

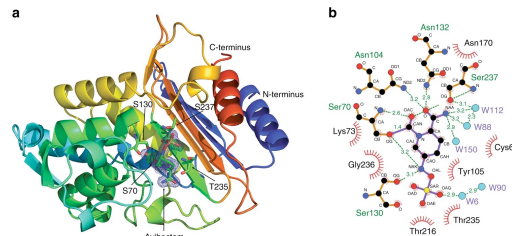
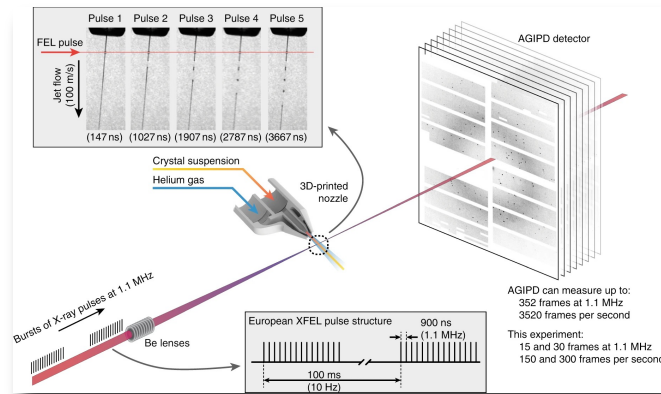
SPB/SFX Update

Adrian Mancuso
SPB/SFX
Leading Scientist

Schenefeld, 12 May 2022



Reminder: Science cases at SPB/SFX

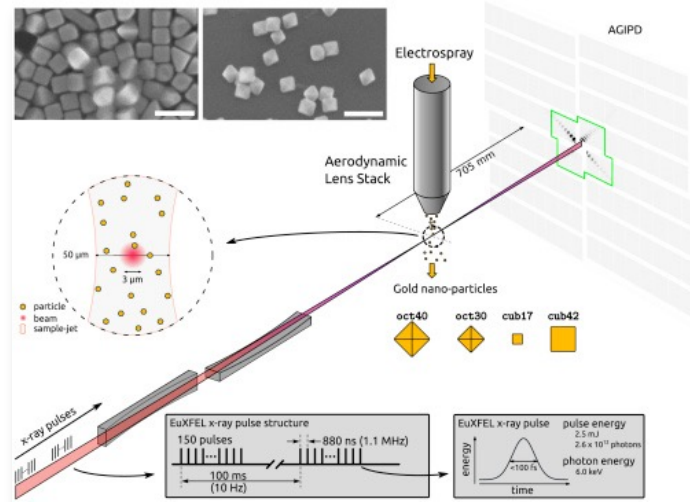


Serial Crystallography

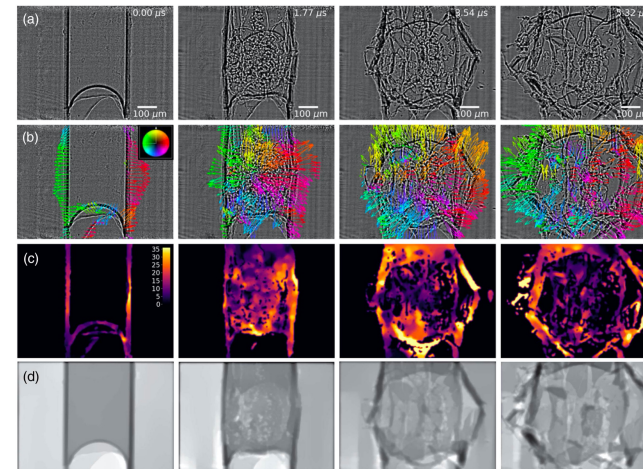
Small Angle Scattering

IN PROGRESS

Includes time-resolved studies



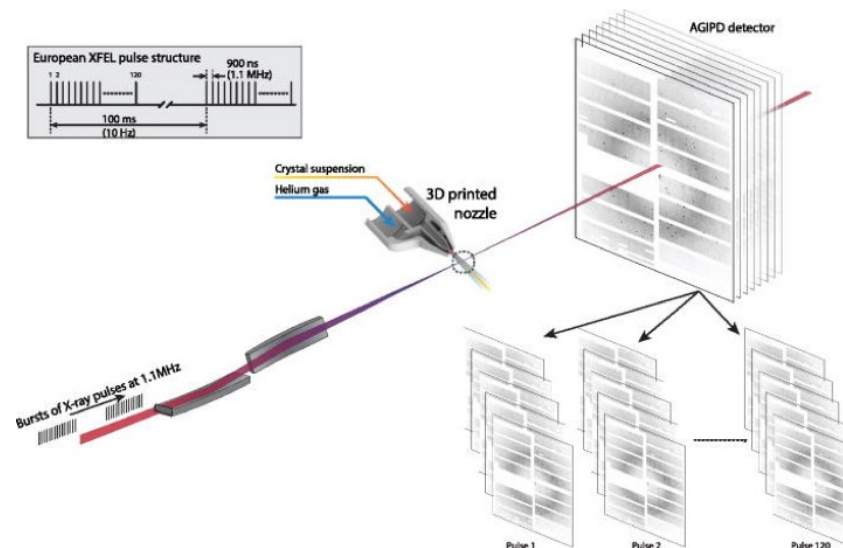
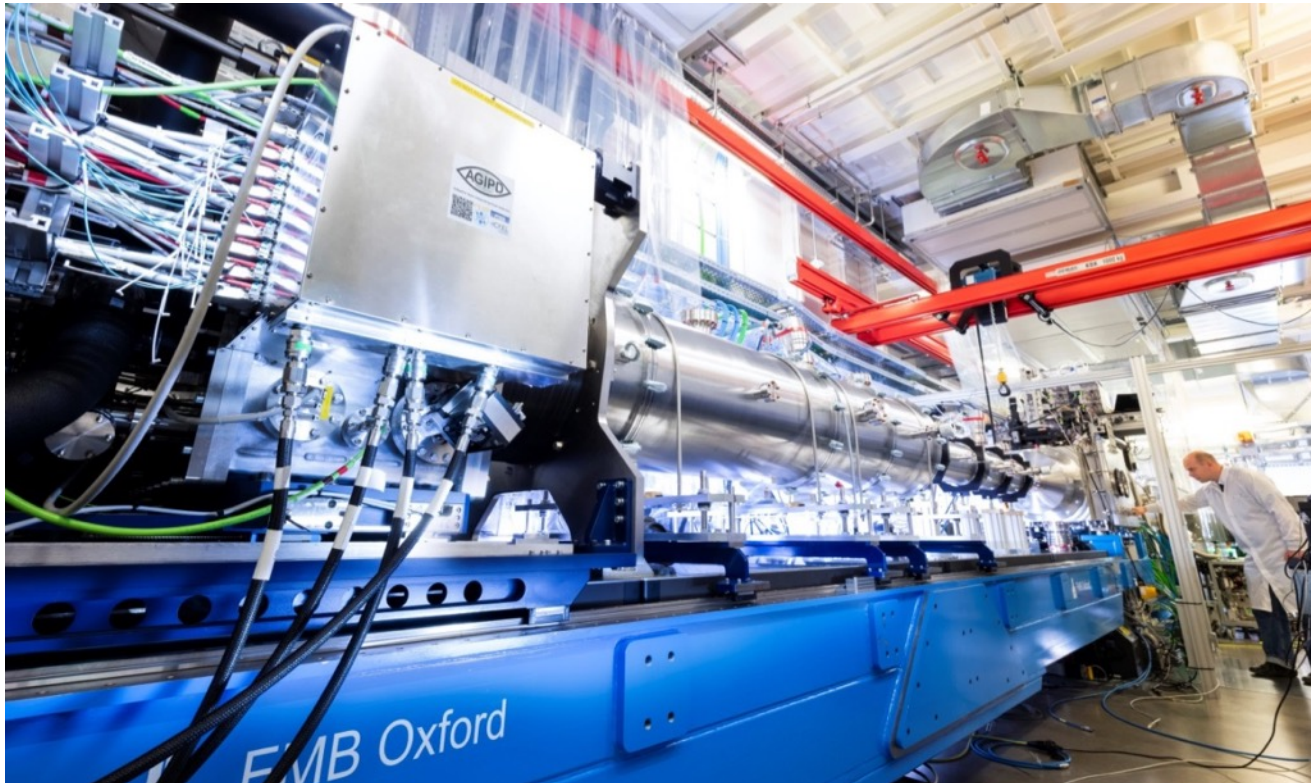
Single Particle Imaging



Megahertz microscopy (up to 24 keV)

Reminder: SPB/SFX Instrument layout

- < 6 keV to ~15 keV
- ~3 μm and 300 nm spot sizes
- 1 Mpx AGIPD
- MHz rep rate capable
- Optical pump laser
- Timing tool & more...

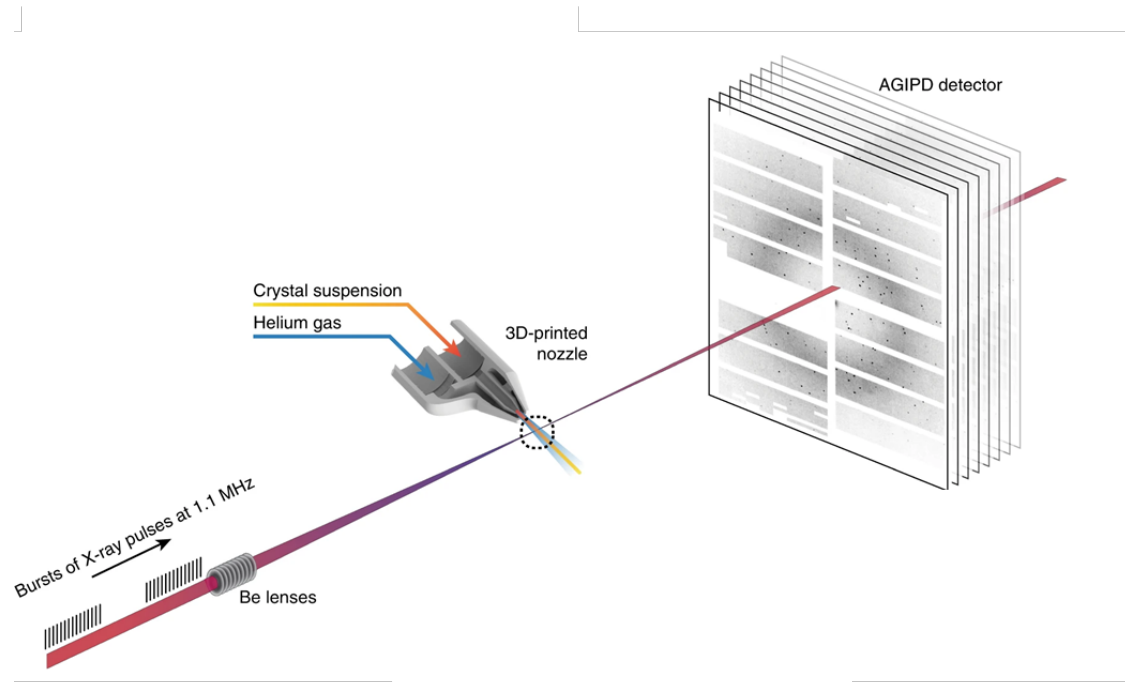


• Mancuso et al., The [SPB/SFX] instrument at the European XFEL: initial installation, *Journal of Synchrotron Radiation*, 26, pp. 660-676 (2019)

Sample delivery for SFX – 3D-printed Gas Dynamic Virtual Nozzles (GDVNs)

Standard GDVN

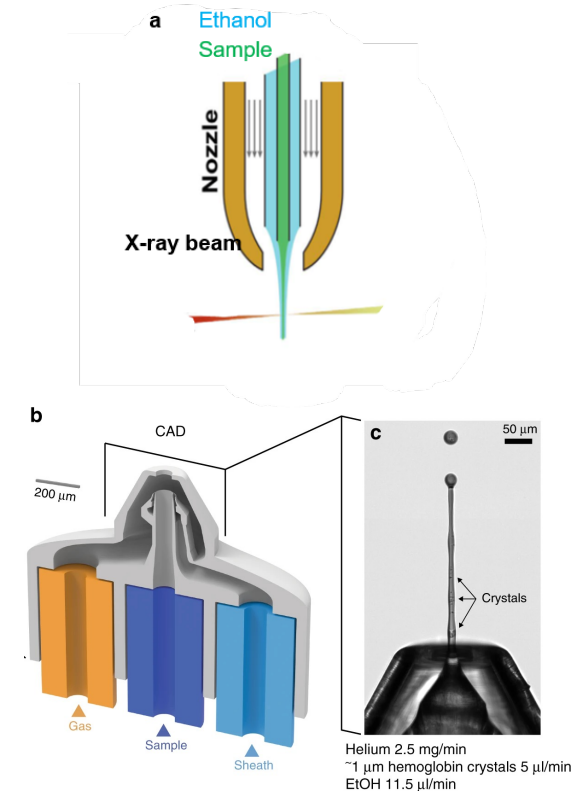
- Sample (crystal suspension) is focused by Helium gas



Modified from Wiedorn *et al* (2018). Nat. Commun. 9, 4025.

Double-flow focusing nozzles (DFFN)

- Outer jet (Ethanol) focused by Helium stabilizes inner jet (Sample)



Oberthuer *et al* (2017) Scientific Reports 7:44628
 Knoska *et al* (2020). Nat. Commun. 11, 657.

Still pretty new: Protein crystal screening (PCS) beamtimes at SPB/SFX

- Two step procedure with users on-site
 - 1. part: Injection tests in the user labs
 - 2. part: Beamtime at the SPB/SFX instrument (~3 hours)
- In case sample is not jettable, sample will be considered for PCS beamtime in the next run
- Injection performed and nozzles (GDVN and DFFN) provided by SEC Group
- Data collection performed by SPB/SFX group with standard parameters
- Simplified proposal form
- Probably a better chance of beamtime than a regular proposal
- For further information, please contact Katerina Dörner (SEC) prior to proposal submission:
katerina.doerner@xfel.eu

Available parameters

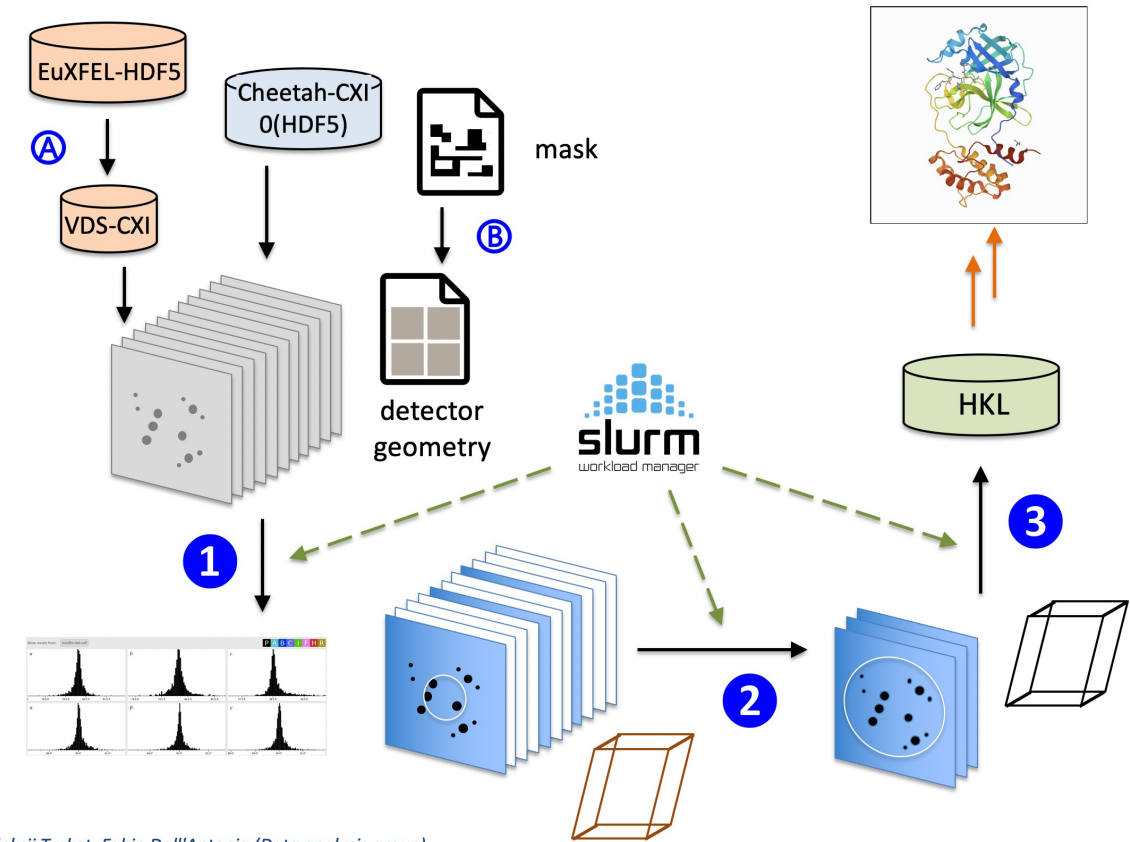
Photon beam parameters		
Photon energy	6 - 12 keV	Up to 15 keV potentially available
Pulse energy	≥2 mJ	Typical at 9.3 keV
Photons per pulse (at source)	~1 x 10 ¹²	Derived from previous two fields (@ 9.3 keV)
Pulse duration	25 fs	Estimated
Focal spot size (FWHM)	~ 3 μm < 200 nm	Two KB mirror systems available
Photons / μm ² (at sample)	> 10 ¹⁰	Derived. Includes abs, expected spot size range.
Train repetition rate	10 Hz	
Intra-train repetition rate	1.1 MHz	(4.5 MHz, 100 kHz, some quasi-arbitrary patterns)
ΔE/E	~0.2%	Estimated
No. of bunches per train	≤352	Some quasi-arbitrary patterns possible.
Sample delivery systems: In vacuum (upstream, 1 Mpx AGIPD) and in-helium (downstream, roadrunner, Jungfrau 4M)		
Liquid jet injector rod	½" nozzle rod with M9x1 mm fine thread nozzle mount compatible with the CXI nozzle rod at LCLS (MPI design), 1200 mm in length. Additionally, 25mm nozzle rod with M23 fine thread.	
Sample injection nozzles (GDVN and DFFN)	3D printed nozzles to produce μm-sized liquid jets. Other nozzle types also possible. Nozzles can be supplied by the SEC group. Please consult with the SEC group prior to proposal submission.	
High viscosity liquid jet	Mounted on nozzle rod. ASU or EuXFEL design	
Aerosol injector	Aerosol produced by electrospray. Other nebulizers also possible	
Fixed target sample holder	Various available. Please consult with instrument scientists prior to proposal submission.	
Pressure systems	HPLC pumps, syringe pumps, gas-pressurised sample reservoirs	

Please contact us for further details:
spb.sfx@xfel.eu

AGIPD 1 Mpx detection properties		
Number of pixels	1024 x 1024	4 quadrants, each 512 x 512 pixels
Pixel size	200 μm x 200 μm	
Minimum sample–detector distance*	~129 mm	Maximum 200 mm stroke
Resolution at edge @ 9.3 keV	< 2 Å	At minimum distance from sample
Max sample-detector distance	~ 5.5 m	
Hole size	8 mm. Possibly ~5 mm—large	

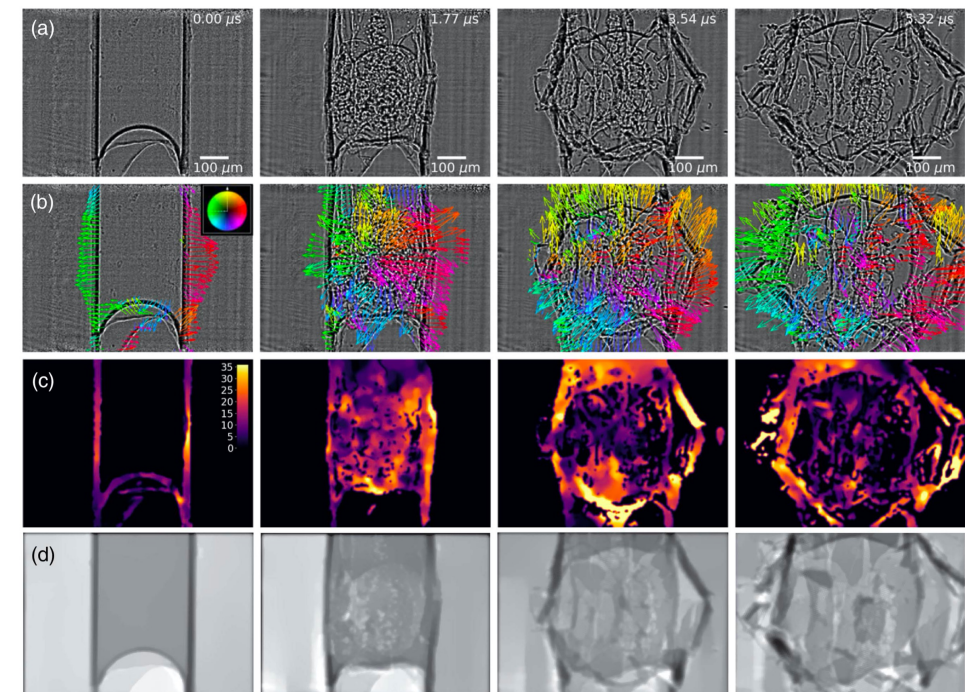
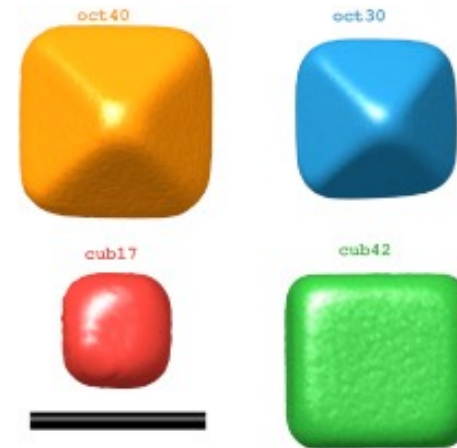
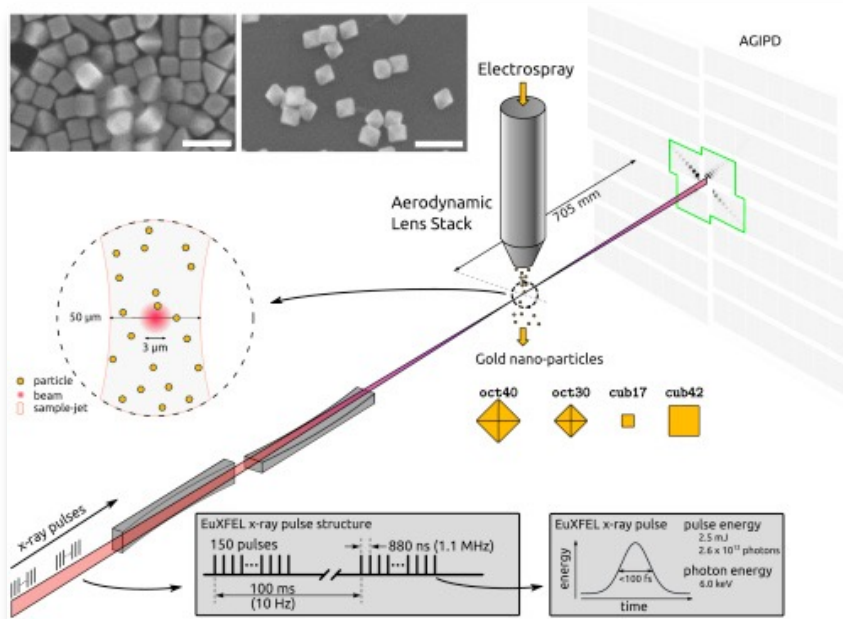
Semi automatic SFX pipeline

- Starting from HDF5 data sets in EuXFEL or Cheetah/CXI format, diffraction images are processed in 3 steps using CrystFEL tools, embedded to a workflow with SLURM interface for distributed computing.
- (1) Initial crystallographic peak-finding and indexing of all detector images, followed by graphical determination of a crystal unit cell.
- (2) Peak-finding and indexing in a low-scattering-angle detector area using the preliminary unit cell, followed by selection of the indexable image subset ("crystal hit frames") and unit cell refinement.
- (3) Peak-finding, indexing and pixel intensity integration at predicted positions on a high-scattering-angle area using only the diffraction image subset, plus the refined unit cell. Crystallographic scaling and intensity averaging yields a unique reflection data set, suited to reconstruct the macromolecular structure (not yet part of the pipeline).
- Preparative steps like (A) automatic conversion of EuXFEL data to the required CXI format in a "virtual" data set or (B) optional import of pixel masks into the detector geometry description file are also supported.



Oleksii Turkot, Fabio Dall'Antonia (Data analysis group)

SPI of material nanoparticles & MHz microscopy also available



Single Particle Imaging of material NPs, also TR

Megahertz microscopy (up to 24 keV)

Headline conclusions & contact details

- Low viscosity SFX of small crystals and time-resolved systems (optical pump or mixing) becomes more and more robust
 - Still requires some volume of sample for large data sets
 - Crystal screening offers a lower barrier to entry
- Other experiment modalities are also increasing in robustness and throughput
- Don't hesitate to contact us to discuss your proposal ideas

https://www.xfel.eu/facility/instruments/spb_sfx/index_eng.html

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- spb.sfx@xfel.eu
- katerina.doerner@xfel.eu for screening proposals



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