Scientific Highlights @ P10 PETRA III

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Beschleuniger Betriebsseminar 2011
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HELMHOLTZ
GEMEINSCHAFT

DESY

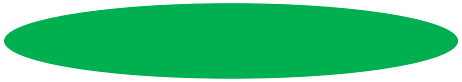
Übersicht

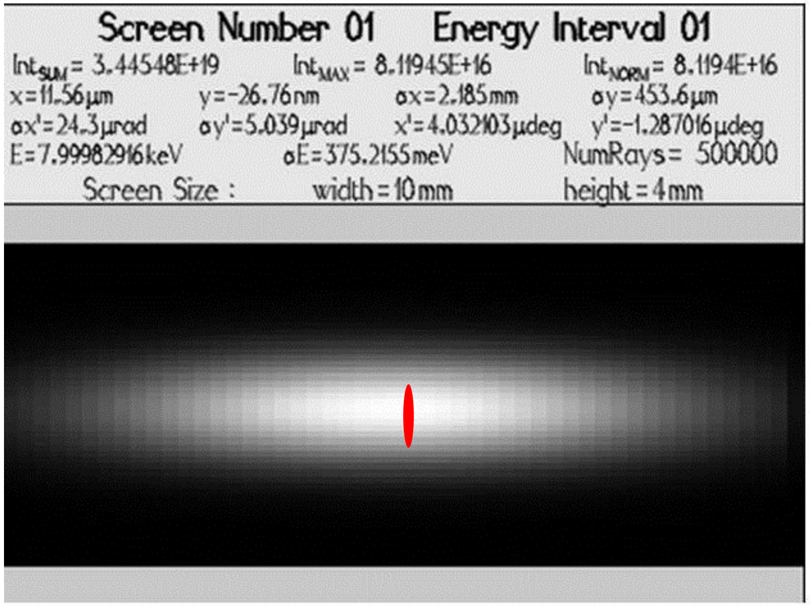
- **Warum eine Kohärenz Beamline @ PETRA III?**
- **Einführung zu P10**
- **Wissenschaftliche Aktivitäten bei P10**
  - **Coherent diffractive imaging (CDI)**
  - **X-ray photon correlation spectroscopy (XPCS)**
- **Weitere wissenschaftliche Beispiele**
- **Zusammenfassung**

Warum eine kohärente Beamline bei PETRA III?

Der kohärente Fluss ist proportional zur Brillianz: $F_c \sim \lambda^2/2 \cdot B$

Low beta Quelle: ~14 x 84 μm^2 (FWHM)





Transverse coherence length: $\xi_{v,h} = {}^{1}/_{2\sqrt{\pi}}\ {}^{\lambda R}/_{(2.35\ \sigma_{v,h})}$

$\xi_{v,h}$ ~ 270 x 45 μm^2 (FWHM)

(@ 90m, 8keV)

Der kohärente Anteil von PETRA III ist grösser als an anderen Synchrotrons (e.g. ESRF, APS, …)!

Kohärenz Beamline P10 von PETRA III: Einführung

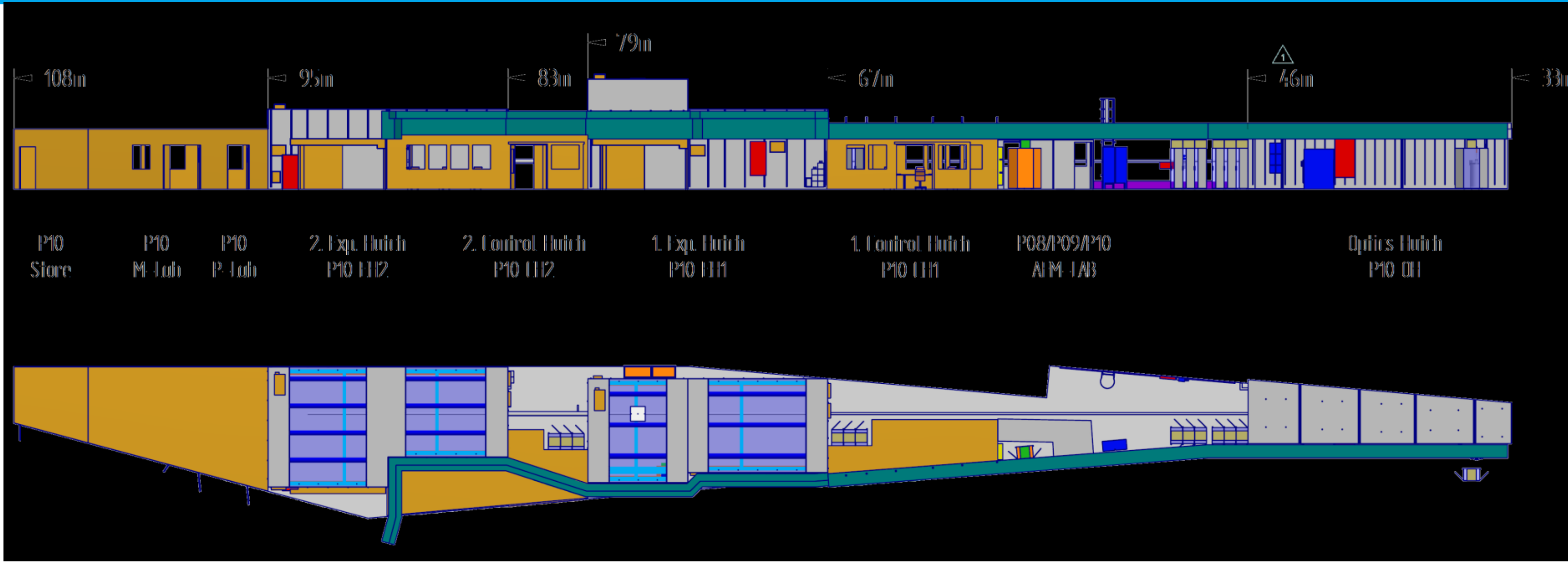
The Coherence Beamline P10 specializes in facilitating coherent x-ray scattering techniques in the medium-hard x-ray range (5—20keV). Scientifically the aim is to investigate structures and dynamics on nanometer length scales. The two main experimental techniques are X-ray Photon Correlation Spectroscopy (XPCS) and Coherent Diffraction Imaging (CDI).

Theoretical longitudinal coherence length and coherent flux @ P10

$\Delta\lambda/\lambda$	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
$3 \cdot 10^{-5}$ [Si(311)]	5.0μm	$6 \cdot 10^{10}$	8keV
$2 \cdot 10^{-3}$ [pink beam, 3rd harmonic]	0.054μm	$1.4 \cdot 10^{12}$	12keV

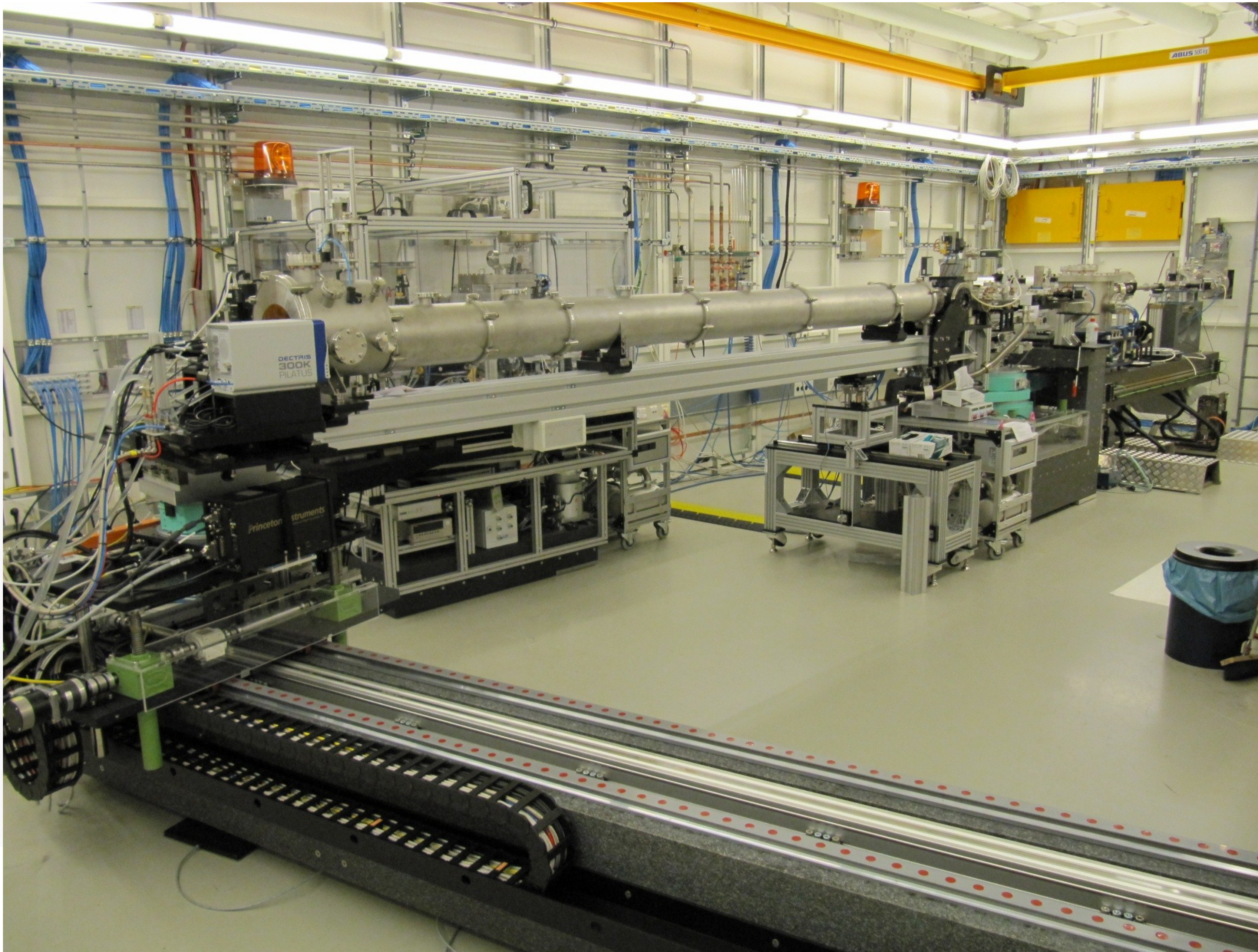
However, without focusing only a tiny fraction (~1/1000) is usable!!!

Kohärenz Beamline P10: Übersicht

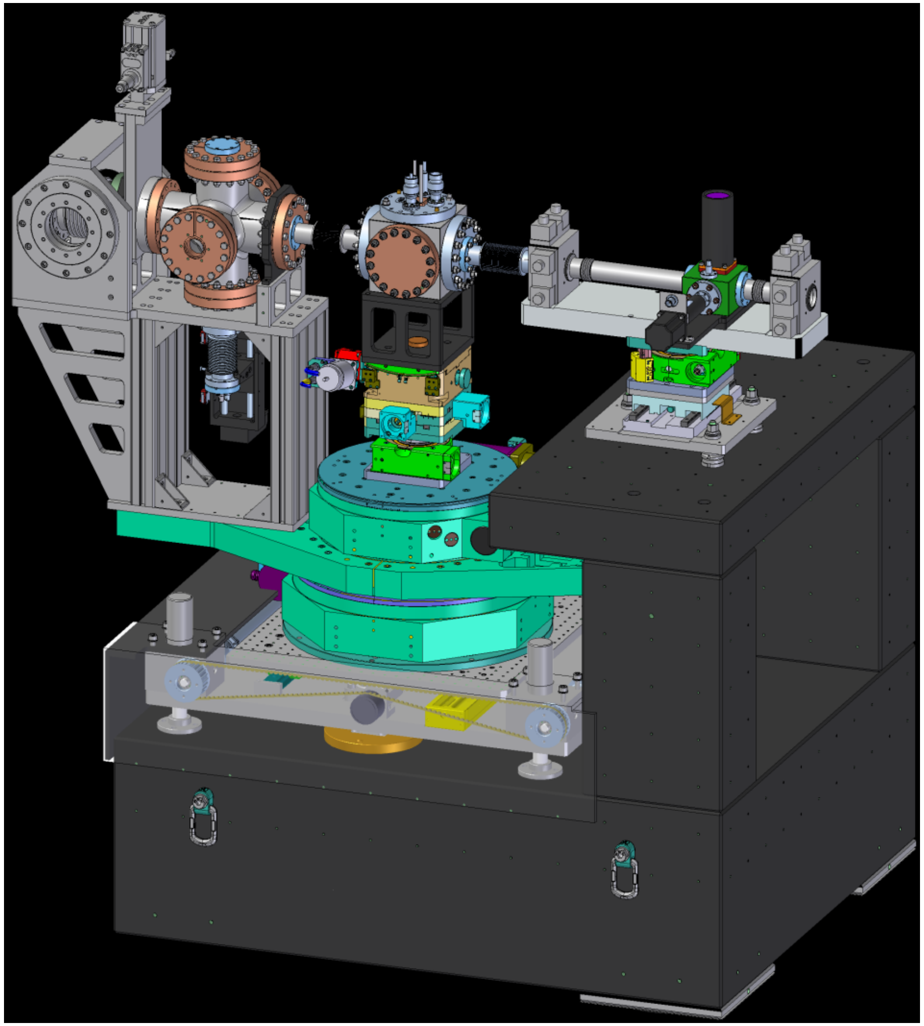


- 1 x Optikhütte
- 2 x Experimentelle Hütten (12m)
- 2 x Kontrolräume
- 1 x Probenpräparationsraum
- 1 x Mechanisches Labor
- 1 x Elektronisches Labor
- 1 x AFM Labor

2. Experimentierhütte EH2 bei P10

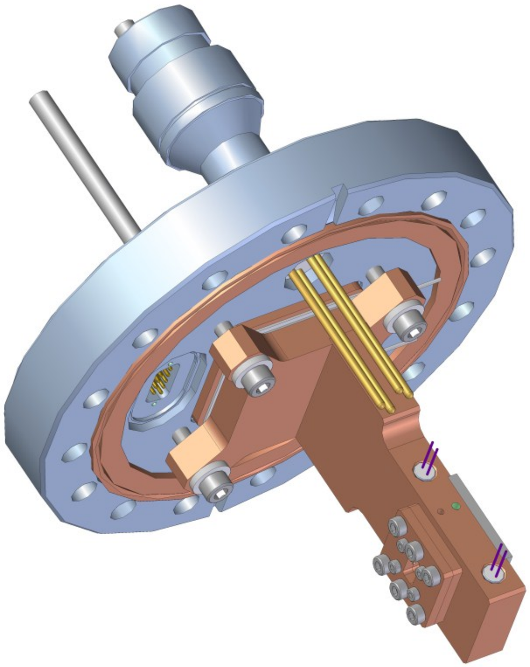


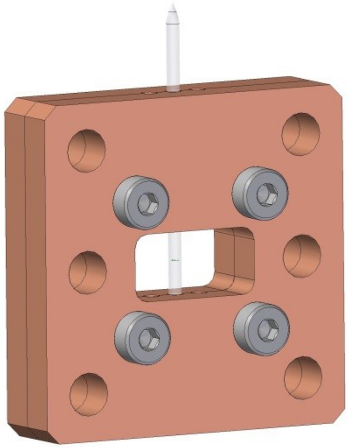
Das Standardsetup von P10: Übersicht



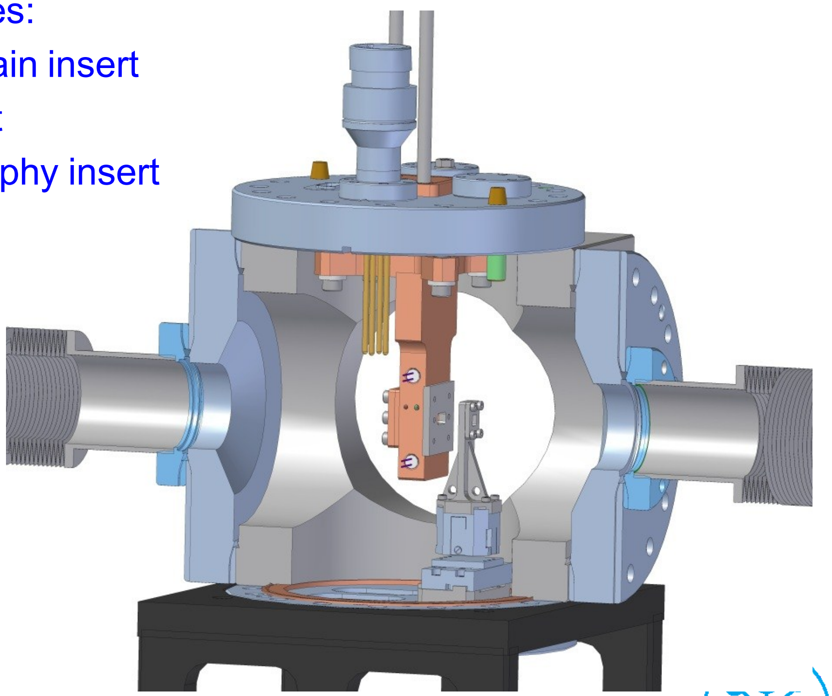
- The standard setup consists of a Huber 4-circle diffractometer sitting on a heavy granite based table. In its final configuration it will be possible to scatter horizontally to ~30°.
- The samples will be placed into a DN100 cube. Different experiments can be easily integrated by designing independent inserts for this cube.
- It is possible to operate this setup fully vacuum integrated. If needed the vacuum environment can be replaced by a large variety of other setups.
- The standard setup will exhibit a sample to detector distance of 5.0m. Flight path as well as the multi-purpose detector holder will sit on 3m long translation stages.

Das Standardsetup: Experimentelle Einsätze

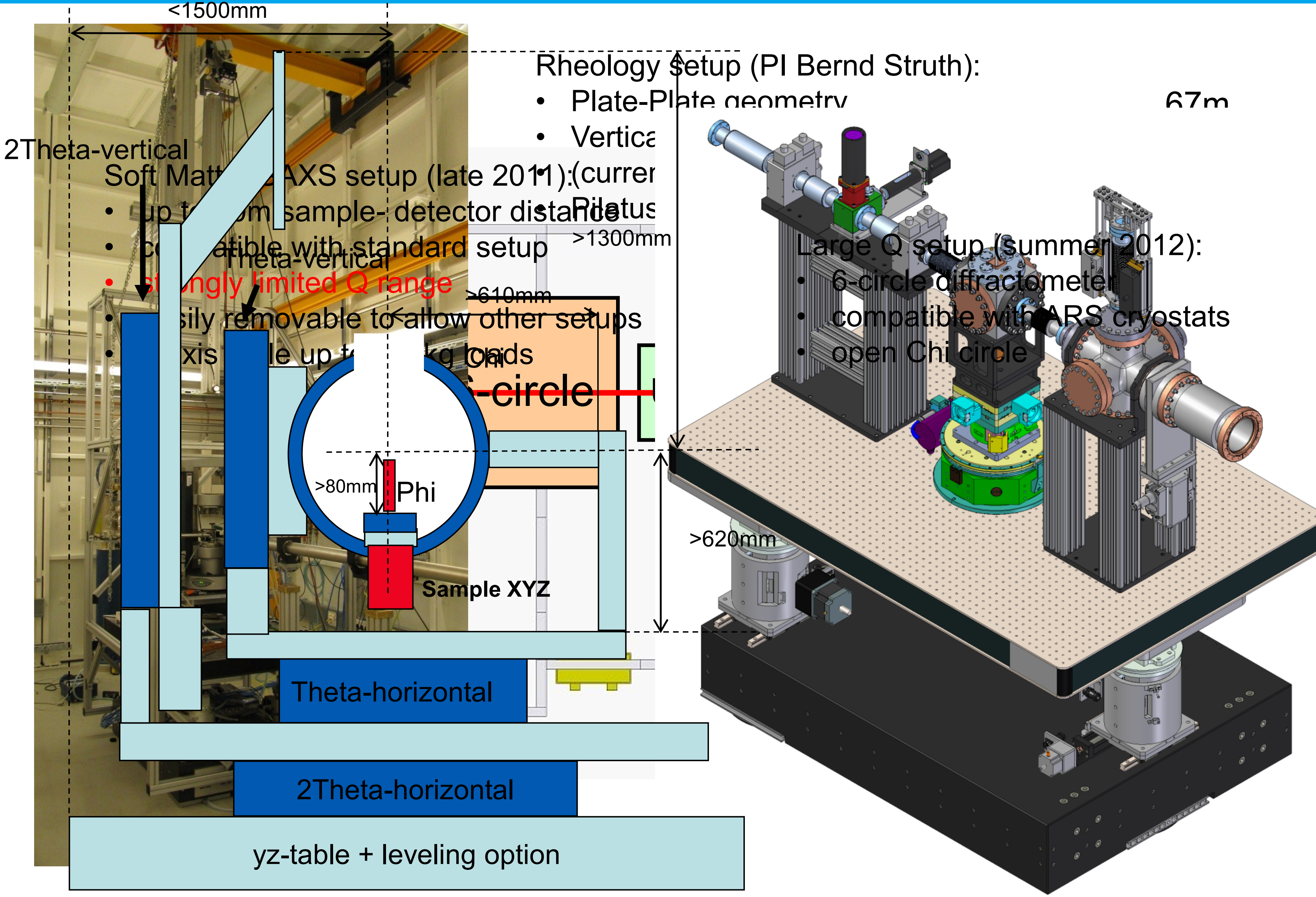




- SAXS and Reflectivity inserts with a heating and cooling option based on Peltier elements and resistive heaters covering the temperature range from -30°C — 200°C.
- SAXS and Reflectivity inserts with a combination of cryogenic cooling and resistive heaters covering the temperature range from -150°C — 50°C.
- 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
  - …



1. Experimentierhütte EH1 von P10

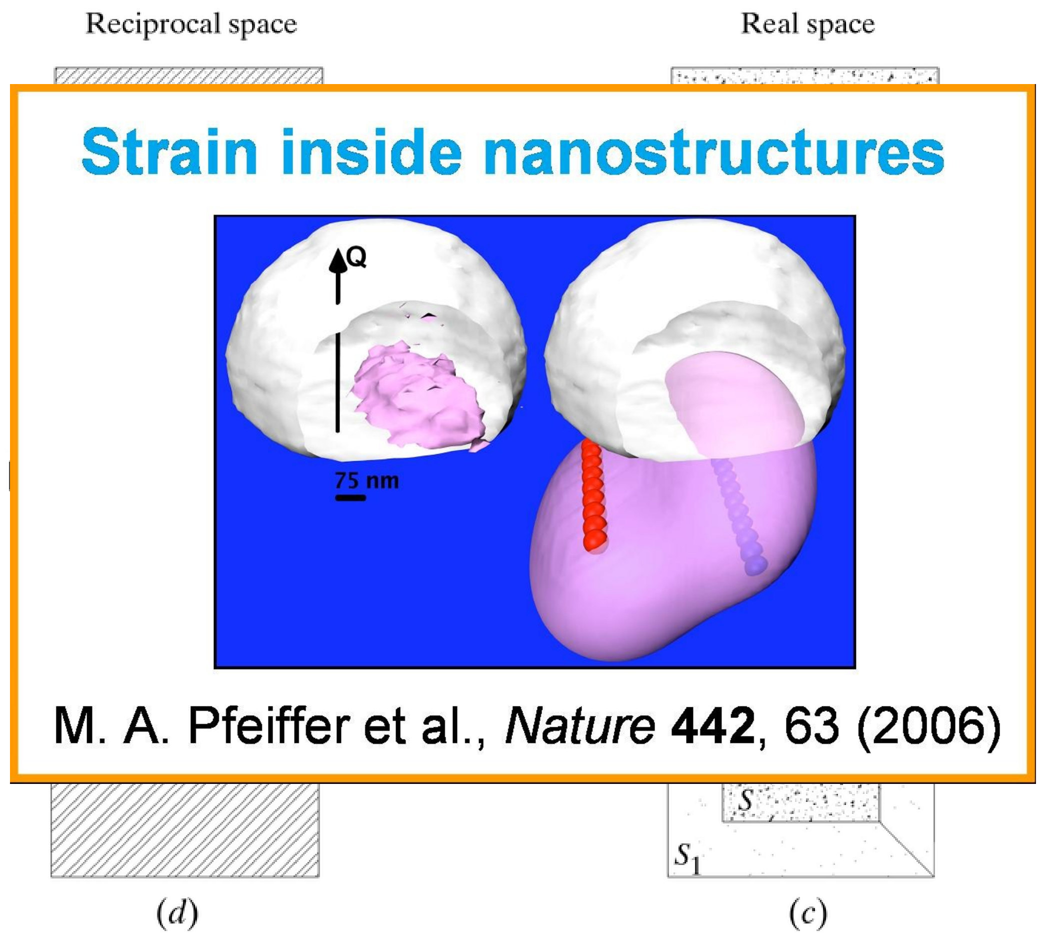


Coherent Diffraction Imaging

High resolution images of small structures

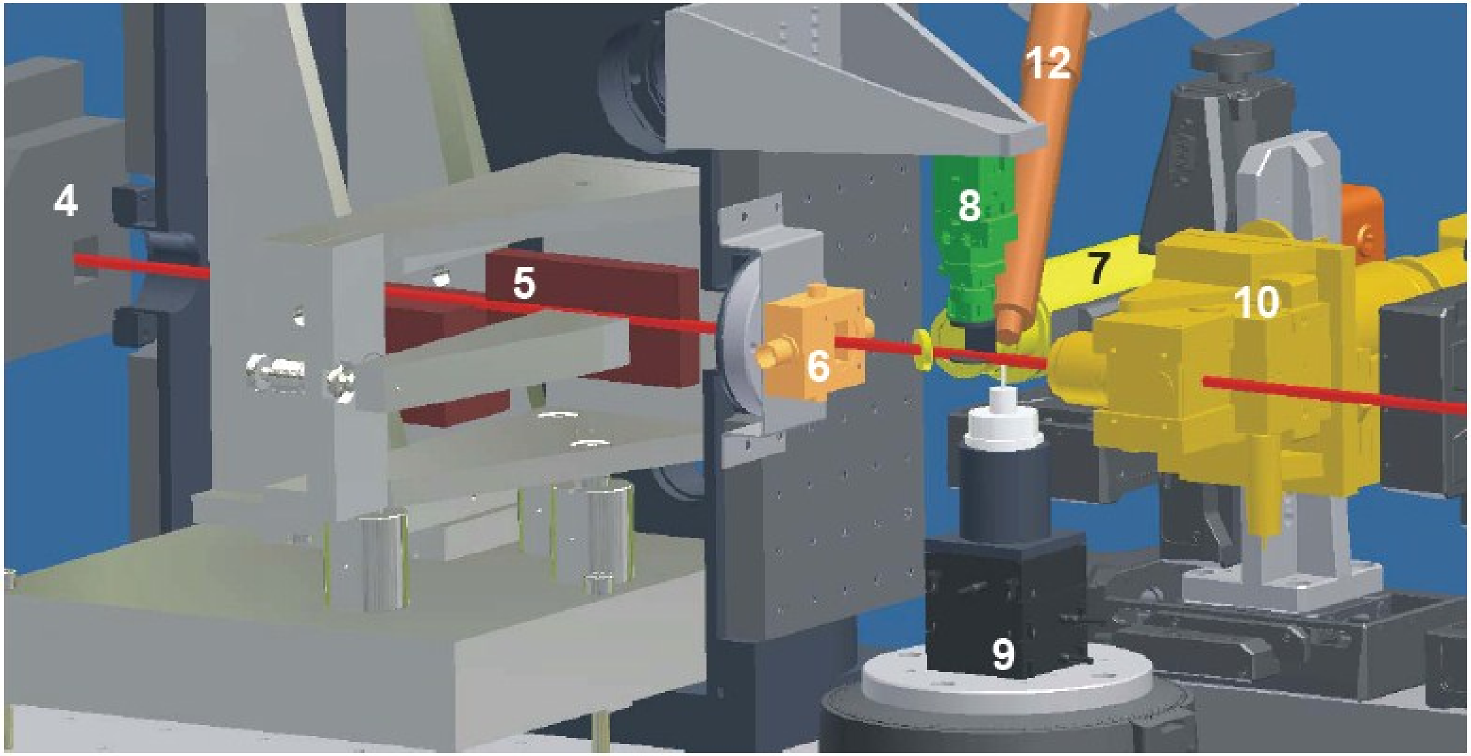
Coherent diffraction imaging techniques aim to reconstruct the real-space structure of objects from its diffraction pattern (reciprocal space) by the use of constraints and phase-retrieval algorithms (e.g. Gerchberg-Saxton-Fienup).

- Ptychography
- Holographic imaging
- …



The nanofocus / waveguide setup

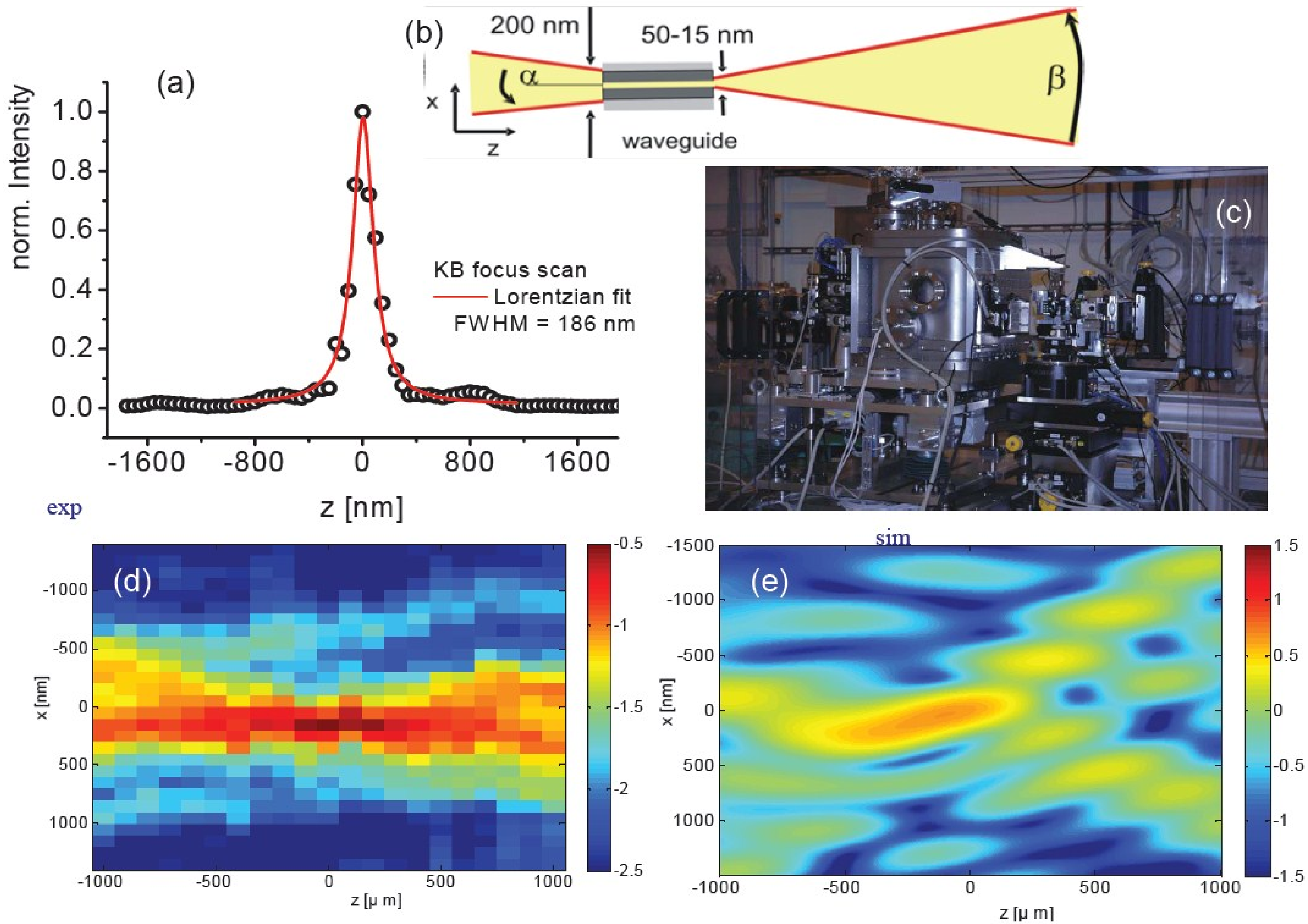
The Göttingen iBox endstation @ P10/PETRA III for Holo-Tomography



→ 11

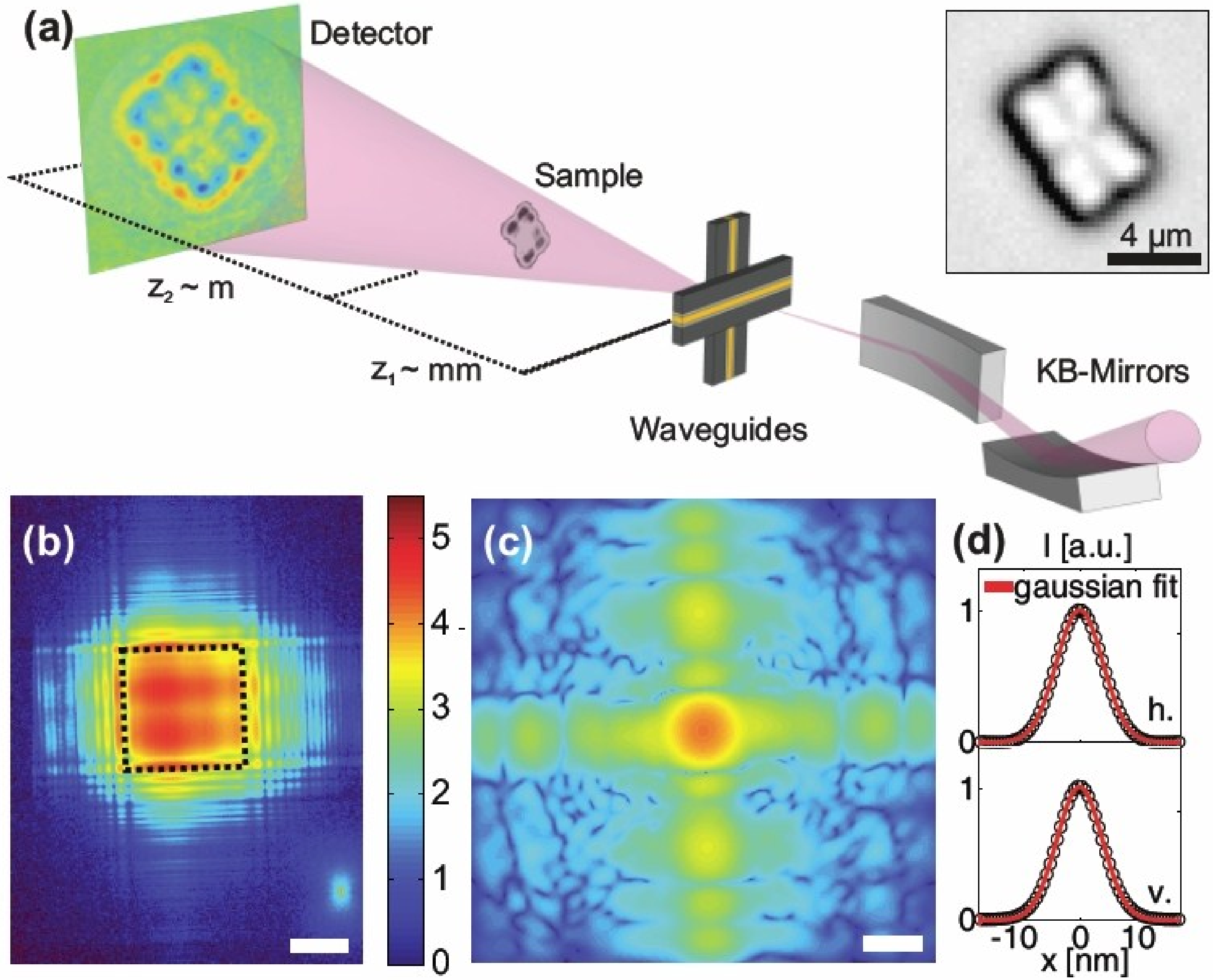
4. Scatterless Slits
5. Kirkpatrick-Baez mirrors
6. Monitor
7. Front side microscope
8. Waveguide motions
9. Sample motions
10. Back side microscope
11. Detector bench
12. Cryogenic cold stream

The nanofocus / waveguide setup: First results

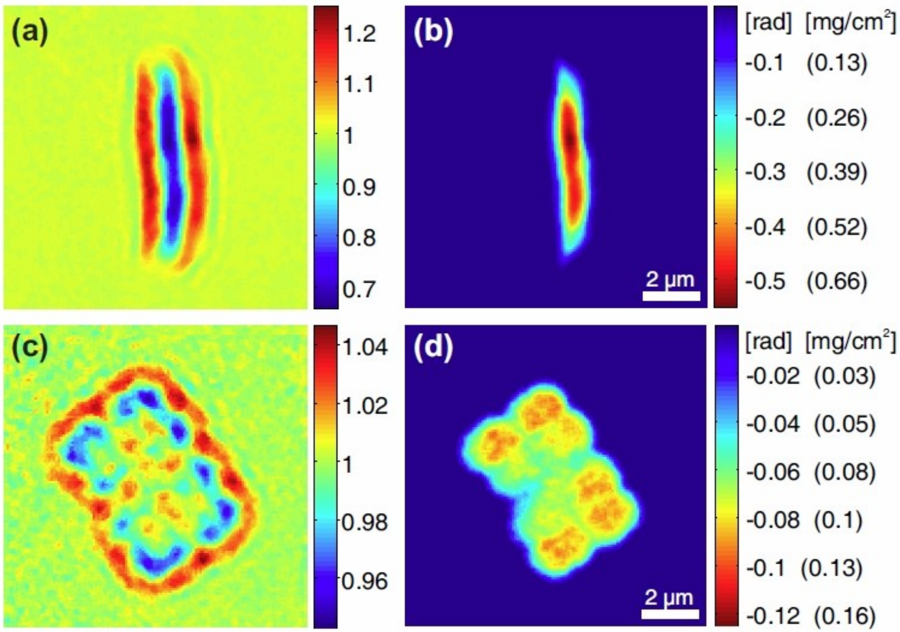


(Coherent) Flux before waveguide ~ 1—5 $\cdot 10^{10}$ photons/s

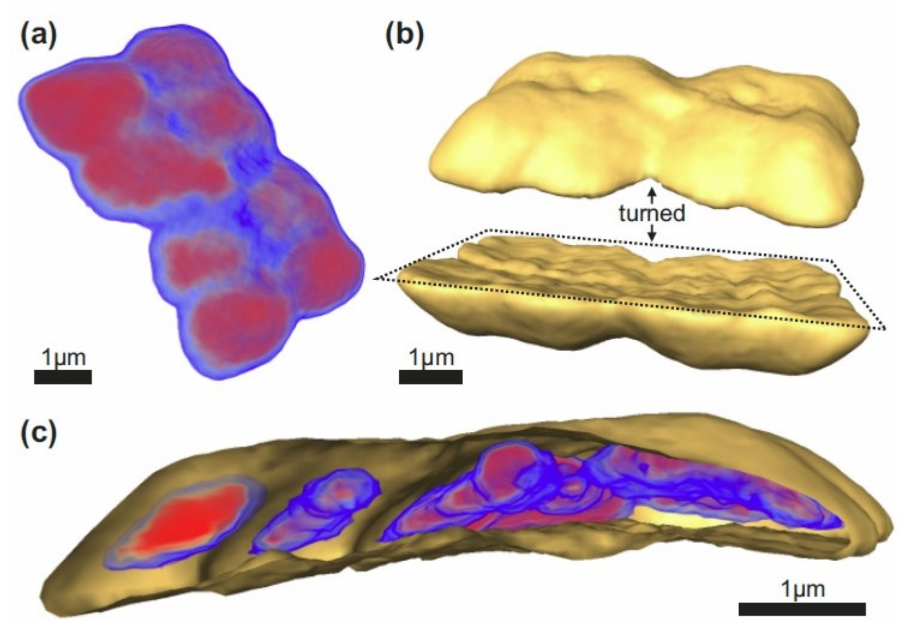
CDI of cells of bacterium ‘Deinococcus radiodurans‘: Schematics



CDI of cells of bacterium ‘Deinococcus radiodurans‘: Results



Low dose experiments on D. radiodurans



X-Ray Photon Correlation Spectroscopy

XPCS is an extension of Dynamic Light Scattering and it utilizes coherent x-rays to study slow (collective) dynamics at small length scales.

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

- 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

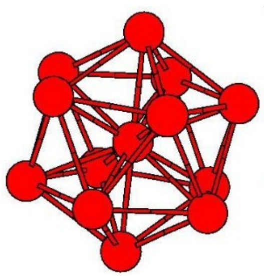
$$g_2(\vec{Q},t) = \frac{\left\langle I(\vec{Q},t)I(\vec{Q},t+\tau)\right\rangle_\tau}{\left\langle I(\vec{Q},\tau)\right\rangle_\tau^2}$$

Coherent Scattering: Scientific opportunities

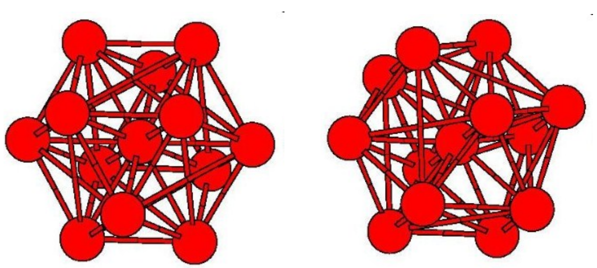
Hidden local symmetries in disordered matter

Can coherent x-ray scattering help to gain a better understanding of the glass transition?

Two popular glass forming scenarios are:
(a) general tendency towards icosahedral order, but locally favored structures cannot fill space
(b) general tendency towards crystalline order, but frustration effects prevent crystallization due to locally favored structures



icosahedral structures can not fill space
but may be energetically favored in liquids
"locally favored structures (lfs)"



fcc and hcp structures can fill up space
and form crystals

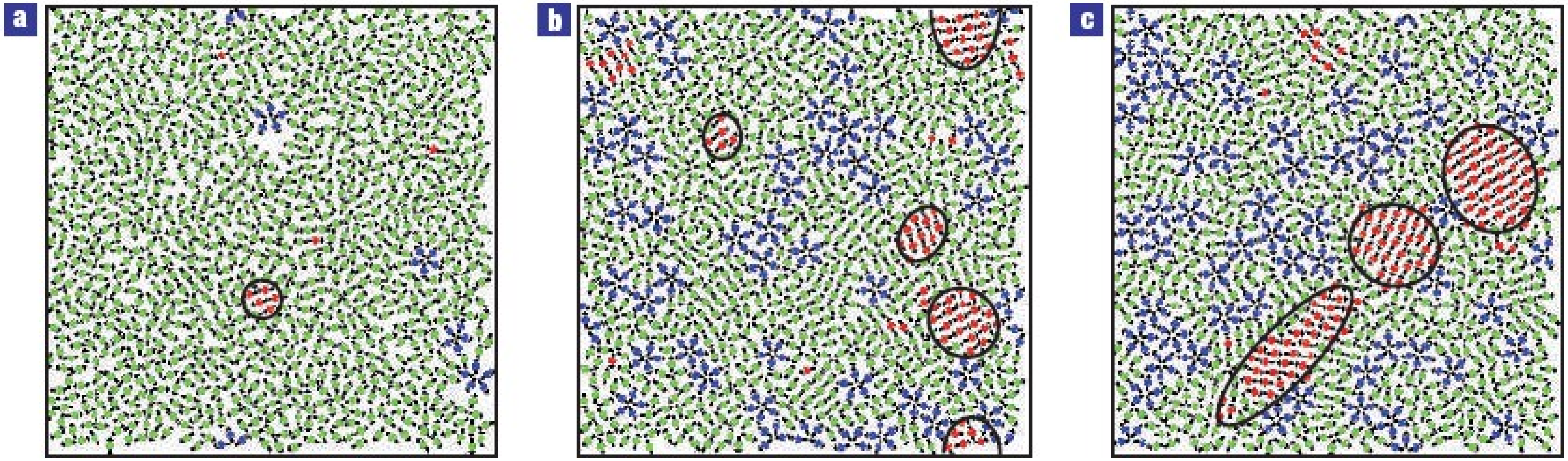
P. Wochner et al., *PNAS* **106**, 11511 (2009)

Coherent Scattering: Scientific opportunities

Hidden local symmetries in disordered matter

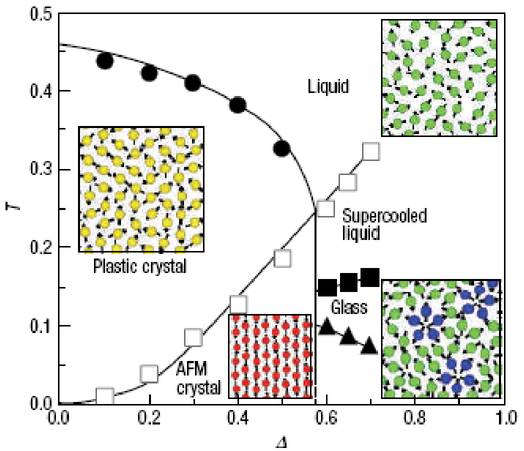
MD simulations – snapshot of liquid structures

Shintani & Tanaka *Nature Physics* 2, 200 (2006).



$$T_r = \frac{k_B T}{\epsilon}$$

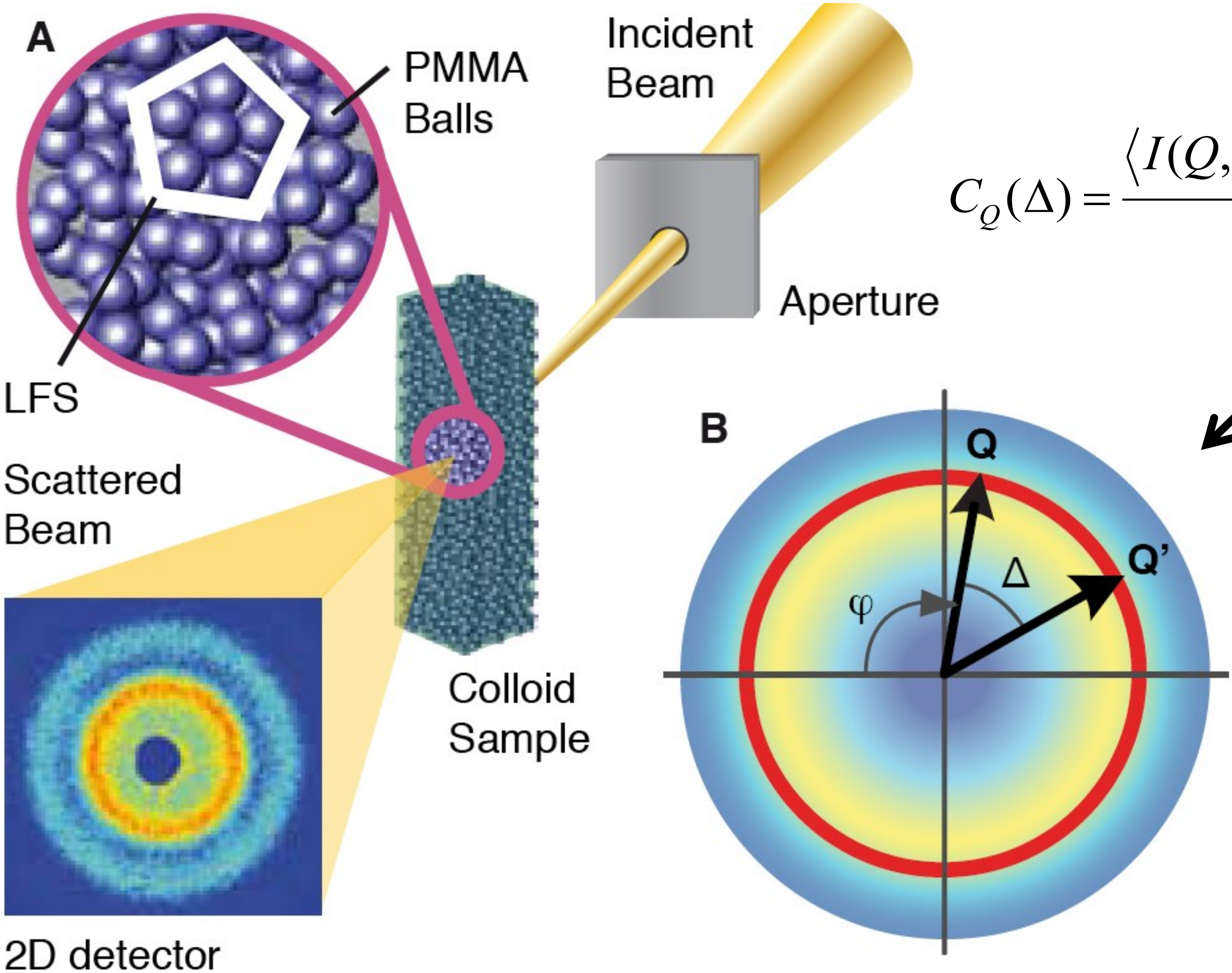
- Dynamic heterogeneity
- Slower regions ➔ higher degree of order
- characteristic length and the lifetime increase on cooling.



Tanaka et al. Nature Mat. 9, 324, 2010

Coherent X-Rays: Scientific opportunities

Hidden local symmetries in disordered matter



$$C_Q(\Delta) = \frac{\langle I(Q,\varphi) I(Q,\varphi+\Delta)\rangle_\varphi - \langle I(Q,\varphi)\rangle_\varphi^2}{\langle I(Q,\varphi)\rangle_\varphi^2}$$

Fourier Transformation

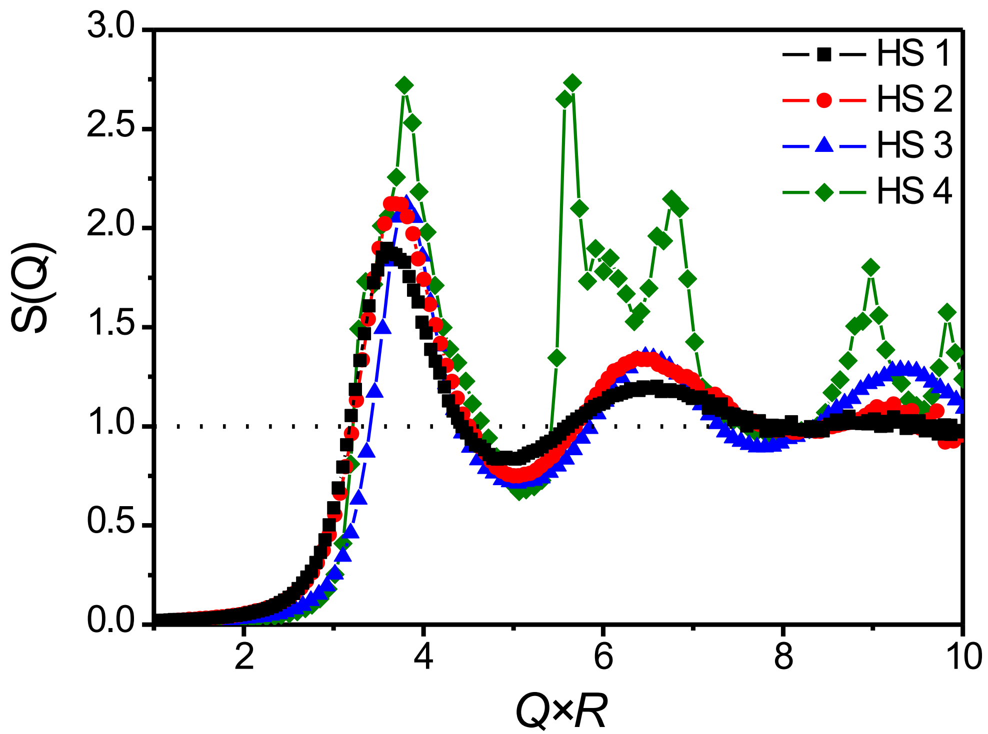
$$\hat{C}_Q(l)$$

Ensemble average

$$\langle \hat{C}_Q(l) \rangle$$

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

XCCA @ P10



$$S(Q) = \frac{I(Q)}{P(Q)}$$

S(Q) : structure factor

I(Q) : intensity

P(Q) : particle form factor

Sample	R	ΔR/R	ϕ	$S(Q_{max})$	Q_{max}
	nm	%	vol fraction		nm^{-1}
HS 1	71.0	16.6	0.56	1.9	0.051
HS 2	92.5	10.4	0.57	2.2	0.040
HS 3	126.6	6.6	0.56	2.1	0.030
HS 4	126.6	6.6	0.52	2.7	0.028

HS 1 – HS 3: Glassy phase

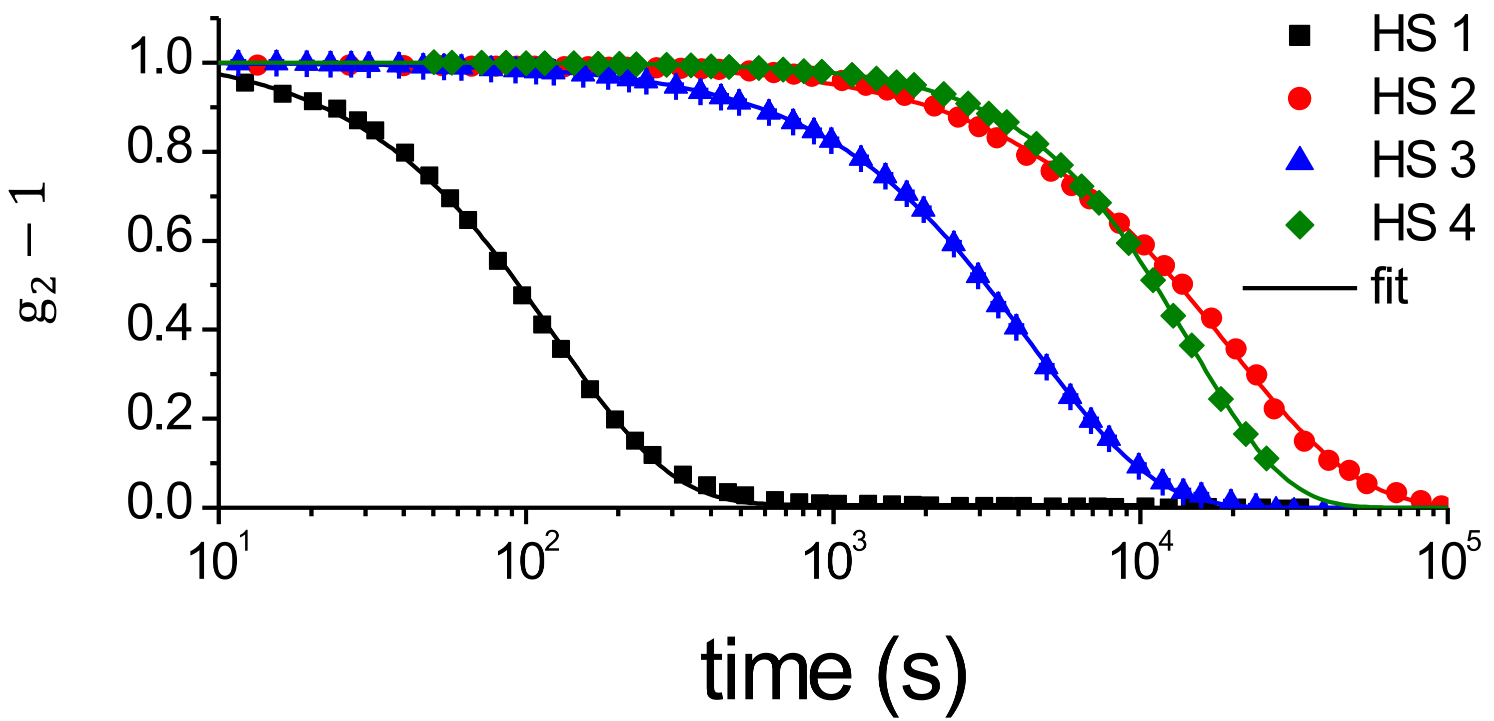
HS 4: Partially crystalline

XCCA @ P10

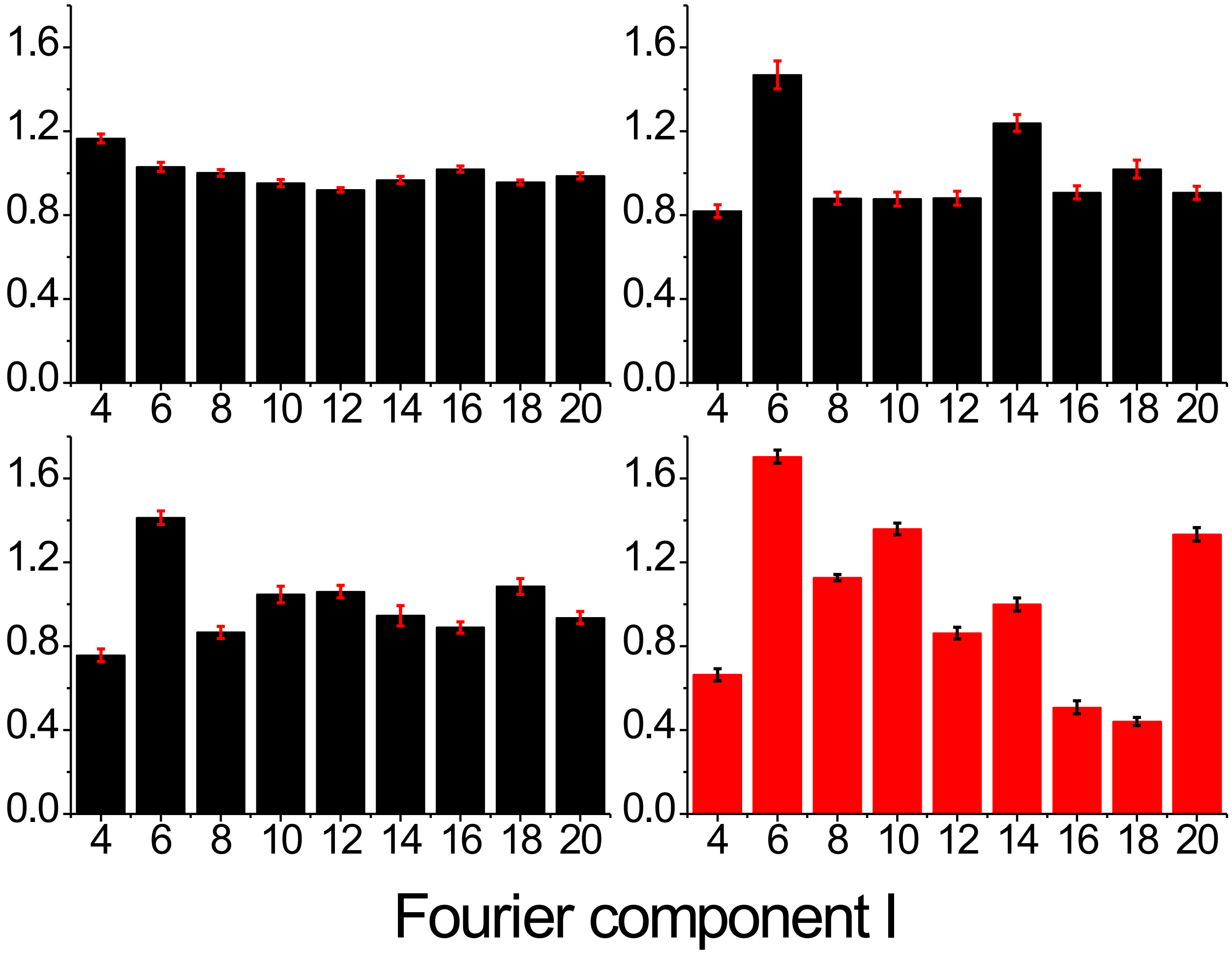
X-ray **P**hoton **C**orrelation **S**pectroscopy

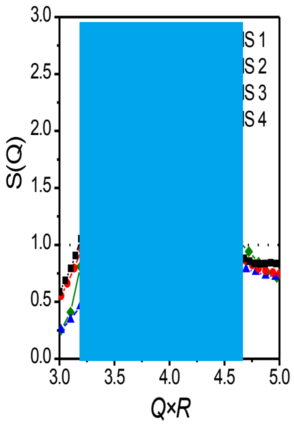
$$g_2(Q,\tau) = \frac{\langle I(Q,t)\cdot I(Q,t+\tau)\rangle}{\langle I(Q,t)\rangle^2}$$

Measured at Q_{max}



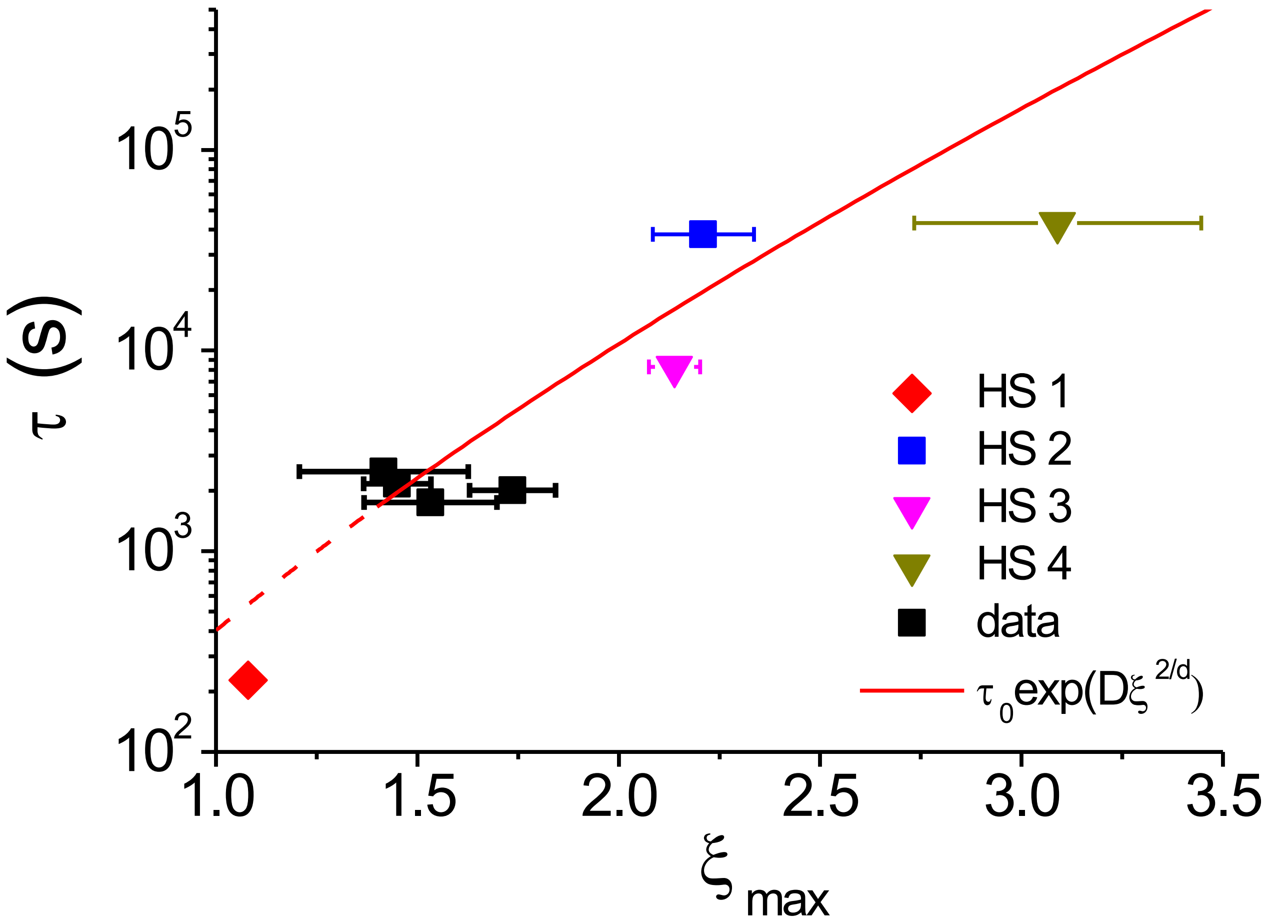
XCCA @ P10


1.6
1.2
0.8
0.4
0.0
4 6 8 10 12 14 16 18 20
Fourier component l


3.0
2.5
2.0
1.5
1.0
0.5
0.0
S(Q)
S 1
S 2
S 3
S 4
3.0 3.5 4.0 4.5 5.0
Q×R

XCCA: Relaxation time and bond-order

The more ordered – the slower



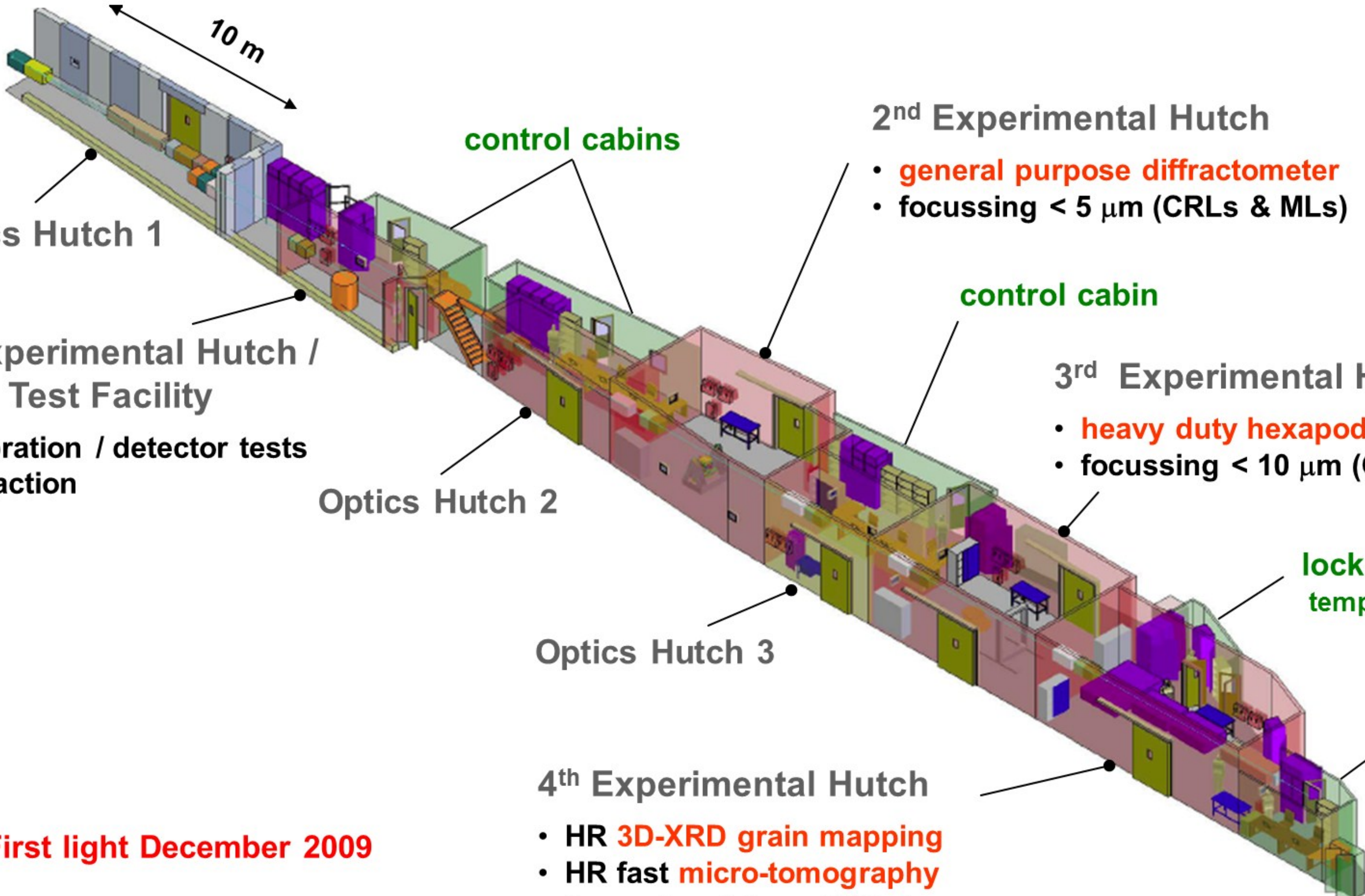
D denotes the fragility index (here *D*=6)

τ_0 the microscopic time (here τ_0 = 1).

d the spatial dimensionality (here, 2/d=0.63)

➔ This relation supports the scaling argument.

More scientific examples: P07



Optics Hutch 1

1st Experimental Hutch / Test Facility
- calibration / detector tests
- diffraction

control cabins

Optics Hutch 2

2nd Experimental Hutch
- general purpose diffractometer
- focussing < 5 µm (CRLs & MLs)

control cabin

Optics Hutch 3

3rd Experimental Hutch
- heavy duty hexapod 1 t
- focussing < 10 µm (CRLs)

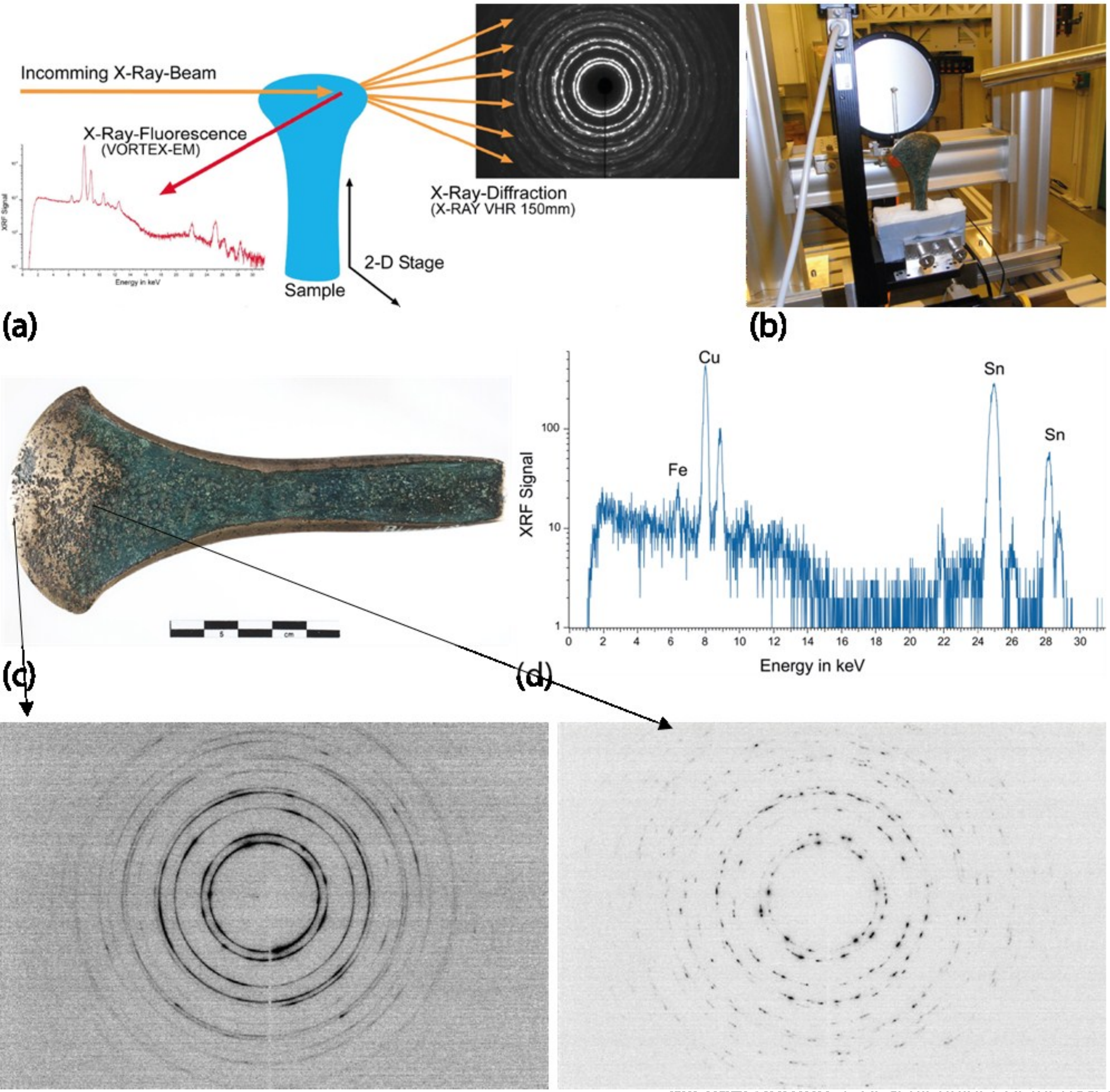
lock room
temperature

control cabin

4th Experimental Hutch
- HR 3D-XRD grain mapping
- HR fast micro-tomography
- focussing < 1 µm (CRLs)

First light December 2009

More scientific examples: P07



aim: how developed metallurgy in the bronze age

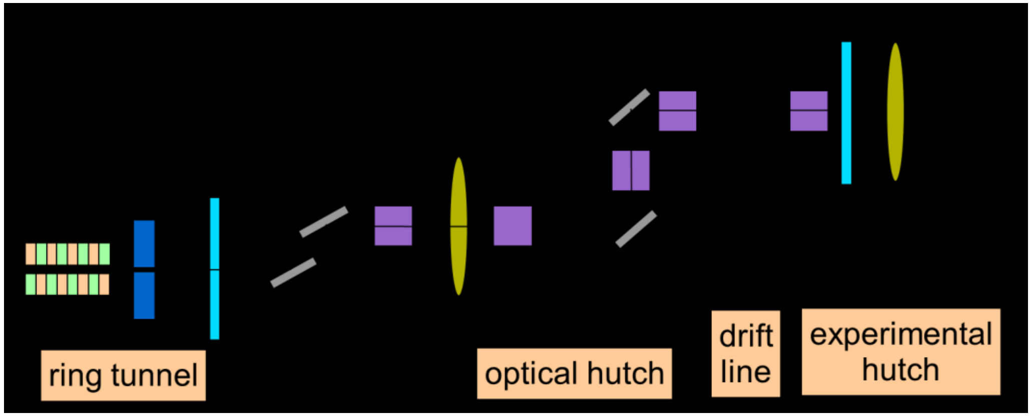
result: characterization of surface treatment

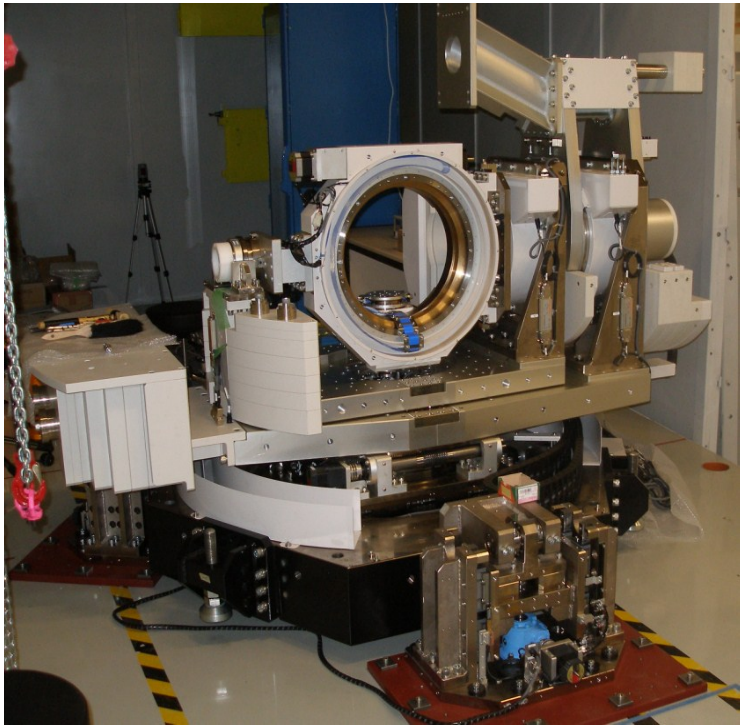
91 keV, CRL 10 x 10 µm², 600 x 600 µm², Photonic Science VHR 30 µm pixels, VORTEX EM Fl-detector

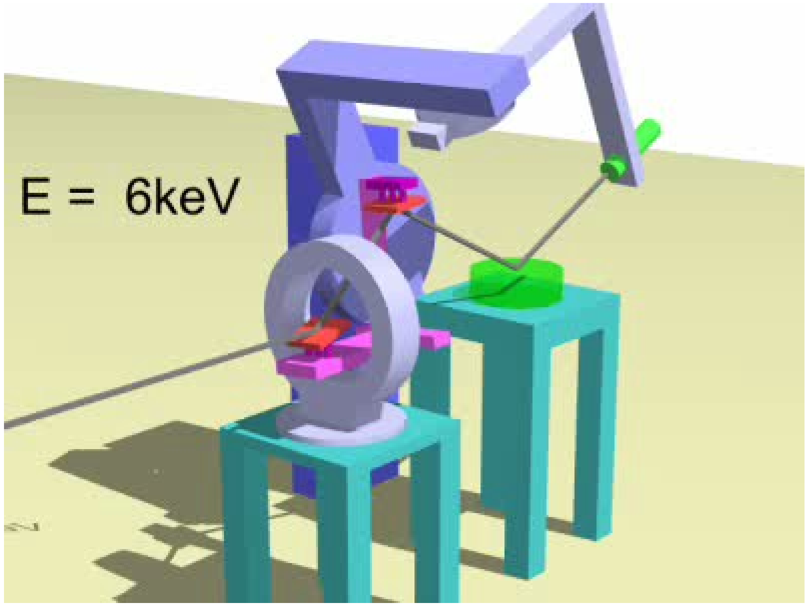
The Axe of Blunk (1600 BC) from the Archaeological State Museum Gottorf Castle, Schleswig.

Courtesy: L. Glaser, M. Freudenberg

More scientific examples: P08





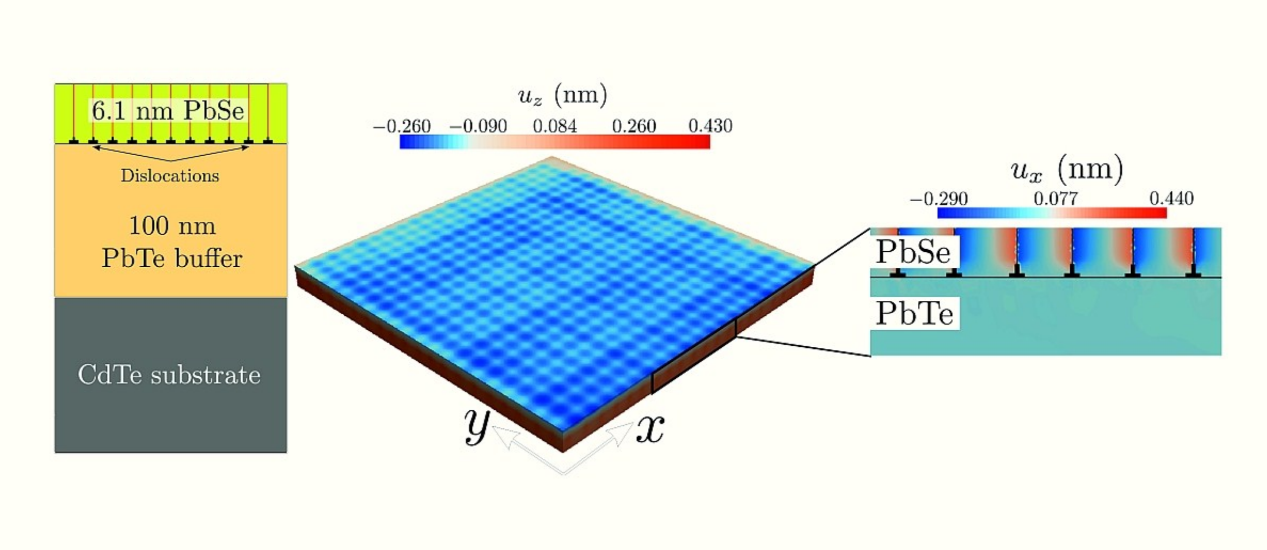


Two experimental setups:

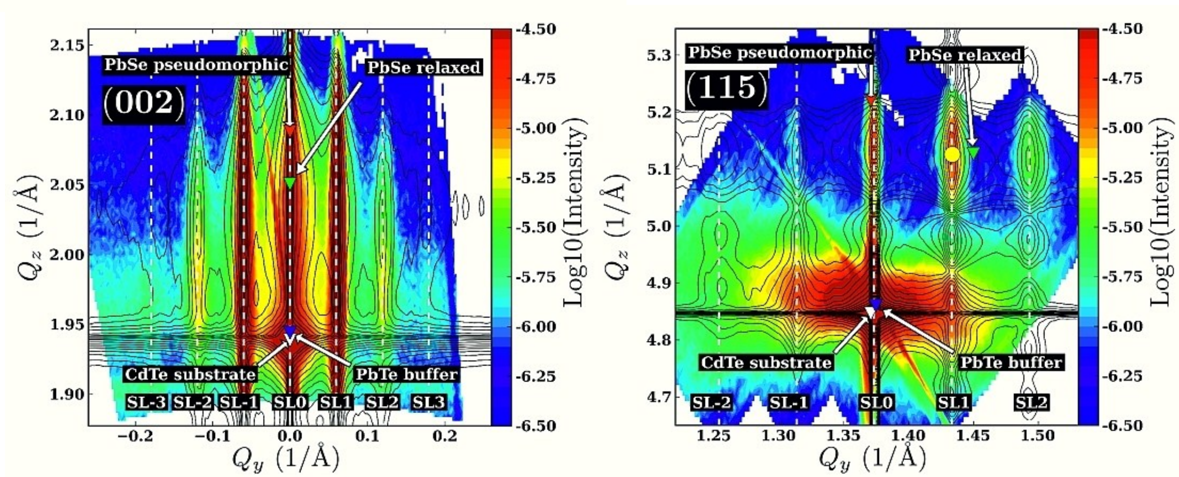
a) High resolution difraction (top)

b) Liquid surface scattering (left)

More scientific examples: P08



Periodic dislocations in thin PbSe films



Wintersberger et al. Appl. Phys. Lett. 96, 131905 (2010)

Zusammenfassung

- PETRA III bietet großartige wissenschaftliche Möglichkeiten an 14 spezialierten Undulator Beamlines!
- Alle diese Beamlines benötigen eine ‘perfekt’ laufende Maschine um ihr volles Potential auszuschöpfen!
- P10 fördert die Entwicklung und Ausnutzung kohärenter Streumethoden
- Kohärente Streumethoden sind eine neue Möglichkeit um interessante wissenschaftliche Fragestellungen anzugehen

Acknowledgements

- All members of the Coherence Beamline
- HASYLAB Coherent Scattering Group
- PETRA III project team
- HASYLAB optics group

Thank you for your attention!

More scientific examples: P09

Resonant Scattering and Diffraction Beamline P09

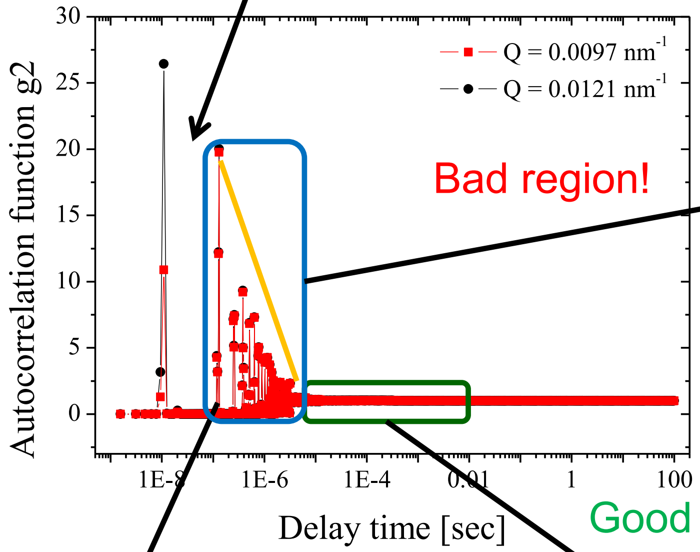
- Energy range: 2.7 – 24 keV
- Variable incident polarization and analysis of scattered polarization (rotated linear or circular)

- 8-circle (Psi) diffractometer for resonant scattering and diffraction experiments
- Horizontal or vertical scattering geometry

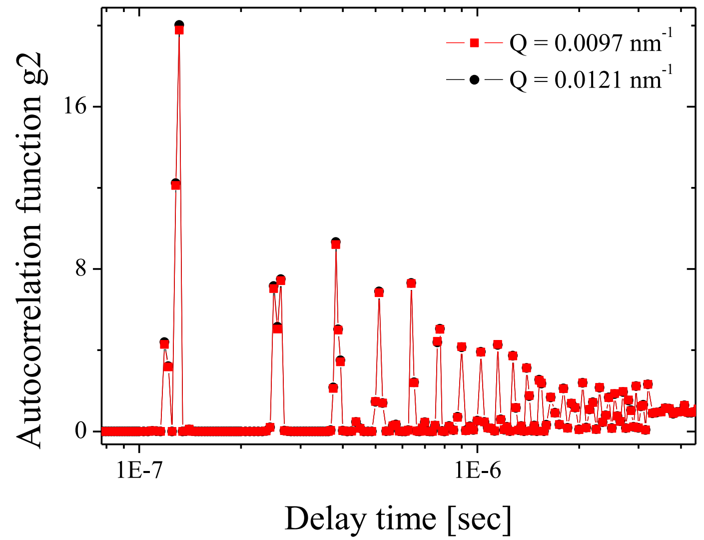
- Low temperatures down to 1.7 K
- Point detectors and area detector used quasi simultaneously

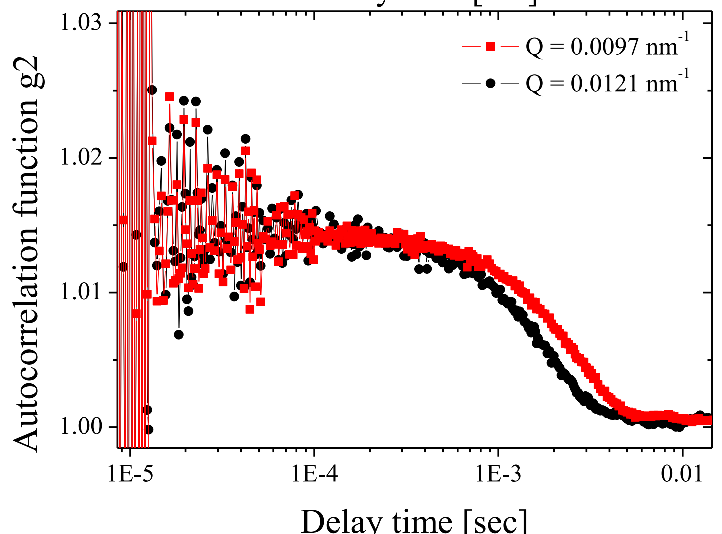
Why different bunch modes?

Interesting region for P01 & P04 experiments



Good region for P10





Peak at 1.29E-7 sec

S = v/t

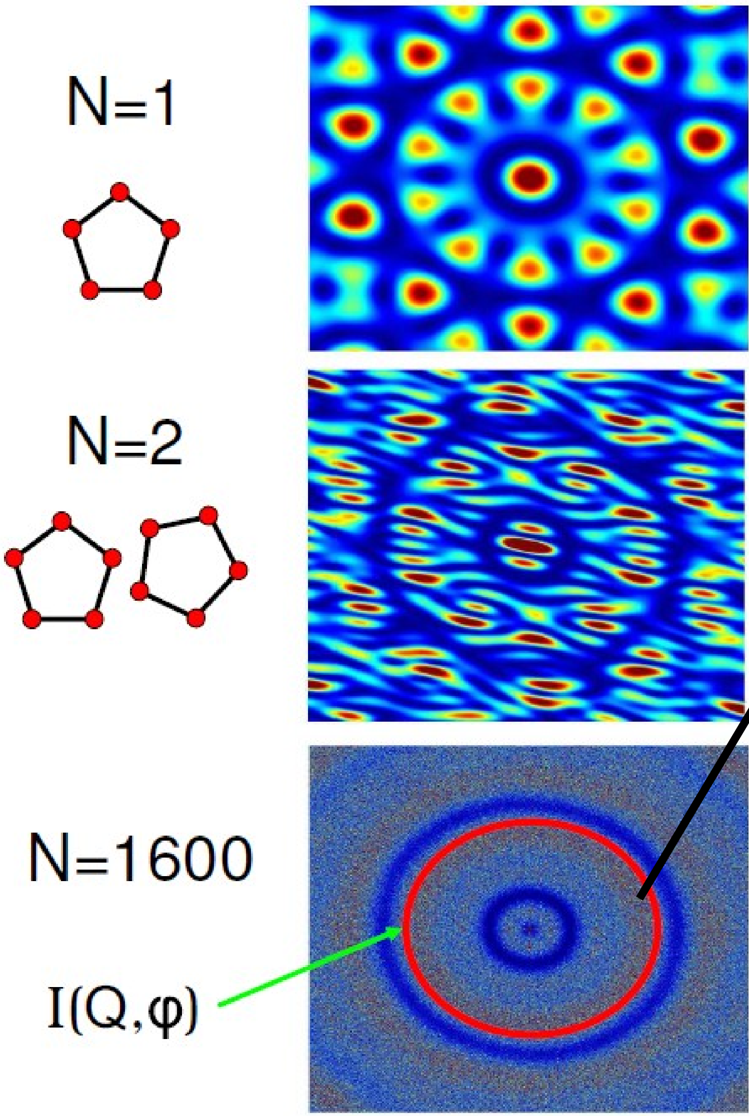
v = c

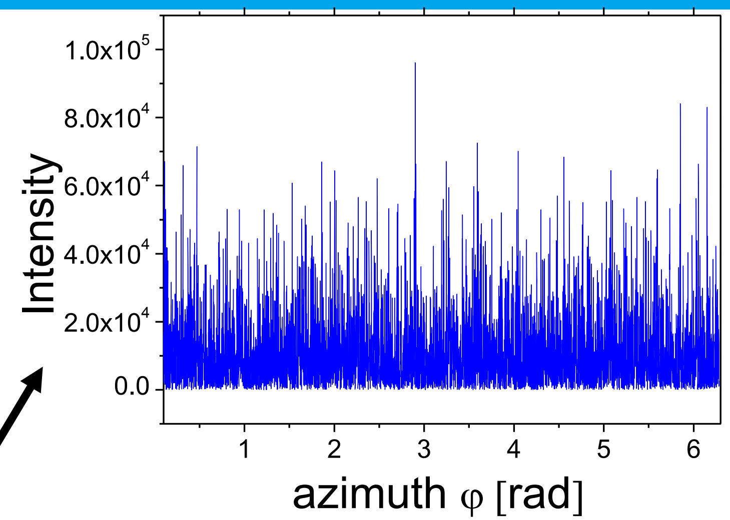
= 3.0E8 m/sec x 1.29E-7 sec

= 38.7 m

2304 m / 38.7 m = **59.7 Bunches**

Simulations of Simple Model Disordered Structures





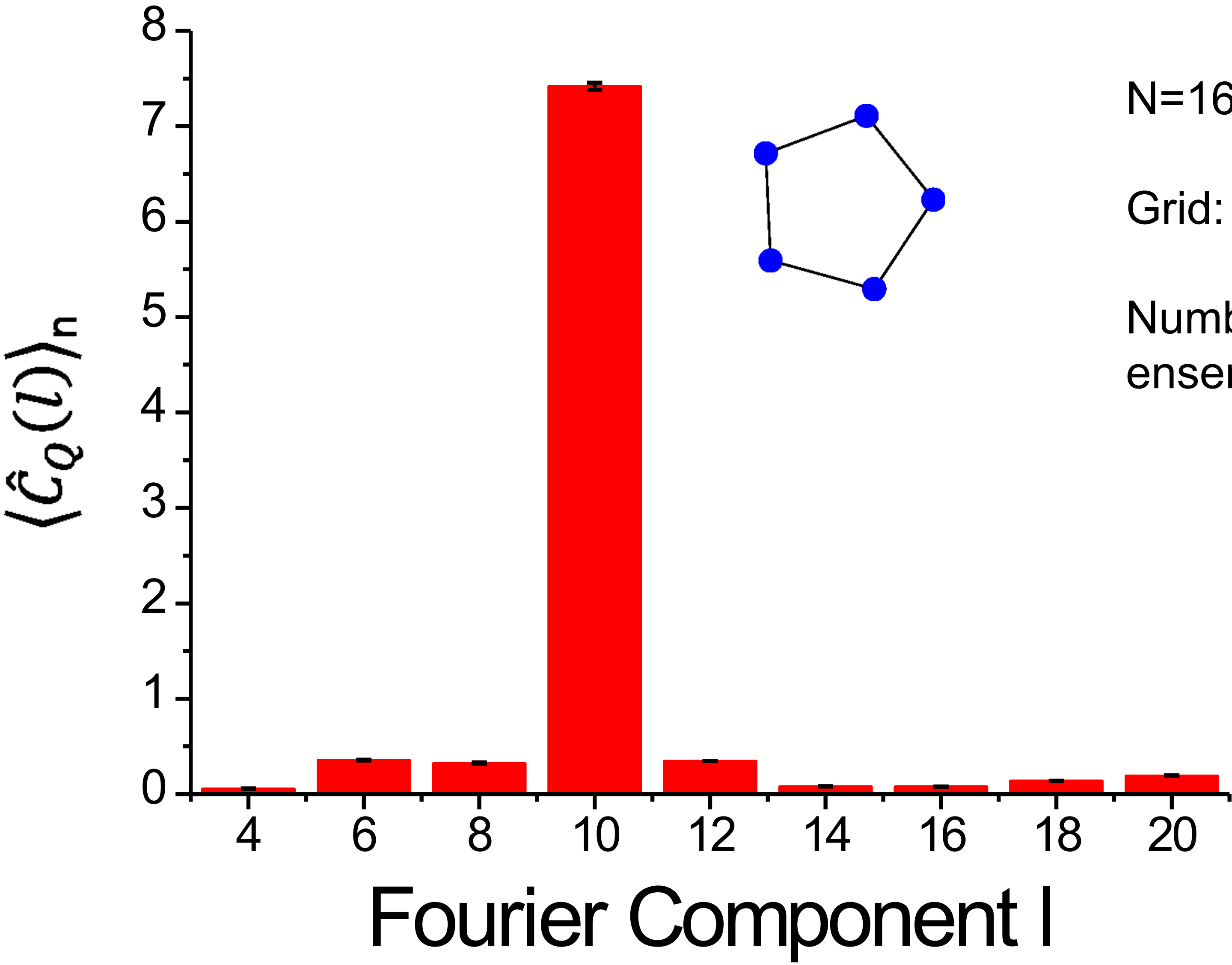
Fourier transform (FT) of $C_Q(\varphi)$

$$\hat{C}_Q(l)$$

average over *n* different ensembles

$$\langle \hat{C}_Q(l) \rangle_n$$

Simulations of Simple Model Disordered Structures

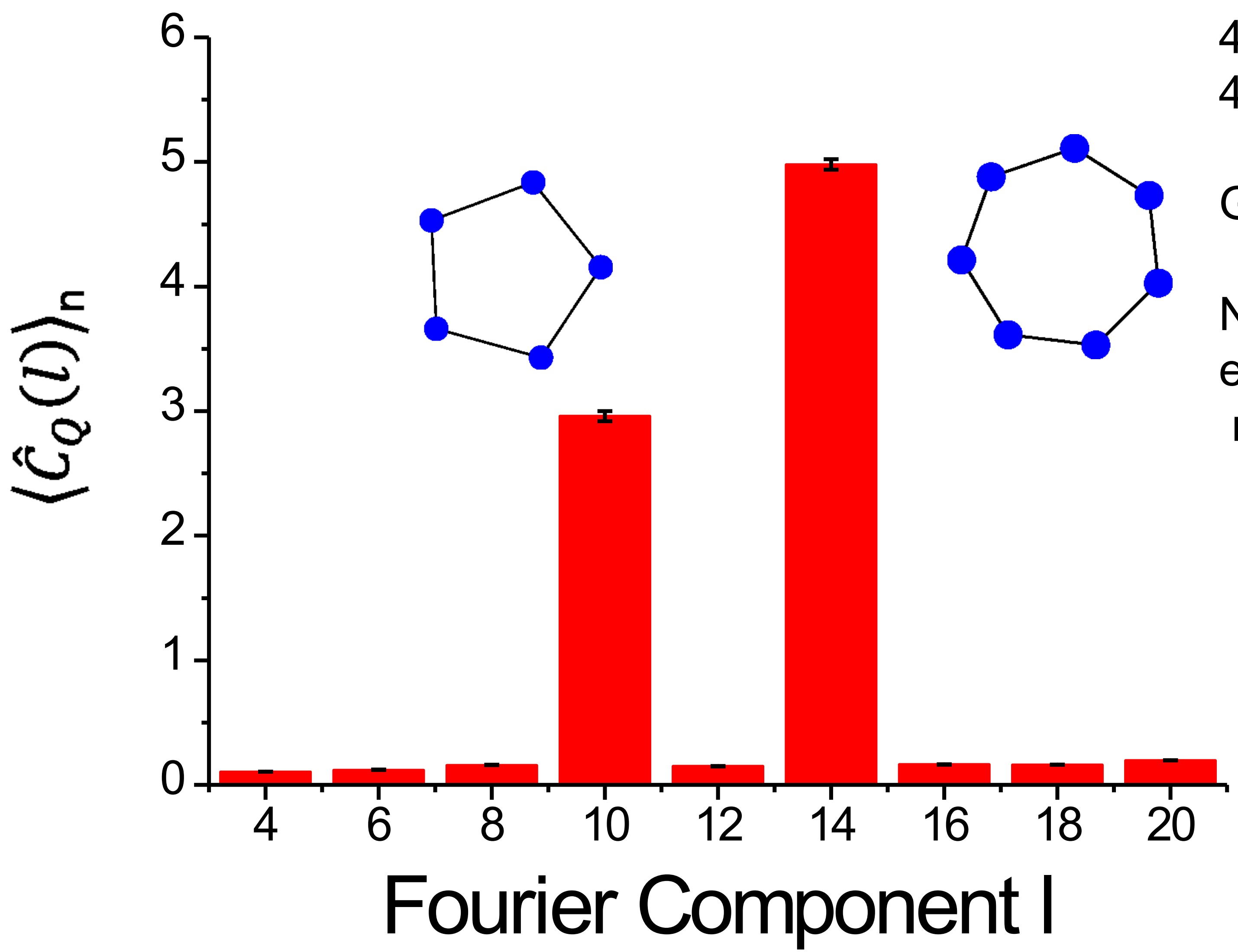


N=1600 Pentagons

Grid: 3000x3000

Number of different ensembles: n= 1000

Simulations of Simple Model Disordered Structures



400 Pentagons
400 Heptagons

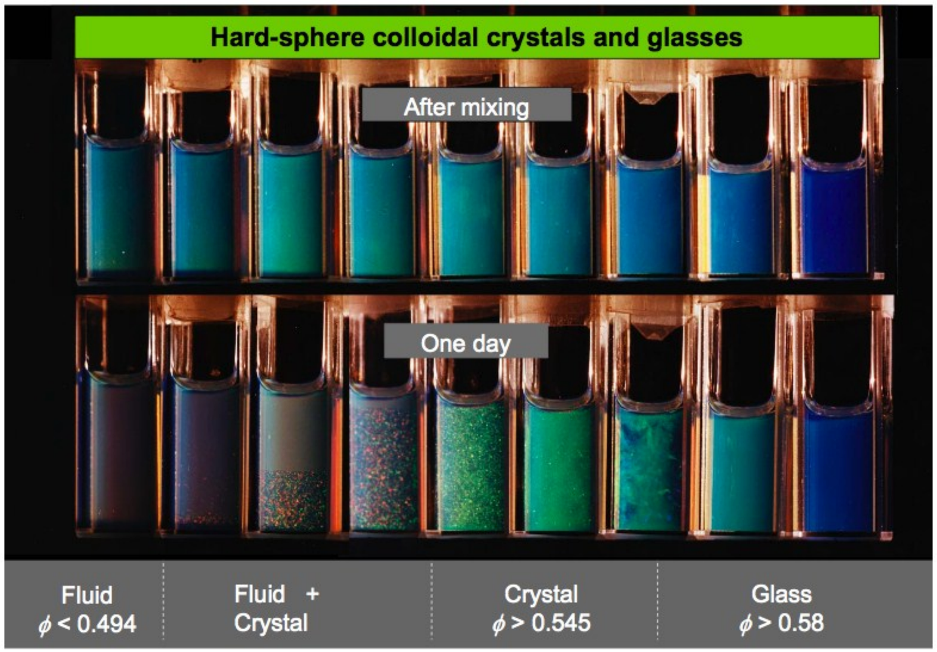
Grid: 3000x3000

Number of different ensembles:
n= 1000

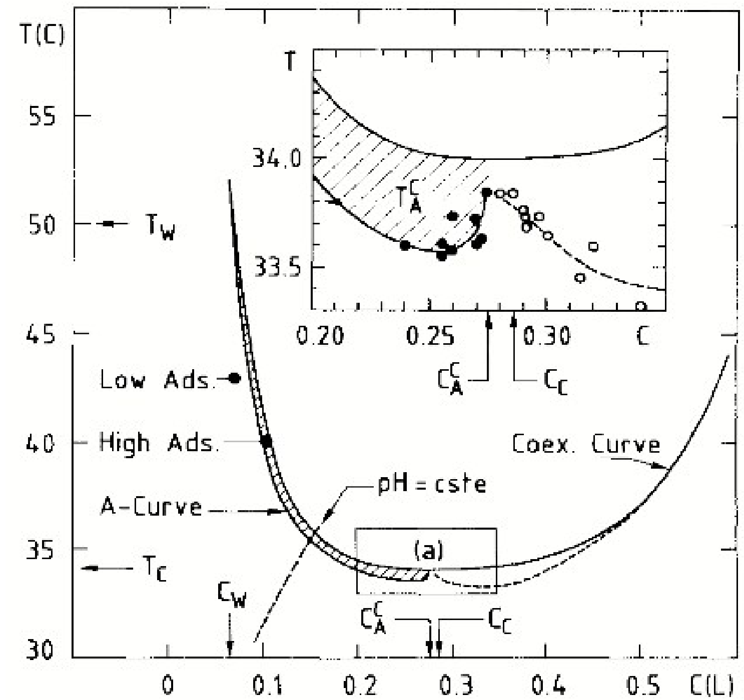
Coherent X-Rays: Scientific opportunities

1) How a liquid becomes a glass both on cooling and on heating!

Phase transitions in colloidal suspensions



Silica spheres in water-lutidine



D. Beysens & D. Esteve, Phys. Rev. Lett. 54 (1985) 2123.
V. Gurfein, D. Beysens & F. Perrot, Phys. Rev. A 40 (1989) 2543.
D. Pontoni, T. Narayanan, J-M. Petit, G. Grubel, and D. Beysens, PRL 90, 188301 (2003).

Coherent Scattering: Scientific opportunities

1) How a liquid becomes a glass both on cooling and on heating!

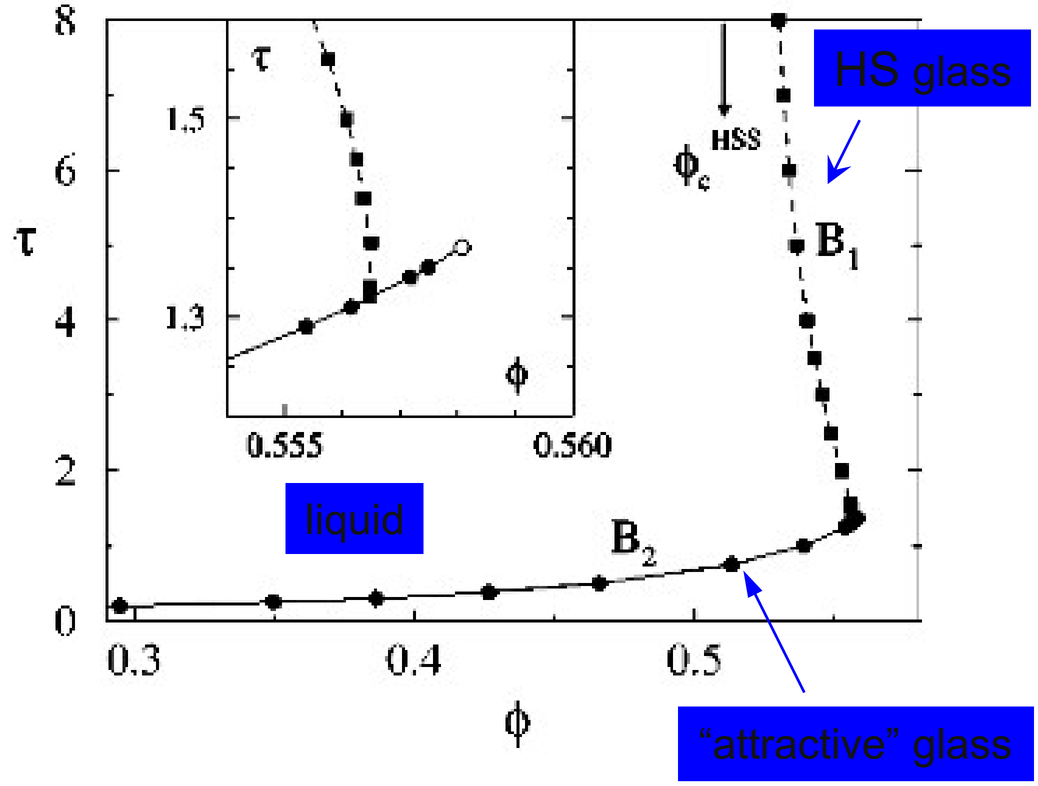
Predictions of MCT

• A colloidal glass with hard-sphere (HS) repulsions may be melted by switching on a short-ranged attractive interaction.

• Such a melted glass may be re-vitrified upon further increase in the attraction.

• There can exist a sharp transition between a HS glass and an “attractive” glass, accompanied by a sudden change in elastic properties.

• Density fluctuations decay logarithmically versus time, in the liquid where attractive and repulsive arrest mechanisms compete.

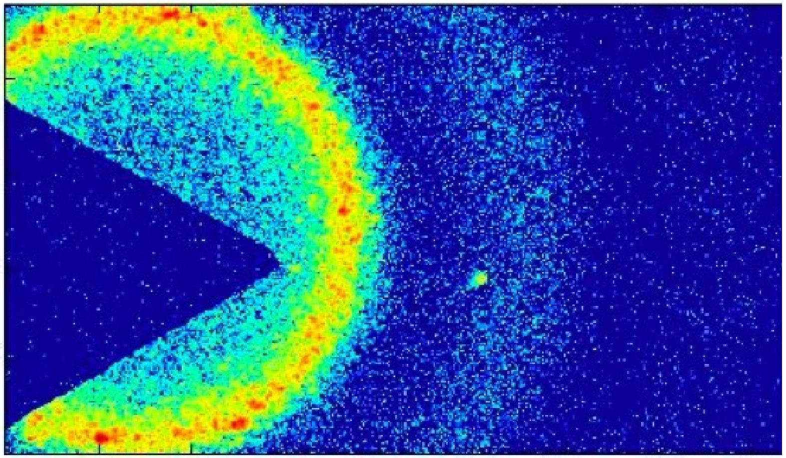


Mode coupling theory phase diagram for sticky hard spheres plotted vs. stickiness (τ) and volume fraction (ϕ) From L Fabbian, W Götze F Sciortino, P Tartaglia, F Thierry, Phys. Rev. E 59, R1347 (1999).

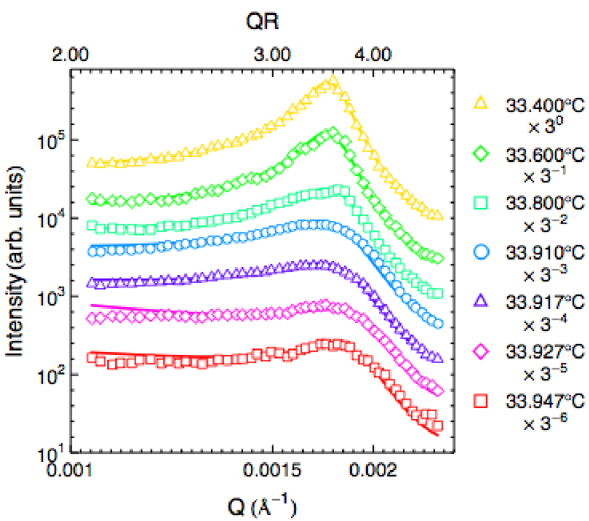
Coherent Scattering: Scientific opportunities

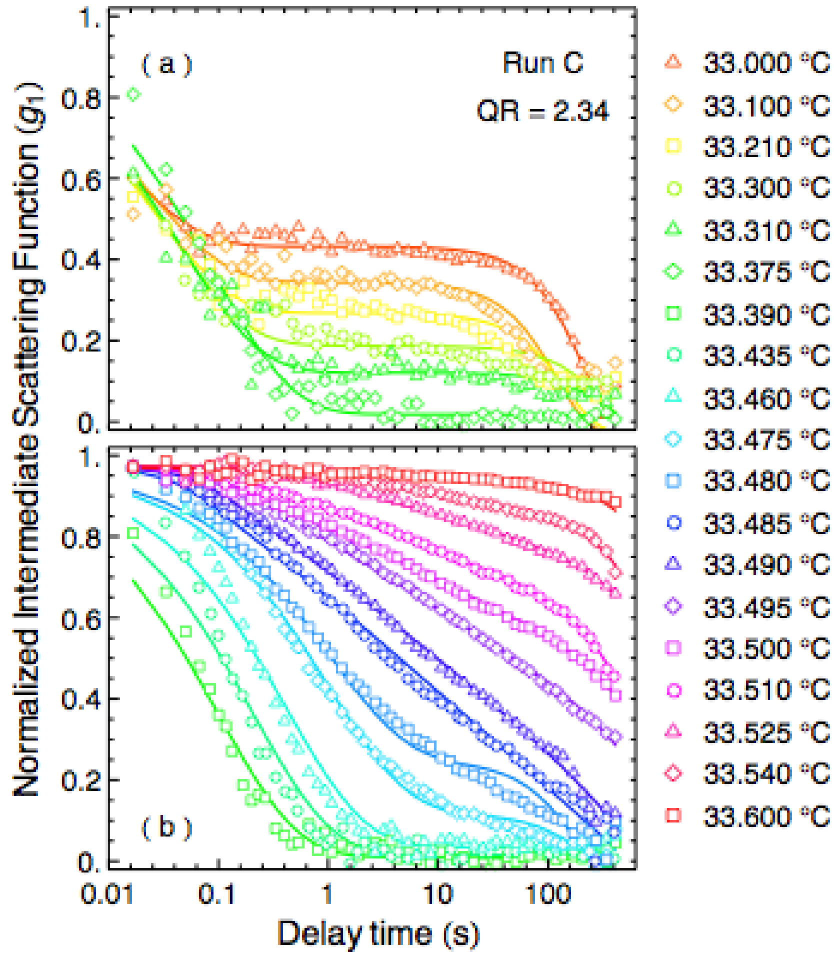
1) How a liquid becomes a glass both on cooling and on heating!

Coherent small-angle x-ray scattering (Coherent SAXS)



150 ms exposure -- 200 nm radius silica spheres





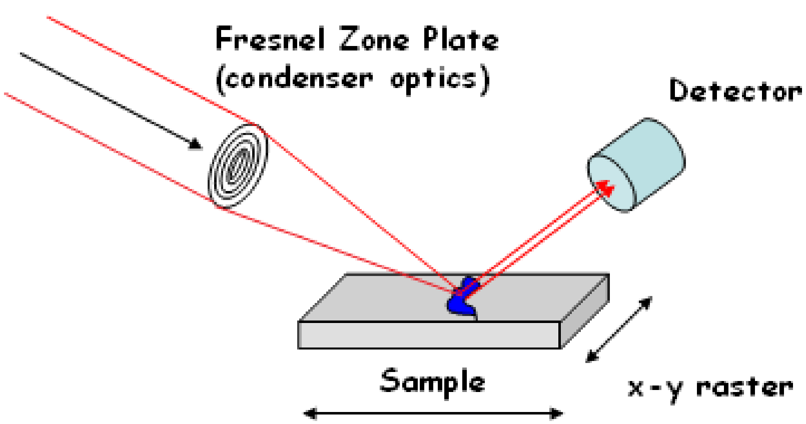
Xinhui Lu, S. G. J. Mochrie, S. Narayanan, A. Sandy, M. Sprung, PRL 100, 045701 (2008)

Coherent Scattering: Scientific opportunities

2) Direct measurement of antiferromagnetic domain fluctuations

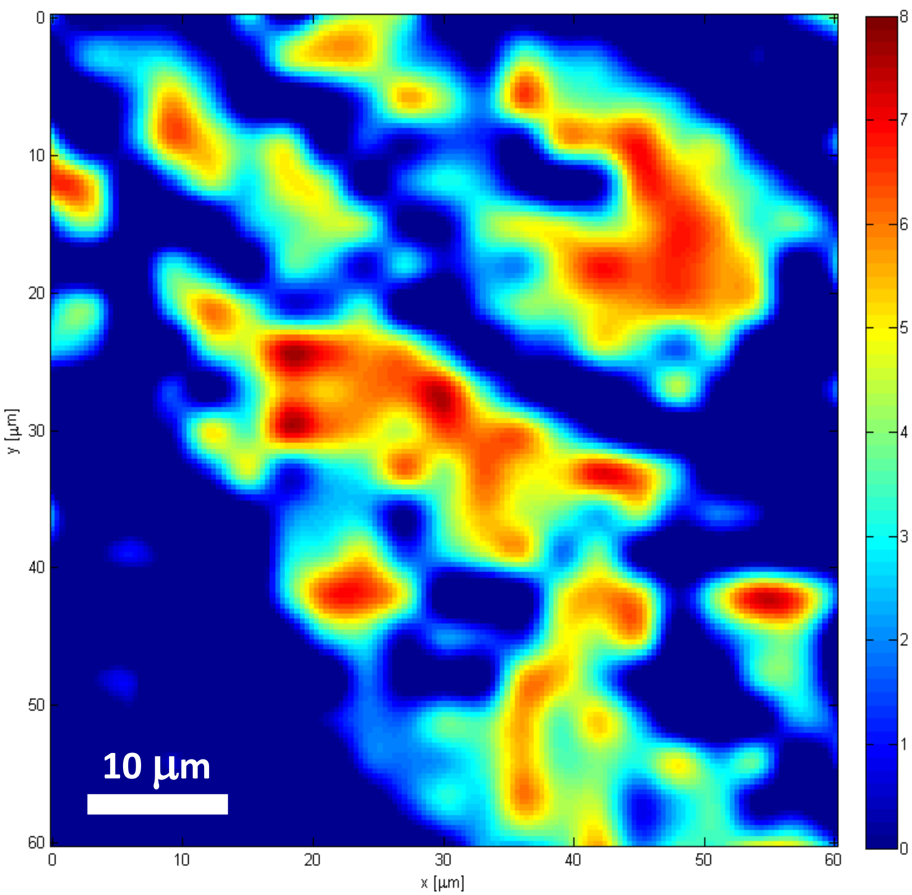
Microscopic Magnetic Domains in Chromium:

Scanning X-ray Microscopy:



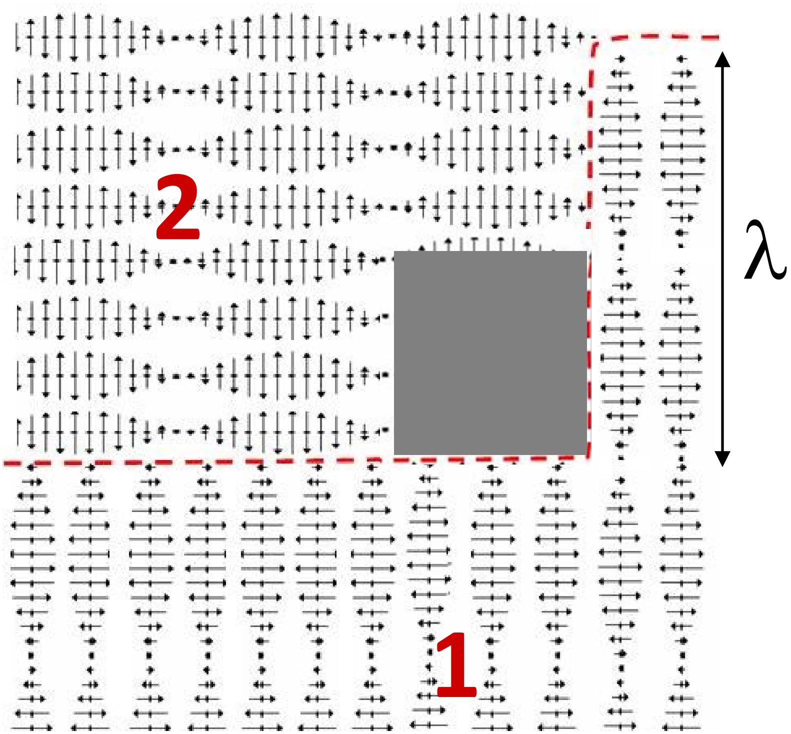
- bulk probe (micron-sized penetration depth)
- spin, charge, lattice and chemical sensitivity

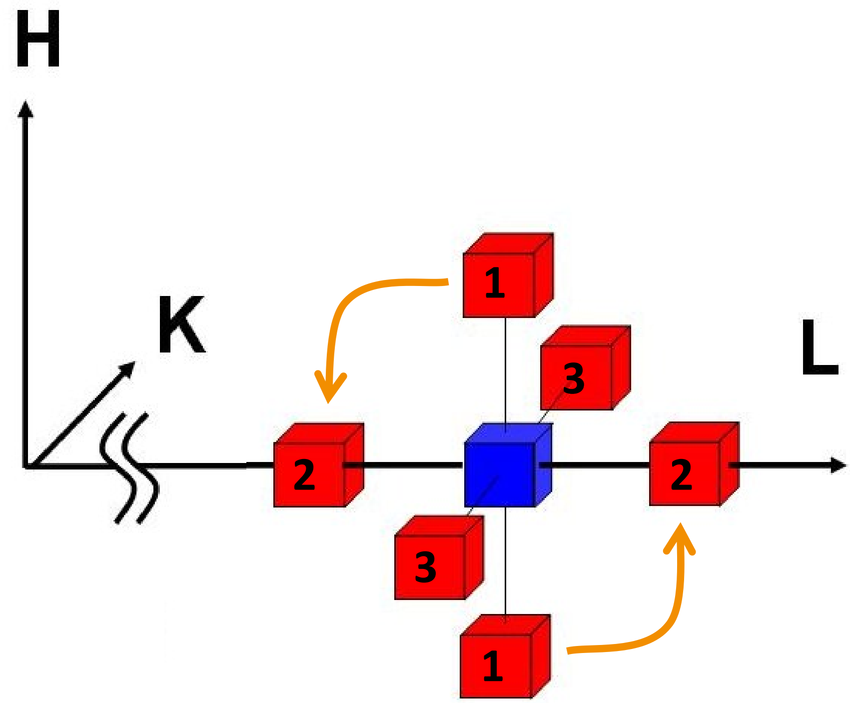
[0, 0, 2-2δ] Charge-density wave satellite



Coherent Scattering: Scientific opportunities

2) Direct measurement of antiferromagnetic domain fluctuations





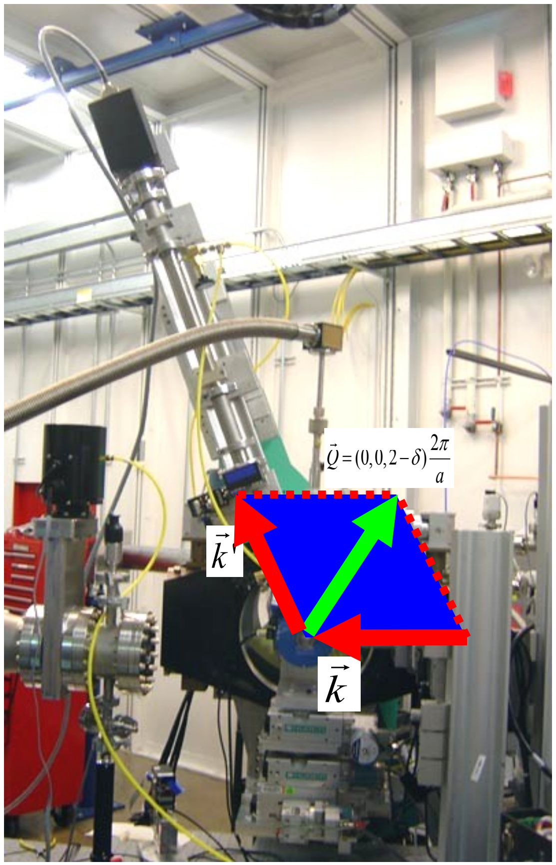
Real Space:
elemental switching block
(V=(λ/2)³, λ=3-4 nm)

Momentum Space:
transfer of intensity from satellites 1 to 2 due to switch

Coherent Scattering: Scientific opportunities

2) Direct measurement of antiferromagnetic domain fluctuations

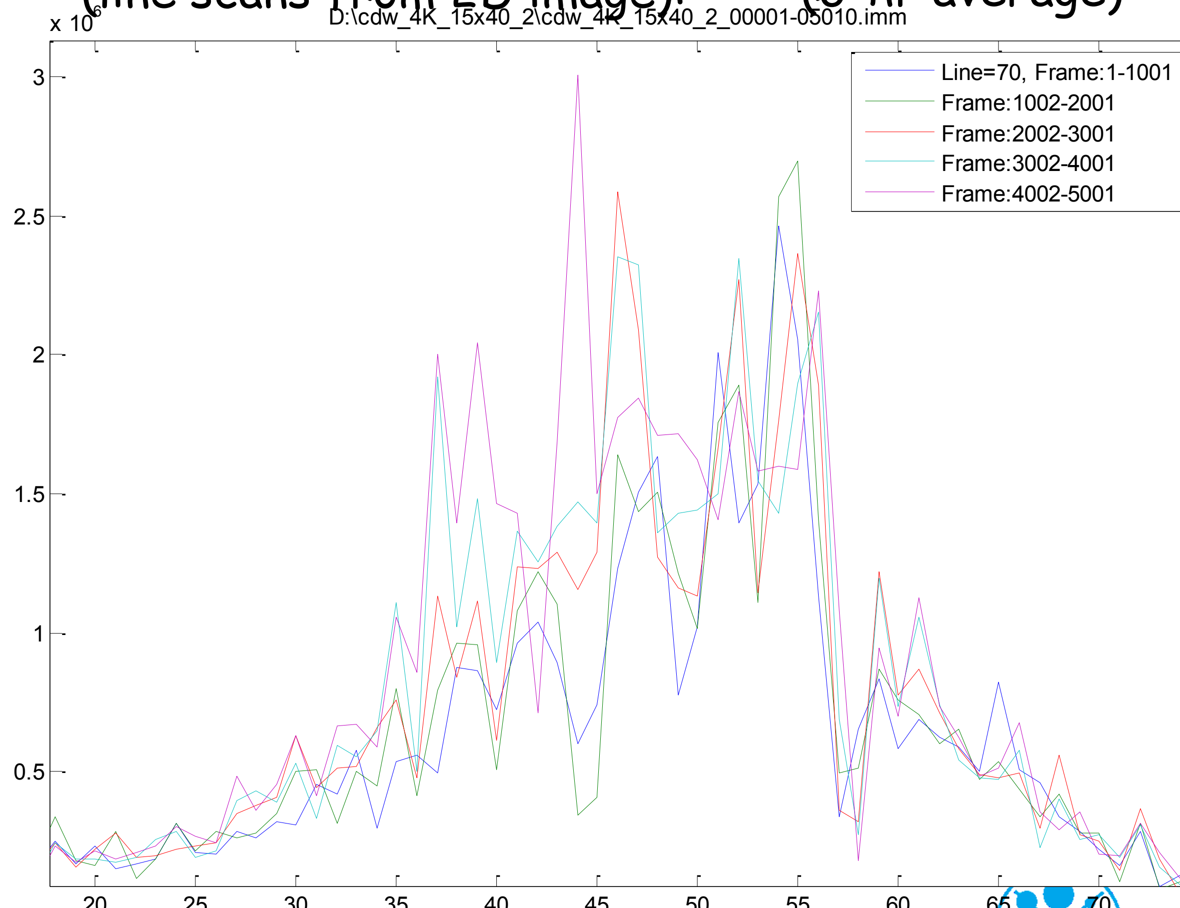
Experimental setup @ 8ID-G



Quasi-static Bragg speckle (line scans from 2D image):

Charge Density Wave (at 4K; 6 hours total)

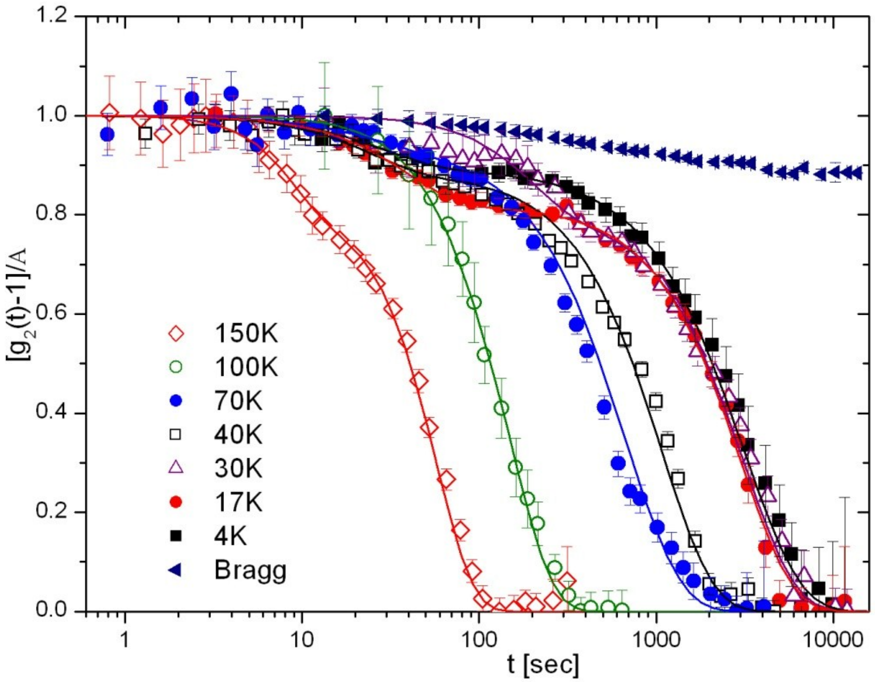
X-ray Bragg speckle (6-hr average)



Coherent Scattering: Scientific opportunities

2) Direct measurement of antiferromagnetic domain fluctuations

XPCS data



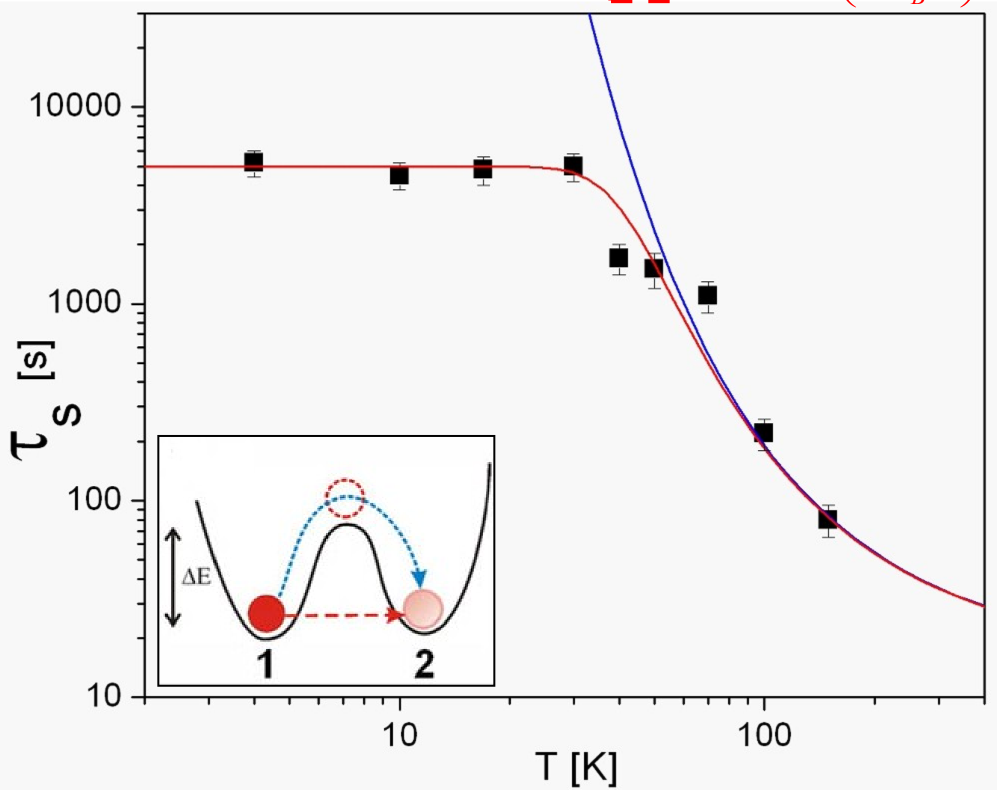
O. G. Shpyrko et al., *Nature* **447**, 68 (2007)

Classical Arrhenius model

$$\tau_S^{-1}(T) = \tau_R^{-1} \exp\left(-\frac{\Delta E}{k_B T}\right)$$

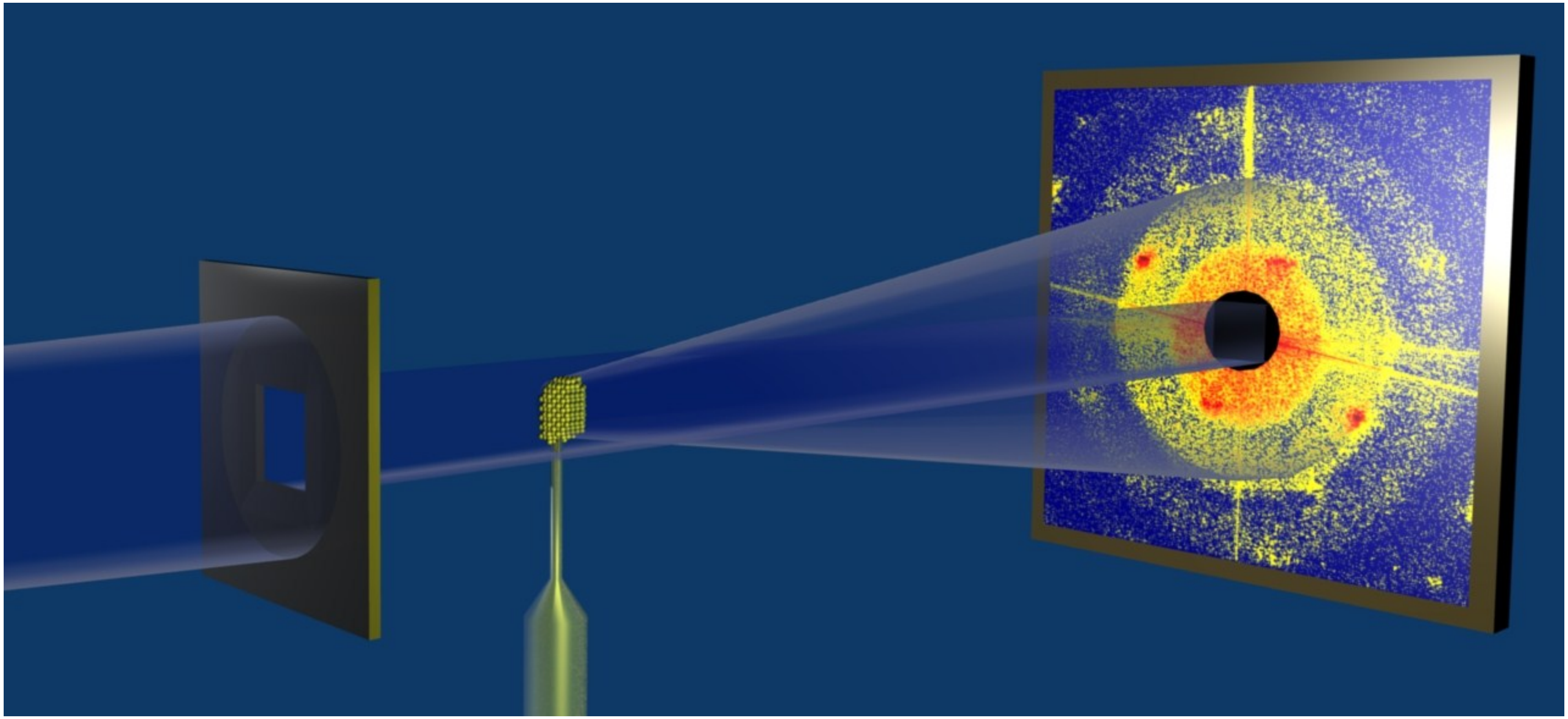
Quantum Tunneling model:

$$\tau_S^{-1}(T) = \tau_{QM}^{-1} + \tau_R^{-1} \exp\left(-\frac{\Delta E}{k_B T}\right)$$



Michael Sprung

Real space images of colloidal crystals



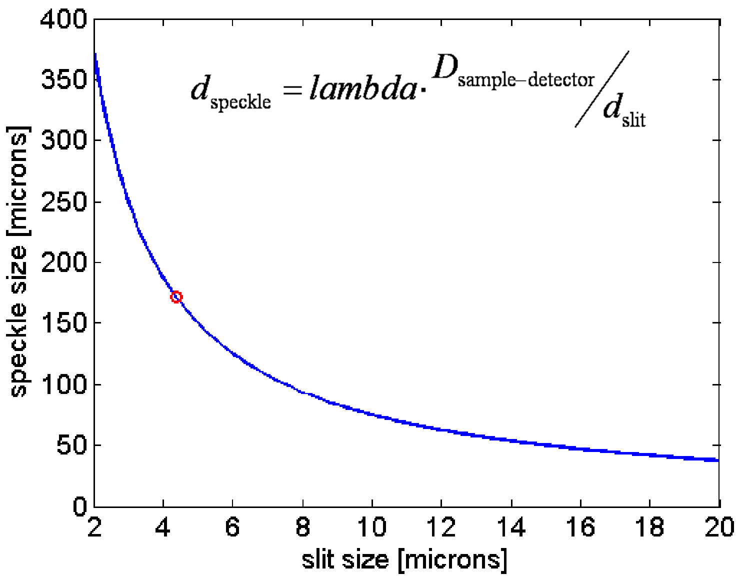
Group of I. Vartaniants/DESY
Experiment led by J. Gulden

Goal of the experiment is to get a full 3D image of colloidal crystals

Energy: 7.9 keV
Samples: colloidal crystals below 10 µm size
Flux: ~10^8 on 10×10 µm^2 spot
Sample detector distance: 5 m
Detector: 20×20 µm^2 pixel size, 1340×1300 pixels
Maximum resolution: 60 nm

Optimizing the combination Pilatus–Standard Setup

Pilatus pixel size is very large!
→ beam size has to be small
to produce large speckles!



→The beam size should be ~4µm for a sample—detector distance of 5.0m
→ Focusing is needed to use the full coherent flux !
→ The loss would be 1/3750!!!

The vertical transversal coherence length is 6x larger than the horizontal one at P10!
→ prefocusing only the vertical direction
→ match vertical and horizontal coherence length at the position of the secondary lens!