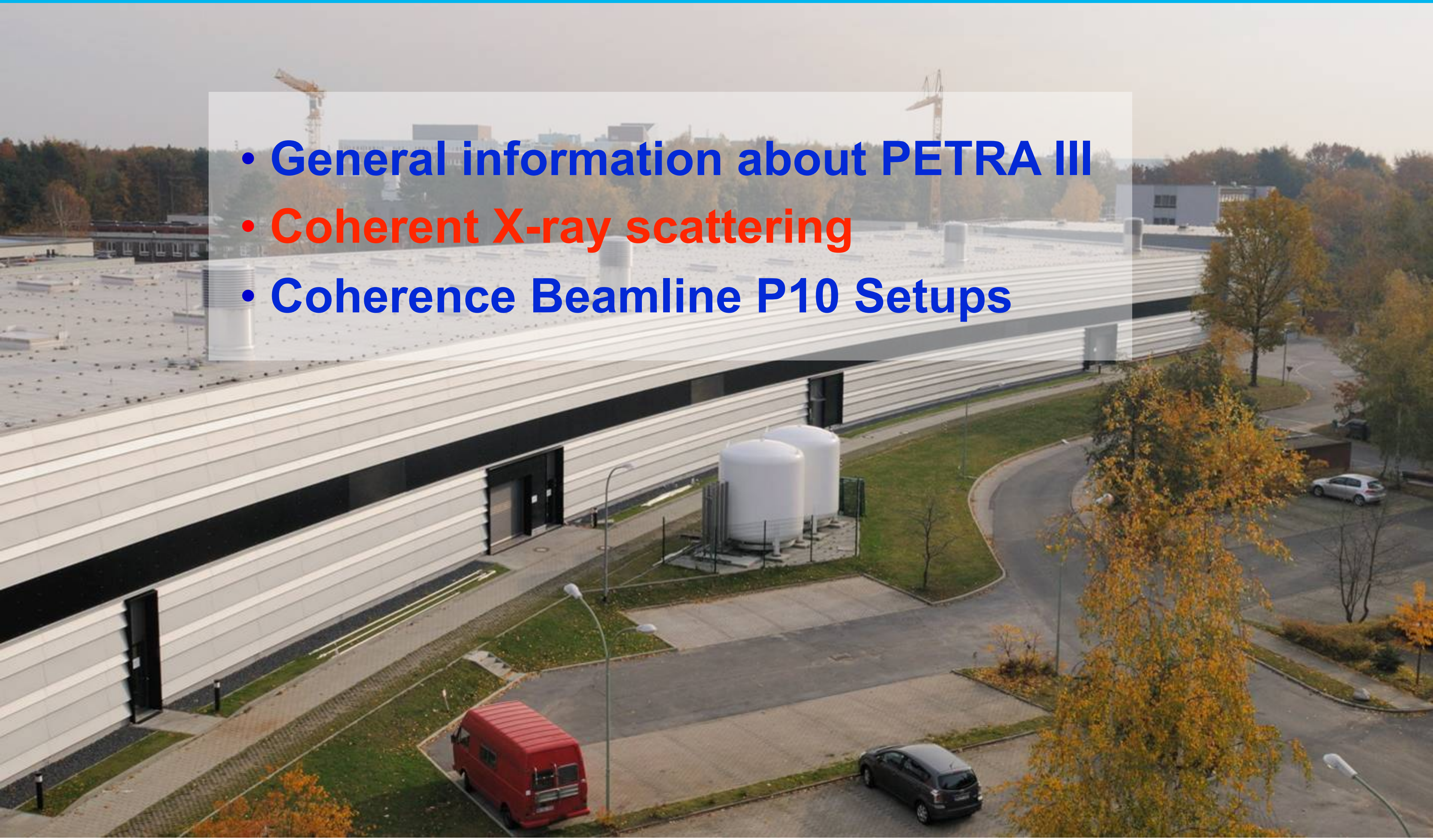
- Single 1m thick concrete slab as floor for the experimental hall
- Walls and roof are decoupled from the floor
- Beamlines are ~100m long (but very thin slices!)

The PETRA III synchrotron expands!

- 02/2014 – 04/2015 shutdown of PETRA III
  - 2 new experimental halls are constructed
    - 5 new undulator beamlines per hall
- i.e. PETRA III expands from 14 to 24 beam lines

New beamlines operated by DESY, HZG, MPI
& foreign countries (Sweden, Russia, India)

Overview



- General information about PETRA III
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Why coherence beamlines at brilliant X-ray sources?

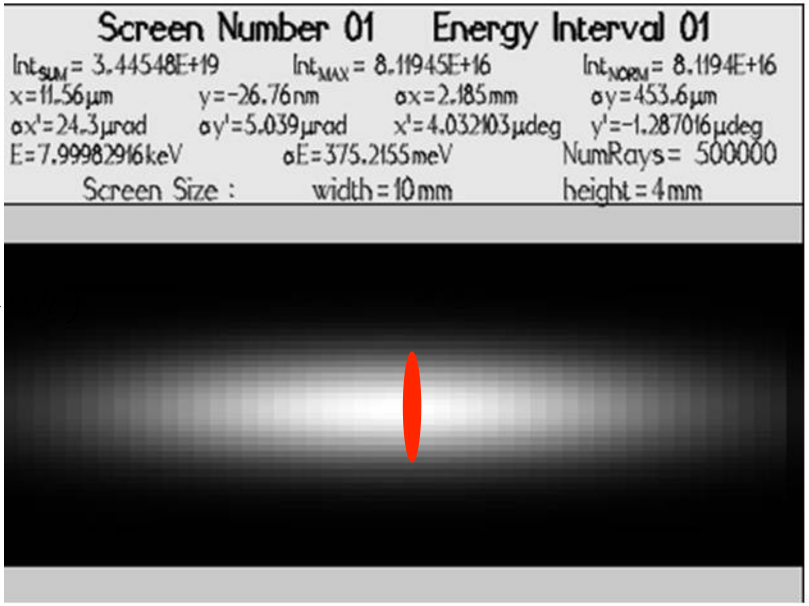
The coherent flux is proportional to the brilliance: $F_c \sim \lambda^2/4 \cdot B$

Low beta source: ~14 x 84 μm² (FWHM)

Transverse coherence length: $\xi_{v,h} = 1/2\ \lambda R/(2.35\ \sigma_{\ldots}$

$\xi_{v,h}$ ~ 480 x 80 μm² (FWHM)

(@ 90m, 8keV)



The coherent fraction @ PETRA III is ~1-2% in the medium hard x-ray range!

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Requirements for coherent scattering experiments

Plenty of coherent flux ($F_{coh} \sim \lambda^2/4\ B$)

Coherent illumination of the sample:

→ Illuminating beam size must be smaller than the transverse coherence length $\xi_t = 1/2\sqrt{\pi}\ \lambda R/2.35\sigma$

→ Path length difference within sample must be smaller than the longitudinal coherence length $\xi_l = \lambda(\lambda/\Delta\lambda)$

Transmission: $\xi_l > 2W \sin^2\theta + d_{slit} \sin 2\theta$ Reflection: $\xi_l > 2/\mu \sin^2\theta$

Speckle resolving detectors:

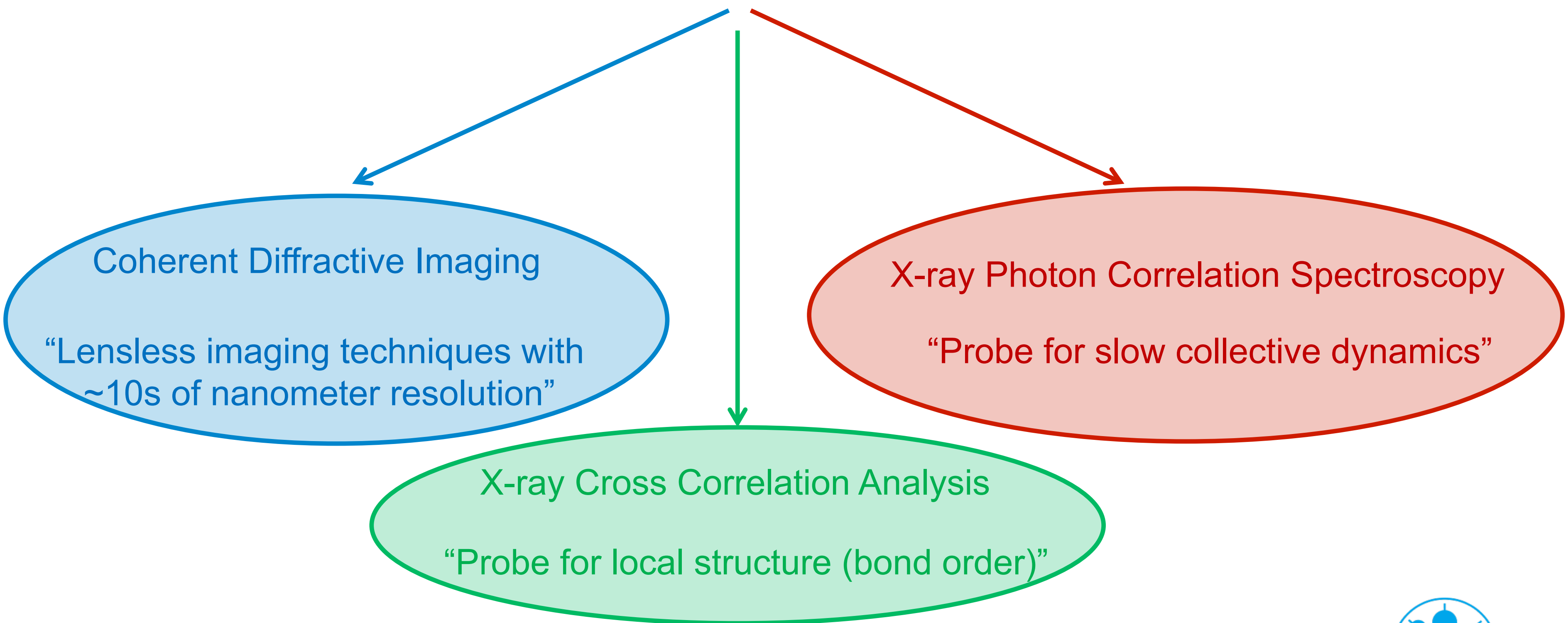
→ Spatial resolution: the detector pixel *p* size must be smaller than the 'speckle' size $p < s = \lambda D/d_{slit}$

!!!Focusing!!!

→ Time resolution: The frame rate of the detector should be ~10x faster than the dynamic process under investigation (XPCS)

Coherent scattering

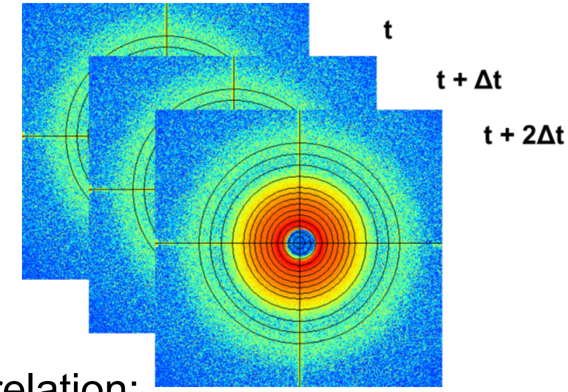
If coherent light is scattered from a disordered system it gives rise to a grainy (“random”) diffraction pattern, known as ‚***speckle***‘*. Such a speckle pattern is an interference pattern and related to the exact spatial arrangement of scattering objects in the disordered system.*



X-Ray Photon Correlation Spectroscopy (XPCS)

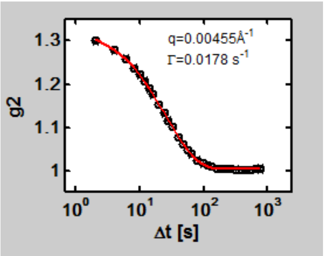
Probe to study slow (collective) dynamics at small length scales

- Disorder yields a speckle pattern … Time evolution of disorder yields a time-varying speckle pattern
- Time autocorrelation of the fluctuating intensity at a particular wave-vector transfer yields characteristic sample fluctuation time (τ) at a particular length scale



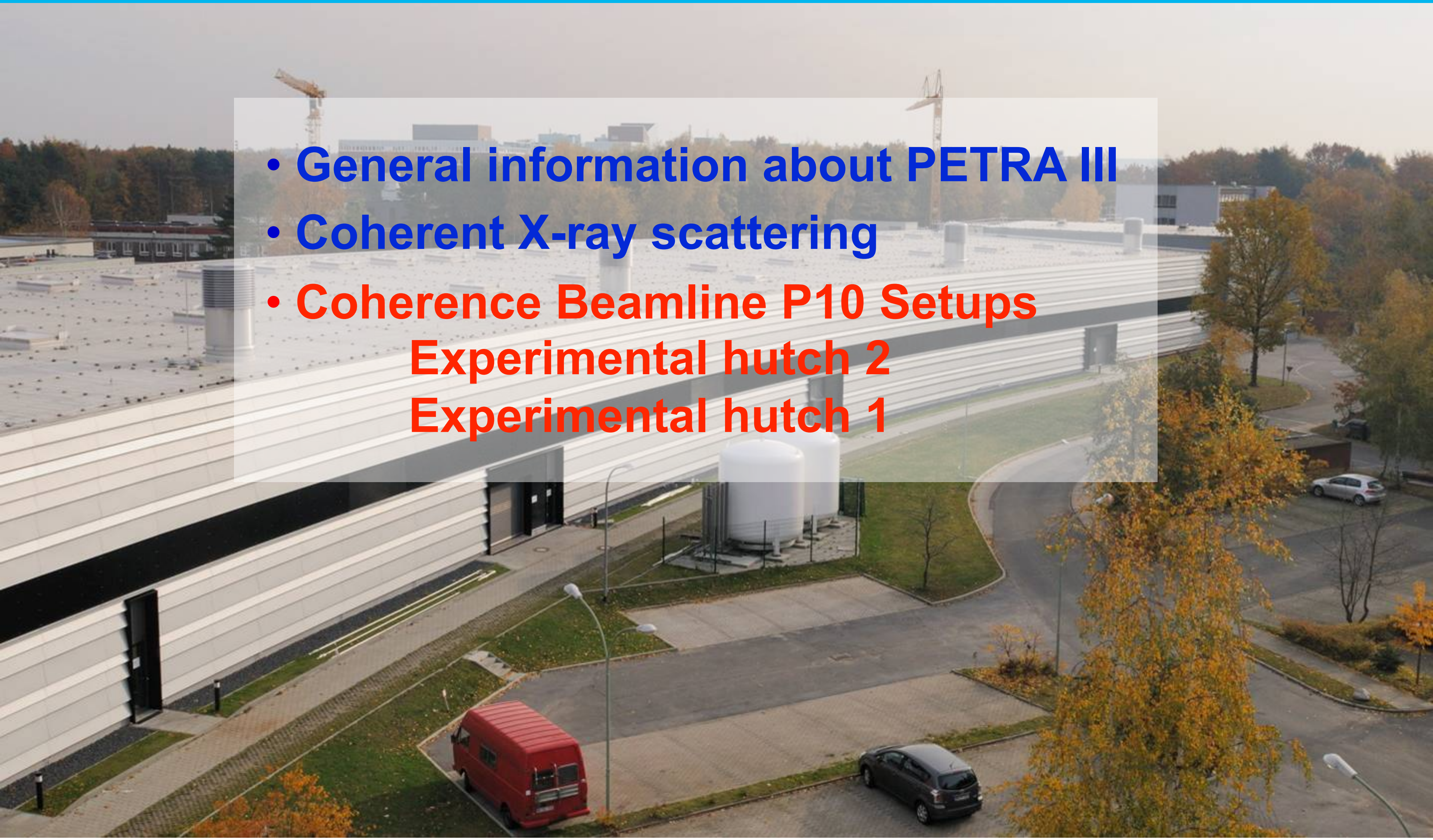
Measured Quantity: $$g_2(\vec{Q},t) = \frac{\left\langle I(\vec{Q},t) I(\vec{Q},t+\tau) \right\rangle_\tau}{\left\langle I(\vec{Q},\tau) \right\rangle_\tau^2}$$

Access to intermediate scattering function *f*(Q,t) via Siegert relation:



$$g_2(Q,t) = 1 + \beta |f(Q,t)|^2$$

Overview



- General information about PETRA III
- Coherent X-ray scattering
- Coherence Beamline P10 Setups
  - Experimental hutch 2
  - Experimental hutch 1

Coherence Beamline P10: Mission & Flux estimates

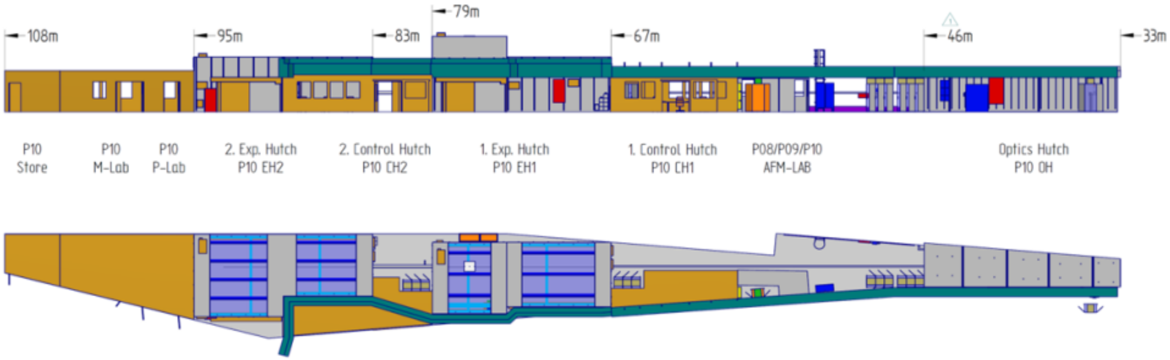
The Coherence Beamline P10 specializes in facilitating coherent x-ray scattering techniques in the medium-hard x-ray range (5—20keV). Scientifically the aim is to investigate structures and dynamics on nanometer length scales. Experimental techniques are X-ray Photon Correlation Spectroscopy (XPCS), X-ray Cross Correlation Analysis (XCCA) and Coherent Diffraction Imaging (CDI).
Additionally, Rheo-SAXS is available for time-resolved measurements.

Theoretical longitudinal coherence length and coherent flux @ P10

Δλ/λ	ξ_l	**Flux$_{coh}$**	**Energy**
$6 \cdot 10^{-3}$ (pink beam, 1st harmonic)	0.025μm	$1.4 \cdot 10^{13}$	8keV
$1 \cdot 10^{-4}$ (Si(111))	1.5μm	$2.3 \cdot 10^{11}$	8keV
$2 \cdot 10^{-3}$ (pink beam, 3rd harmonic)	0.054μm	$1.4 \cdot 10^{12}$	12keV

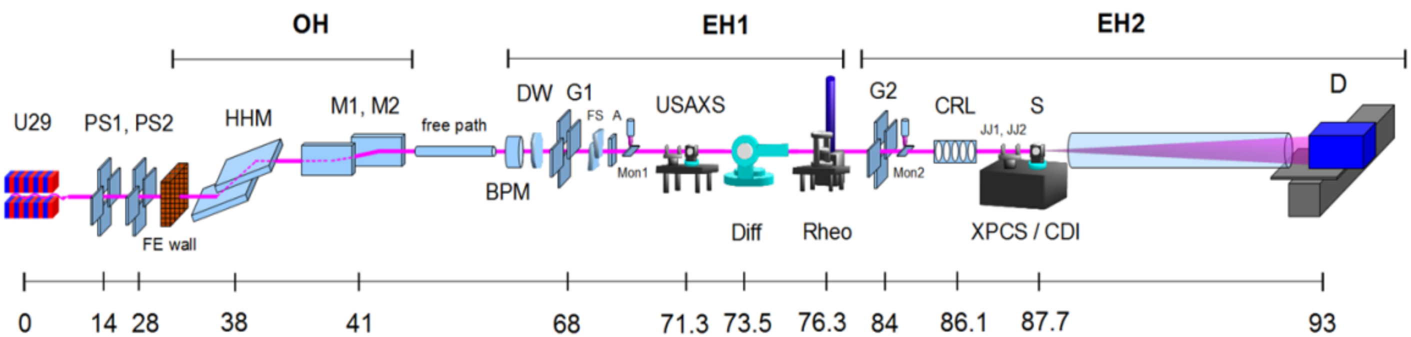
!!!Without focusing only a tiny fraction (~1/1000) is usable!!!

Coherence Beamline P10: The layout & optical scheme



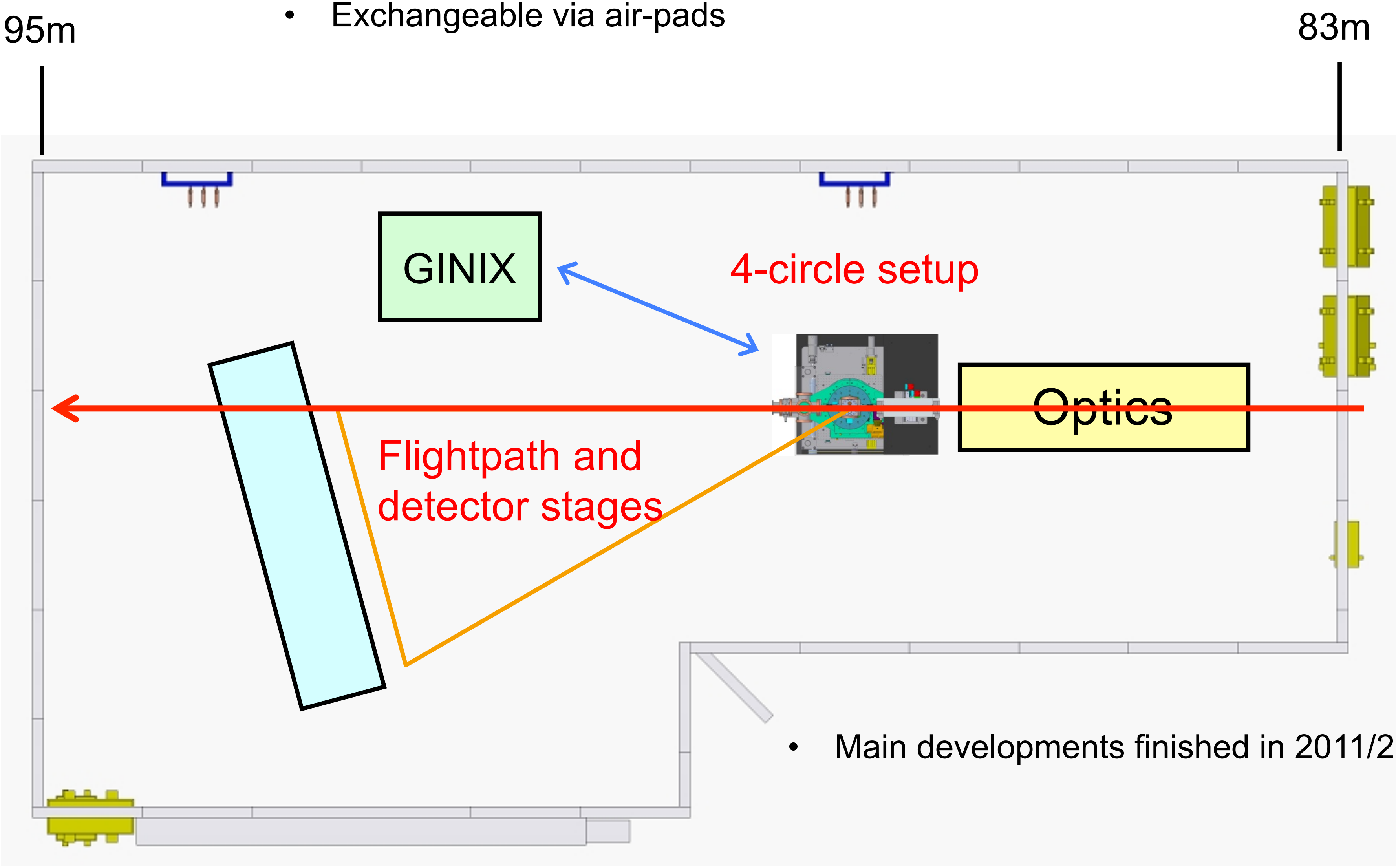
- 1 x Optics hutch
- 2 x Experimental stations
- 2 x Control hutches
- 1 x Sample preparation room
- 1 x Mechanical lab
- 1 x Electronic lab
- 1 x AFM lab

5 independent experimental setups



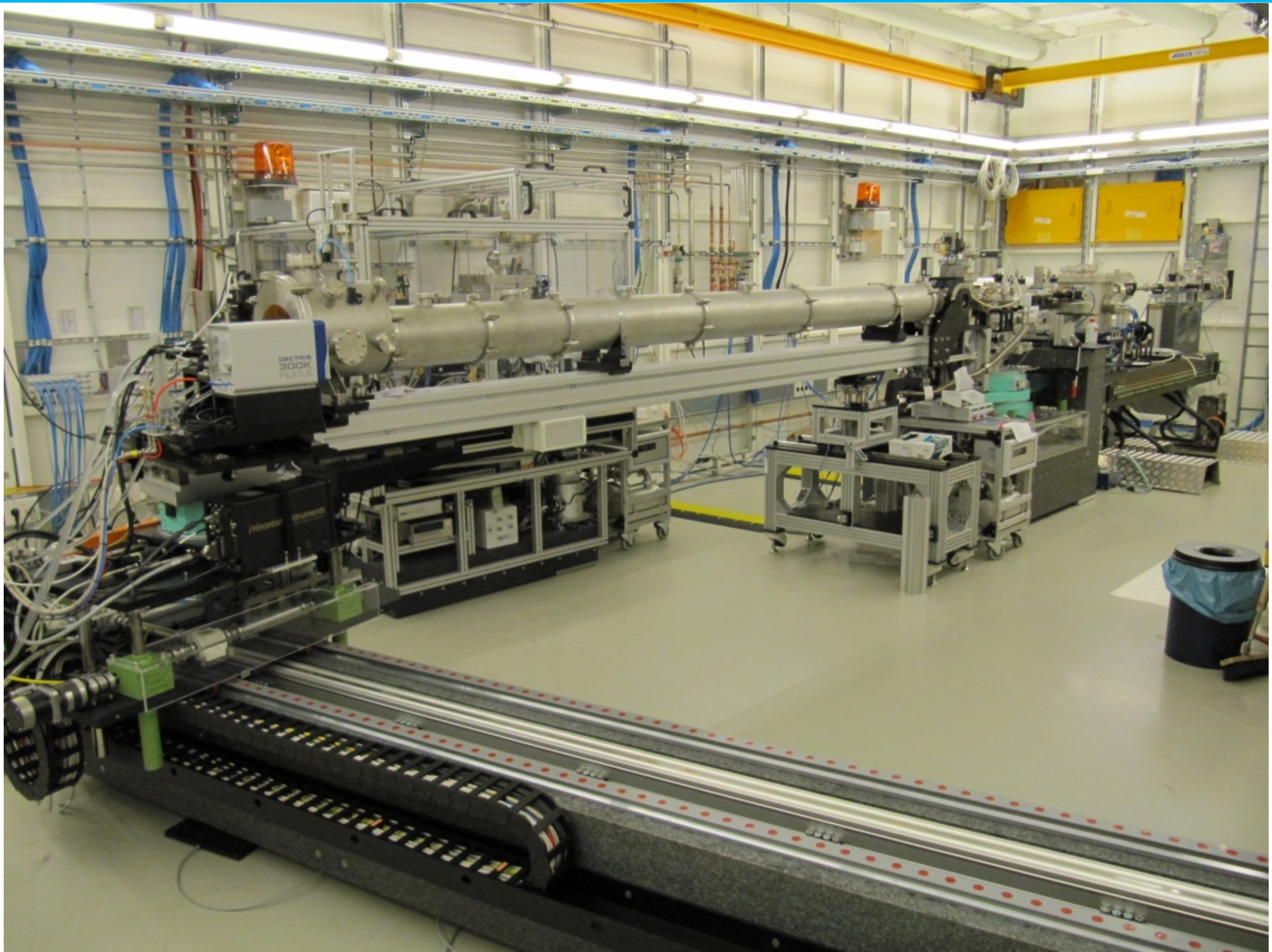
2nd experimental hutch EH2: Schematic overview

- 2 experimental setups using a "common" sample position:
  a) micro-focusing 4-circle setup
  b) nano-focusing GINIX setup (University of Göttingen)
- Exchangeable via air-pads



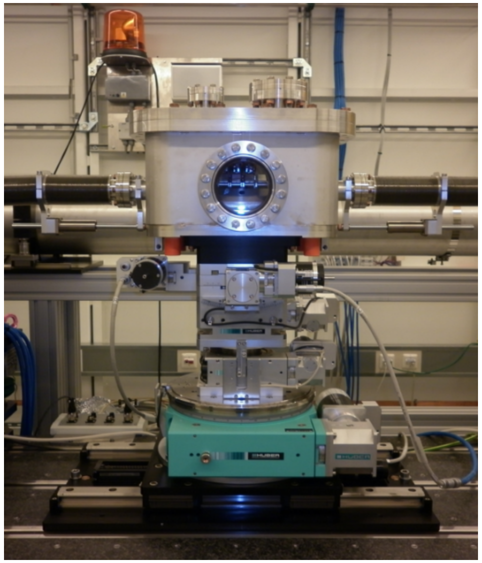
- Main developments finished in 2011/2

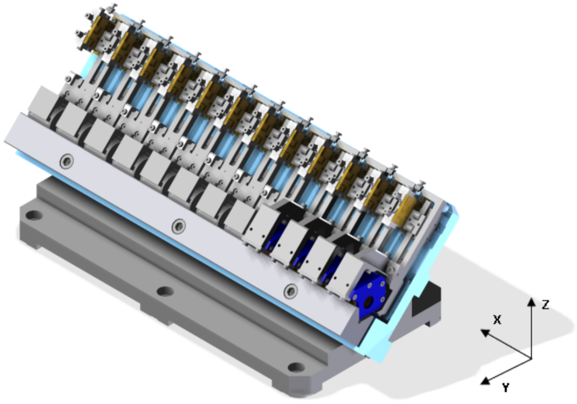
2nd experimental hutch EH2: Actual overview

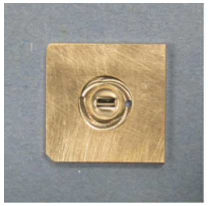


4-circle setup: Micro-focusing optics

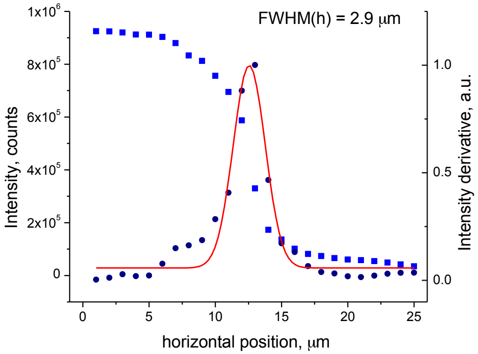
Transfocator for 1D & 2D lenses

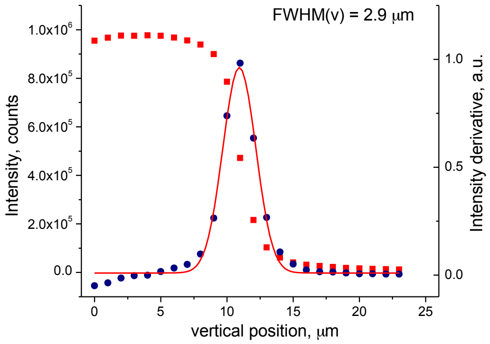


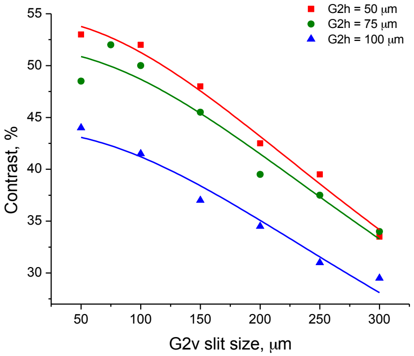




Focal length: ~1.57m
Focal size: ~1-5µm
Energy range: 5-18keV



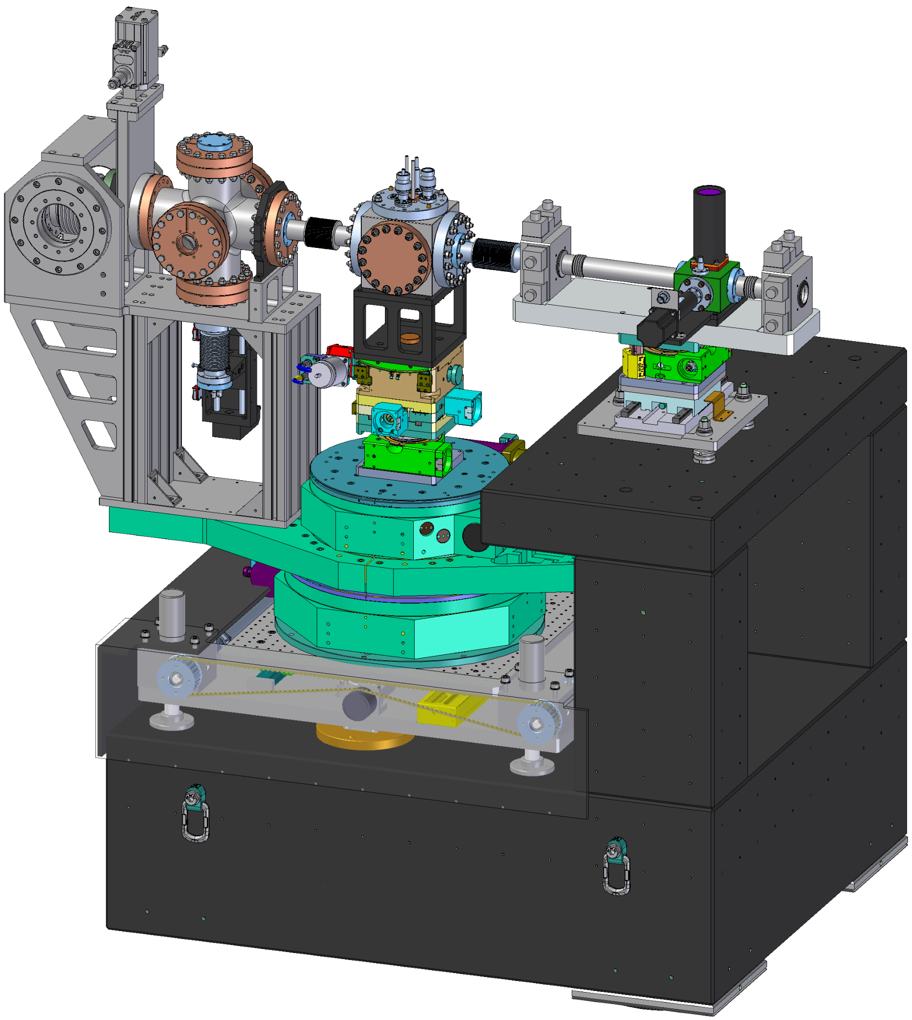




Usable coherent flux: ~1 x 10^{11}cps (G2 @ 150x75µm^2, 80% Transmission)

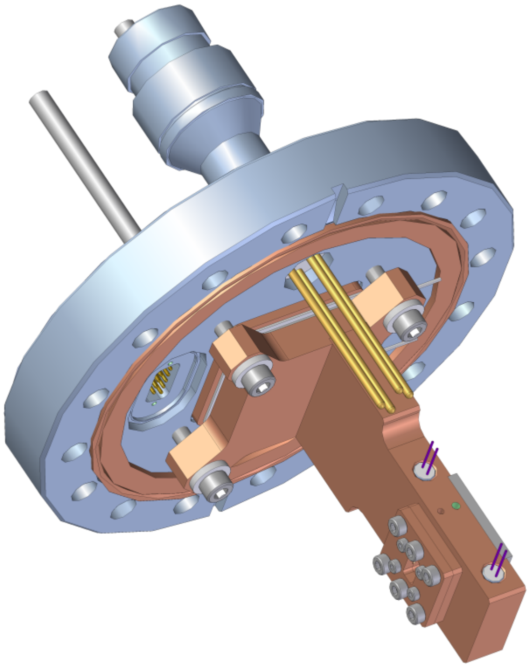
A. Zozulya et al., Optics Express 20, 18967 (2012)

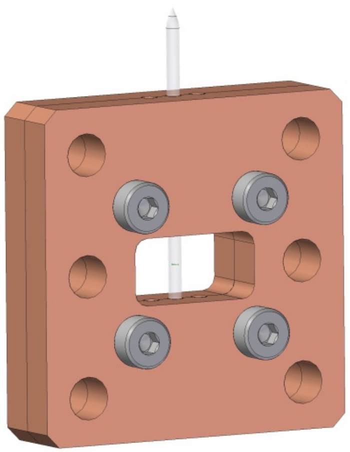
4-circle setup: Sample region / environment



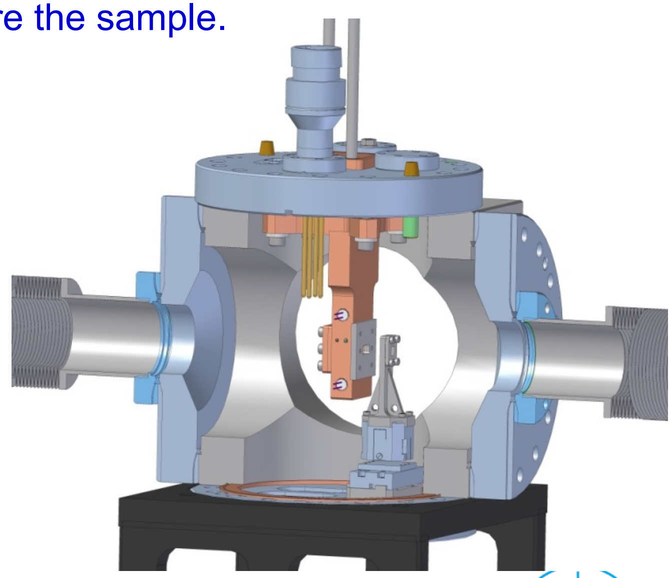
- This setup consists of a Huber 4-circle diffractometer sitting on a heavy granite based table. It is possible to scatter horizontally to 30.0°.
- The samples are placed into a DN100 cube. Different experiments are easily integrated by designing independent inserts for this cube.
- It is possible to operate this setup fully vacuum integrated. The vacuum environment can be replaced by a large variety of other setups.
- The standard setup exhibits a sample to detector distance of ~5.0m. Flight path as well as the multi-purpose detector holder sit on 3m long translation stages.

4-circle setup: Experimental inserts





- SAXS and Reflectivity inserts with a heating and cooling option based on Peltier elements and resistive heaters covering the temperature range from -30°C — 200°C.
- SAXS and Reflectivity inserts with a combination of cryogenic cooling and resistive heaters covering the temperature range from -150°C — 50°C.
- SAXS and Reflectivity inserts with a heating option based on resistive heaters covering the temperature range from 100°C — 500°C.
- CDI setup based on Attocubes (XYZ and Rot Z)
- An independent guard slit insert based on an Attocube YZ translation stage directly before the sample.
- other possibilities:
  - stress-strain insert
  - flow insert
  - ptychography insert
  - …



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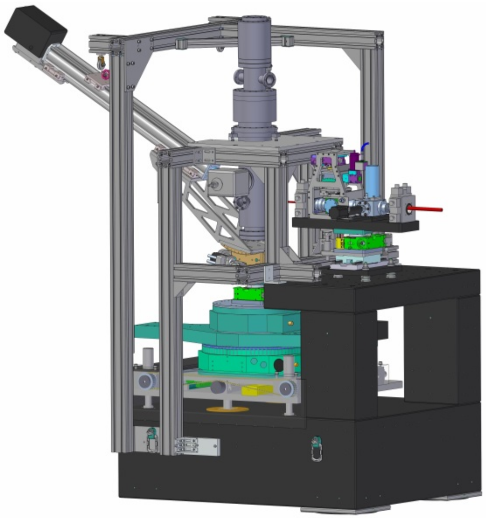
4-circle setup: Flexibility

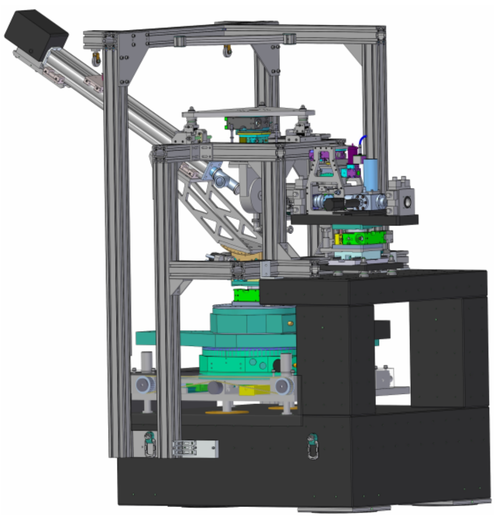
- Variable beam conditions:
  Energy range: 5-18keV
  Beamsizes: 2-3µm^2 (focused; ~80% transmission)
  15x15µm^2 - 50x50µm^2 (unfocused)
  Flux density: 1x10^7 photon/s/µm^2 (unfocused)
  Coherent flux: up to 1-2 x 10^{11} photons/s (8keV)

- Variable setup:
  SAXS / WAXS (<30°) with 5m long flight path
  Multi-detector mount
  Short detector distance options
- Fully vacuum integrated
- Variable sample conditions

Recent Additions:

- Specialized CDI PIEZO scanner (SmarAct based)
- Short focus option (~1x1µm^2)

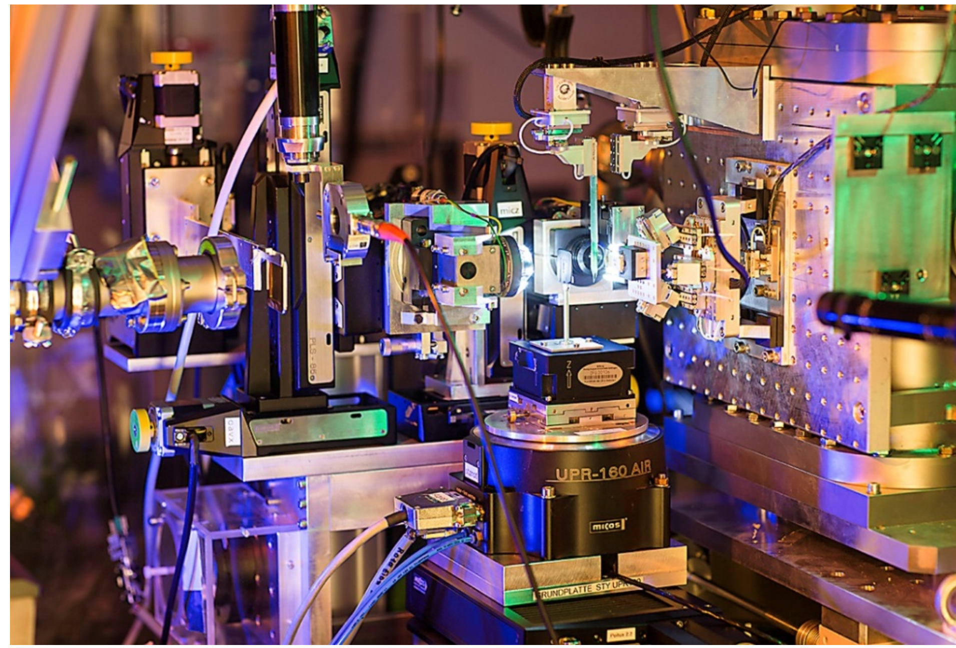


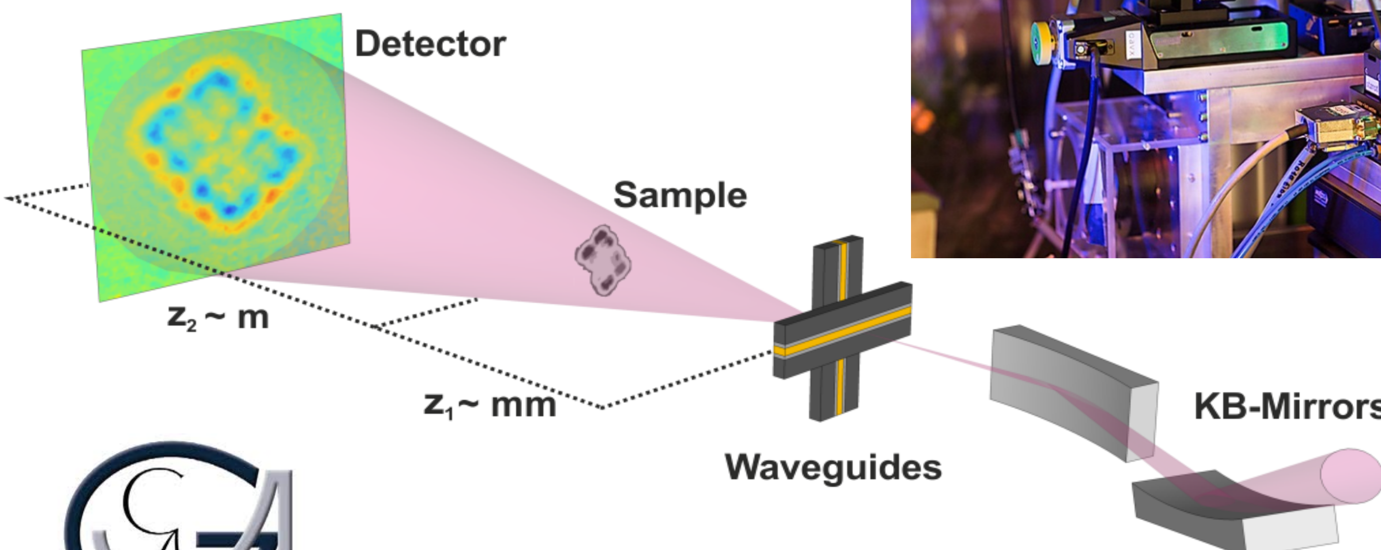


Nanofocus / waveguide setup (GINIX): Purpose

Enable (coherent) SAXS / WAXS studies using nano-focus beams
Enable waveguide propagation imaging techniques
Enable KB focus imaging techniques

Group of Prof. T. Salditt,
University of Göttingen





Kalbfleisch et al., AIP conf. Proc. 2011

Nanofocus / waveguide setup (GINIX): parameters

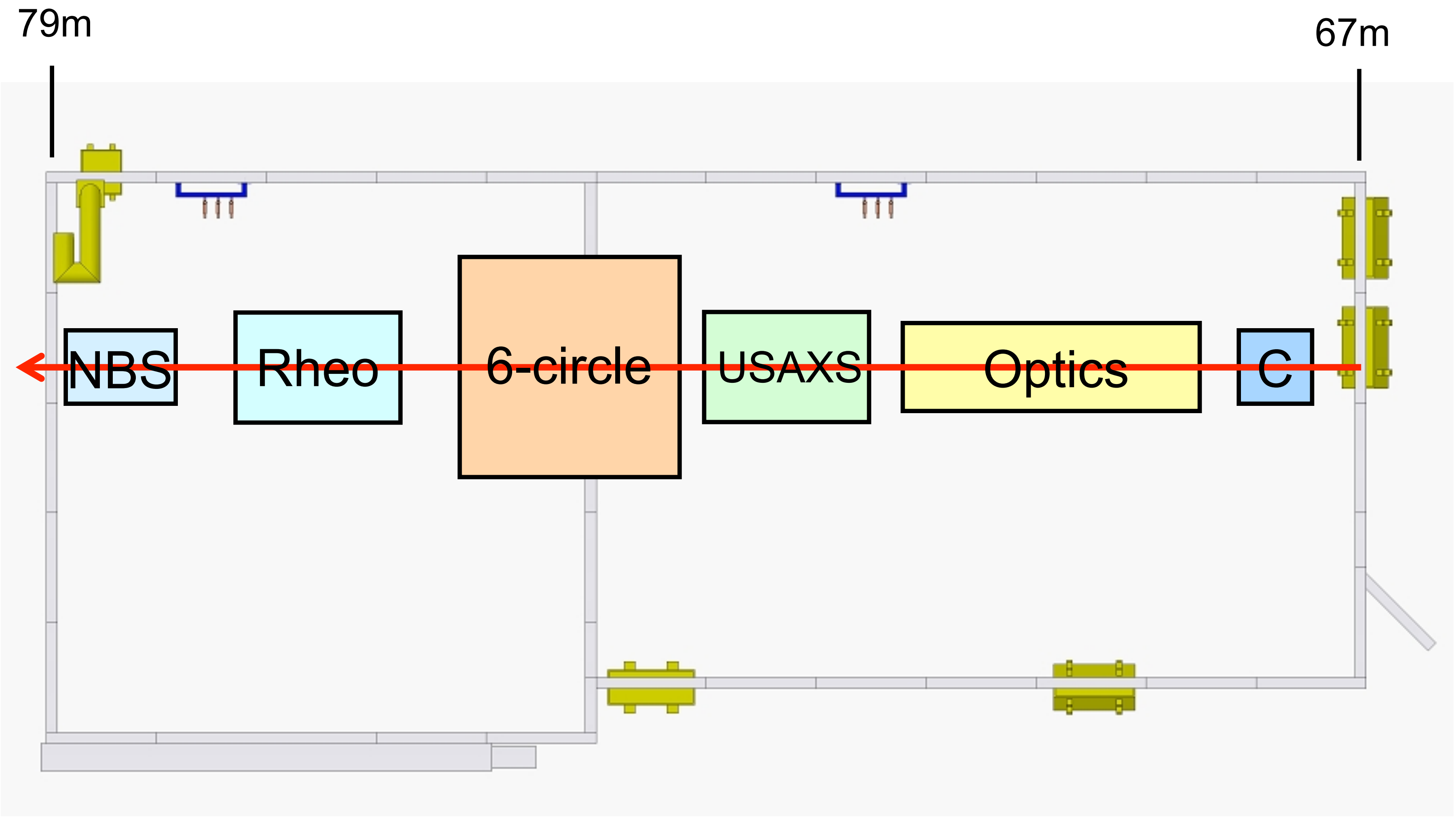
- Situated in EH2 (~87.6m)
- 5-axis IDT table
- UHV KB mirror chamber
  Focus length: 302_vmm; 200_hmm
  Focus size: down to 150_v x $300_h nm^2$
  Flux: up to 5x 10^{10} photons/s
  Cutoff energy: 13.9keV
- Waveguides for 7.9 and 13.8keV
- Flexible accessible setup

- 2nd generation of PIEZO stages

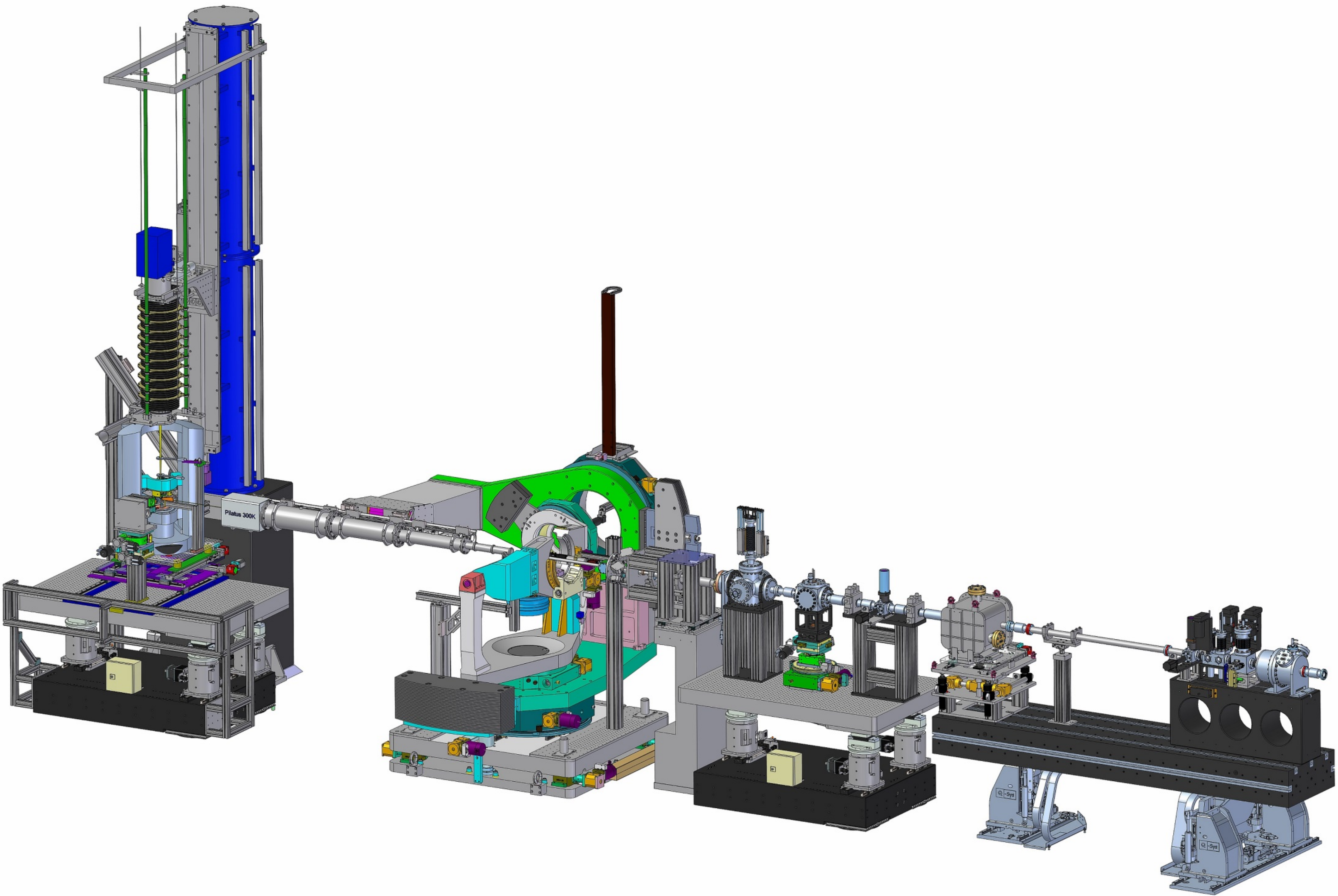
Accomplishments:
- 1st nano focus beam at PETRA III
- 10x10nm² waveguide focus size
- 5x5nm² MLL lens focus size

- Strong sample setup restrictions!!!

1st experimental hutch EH1: Overview



1st experimental hutch EH1: Overview



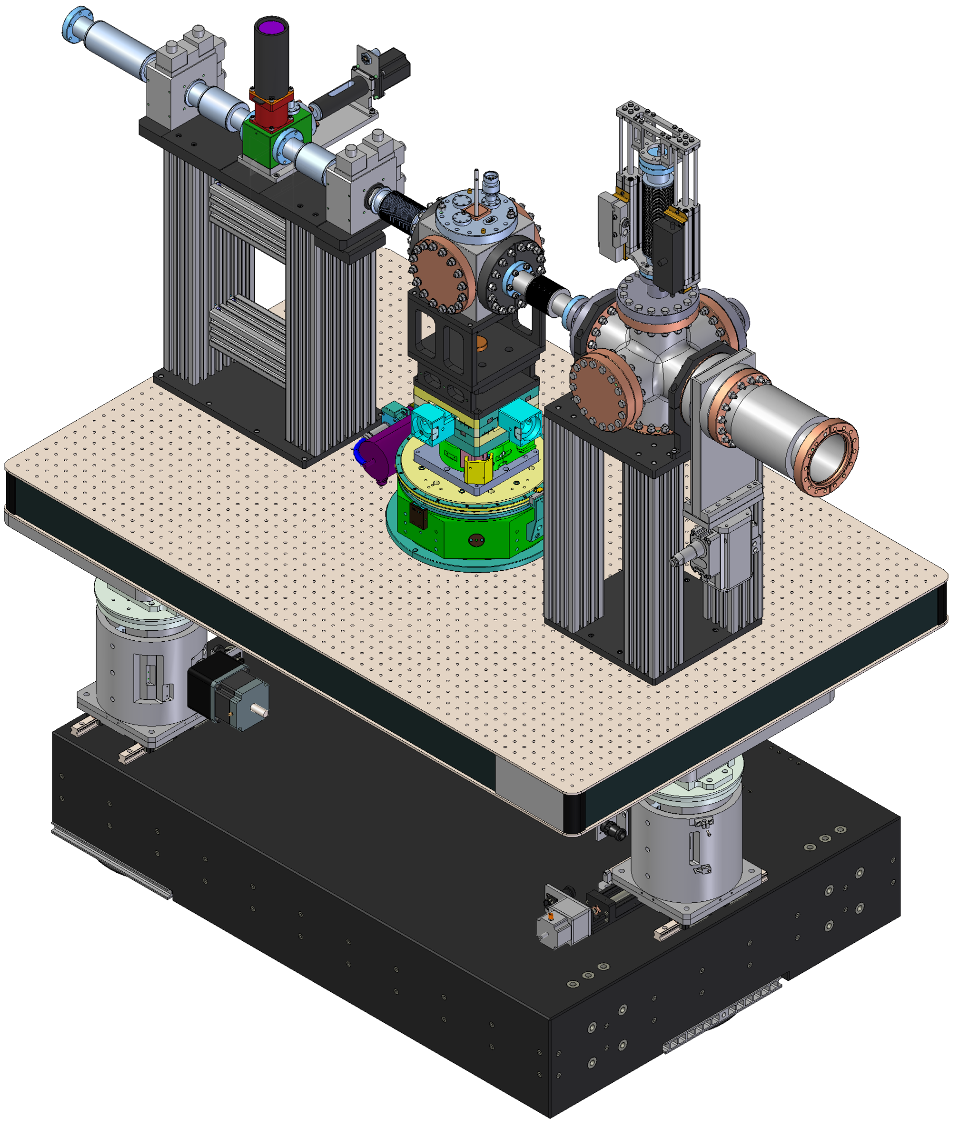
1st experimental hutch EH1: USAXS setup

Soft Matter SAXS setup:

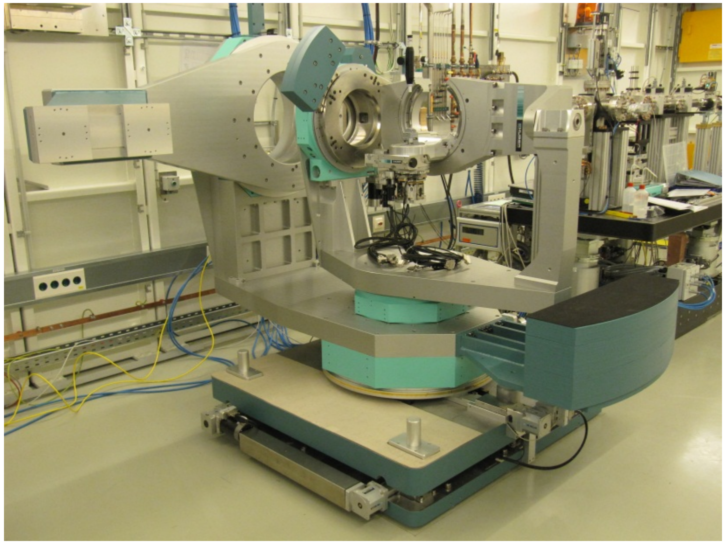
- up to 21.5m sample-detector distance
- compatible with 4-circle setup
- easily removable to allow other setups
- 5-axis table up to 500kg loads
- strongly limited Q range

Possibility to resolve speckles using “large” unfocused beams with a relatively low flux density
→ beam damage reduction

Micro-focus position for
6-circle diffractometer & rheology setup



1st experimental hutch EH1: 6-circle diffractometer

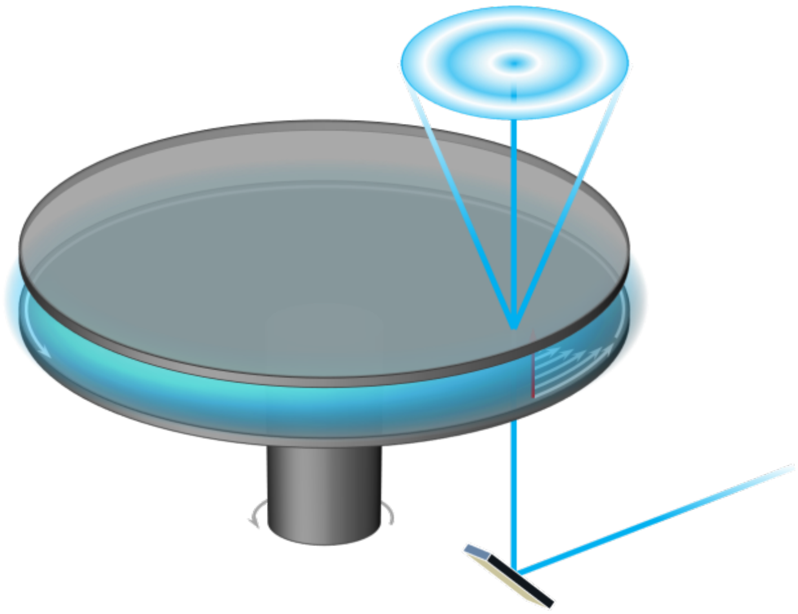


- 6-circle diffractometer
- compatible with P09 6-circle
- compatible with ARS cryostats
- shared flow cryostat with P09
- open Chi circle
- micro-focus from USAXS setup
- > 2m sample – detector distance

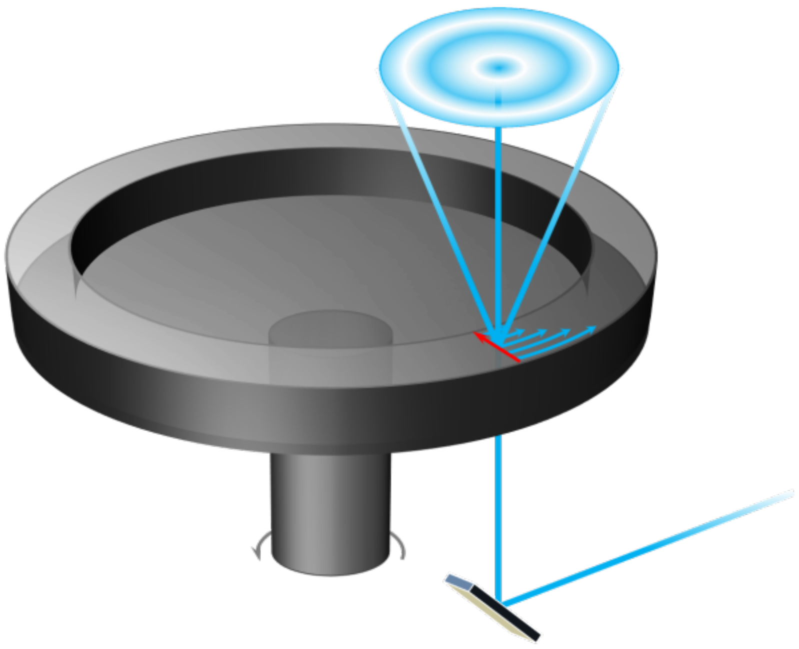
n	Alias	symbol	Motor name	Note	Restrictions
1	delta (2theta_v)	δ	E1_mot66	(+) left	-10º : +190º
2	gamma (2theta_h)	γ	E1_mot65	(+)	-10º : +170º
3	omega (theta_v)	ω	E1_mot53	(+) left	±180º
4	mu (theta_h)	μ	E1_mot54	(+)	±180º
5	chi	ζ	E1_mot57	(+)	-5º : +95º
6	phi	φ	E1_mot52	(-)(o) left	±180º

Vertical X-ray Rheology

Plate- Plate cell



Couette cell

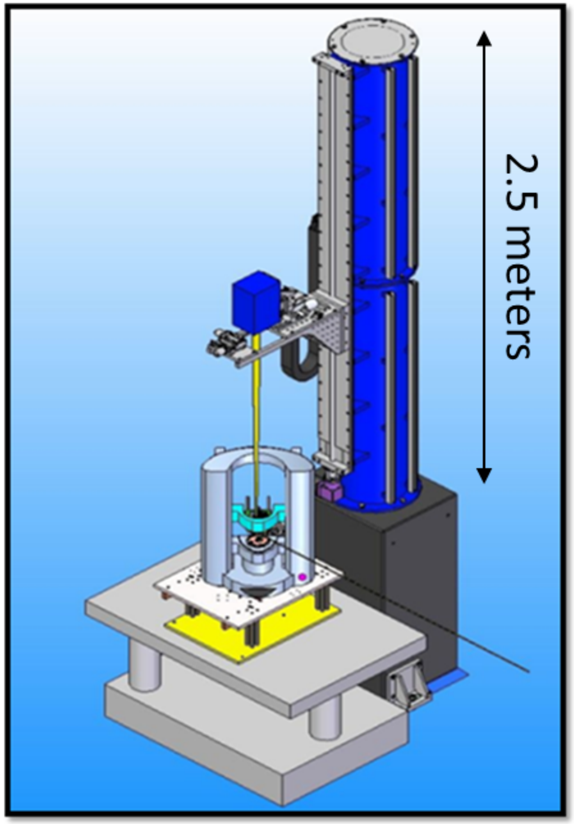


B. Struth et al, *Langmuir*, Feb. 2011.

P. Lettinga et al, *unpublished*

1st experimental hutch EH1: Rheology setup

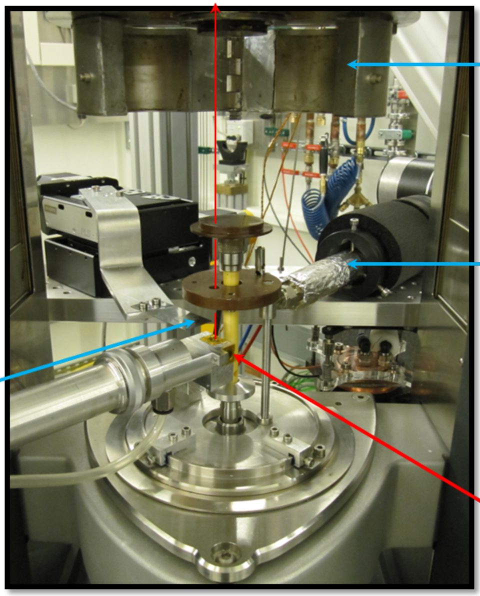
„acrobatic" setup | **virtual setup** | **current setup**

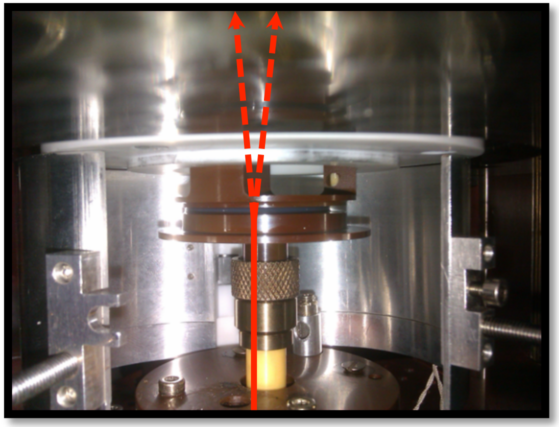


B. Struth et al, *Langmuir*, Feb. 2011.

1st experimental hutch EH1: Rheology setup

based on "inverted" Haake MARS II rheometer





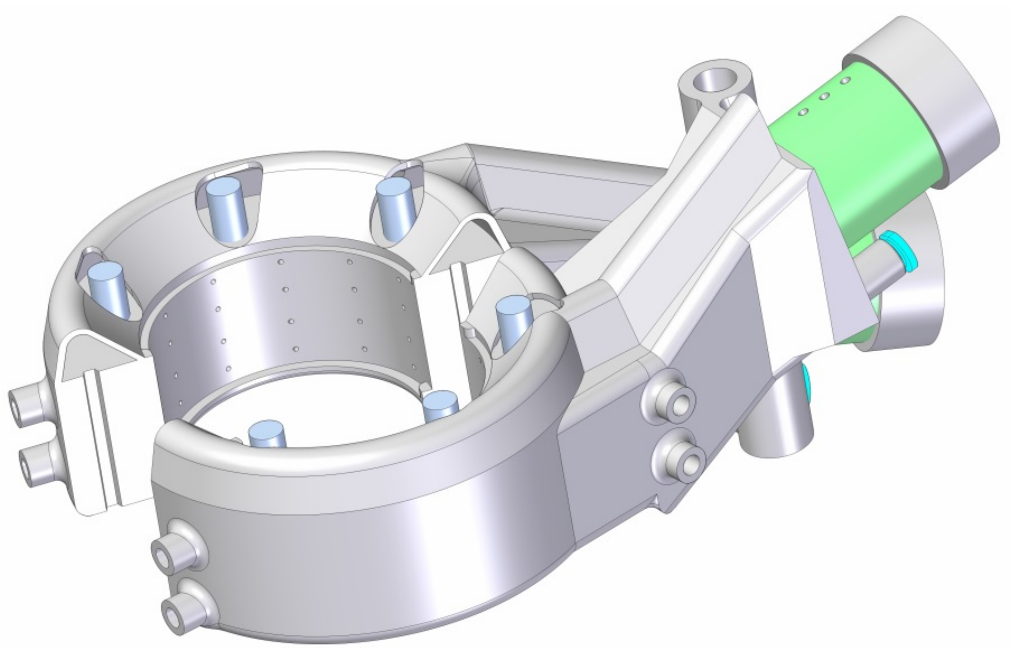
Rheology setup:

- plate-plate, plate-cone & Couette geometries
- vertical scattering geometry
- currently mostly incoherent TR scattering
- Pilatus detector / Lambda detector
- High inherent background

B. Struth et al, *Langmuir*, Feb. 2011.

P10 Rheometer: Sample environment

New sample environment
(by E. Stellamanns)



- Up to 200ºC
- Humidity control posible
- 3D Print

- Addon:
  Optical probe of the sample

Summary and Conclusions

- New X-ray sources are well suited to work with small beams and coherent x-rays
- A large variety of scattering experiments can be done coherently
- Coherent and incoherent experiments use similar experimental setups
- XPCS experiments are often flux limited
  → Need to minimize parasitic scattering (windows)
- Detector field of view is strongly limited in order to resolve speckles

Questions

- When does a new setup become a 'supported standard setup'?
- How does one keep rarely used labor intense setups running?
- What are 'standard' shared electronics (Keithleys, Power supplies)?
- How can we integrate user equipment easily into 'fast scanning'?
- Experimental setups at beamlines are mostly compromises and can't provide ultimate performances? E.g. rheometer
- Many sample systems are at beam damage limit for XPCS? How do we incorporate new data collection schemes?

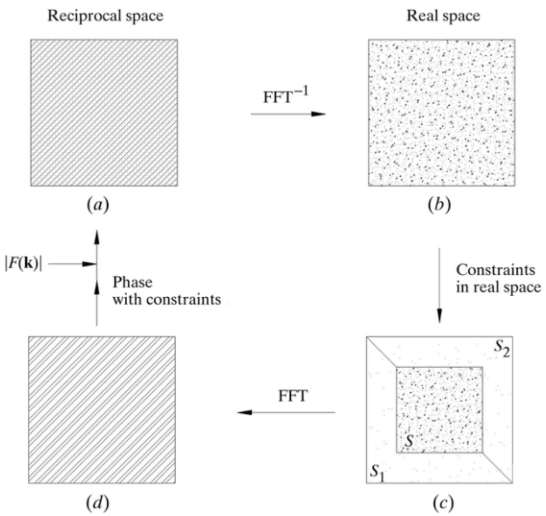
Acknowledgements II

Thank you for your attention!

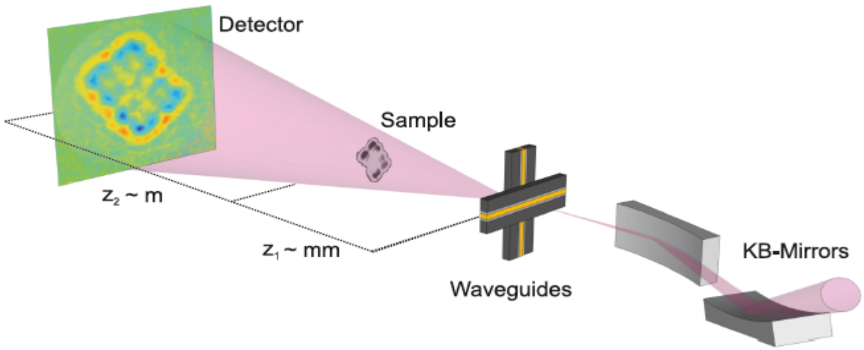
Lensless Imaging

Techniques for medium to high resolution images of small structures

Lensless imaging (coherent diffractive imaging) techniques aim to reconstruct the real-space structure of objects from its diffraction pattern (or hologram) by the use of constraints and phase-retrieval algorithms (e.g. Gerchberg-Saxton-Fienup) or by holographic reconstruction using Fresnel back propagation.

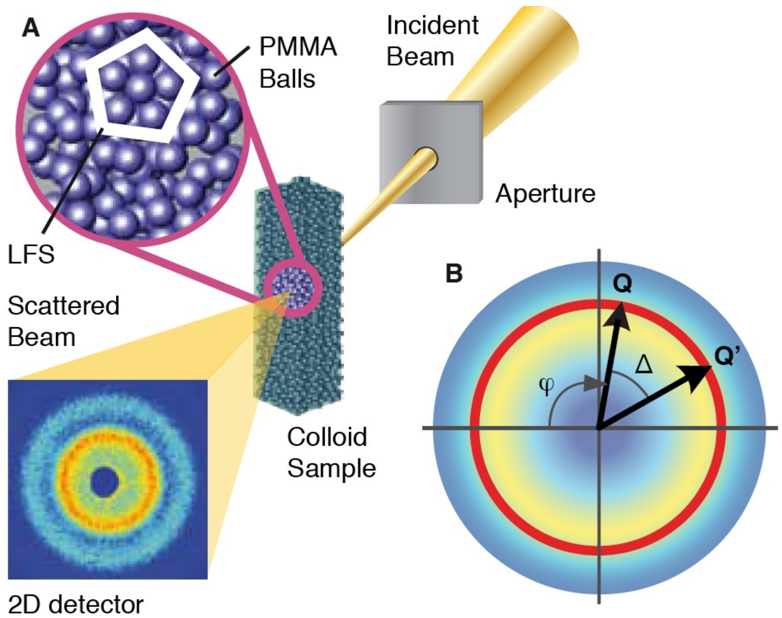


- Ptychography
- Plain-Wave CDI
- Holographic imaging
- Keyhole imaging
- …



X-ray Cross Correlation Analysis (XCCA)

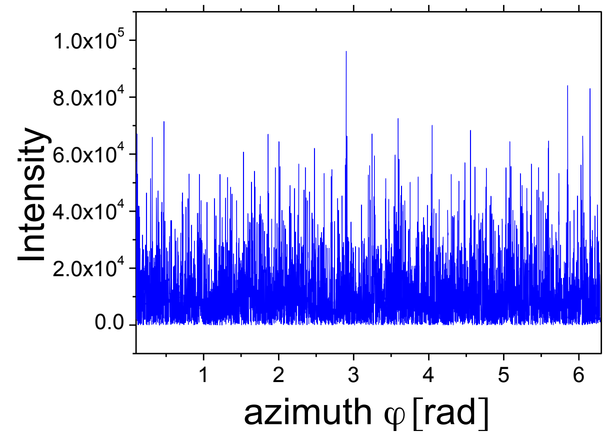
Probe of local (bond order) structure



Orientational Correlation function

$$C_Q(\Delta) = \langle I(Q,\varphi) I(Q,\varphi+\Delta)\rangle - \langle I(Q,\varphi)\rangle_\varphi^2 \,/\, \langle I(Q,\varphi)\rangle_\varphi^2$$

P. Wochner et al. PNAS 106, 11511 (2009)



FT of I(φ): $$\hat{I}(Q,l) = \Re\left(\int_0^{2\pi} I(Q,\varphi) e^{2\pi i l \varphi} d\varphi\right)$$

Variance of $I(Q,l)$: $$\Psi(Q,l) = \left\langle \hat{I}(Q,l)^2 \right\rangle - \left\langle \hat{I}(Q,l) \right\rangle^2$$

M. Altarelli et al. PRB 82, (2010), 104207

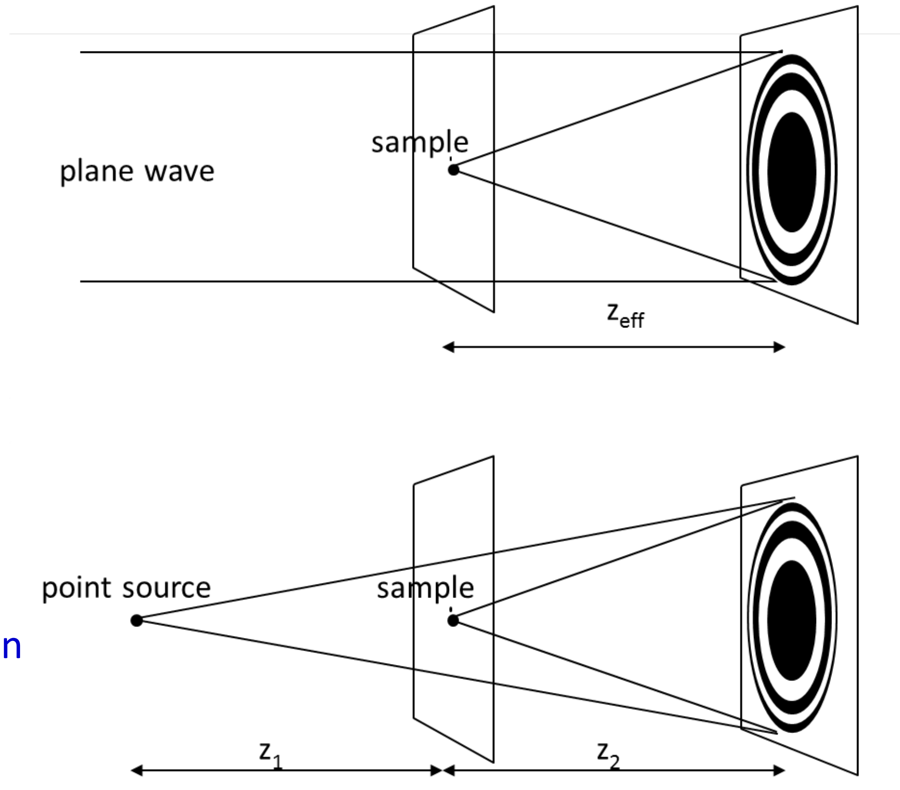
GINIX: Waveguide imaging principle

Fresnel scaling theorem: *an equivalence between parallel and point source illumination*

hologram recorded with a point source corresponds to a hologram recorded with a plane wave at

an effective defocusing distance $$z_{eff} = \frac{z_1 z_2}{z_1 + z_2}$$

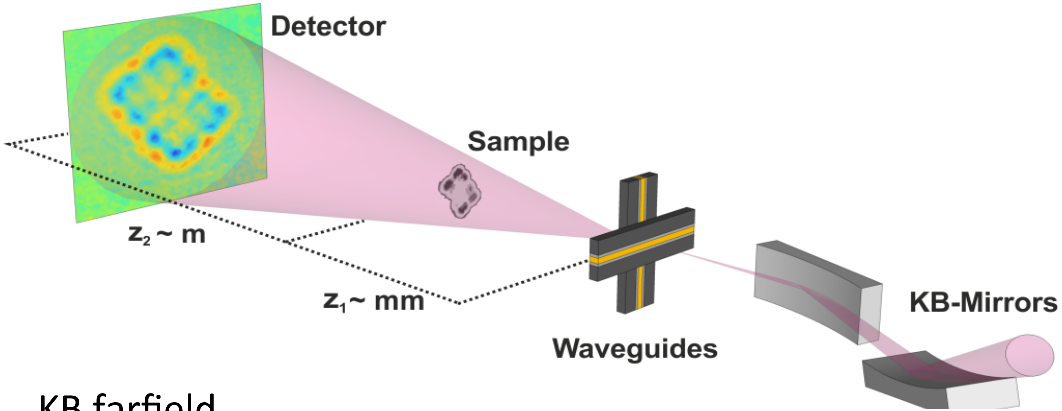
magnified by $$M = \frac{z_1 + z_2}{z_1}$$



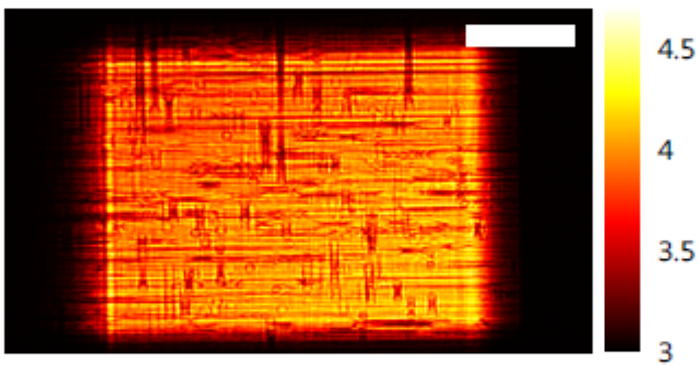
→ magnification allows for a spatial resolution much better as detector pixel size!

→ plane wave setup used for simulations and reconstruction

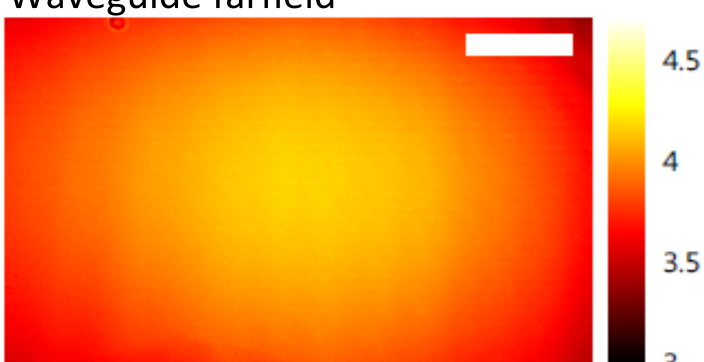
The nanofocus / waveguide setup: Why waveguides?



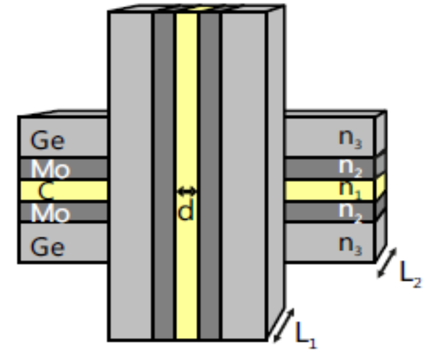
KB farfield



Waveguide farfield



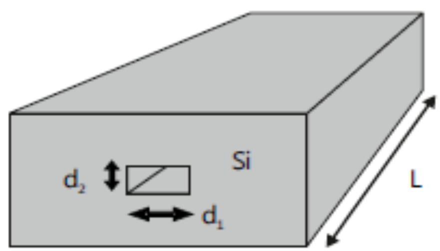
Crossed waveguides



~20 nm x 20 nm; 10^8 -10^9 ph/s; 13-15 keV

Krüger et al. Opt. Express **18**, 13492 (2010)
Krüger et al. J. Synchrotron. Rad. **19**, 227 (2012)

Etched waveguides



~20 nm x 17 nm
2×10^9 ph/s
8 keV

H. Neubauer, Doktorarbeit 2012
J. Haber, Masterarbeit 2013