Science at the MID instrument

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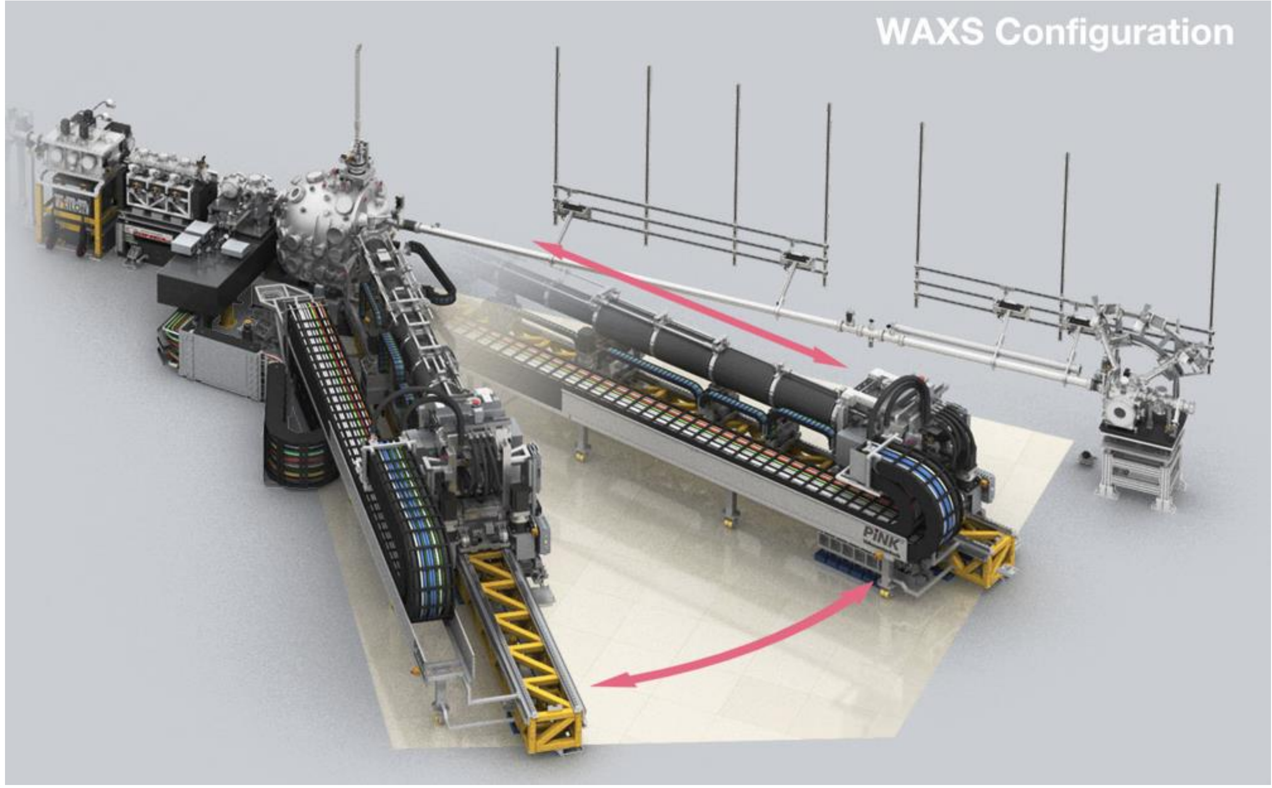
Instrument Scientist at MID

Hamburg, 23.01.2024

Outline

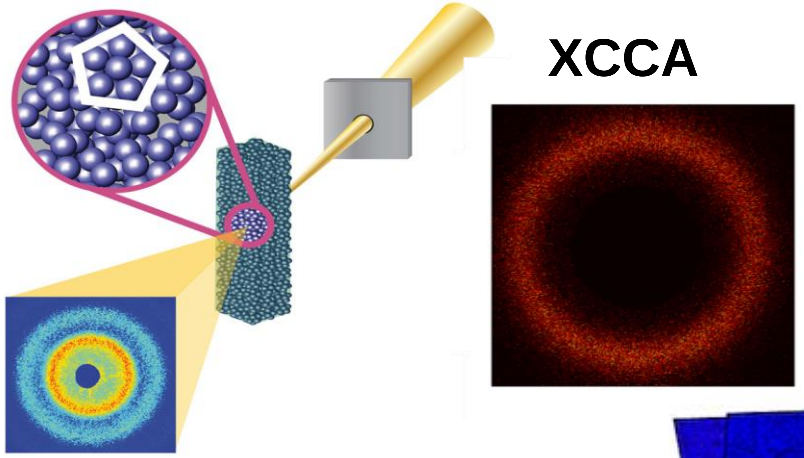
- Introduction to the MID instrument
- X-ray beam parameters and their relevance
  - 4.5MHz operation
  - Hard X-ray self seeding
- Typical measurement techniques and examples for experiments at MID
  - XPCS
  - Imaging (holography)
  - Ultrafast diffraction
  - Mössbauer experiments



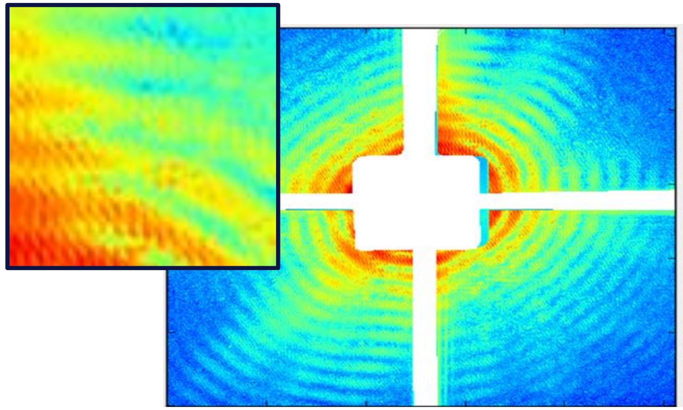
The MID instrument: it all started in 2009…

The Materials Imaging and Dynamics (MID) station aims at the investigation of nanosized **structure** and nanoscale **dynamics** using **coherent hard X-rays**. Applications to a **wide range of materials** from hard to soft condensed matter and biological structures are envisaged

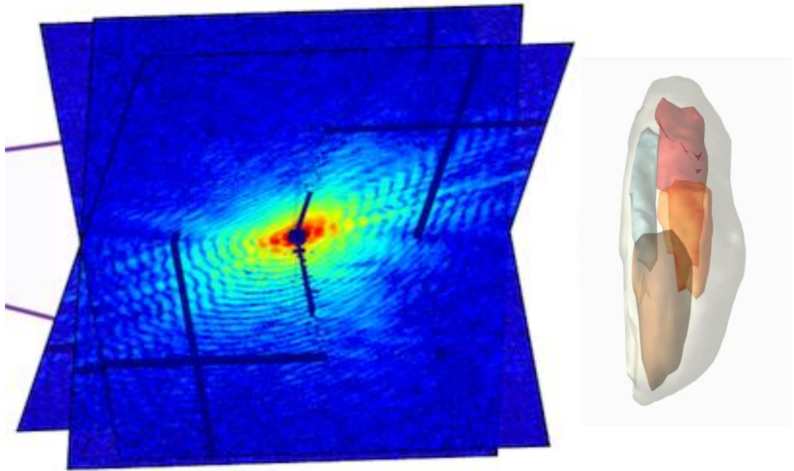
(1st MID workshop, Oct 2009 @ ESRF, Grenoble)



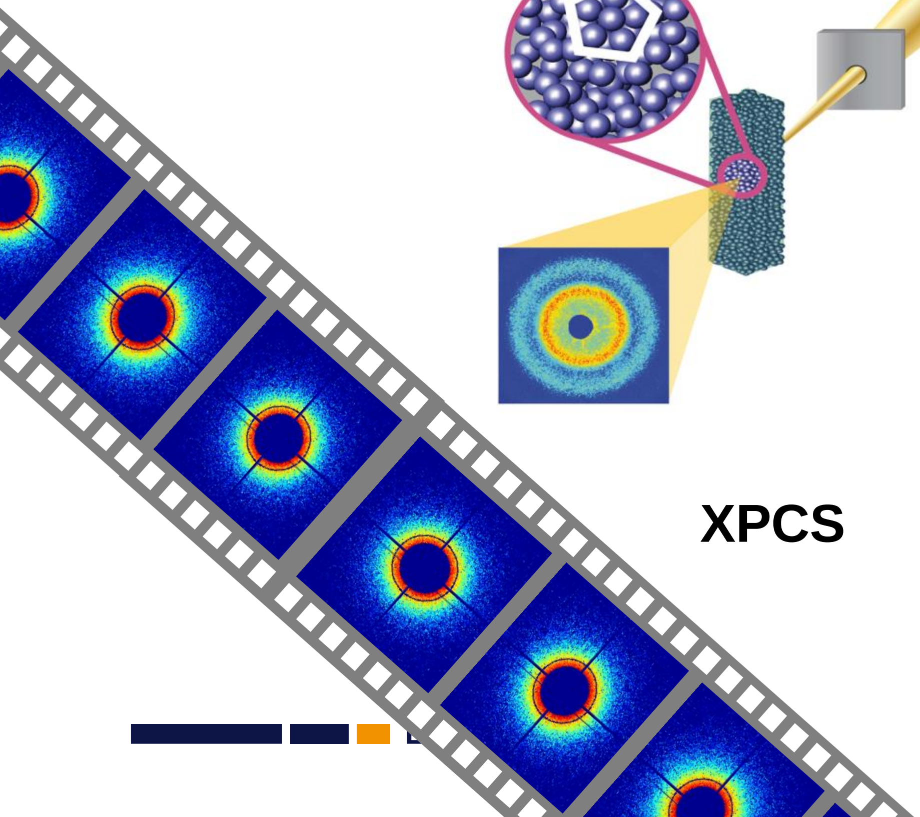
XCCA



Holography



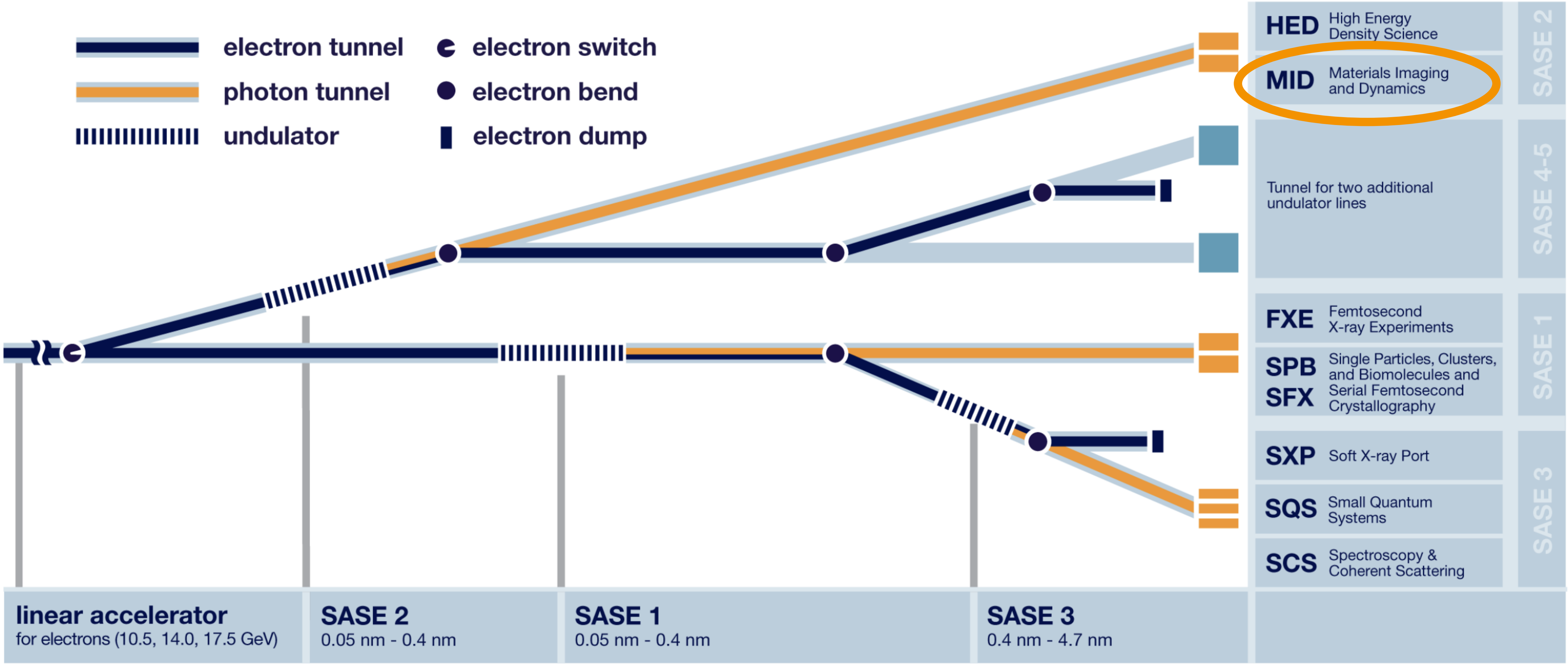
CDI



XPCS

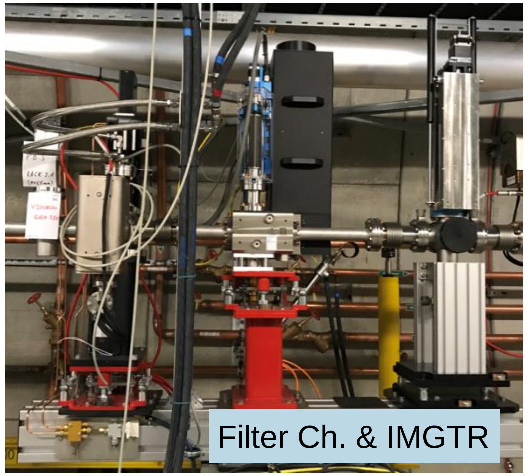
in combination with:
- External pump
- Spectroscopy
- Nano-focusing
- Split-delay techniques

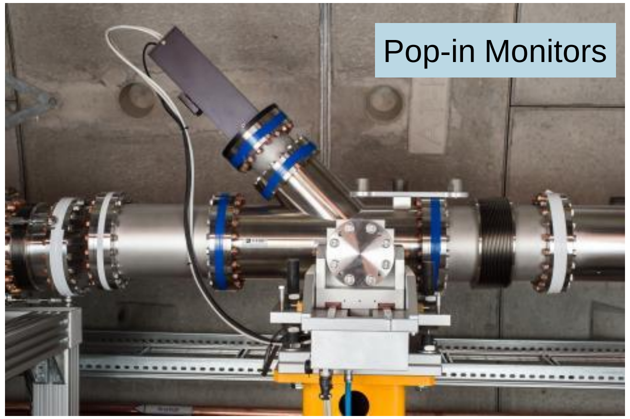
Beamline layout and experiment stations

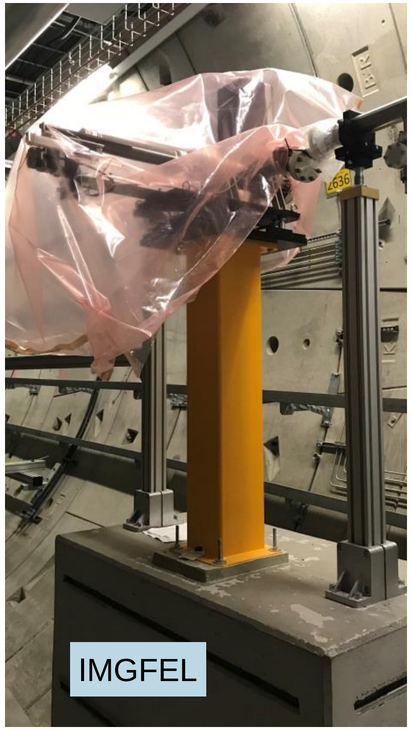


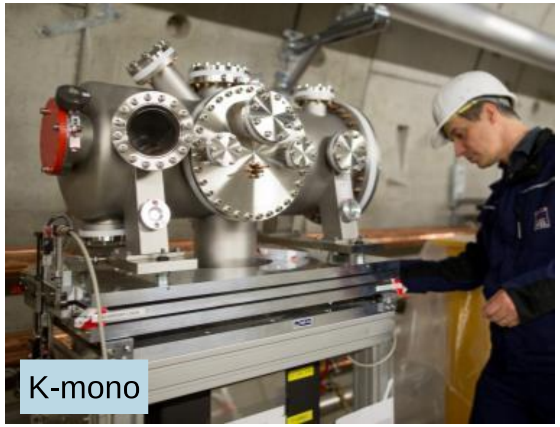
Tunnel diagnostics

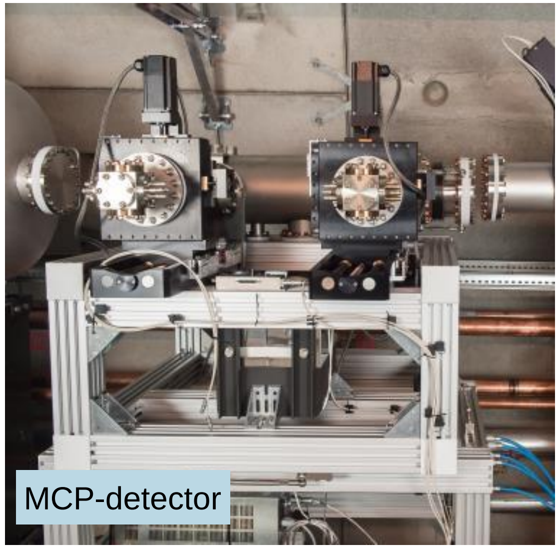
- all SASE2 diagnostics vacuum systems are in the tunnel





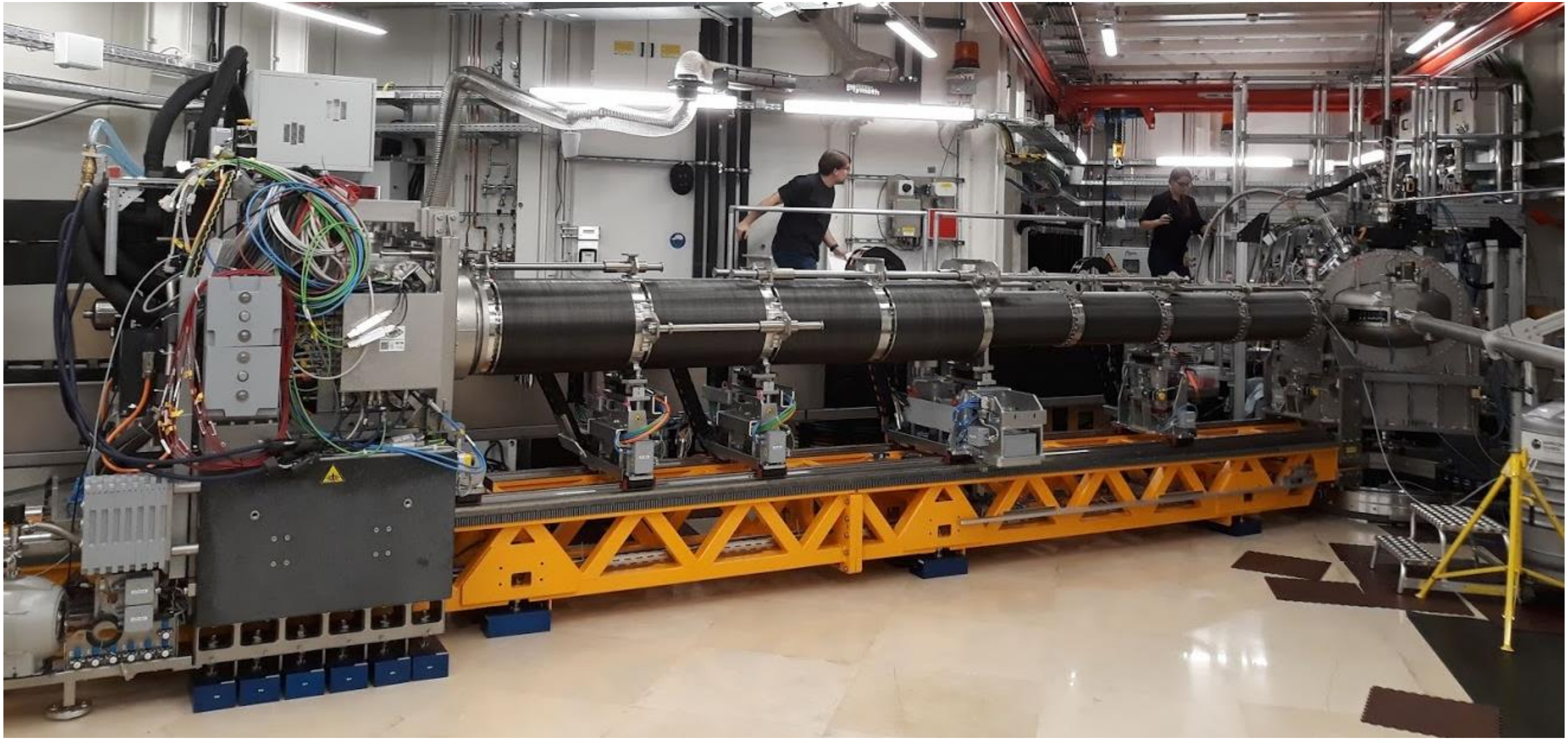






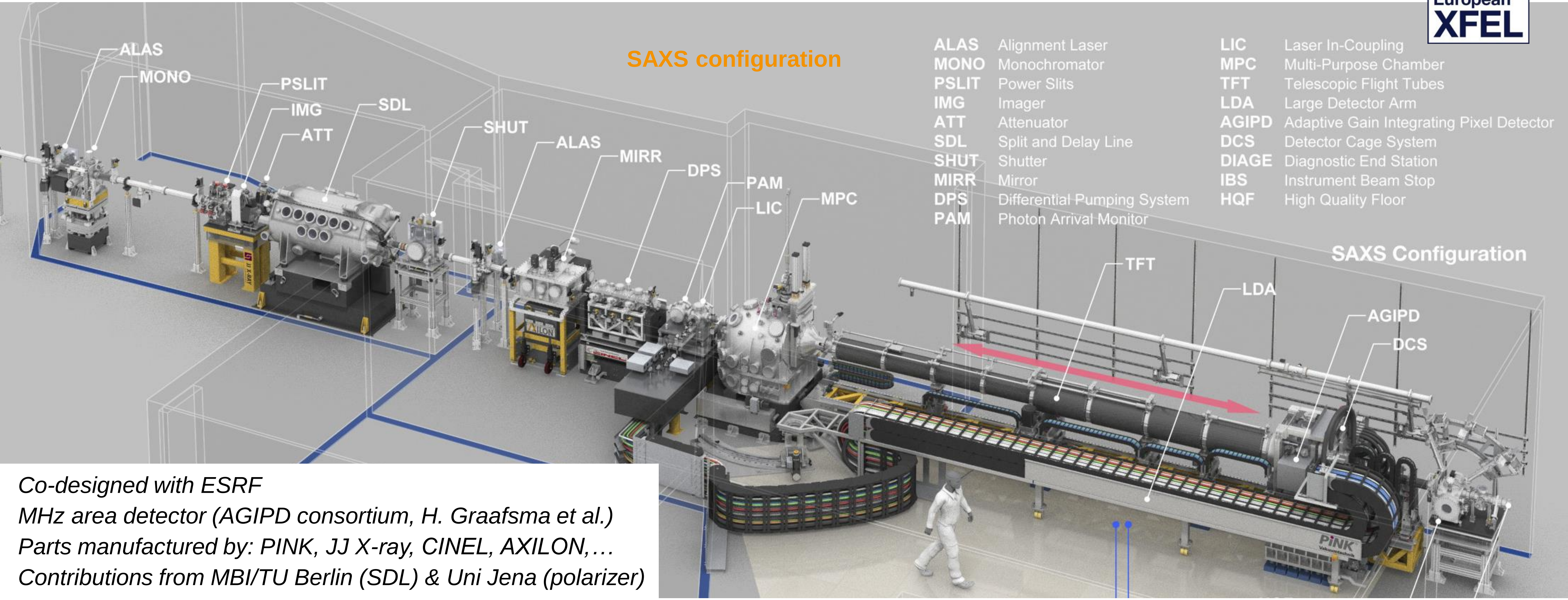
Courtesy of Jan Grünert and the photon diagnostics group

MID: Materials Imaging and Dynamics Instrument



Madsen et al. JSR **28**, 637-649 (2021)

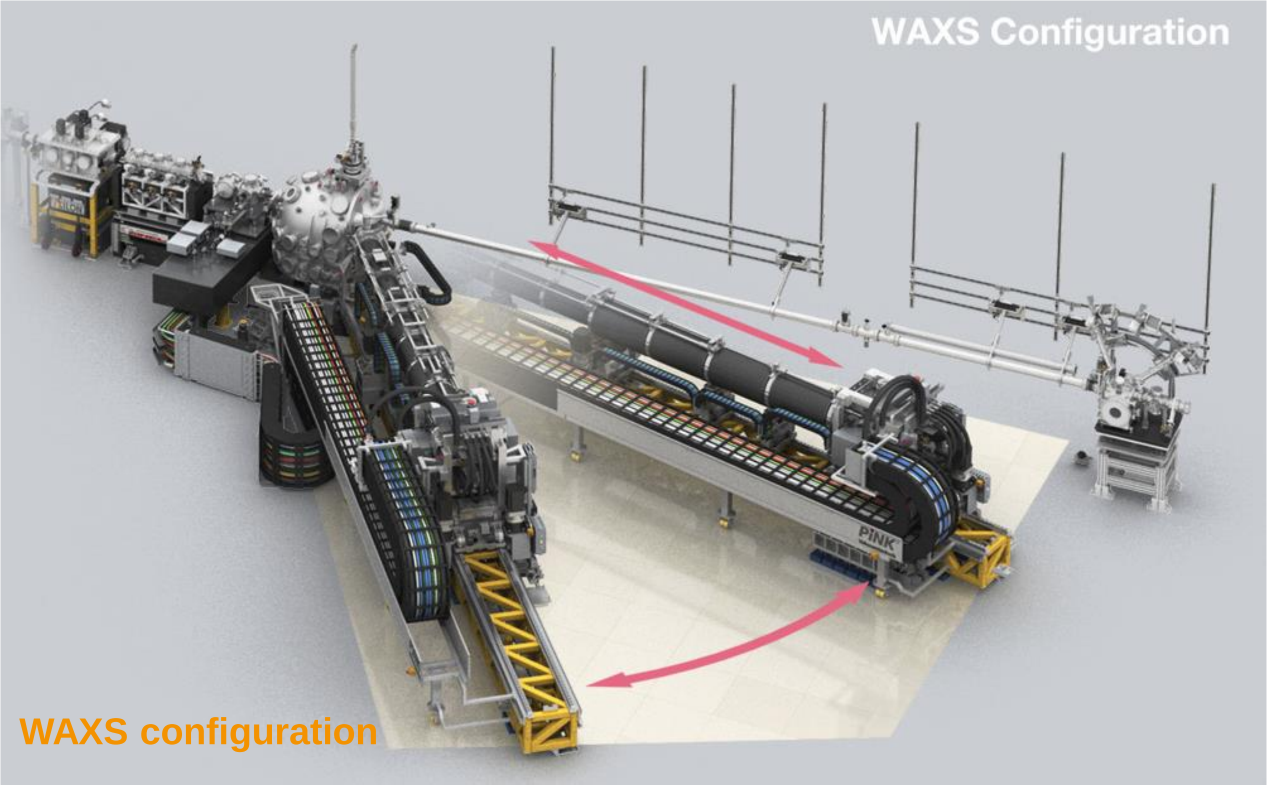
Materials Imaging and Dynamics (MID) experiment



Co-designed with ESRF
MHz area detector (AGIPD consortium, H. Graafsma et al.)
Parts manufactured by: PINK, JJ X-ray, CINEL, AXILON,…
Contributions from MBI/TU Berlin (SDL) & Uni Jena (polarizer)

*Madsen et al. JSR **28**, 637-649 (2021)*

MID overview



Materials Imaging and Dynamics (MID) instrument

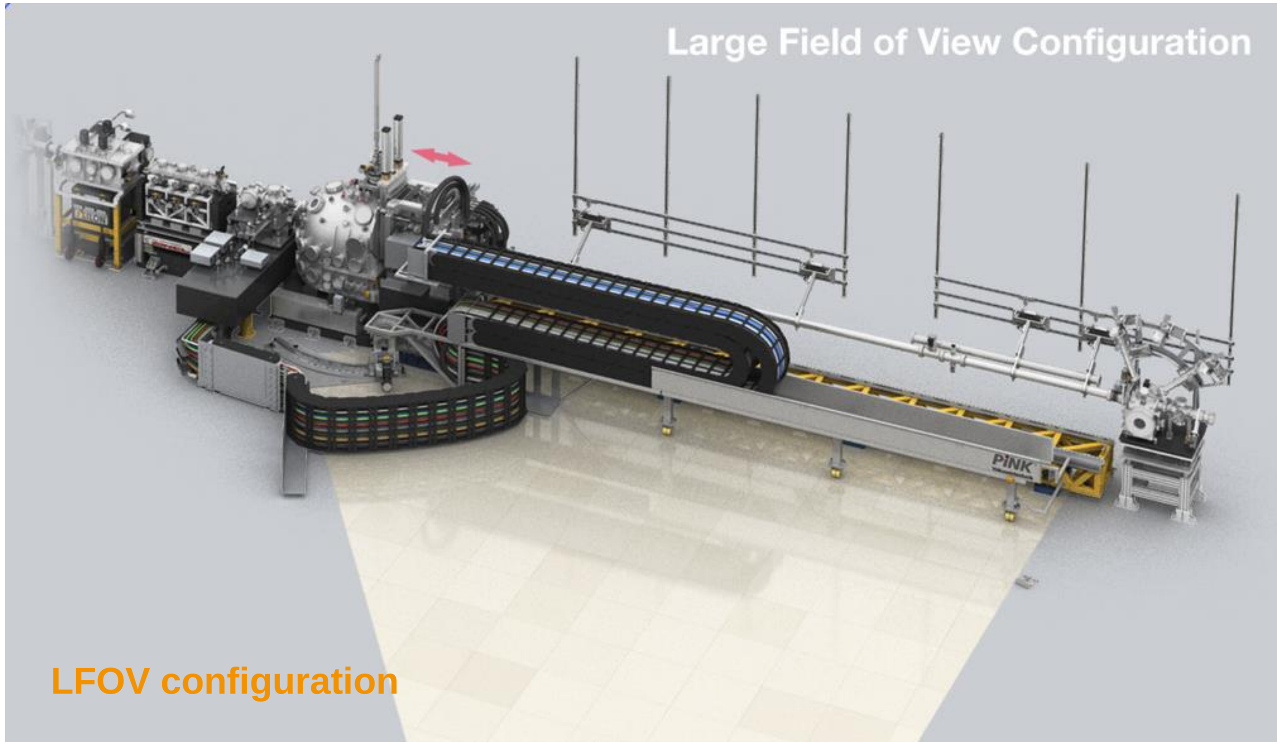
MHz area detector, 10^6 pix of 200 μm size (AGIPD)

Versatile setup, multi-purpose interaction chamber

Windowless (in-vacuum setup) or sample in air

Sample - detector distance 0.2 – 8 m

2θ up to ~50º , 5 - 25 keV



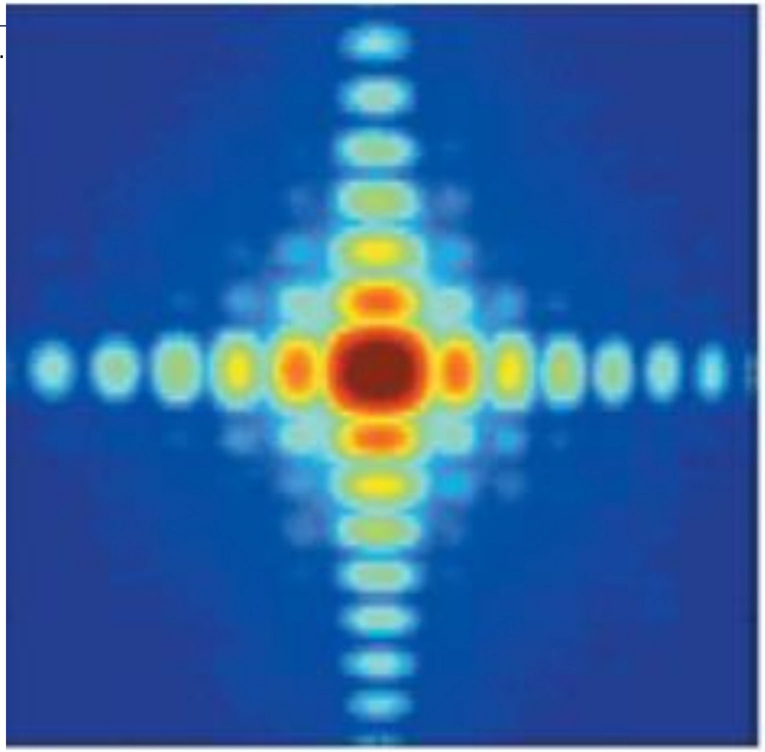
*Madsen et al. JSR **28**, 637-649 (2021)*

Materials Imaging and Dynamics (MID) experiment – Detectors

- AGIPD: 1MPix, 4.5MHz capable detector, pixel size 200um, 352 storage cells (352 images/train)
- ePix and Jungfrau detectors: 0.5MPix detectors (2 modules), 50 and 75um pixel size, 10Hz and 16 storage cells (Jungfrau) with 128kHz.
- Gotthard: 50um pixel strip detector. First generation 0.5MHz capable, next generation up to 4.5MHz (used in spectrometer)
- Diamond solid state ion chambers: 4.5MHz pulse resolved intensity monitors and future position sensitive monitors.

Sztuk-Dambietz, Frontiers in Physics (2024), accepted.

Key experimental techniques used at MID



Making use of the coherence, repetition rate, time resolution and high intensity of the X-rays

- MHz XPCS
  - Small angle scattering geometry (SAXS)
  - Wide angle scattering geometry (WAXS)
  - At a selected Bragg peak in i.e. combination with pump (optical/electrical/magnetic)

- Imaging
  - Holography (full-field technique using cone-beam)
  - (Bragg-) CDI Coherent diffraction imaging

- SAXS/WAXS
  - XRD – powder pattern

- Beam damage is an issue, especially for solid samples
  - But: we can attenuate the beam and can control the intensity on the sample

*Madsen et al. JSR **28**, 637-649 (2021)*

Typical beam parameters at MID and their relevance

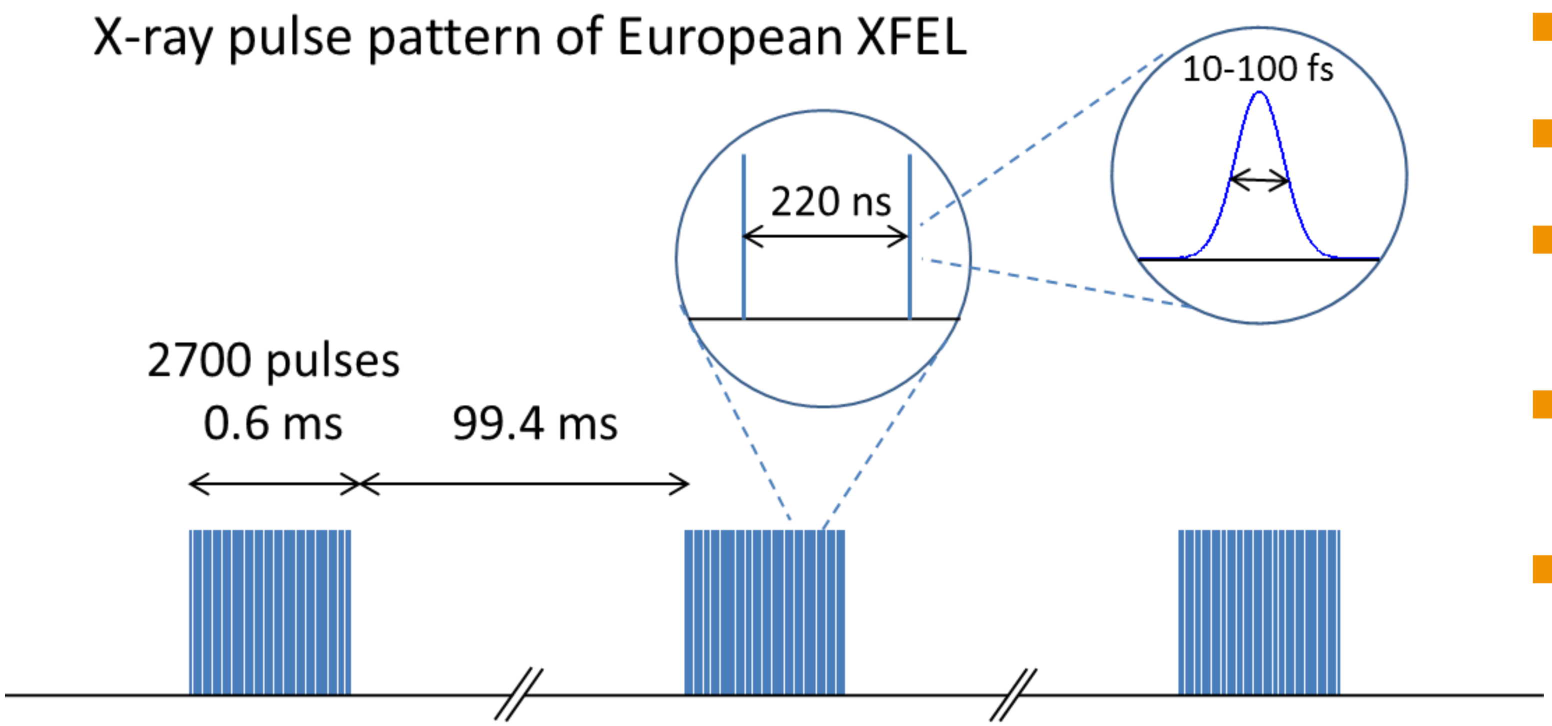
- Hard X-rays 6-25keV
  - Main working point around 9keV with about 1-2mJ or 10^{12} ph/pulse

- Repetition rate up to 4.5MHz
  - essential to investigate the dynamics, provides 220ns as shortest time period between X-ray bunches
  - XPCS does not work in the “diffract and destroy” regime
    - ► Rolling bunch pattern

- Monochromatic beam for temporal coherence: important for wide-angle XPCS and imaging applications
  - Monochromators are challenged by the bunch structure (heatload)
  - Hard X-ray self seeding

- Ongoing developments:
  - Future also harder X-rays (>30KeV)
  - Ultra-short pulses (attoseconds)
  - Two-color mode

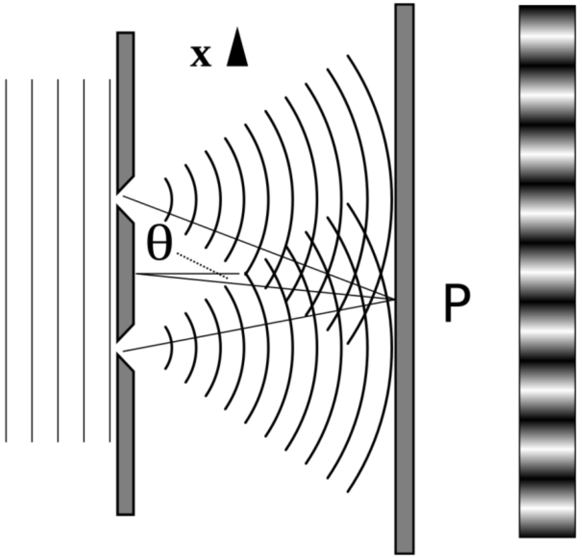
Time structure of European XFEL



- The European XFEL can produce up 27000 X-ray laser flashes per second
- Other XFELs typically operate with 100Hz
- Bunch train repetition rate 10Hz
- Bunch trains: 4.5 MHz total repetition rate
- 10^{12} X-ray photons (~1 mJ) per pulse (<100 fs)
- Pulses within train distributed to all beamlines in a predefined pattern
- Timing structure offers unique measurement opportunities

Coherent scattering and speckles

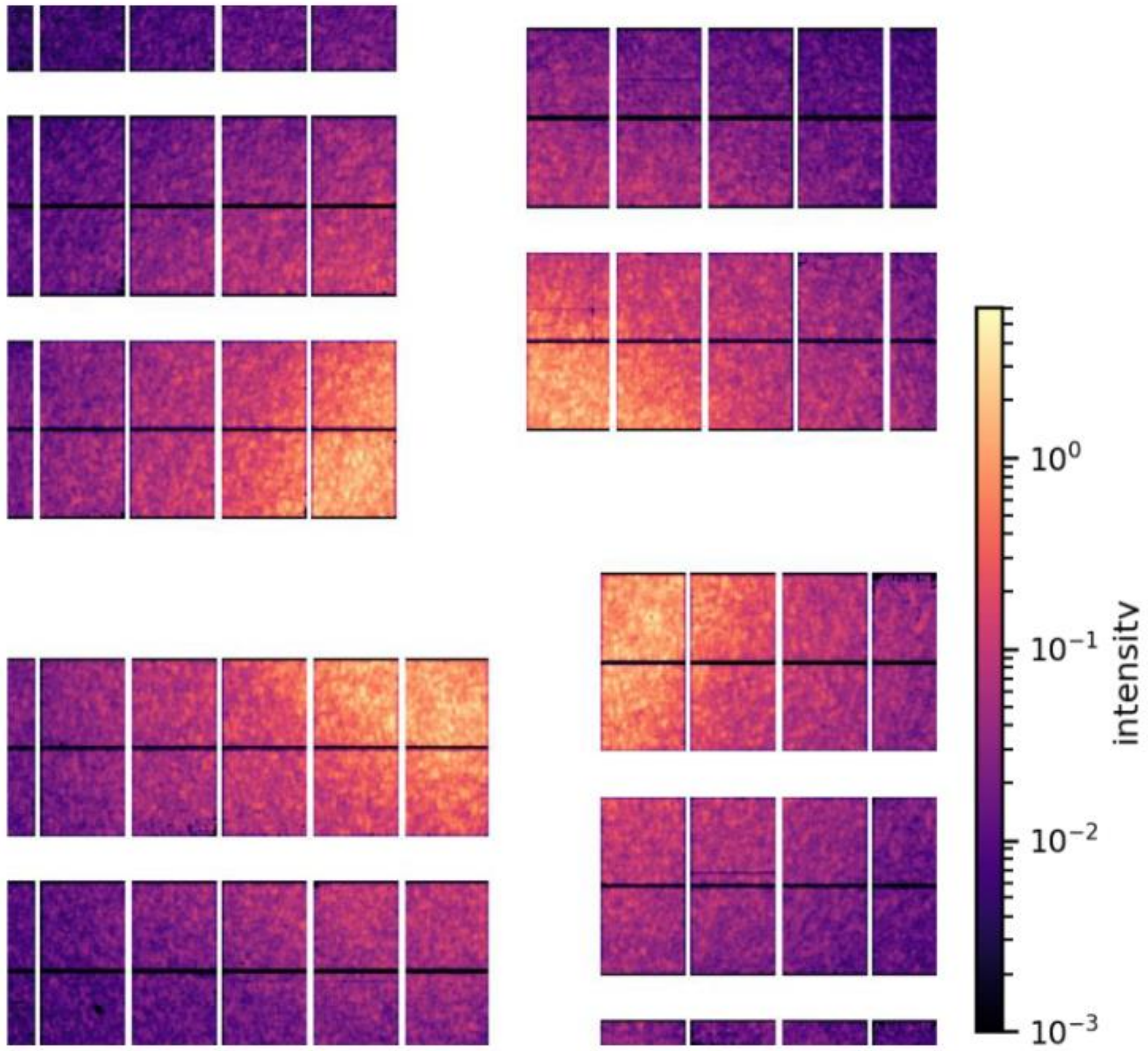
In physics, coherence expresses the potential for two waves to interfere.



Also works with 3D real samples…

https://en.wikipedia.org/wiki/Coherence_(physics)
By Ebohr1.svg: en:User:Lacatosias, User:Stanneredderivative work: Epzcaw (talk) - Ebohr1.svg, CC BY-SA 3.0, https://commons.wikimedia.org/w/index.php?curid=15229922

Speckle pattern (SAXS) on AGIPD

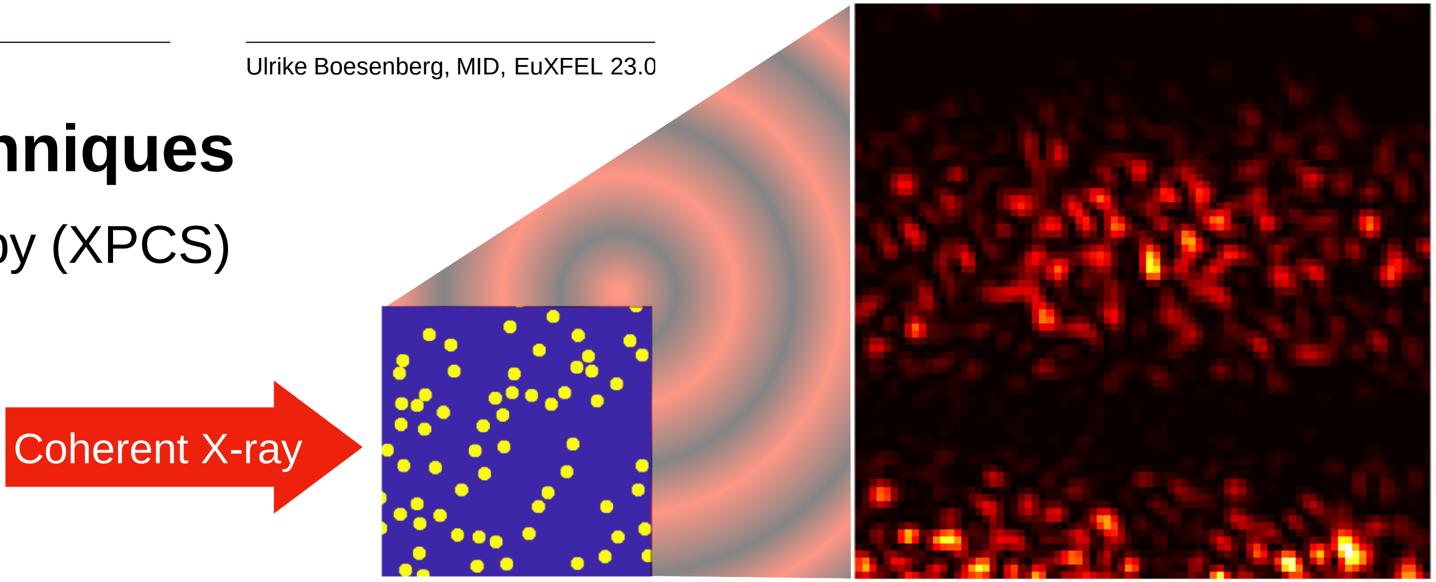


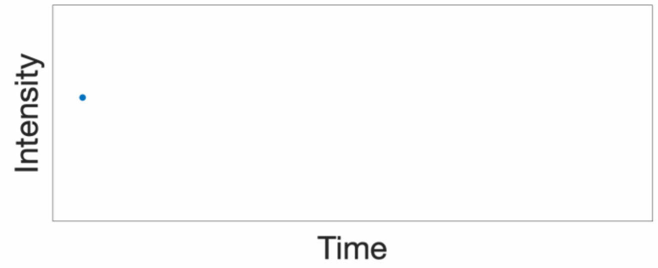
Sample:Vycor (glass with defined air bubbles)

Coherent X-ray scattering techniques

X-ray Photon Correlation Spectroscopy (XPCS)

- Versatile speckle technique investigating dynamics of disordered systems
- The exact spatial arrangement of the system generates an interference speckle pattern





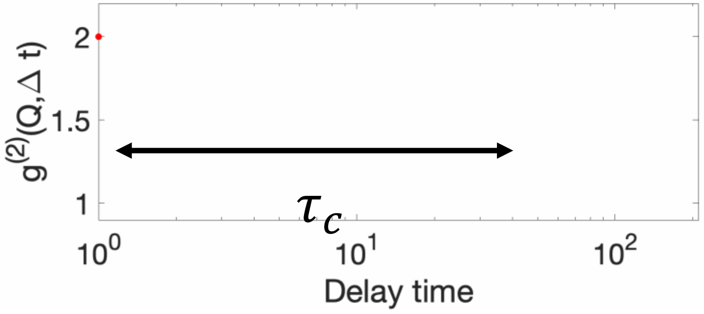
Spatial resolution	Temporal resolution
Photon energy / Wavelength	• **Pulse repetition rate** • **Detector frame rate**

Intensity correlation function

$$g^{(2)}(q,\tau) = \frac{\langle I(q,t)I(q,t+\tau)\rangle}{\langle I(q,t)\rangle^2}$$

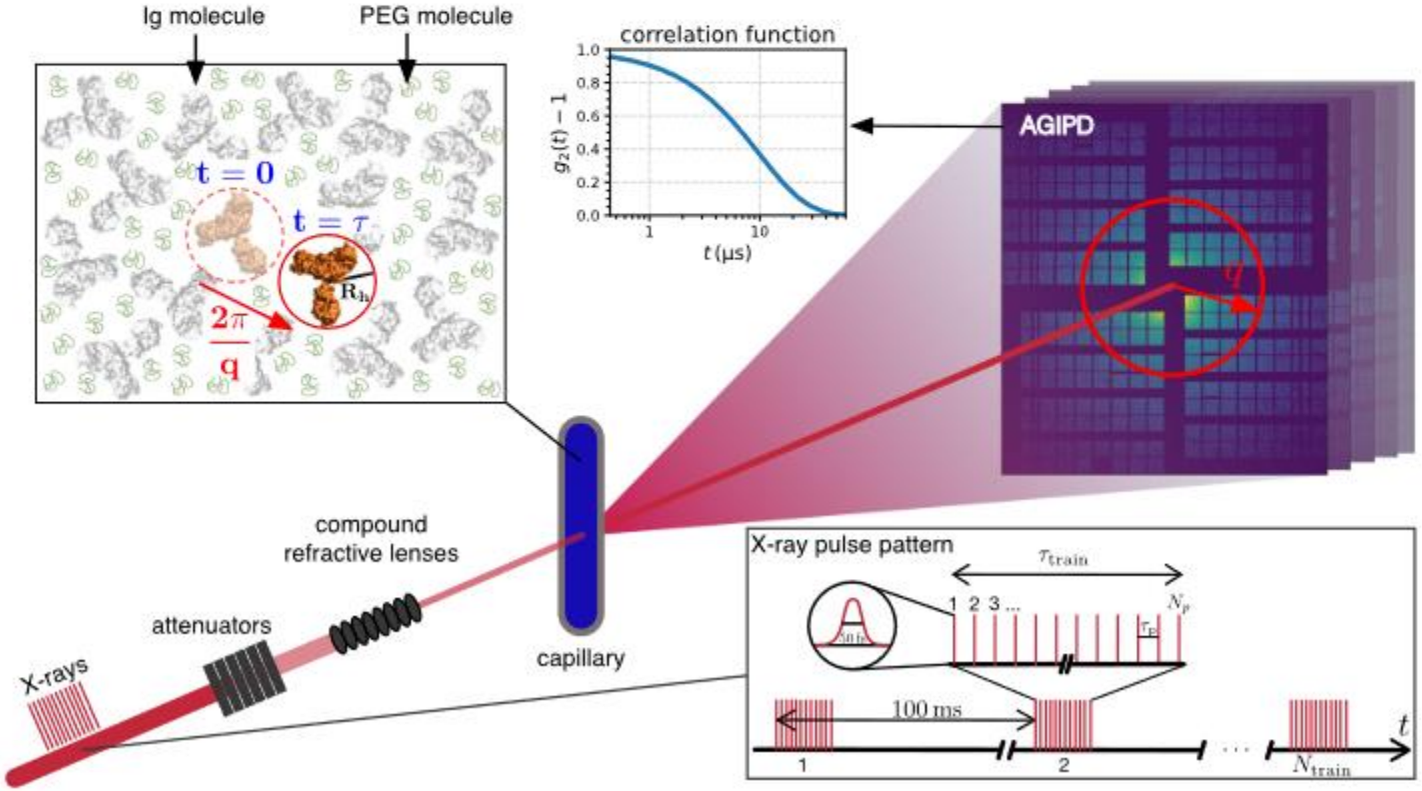
$$= 1 + \beta|f(q,\tau)|^2$$

$$= 1 + \beta exp(-2\tau/\tau_c)$$



Courtesy of Wonhyuk Jo (MID)

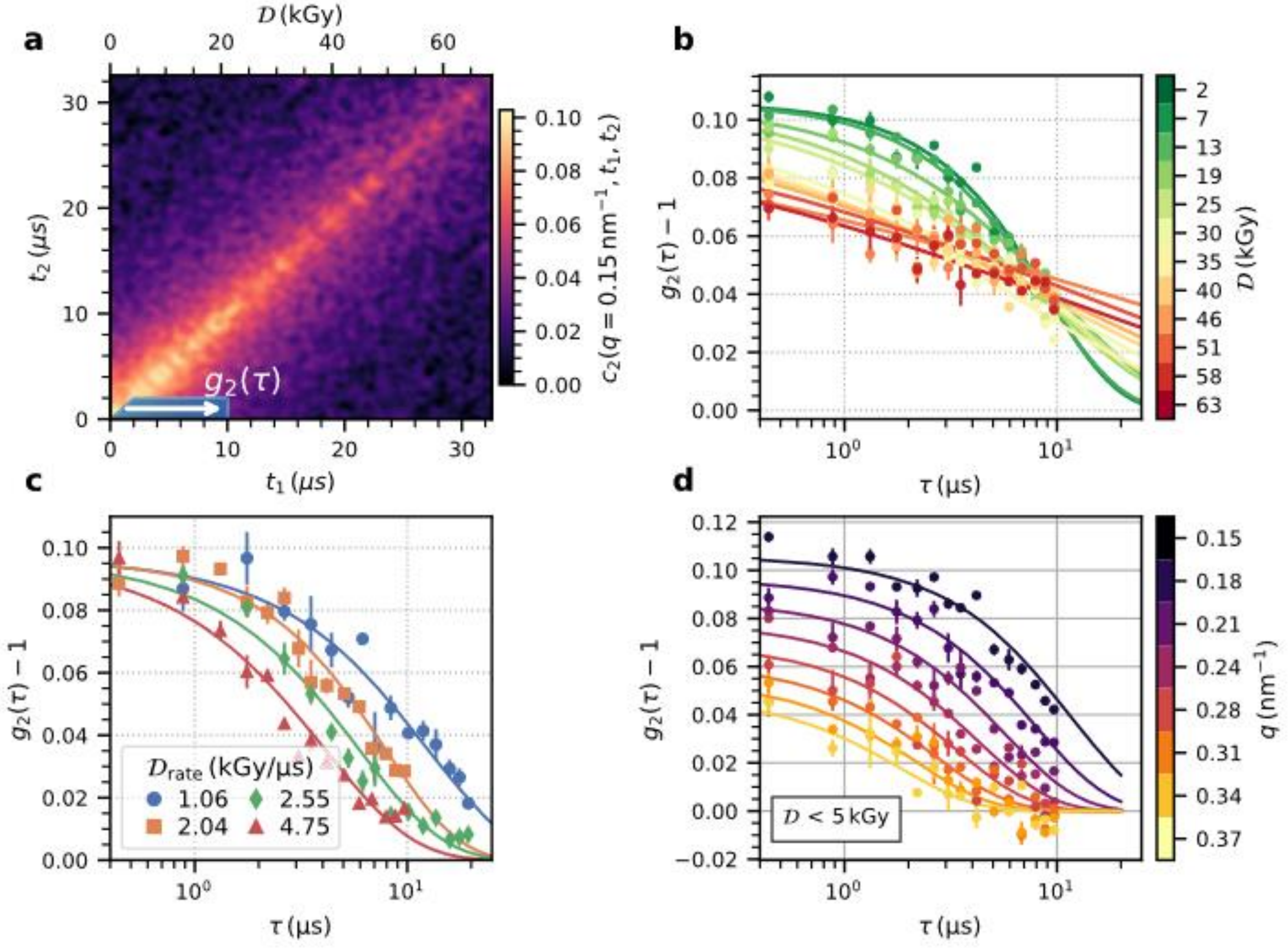
MHz X-Ray Photon Correlation Spectroscopy



- Record a time series of speckle patterns (with coherent X-rays)
- Calculate a time correlation
- Fast dynamics are accessible with the high repetition rate of the EuXFEL

Reiser et al. Nat. Com.(2022)13:5528
Dallari et al. Appl. Sci. (2021)11:8037

MHz XPCS on aggregation of proteins



- Molecular diffusion
- Aggregation of antibody proteins
- Agglomeration dynamics
- Beam induced dynamics – results are dose rate dependent

Fig. 3 | Correlation functions. **a** Two-time correlation function, c_2, of Ig-PEG measured with an average dose rate of 2.04 kGy μs^{-1} for $q = 0.15$ nm^{-1}. **b** Correlation functions for different initial doses ($\mathcal{D}_{\mathrm{rate}} = 2.04$ kGy μs^{-1}, $q = 0.15$ nm^{-1}). **c** Correlation functions with an initial dose below 5 kGy for different dose rates at $q = 0.15$ nm^{-1}. **d** Correlation functions for different momentum transfers fitted with a q-squared dependent relaxation rate ($\mathcal{D}_{\mathrm{rate}} = 2.04$ kGy μs^{-1}). The error bars represent the standard error over pixels and repetitions. Source data are provided as a Source Data file.

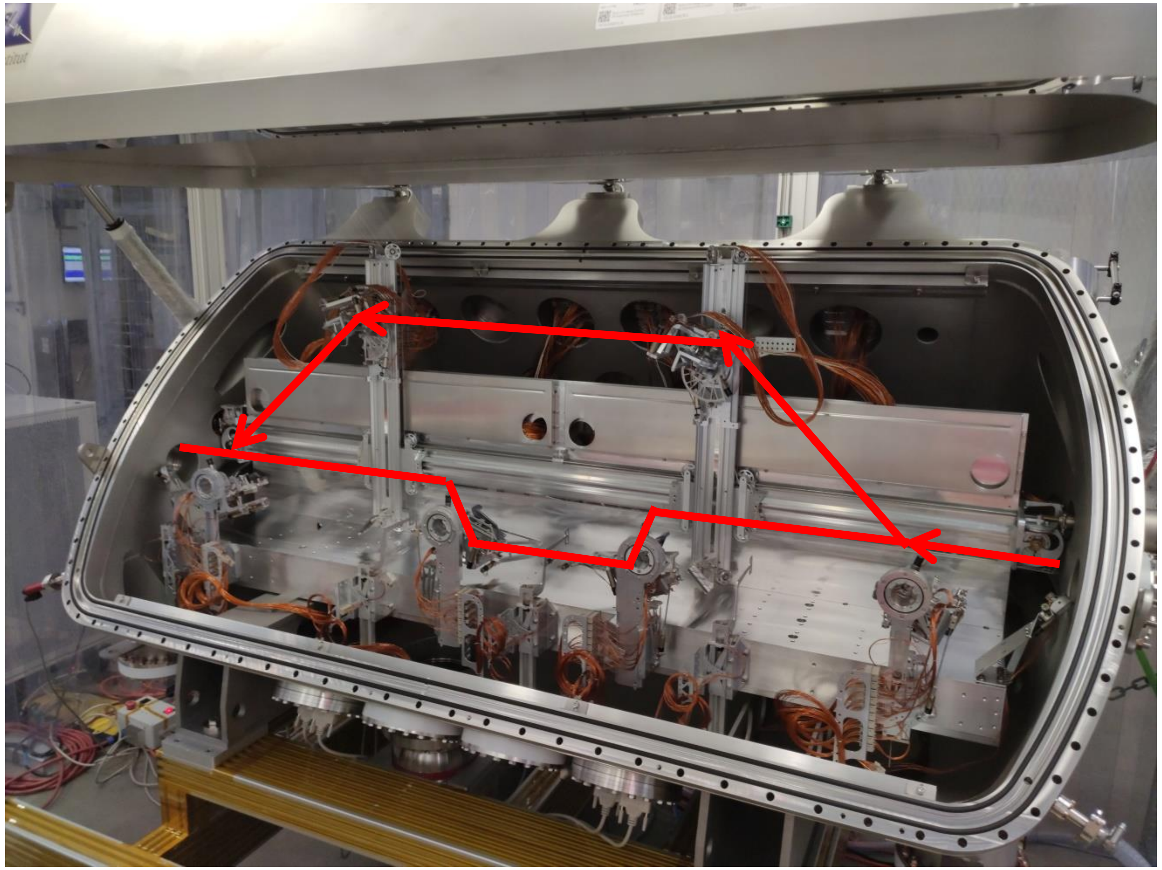
Reiser et al. Nat. Com.(2022)13:5528

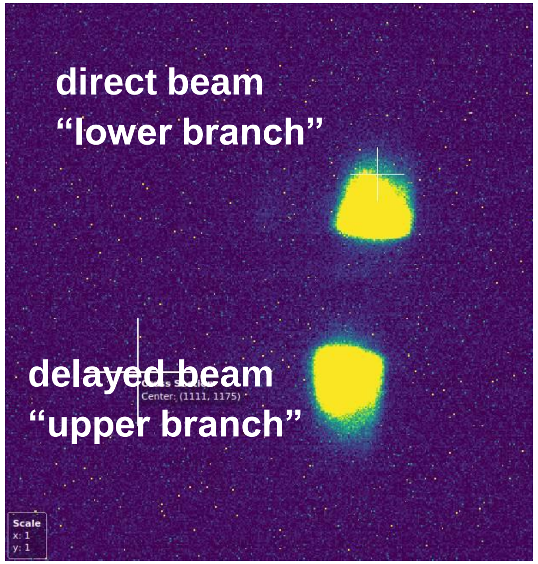
Split – and – delay – line (SDL)

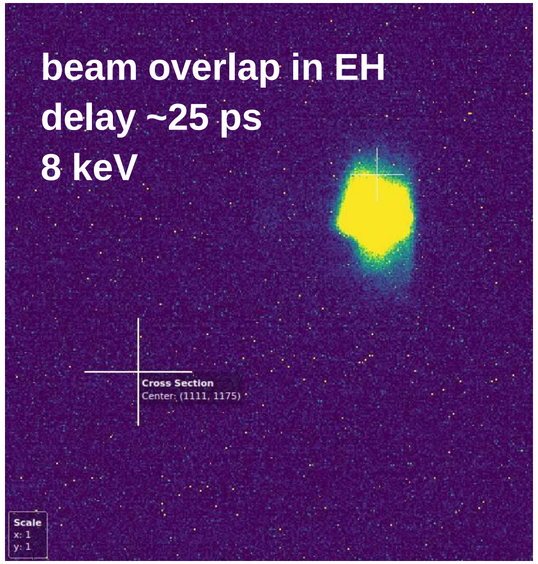
- Ultra short delay (few fs to 800ps) between two X-ray pulses
- X-ray pump – X-ray probe experiments
- 5-10 keV operating range

Bundesministerium für Bildung und Forschung

FEL FSP-302





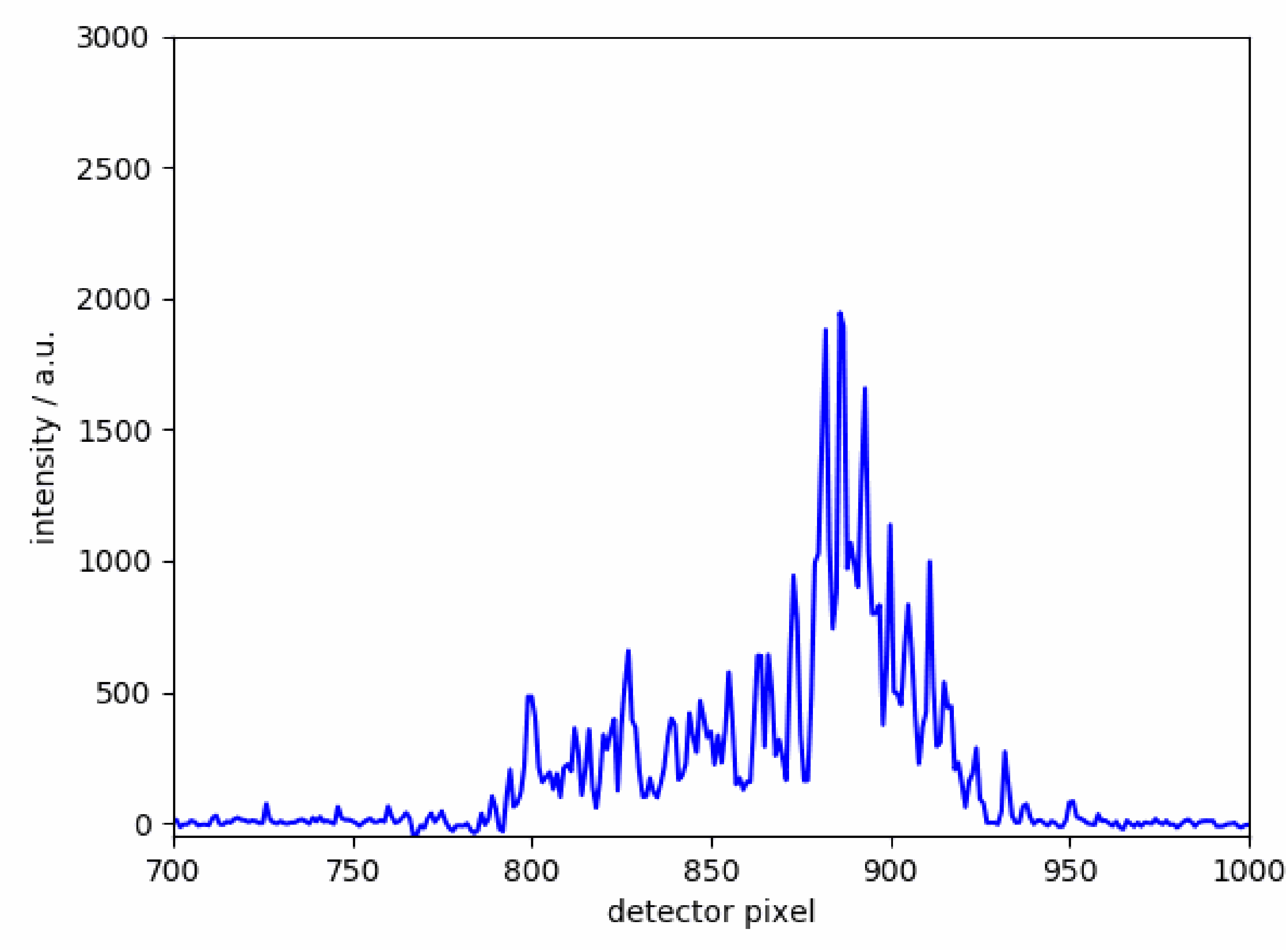


S. Eisebitt (MBI)
W. Lu, B. Friedrich et al. (MID)

W. Lu et al. Rev. Sci. Instrum. ***89*** *(6):063121 (2018)*

Funding via BMBF Verbundforschung under contracts 05K13KT4 and 05K16BC1 is gratefully acknowledged.

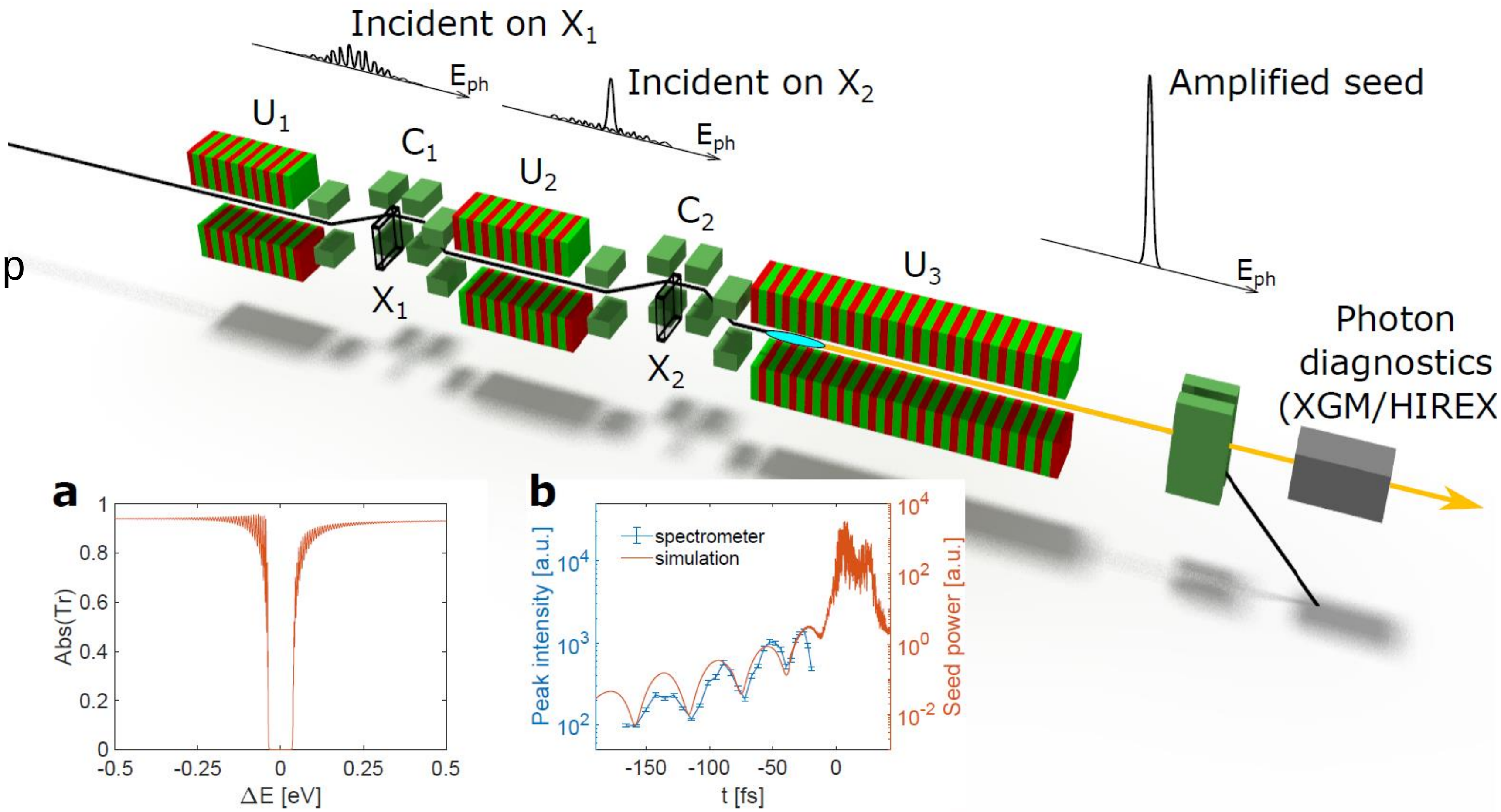
SASE bandwidth



- Temporal coherence is influenced by monochromaticity – important to obtain sufficient contrast
- Focus properties suffer from pink beam when using chromatic X-ray lenses (CRLs)
- Standard Si-monochromators suffer from the high heat load of the burst mode.

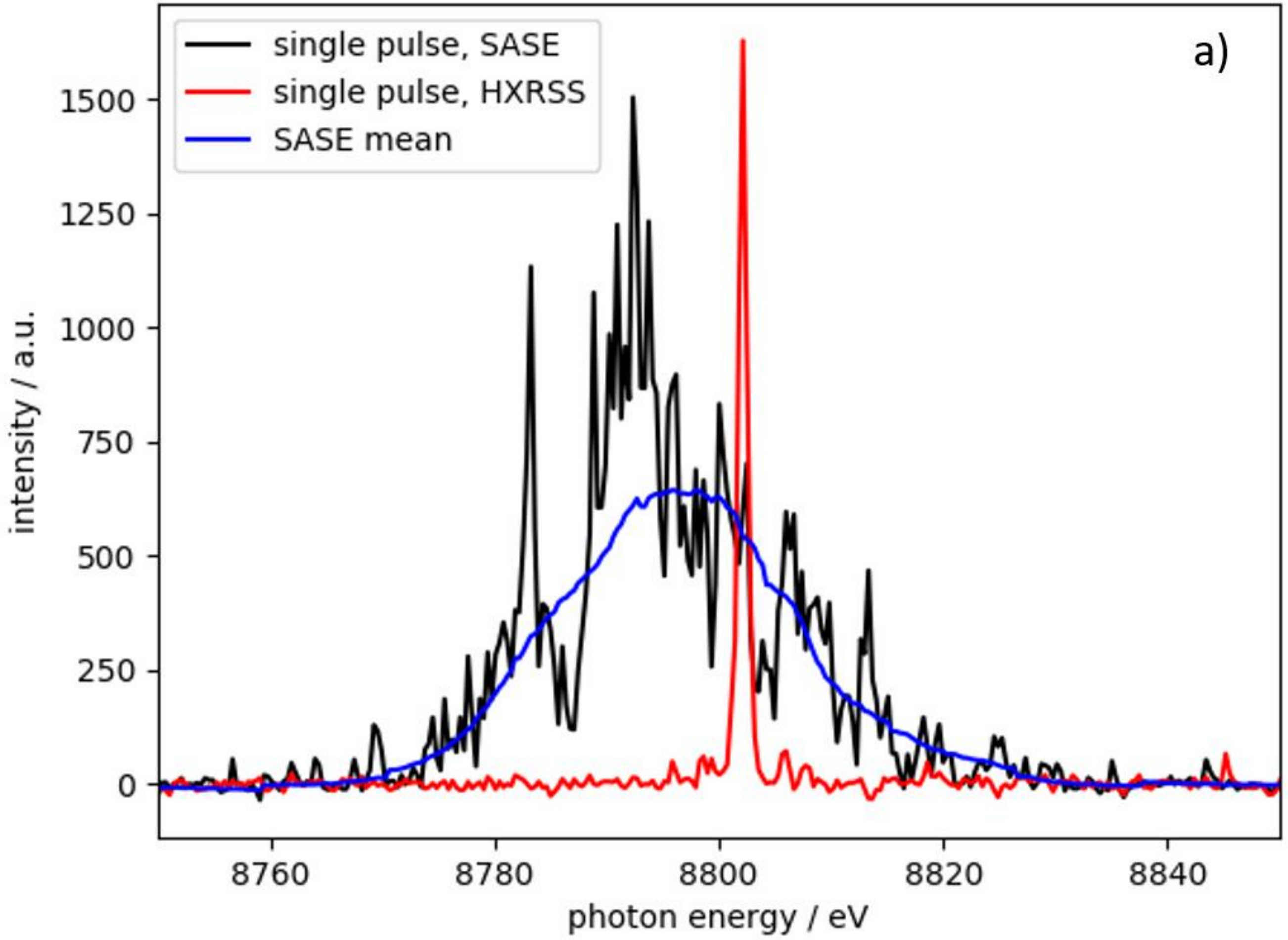
Hard X-ray self seeding at SA2

- Cascaded HXRSS setup developed at SASE-2
- Two diamond crystals inside the undulator



S. Lui et al., Nature Photonics (2023) 17:984–991

Monochromatic beam - HXRSS

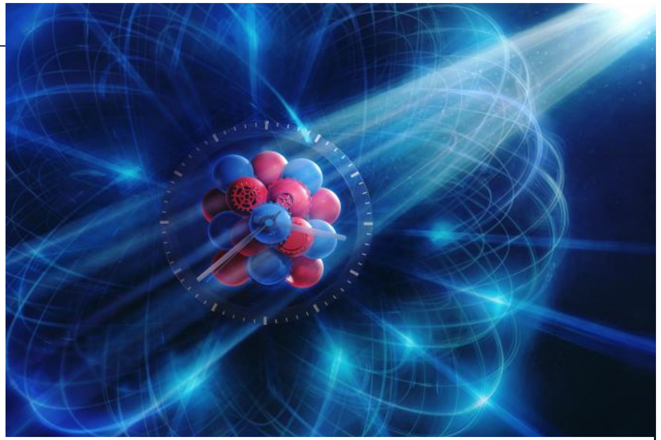


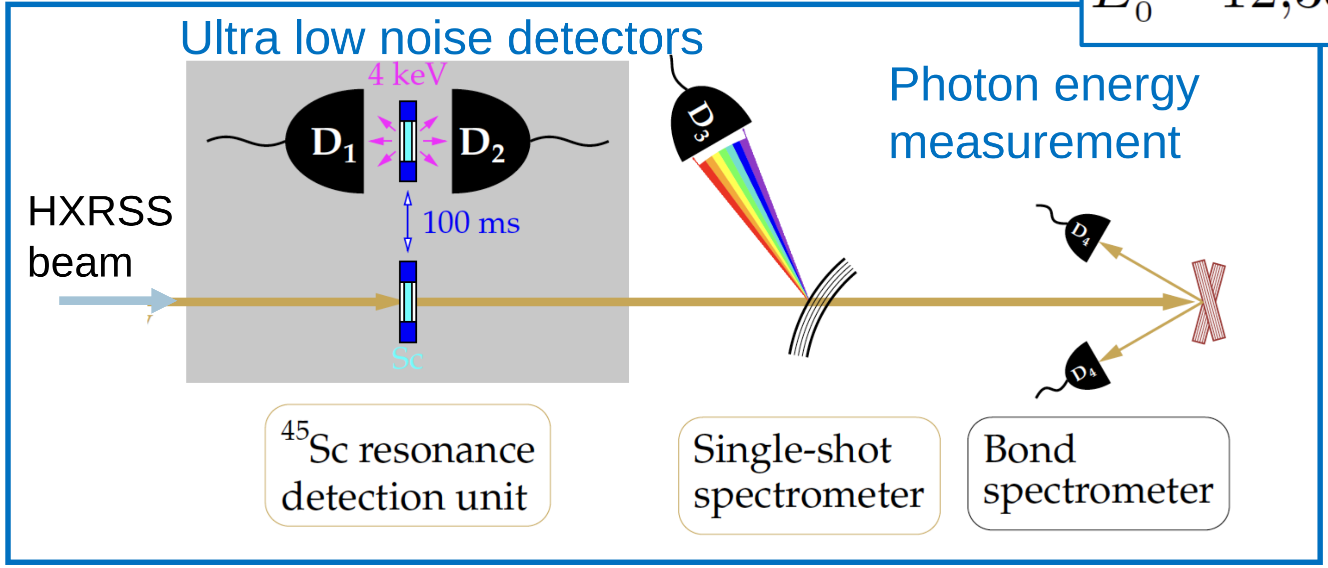
- Temporal coherence is influenced by monochromaticity – important to obtain sufficient contrast
- Focus properties suffer from pink beam when using chromatic X-ray lenses (CRLs)
- Standard Si-monochromators suffer from the high heat load of the burst mode.
- Availability to tune the seeded photon energy

Boesenberg et al., submitted to JSR (2024)

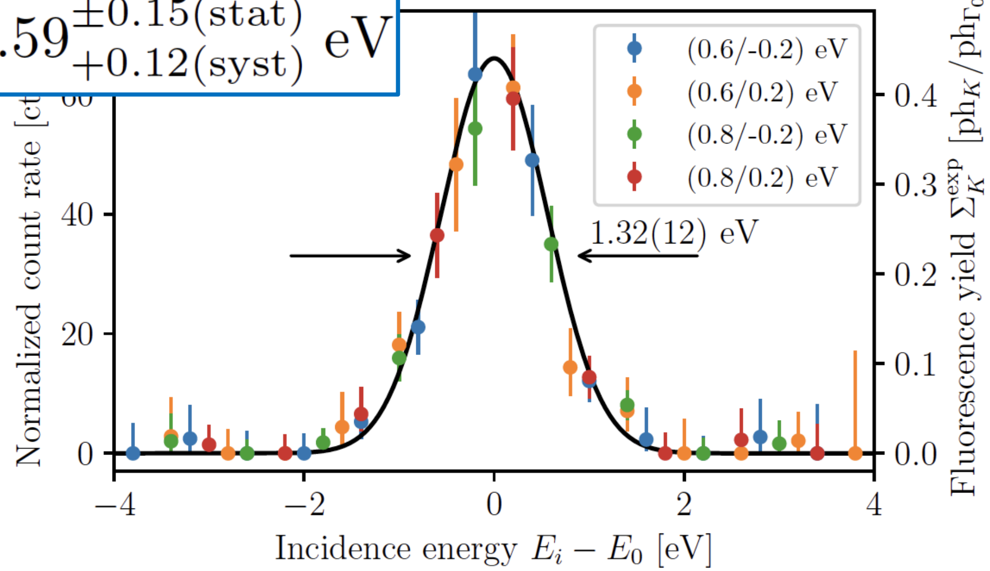
Mössbauer experiments

- Observing the 1.4 feV 45-Sc nuclear resonance for the first time!
- Scanning the seeded line around the expected resonance at 12.4 keV
- Looking for fluorescence in the “incoherent channel”





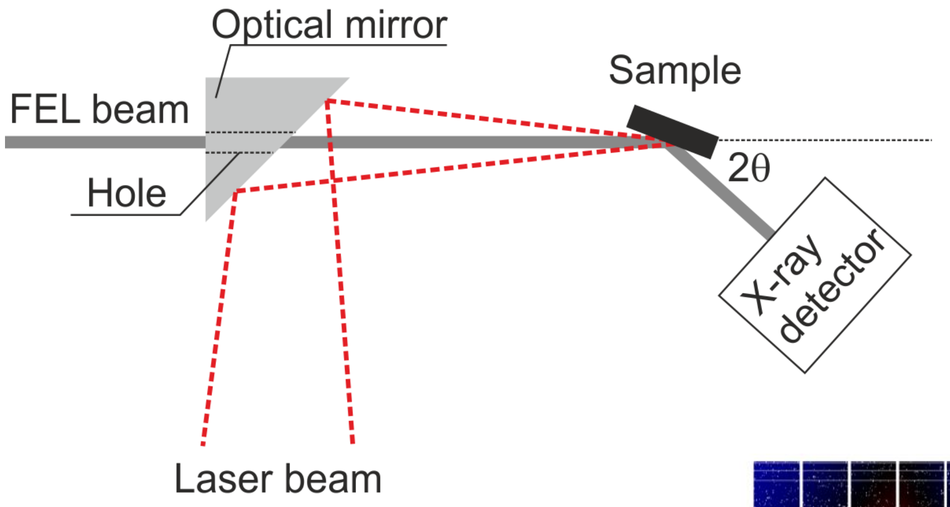
$$E_0 = 12{,}389.59^{\pm 0.15(\mathrm{stat})}_{+0.12(\mathrm{syst})}\ \mathrm{eV}$$



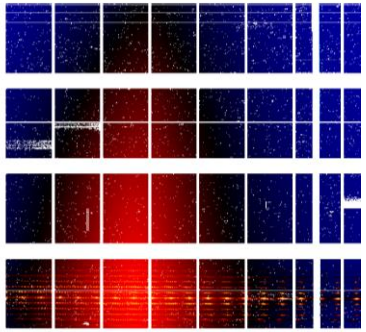
Texas A&M University · Friedrich-Schiller-Universität Jena · Max-Planck-Institut für Kernphysik Heidelberg · Argonne National Laboratory · DESY. · European XFEL

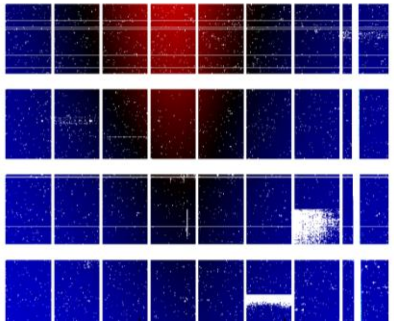
Y. Shvyd‘ko et al., Nature (2023) 622:471

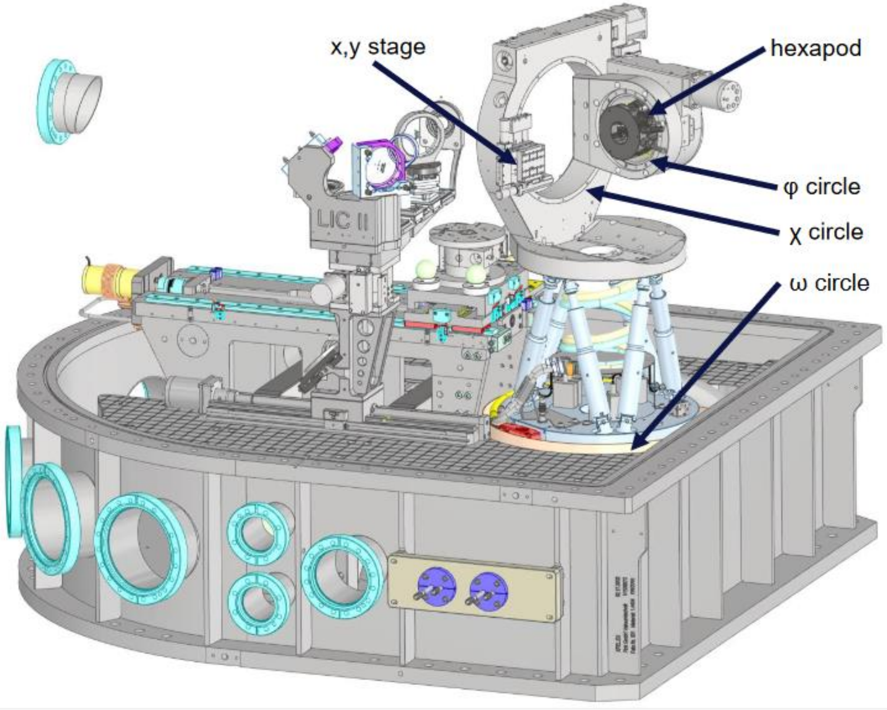
Setup for ultrafast diffraction



- Instrument in WAXS geometry
- Optical pump - X-ray probe
- High resolution Bragg Peak analysis
- Small goniometer setup (in progress)



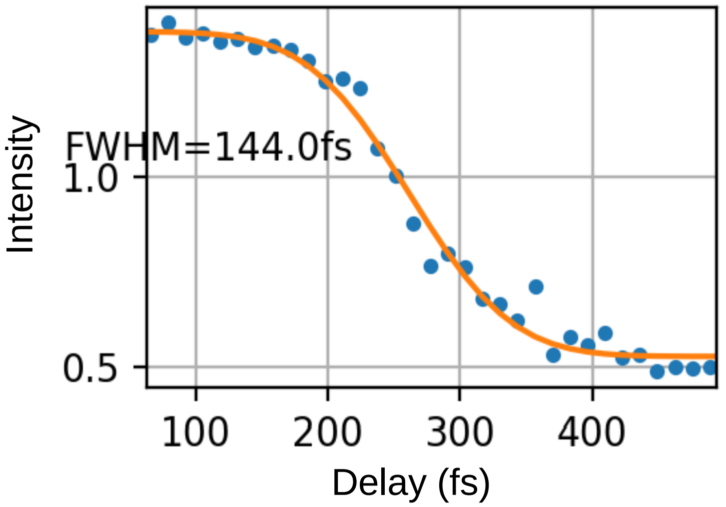


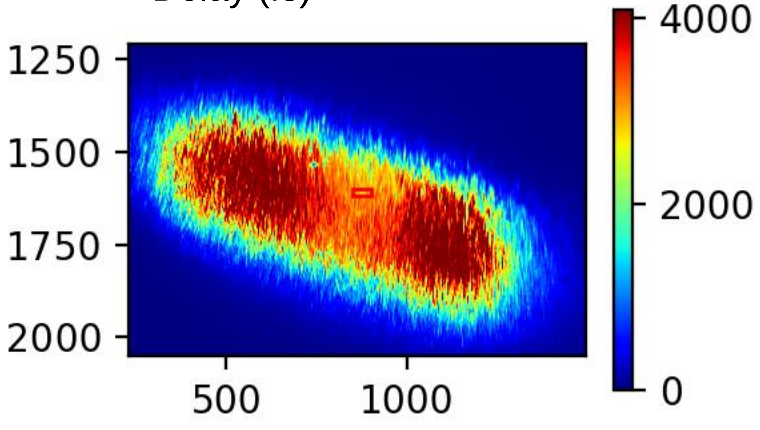


Courtesy of J.-E. Pudell

Pump-probe experiments at MID

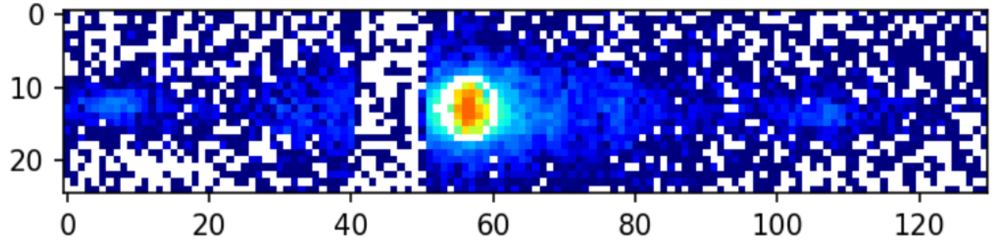
X-ray Pump – Optical probe on YAG

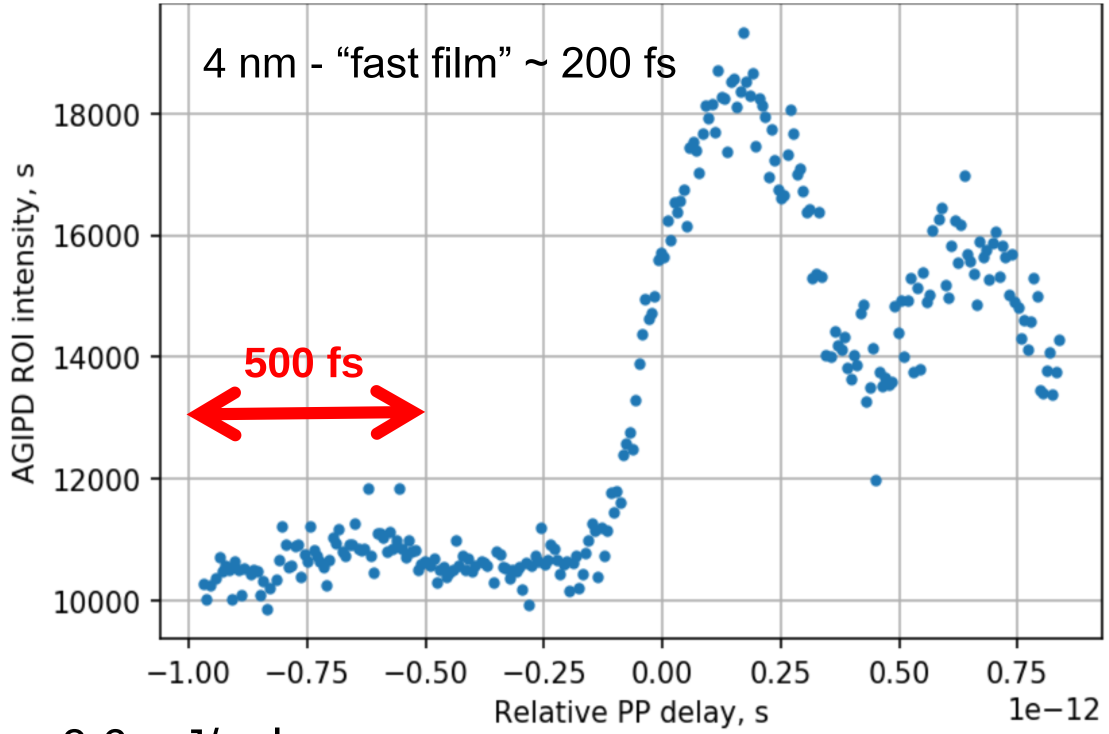




800 nm pump – X-ray probe

SrRuO3/SrTiO3 thin film

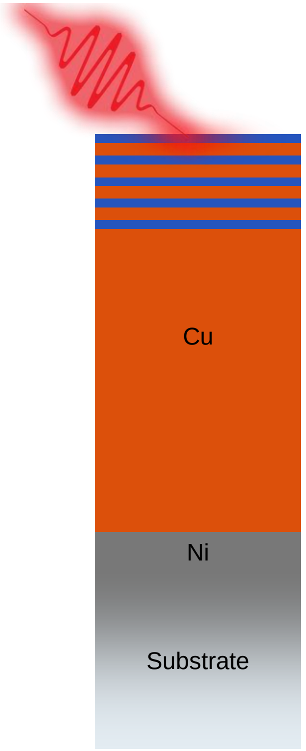


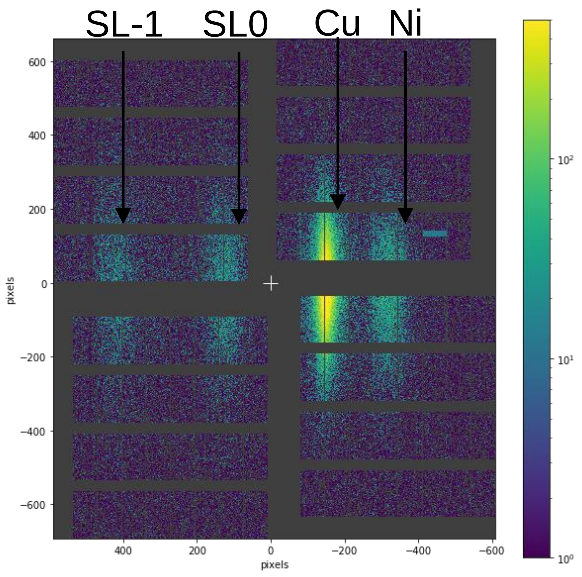


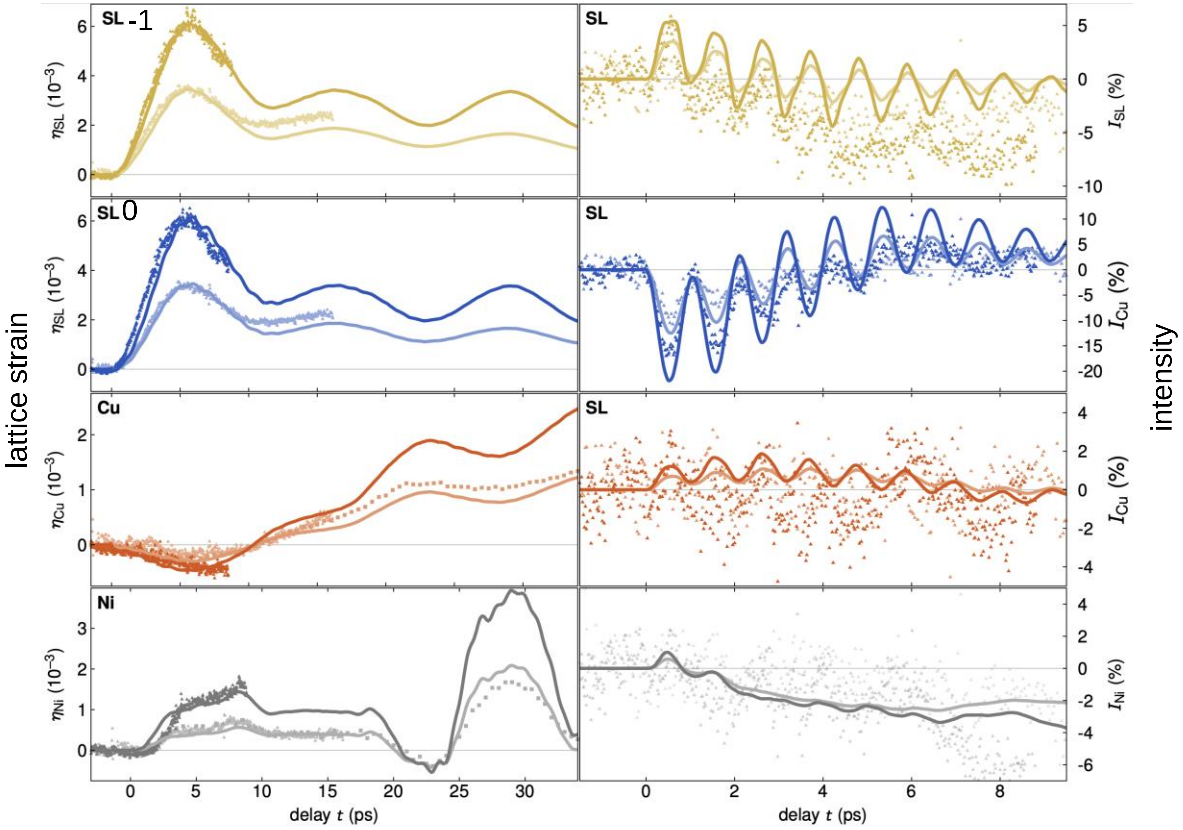
fs laser parameters: 800 nm, ~15 fs, ~150 fs jitter (or less), ~0.6 mJ/pulse
nanosecond laser also available.

Shayduk et al.: APL (2022)120:202203

Ultrafast strain in heterostructures

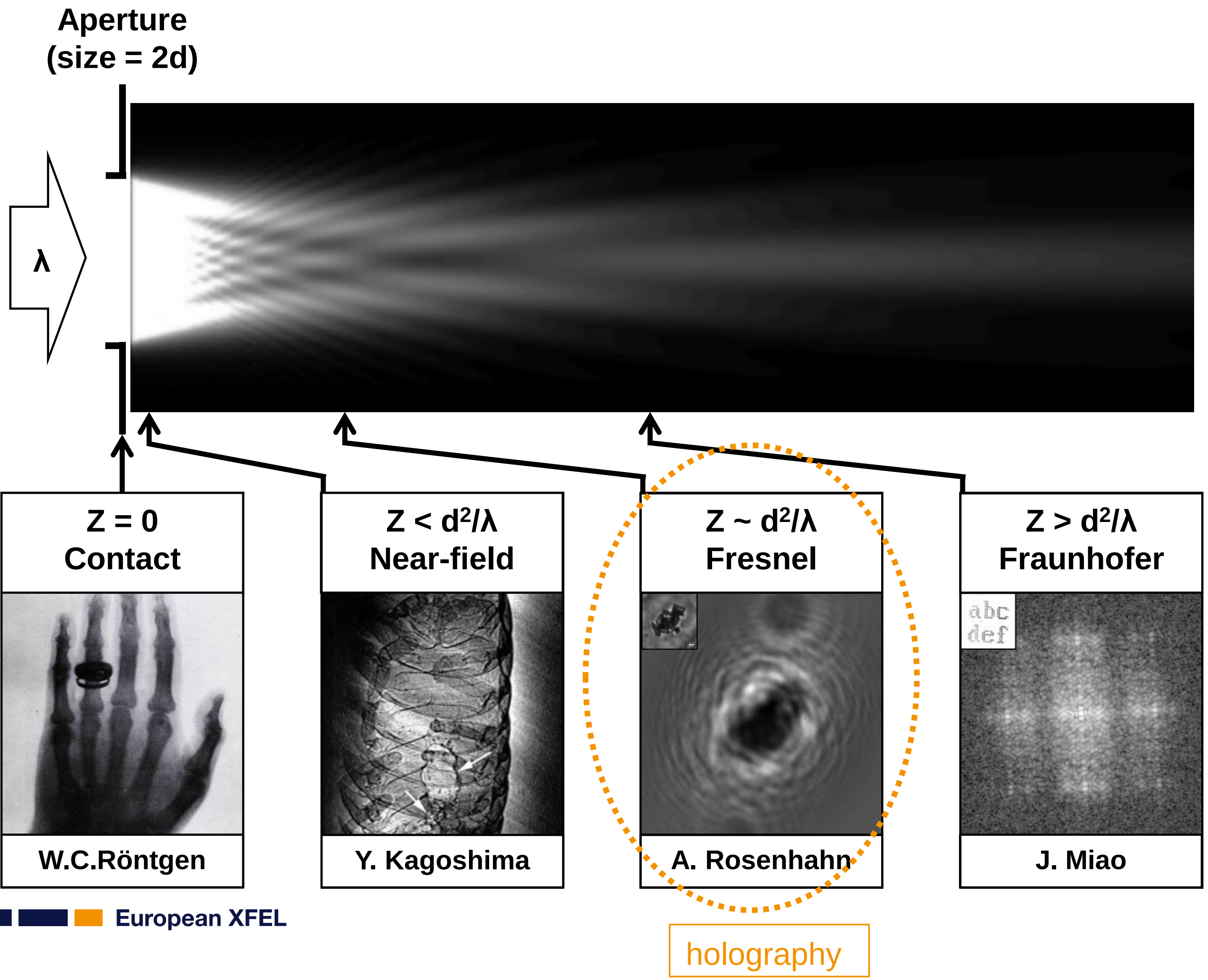






Courtesy of J.E. Pudell (MID)

Propagation of Waves (Fresnel Number)



$$F = \frac{d^2}{Z\lambda}$$

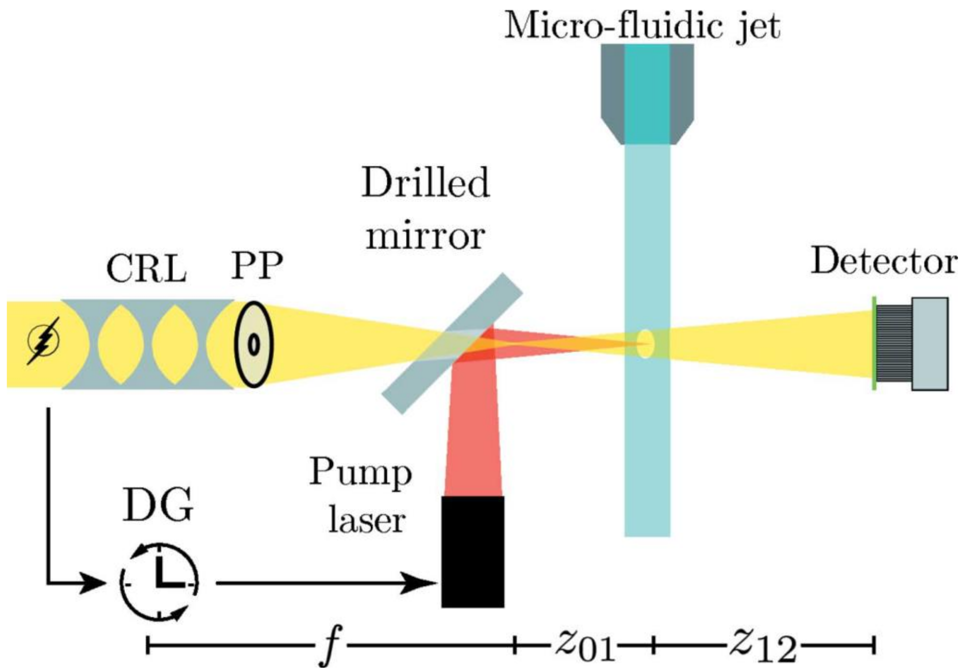
F : Fresnel number

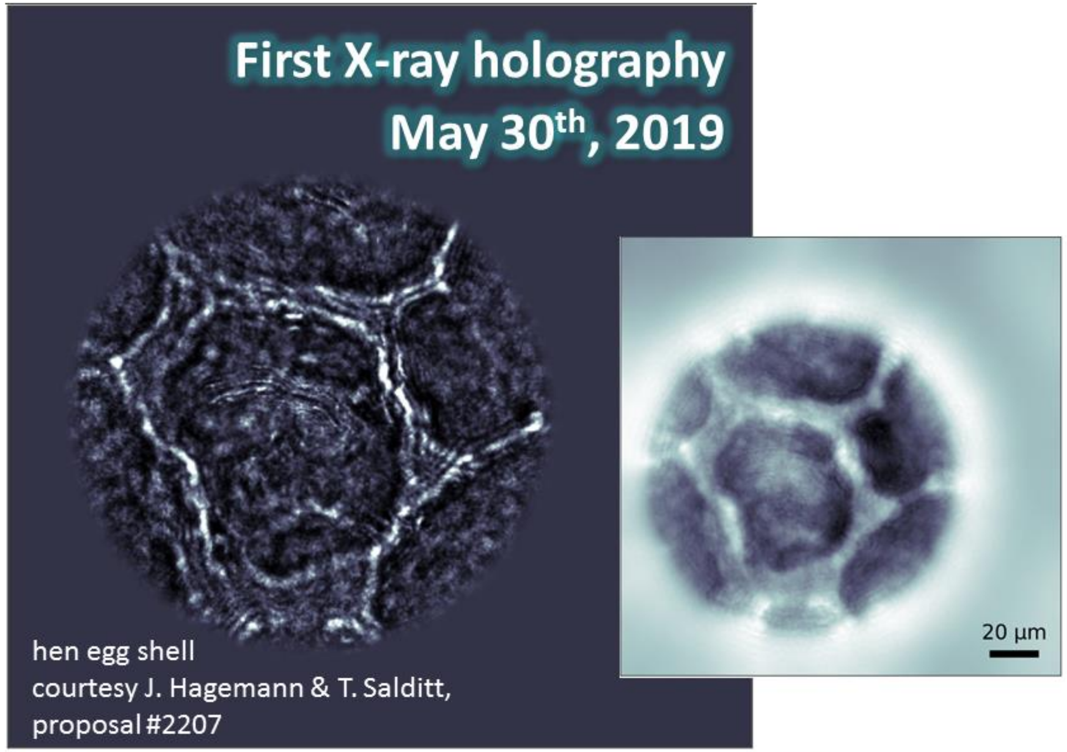
d : size of the aperture

Z : distance of the screen from the aperture

λ : incident wavelength

Single-pulse phase-contrast imaging



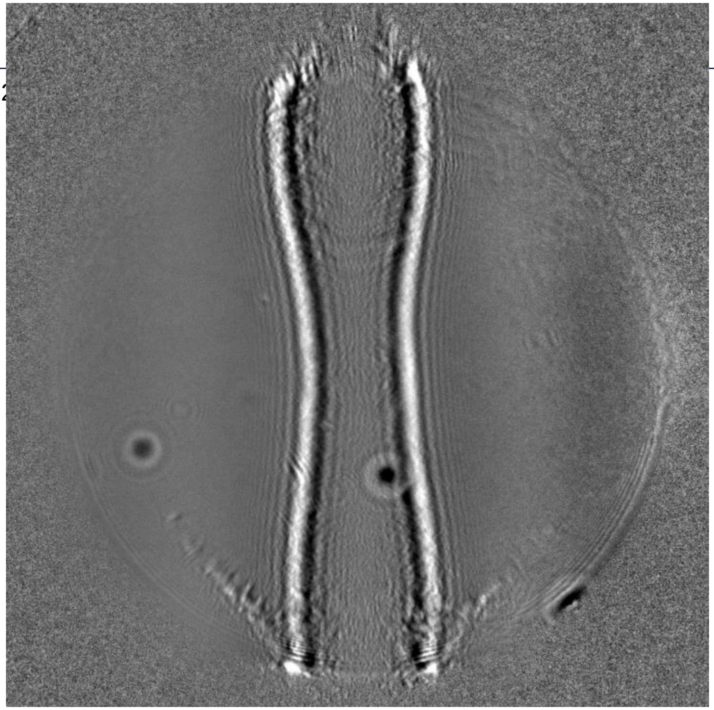


- Create a secondary source by focusing the X-rays.
- Cone-beam geometry allows full-field imaging
- Interference of the probing beam and the scattered waves generate the intensity contrast.

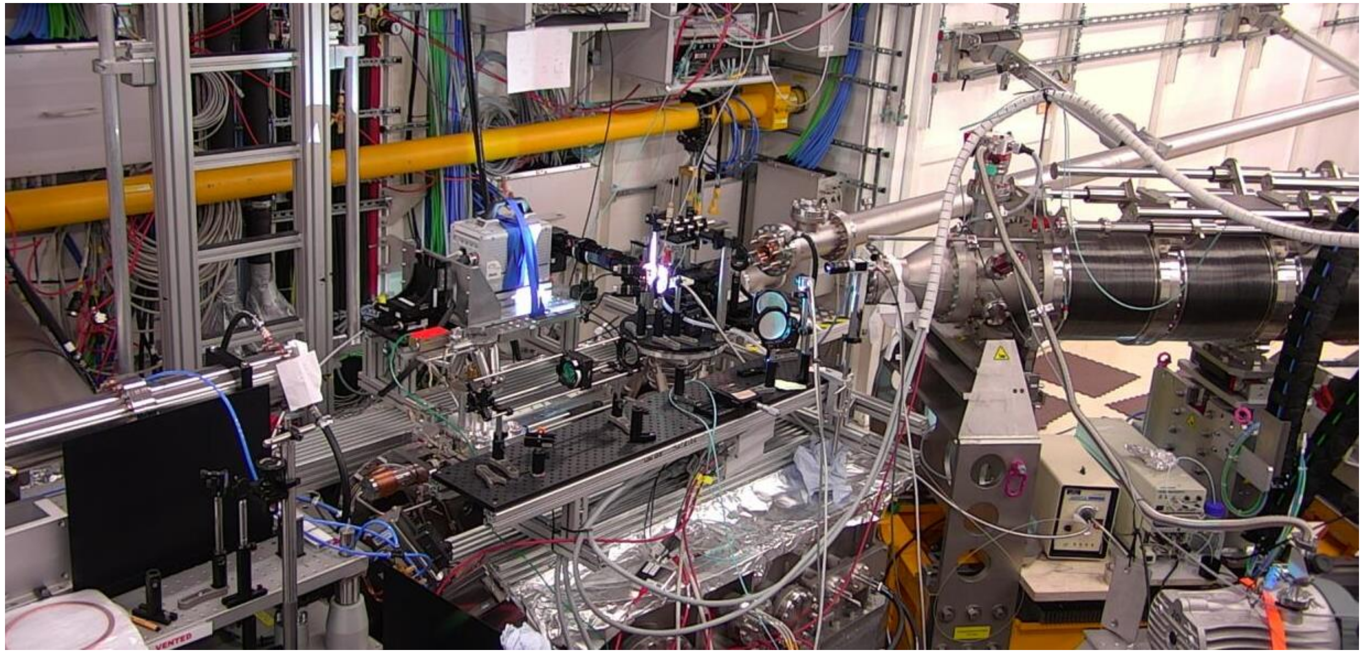
Vassholz et al., Nature Communications(2021) 12:3468; Hagemann et al., J. Synchrotron Rad. (2021). 28, 52–63

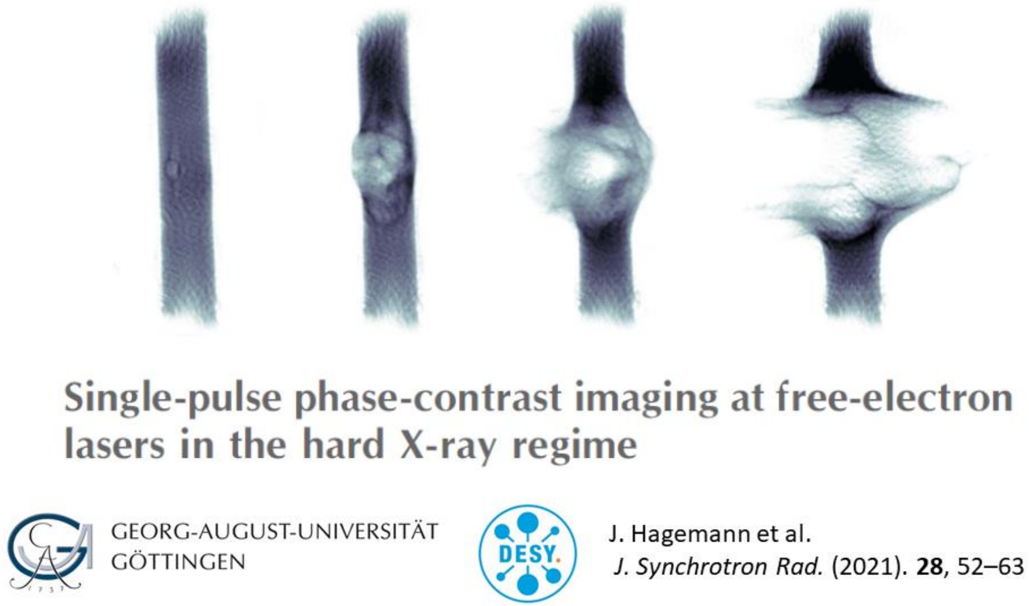
Time-resolved imaging

laminar water jet



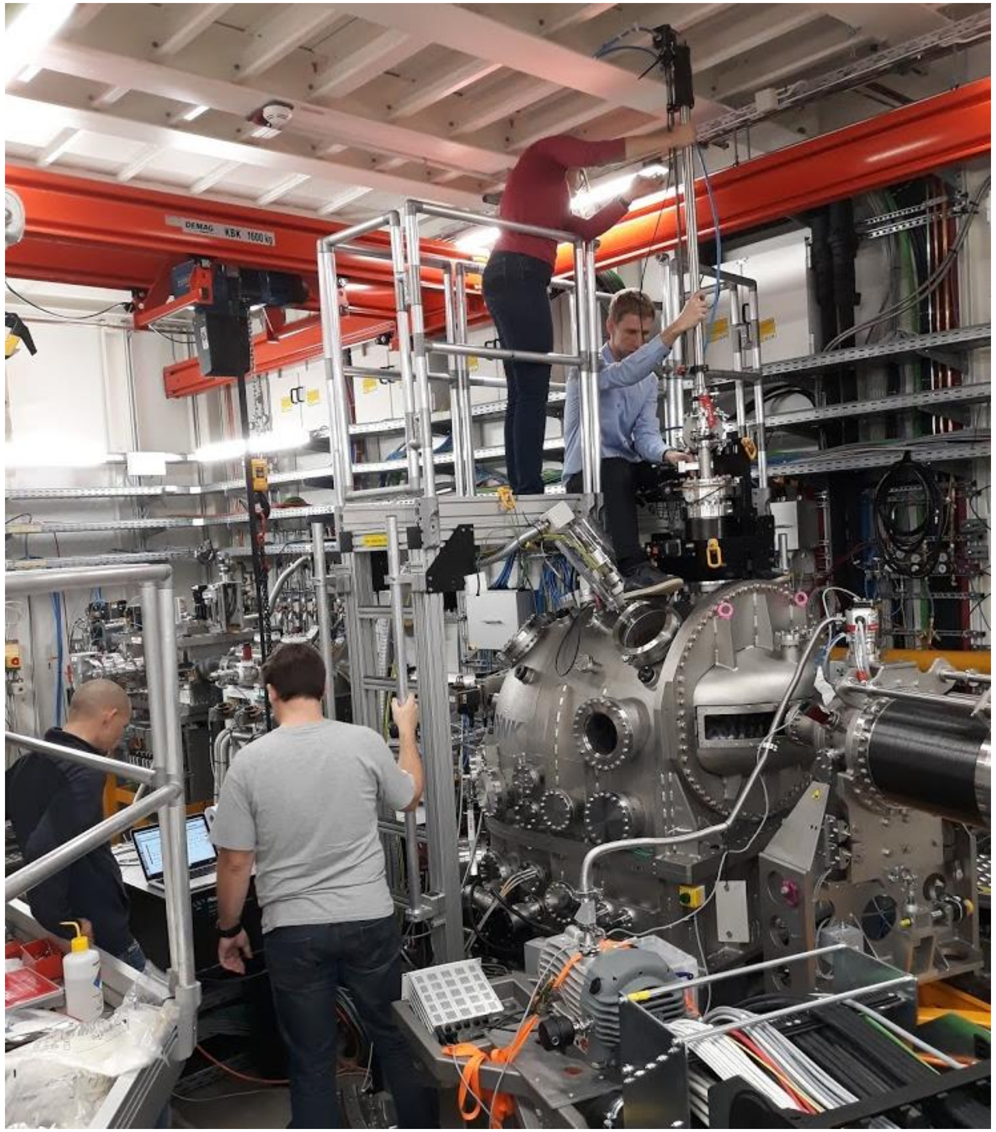
Andor Zyla CCD, 10 Hz, 6.5 um pixel size,
Cone beam hologram @ 14 keV
~100 nm pixel resolution
Combined with optical pump laser-> different delays





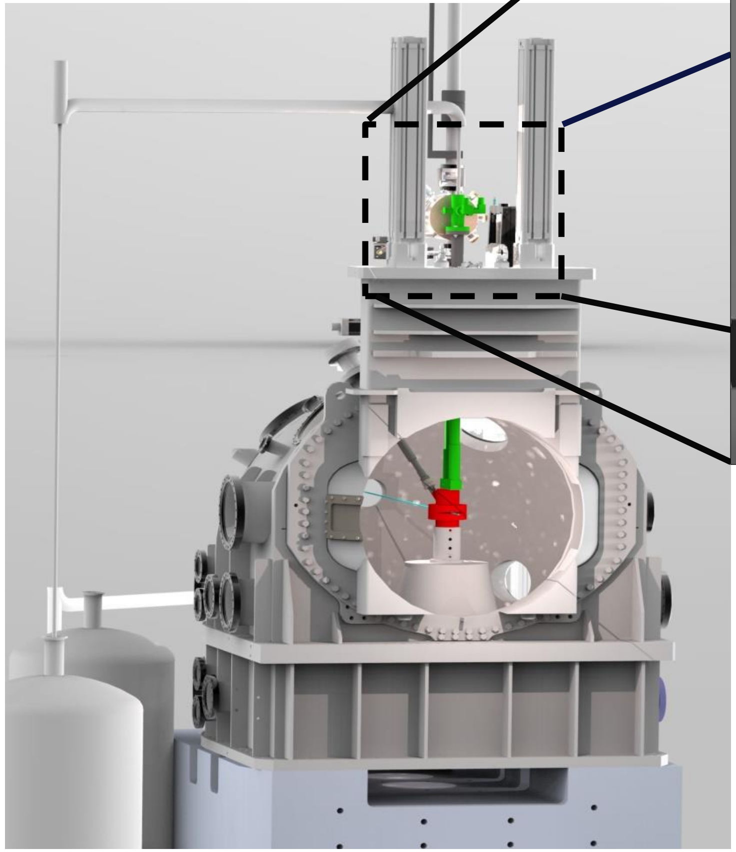
Vassholz et al., Nature Communications(2021) 12:3468; Hagemann et al., J. Synchrotron Rad. (2021). 28, 52–63

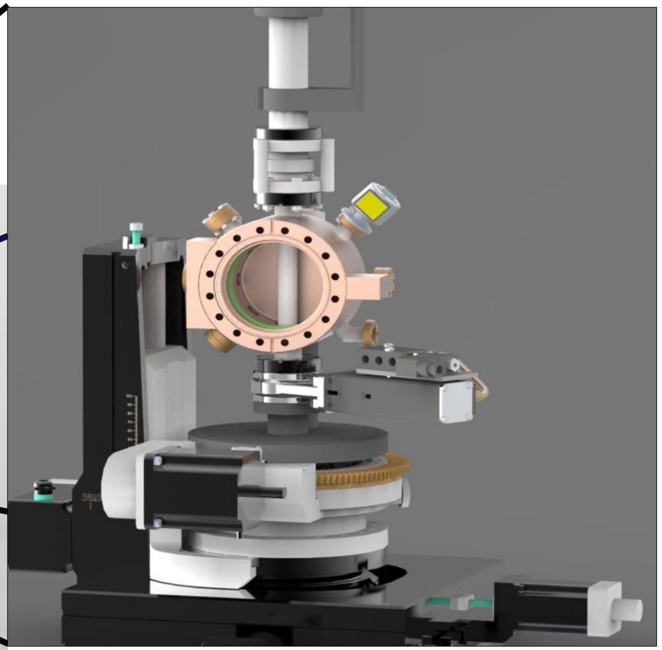
Cryostat and pulsed magnetic field setup (PUMA)

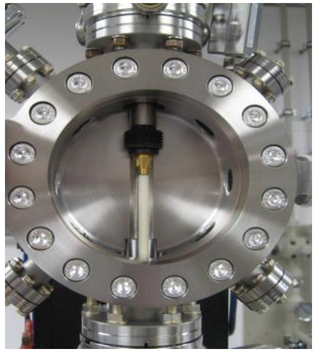


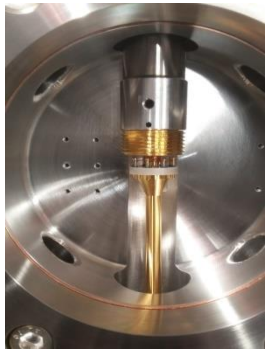
First commissioning Nov 2019

Flow cryostat with load lock



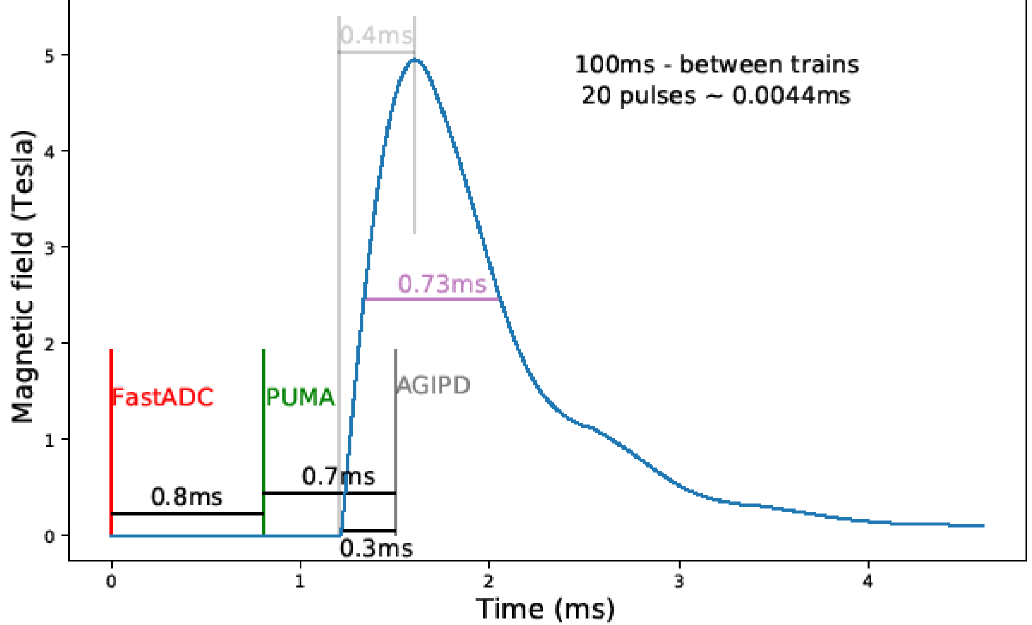


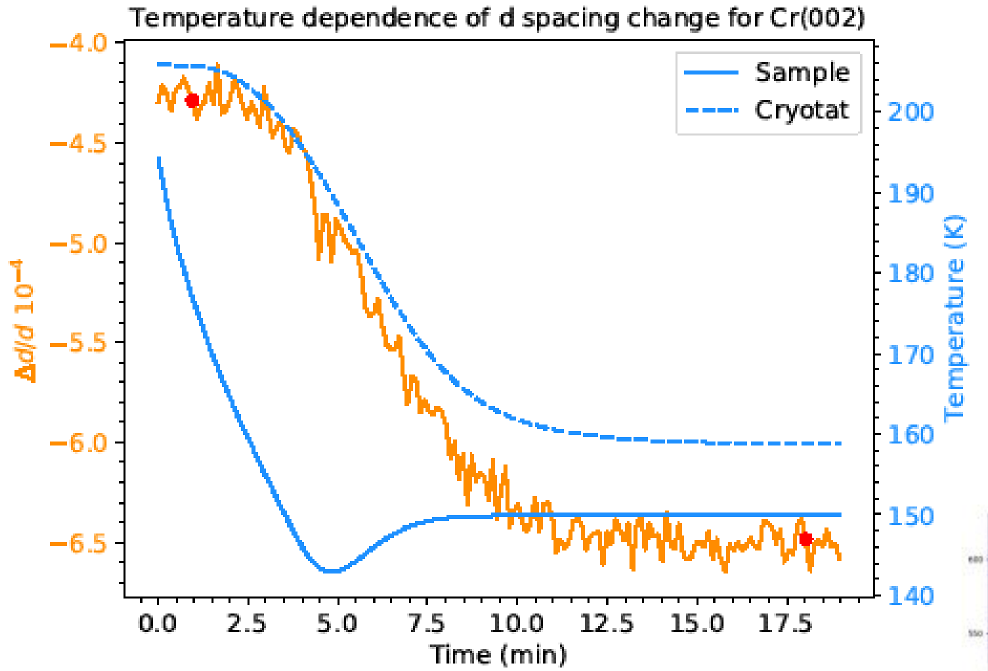


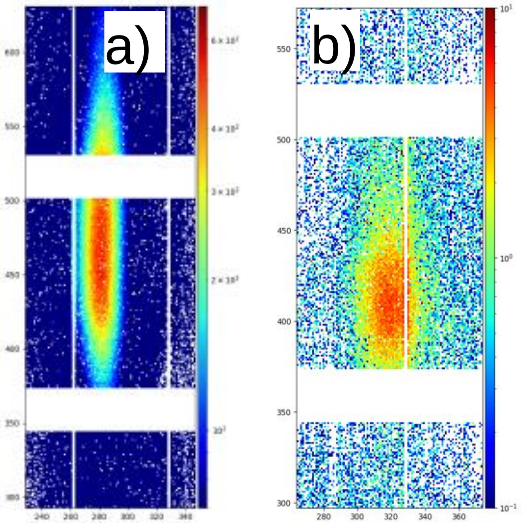


Cryostat and pulsed magnetic field setup (PUMA)

Timing scheme







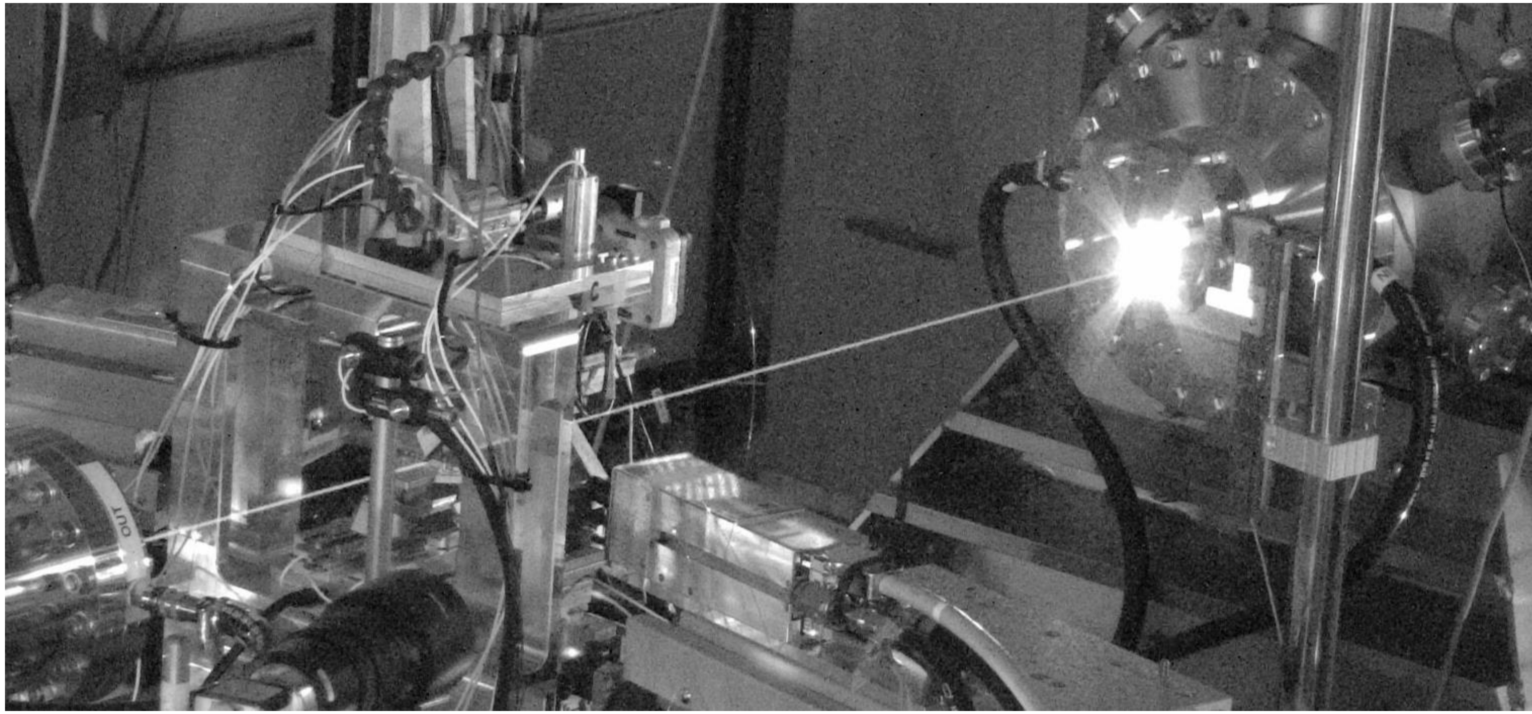
Reflections on AGIPD
a) Cr(002) reflection, b) CDW (002-2δ), where δ=1/27 lattice constant

T_{min} ~ 5K
B_{max} ~ 5T, soon 15T (safety issue…)

K. Kazarian's PhD project
PUMA developed with J. Moore (sample env group)

Conclusions

- Versatile instrument for time-resolved coherent X-ray scattering and imaging
- Beamsize ~250 nm – 1 mm
- 5 - 25 keV
- Windowless, in-vacuum or in-air setup
- MHz integrating pixel detector or high resolution CCD (Hz)
- Optical fs pump laser
- X-ray split-delay line
- Various X-ray optics and beam diagnostics
- Various sample environments



MID, April 2019
2000 pulses/s
9 keV, 1.7 mJ/pulse

MID
Materials Imaging and Dynamics

- And our accelerator team, experts groups and users…