SAMPLE ENVIRONMENTS AT A SYNCHROTRON

Part 2: Sample Environments at MAX IV

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ISSE

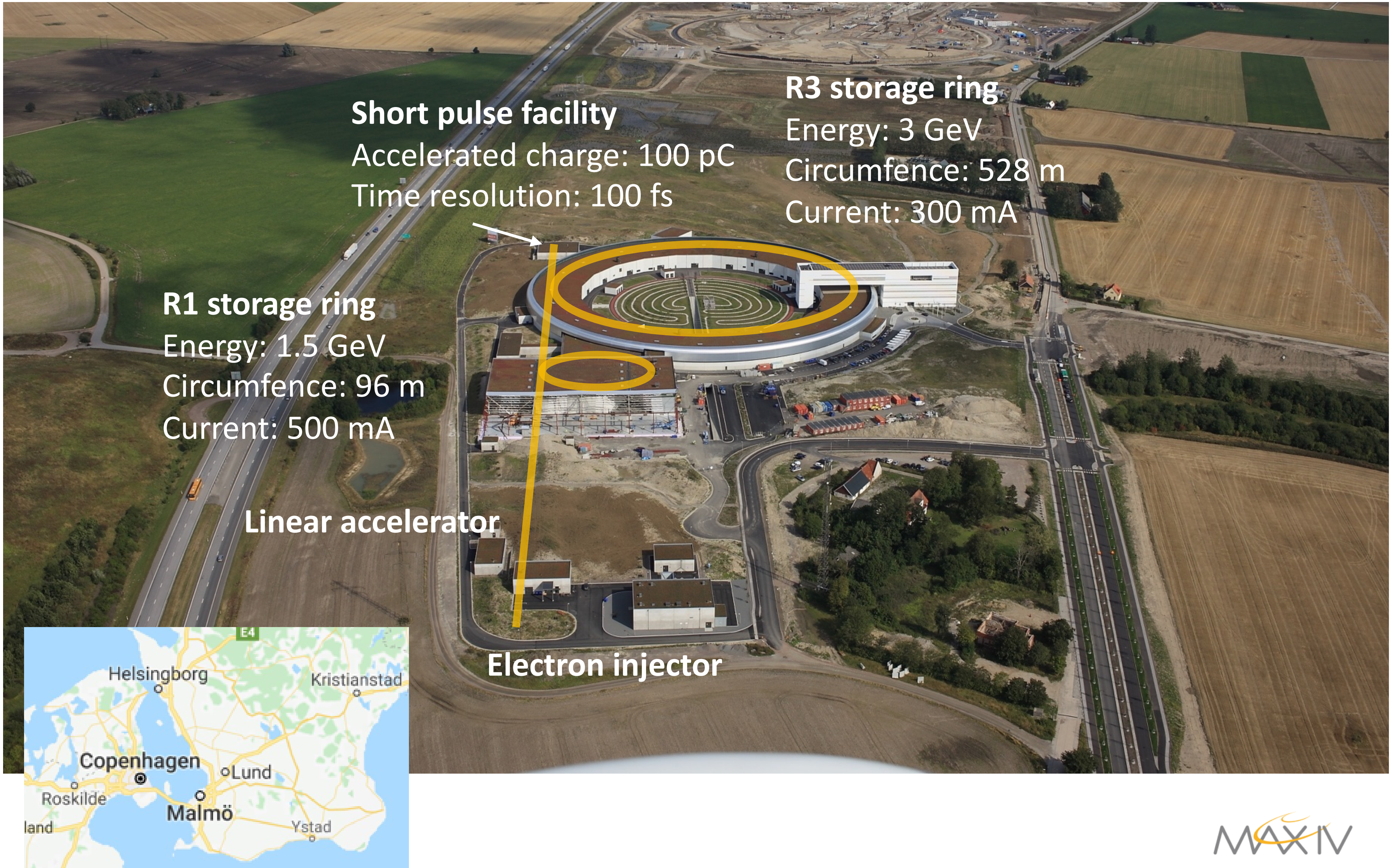
School on Synchrotron Light Sources and their Applications | (SMR 3815), 26 January 2023

MAX IV

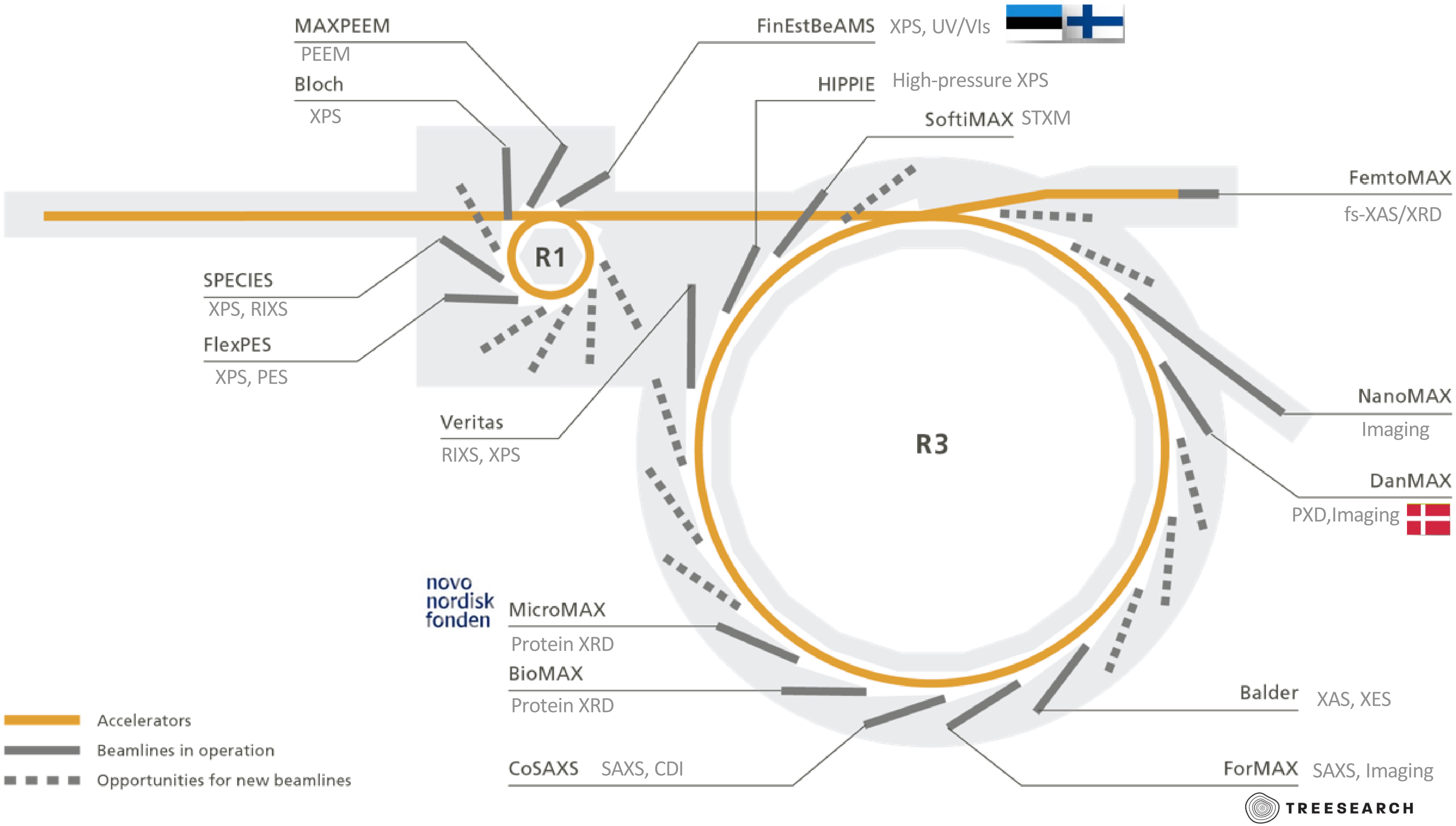
Overview

- Introduction to MAX IV
- Sample-environment support at MAX IV
- Standard sample holders
- Examples
- Conclusion

The MAX IV Laboratory



MAX IV beamlines today



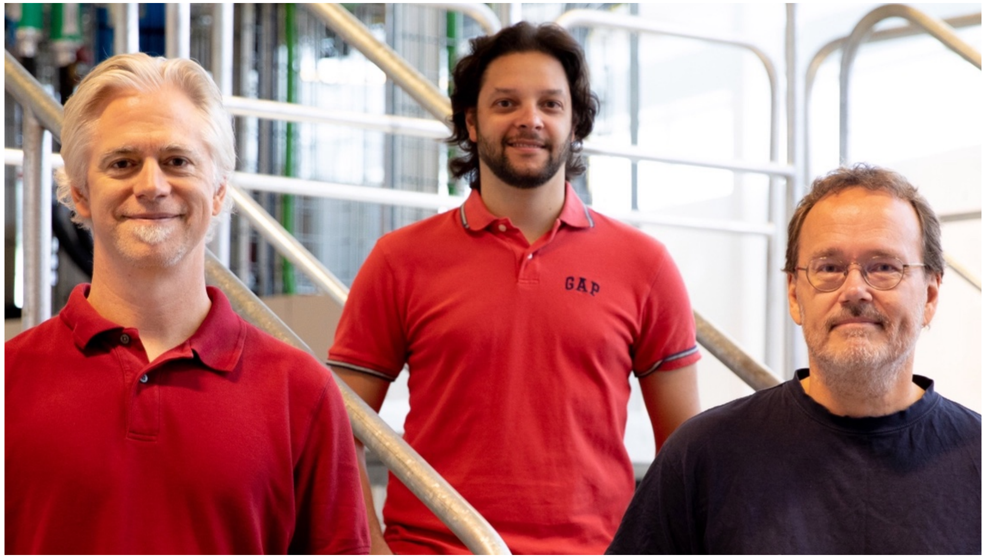
"4th generation" low-emittance storage ring

16 beamlines covering energies from 4 eV to 40 keV.

Sample-environment support at MAX IV

Sample Environment and Detector Support (SEDS)

SEDS support experiments with sample environments, instrumentation, consumables, and support labs.



Left to right: Chris, Artur, and Stefan

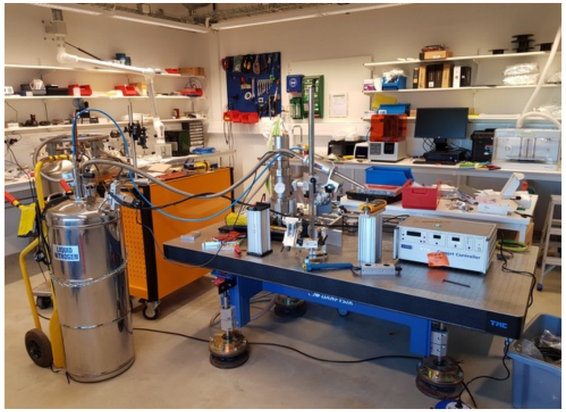
The SEDS team

Stefan Carlson	Team leader
Artur Domingues	Instrumentation and fast prototyping
Christopher Ward	Detector support
Vacant position	Installations and optics support

MAX IV

Support labs operated by SEDS

Sample environment lab



Furnace, cryo, assembly

Test and detector lab

Detector test, X-ray test station

Fast prototyping lab

3D printers, milling, laser cutter

Beamline workshop

24/7: 3D-printers, Drilling machine, Tools

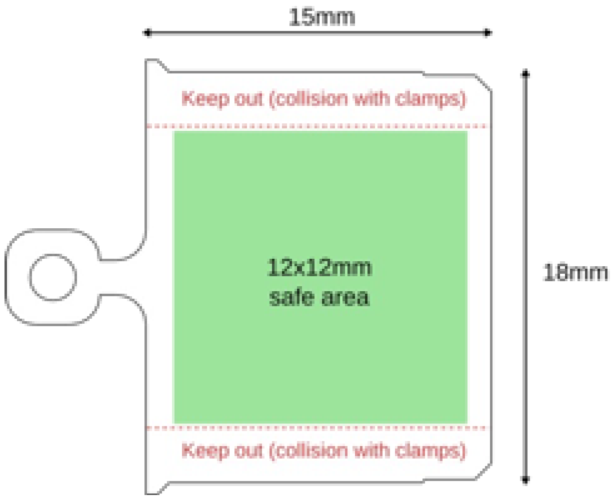
D3 Preplab

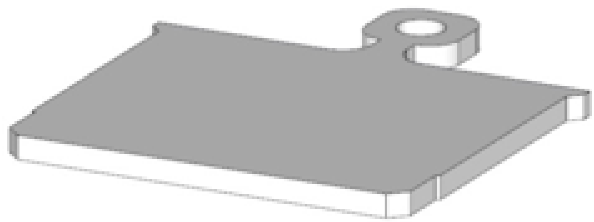
Preparation tools for soft X-ray beamlines

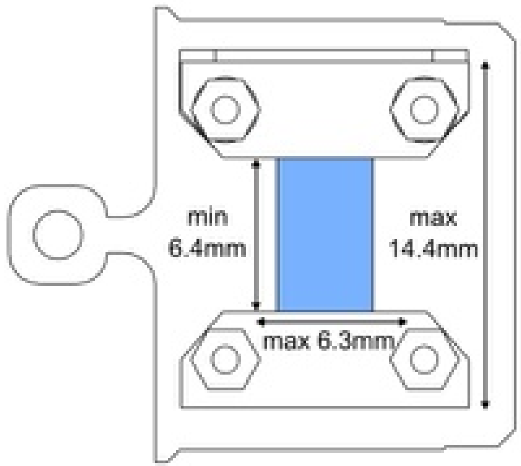
Standard sample holders

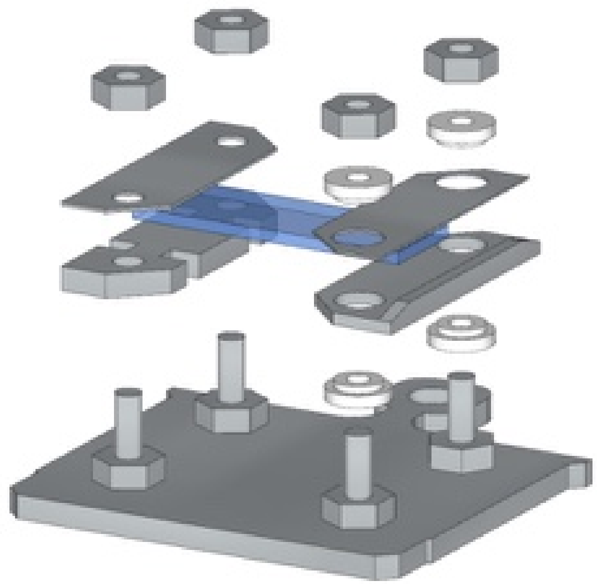
Sample-holder standards at MAX IV – Soft X-rays/UV

Soft X-ray (< 2 keV) surface science: "Flag-style" Omicron sample holder





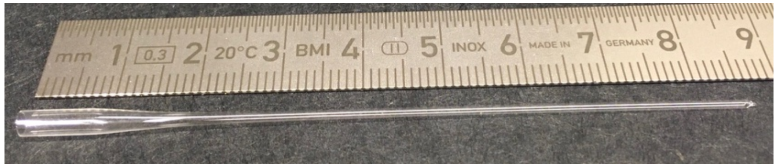




Sample-holder standards at MAX IV – Hard X-rays

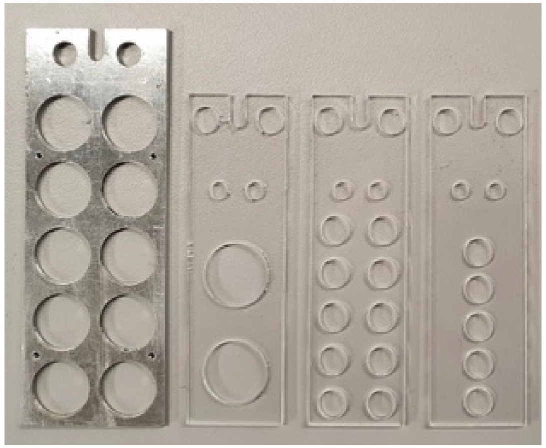
Hard X-ray (> 2 keV) experiments: Several standards for different techniques.

Powder XRD: Capillaries



Crystals (XRD): Glass pins, “Spine” pins

XAS: Frames, tape, capillaries

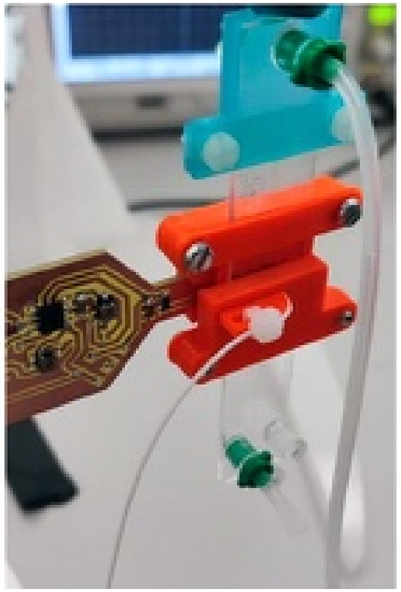


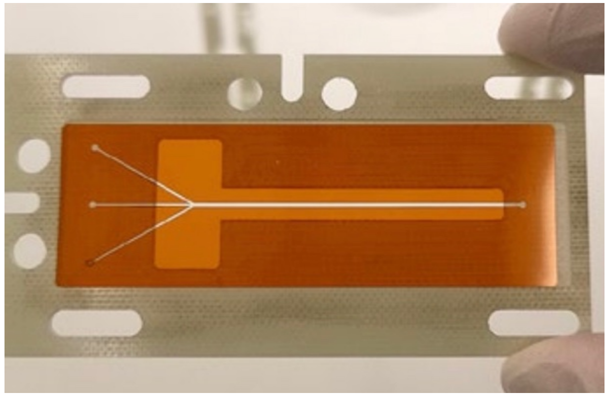
Imaging with nano beams: TEM grids

Examples

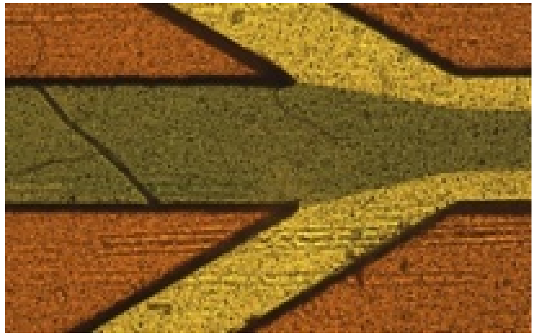
Microfluidics

Project at Balder (XAS) and CoSAXS (SAXS):
Adaptable microfluidic flow-cell platform for sample delivery and fluid mixing.
Create a protective buffer layer around the protein solution by “flow focusing”.
20 – 300 µm channel diameters.

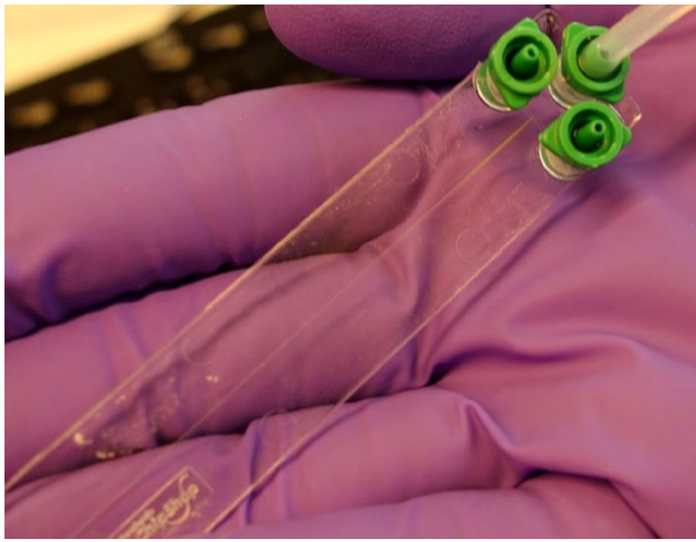




Setup used in lab and at beamlines.



Flow focusing



Commercial microfluidic cell used in the cell platform.

Protein crystallography at BioMAX and MicroMAX

Samples are mounted on "Spine" pins

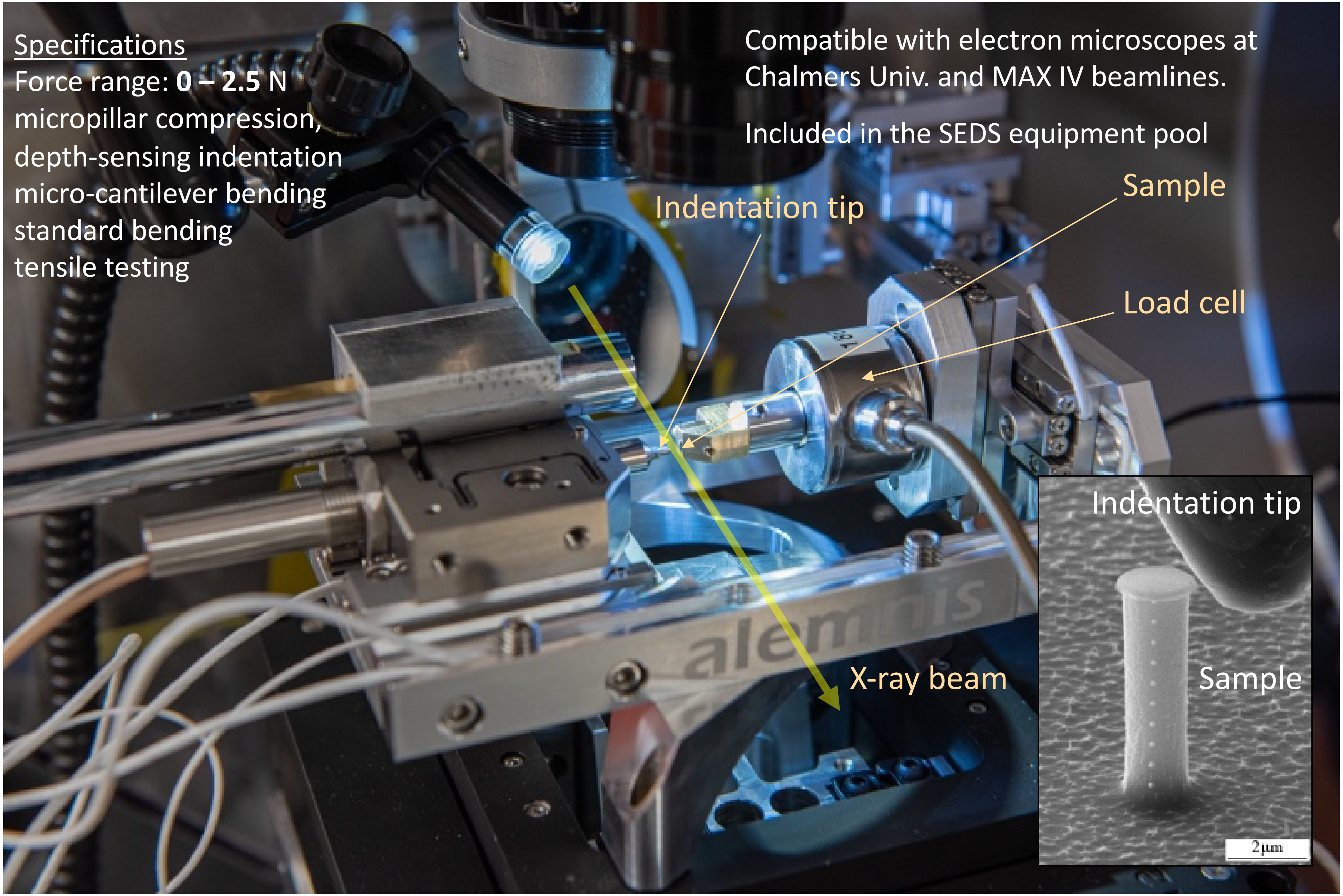
Multiple samples are stored under liquid nitrogen in "Unipucks"

Automatic sample transfer to diffractometer with a robot.

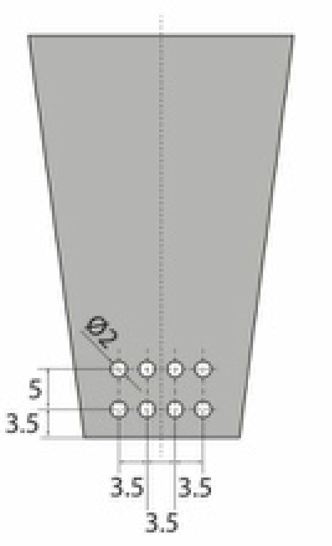
<u>Current development area at MicroMAX is in serial crystallography.</u>

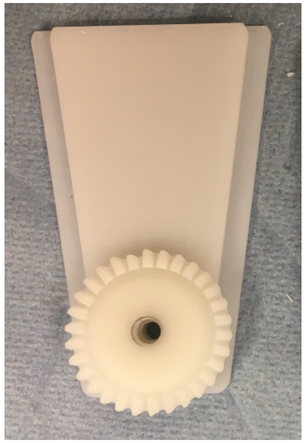
- Many micro-crystals are injected one by one in the X-ray beam.
- Micro- to millisecond exposures are made and complete data sets are stitched together.
- Prevents radiation damage and allows structure determination from micro-crystals.
- Allows room temperature measurements and controlled reactions.

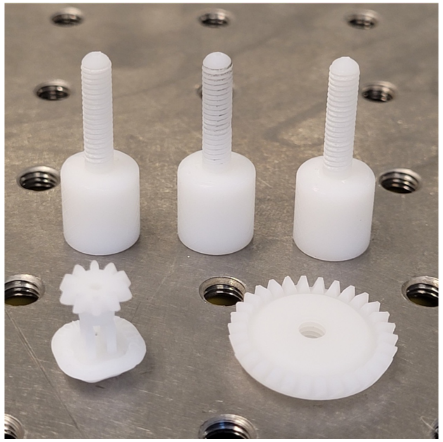
Nanoindentation at NanoMAX



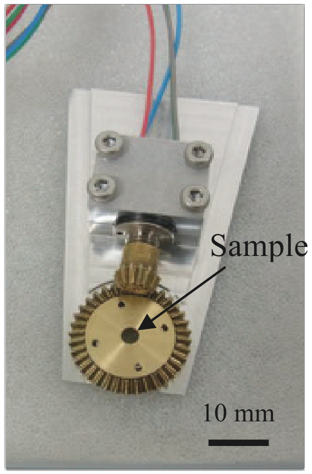
Sample holders for SoftiMAX (STXM)



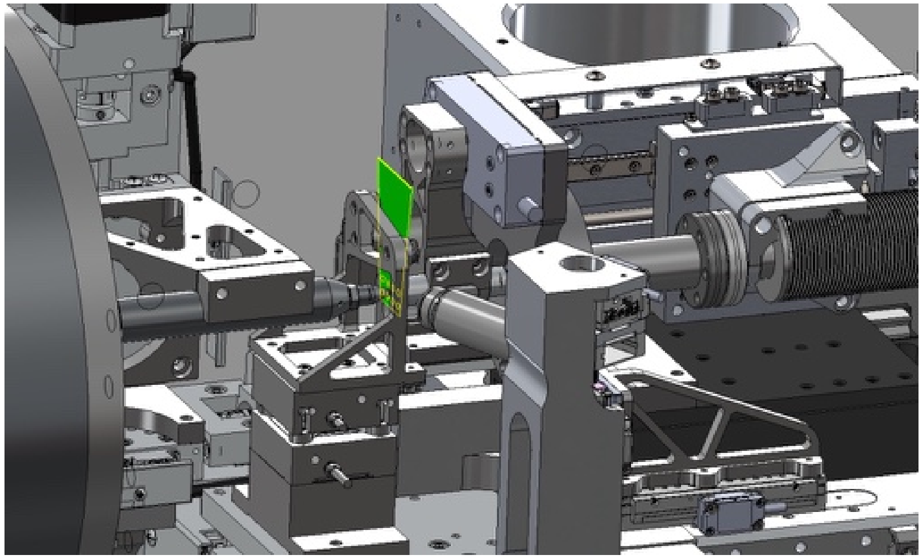




3D printed holder with azimuthal rotation



Takuji Ohigashi et al., AIP Conference Proceedings 1741, 050002 (2016)

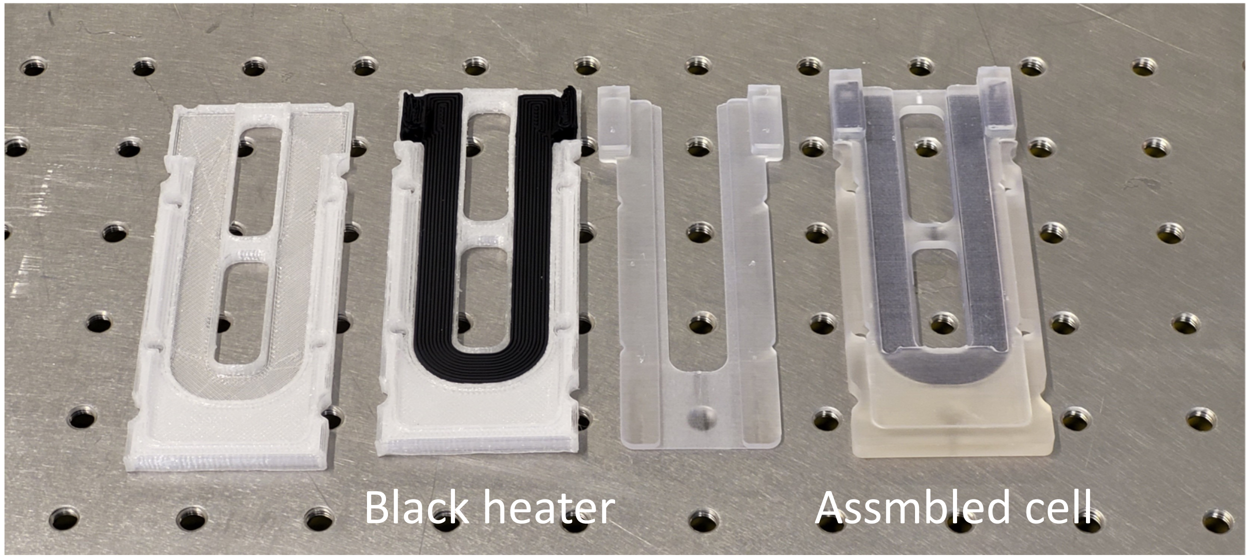


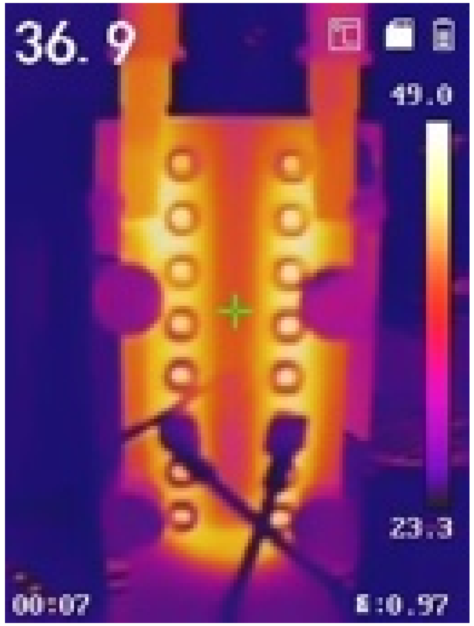
SoftiMAX experiment station. Sample holder marked in green.

Fast prototyping for CoSAXS

SAXS measurements on human-tissue material

Temperature at the sample must be controlled at 37°C





Thermally stabilized cell printed out with electrically conducting plastic heater

<u>Humidity cell project.</u>

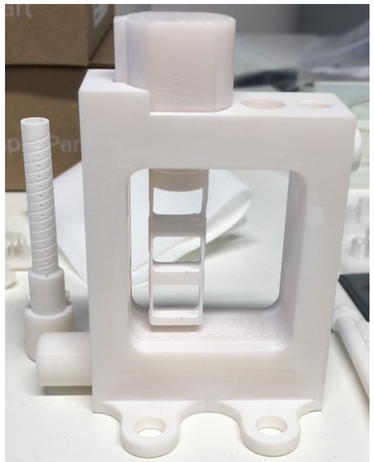
Collaboration between CoSAXS, SEDS, and Malmö University.

Integrated heater inside the sample chamber.

Humidity sensor feedback to humidifier system.

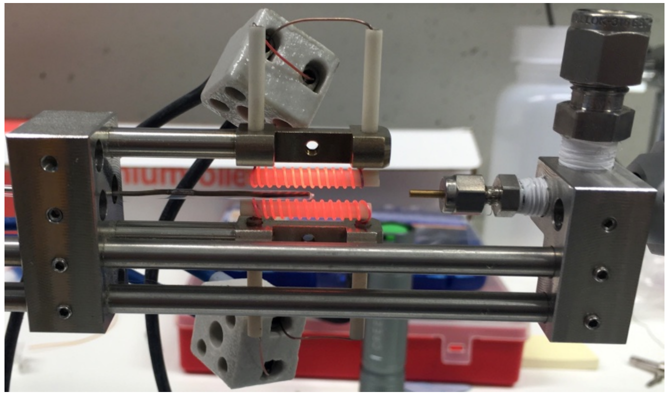
The cell design in inspired by P.A. Pentillä, Cellulose (2020) 27:71–87.

Initial 3D drawings was kindly provided by Peter van der Linden, ESRF.

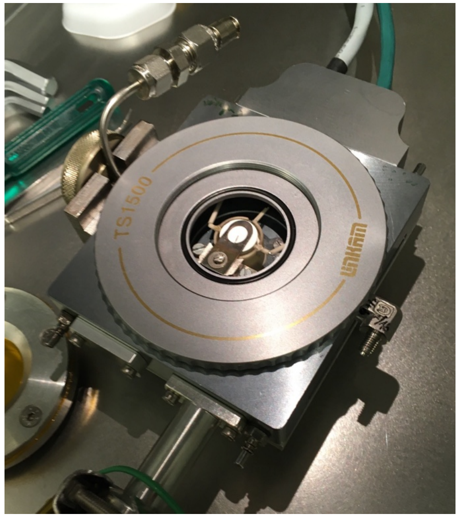


In-situ reaction cells

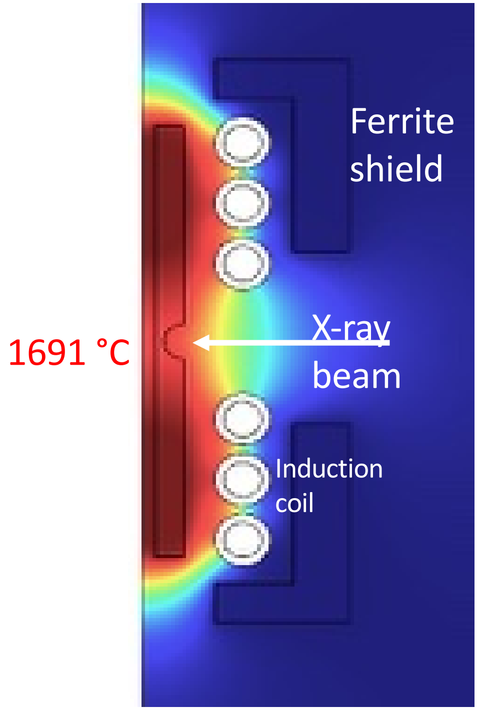
Capillary cell

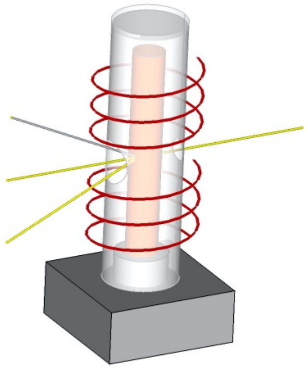


Linkam furnace

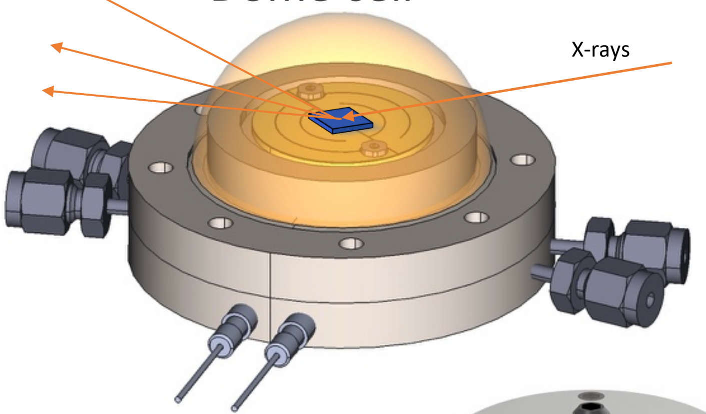


Induction furnace





Dome cell



MAX IV catalysis cell

Diamond-anvil cells

Conclusion

Sample environments are becoming more complex (temperature, pressure, reactive gases and liquids, humidity, magnetic fields, strain/stress…)

Sample environments are becoming more compact due to smaller beam sizes and more weight-sensitive experiment stations.

3D-printing is a great tool in the development of sample environments (fast prototyping).

Laser micromachining and focused ion-beam etching are also becoming more common to prepare samples.

Thank you for your kind attention!

More info on www.maxiv.lu.se

MAX IV