Australian Government

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Anything goes: SAXS/WAXS at the Australian Synchrotron

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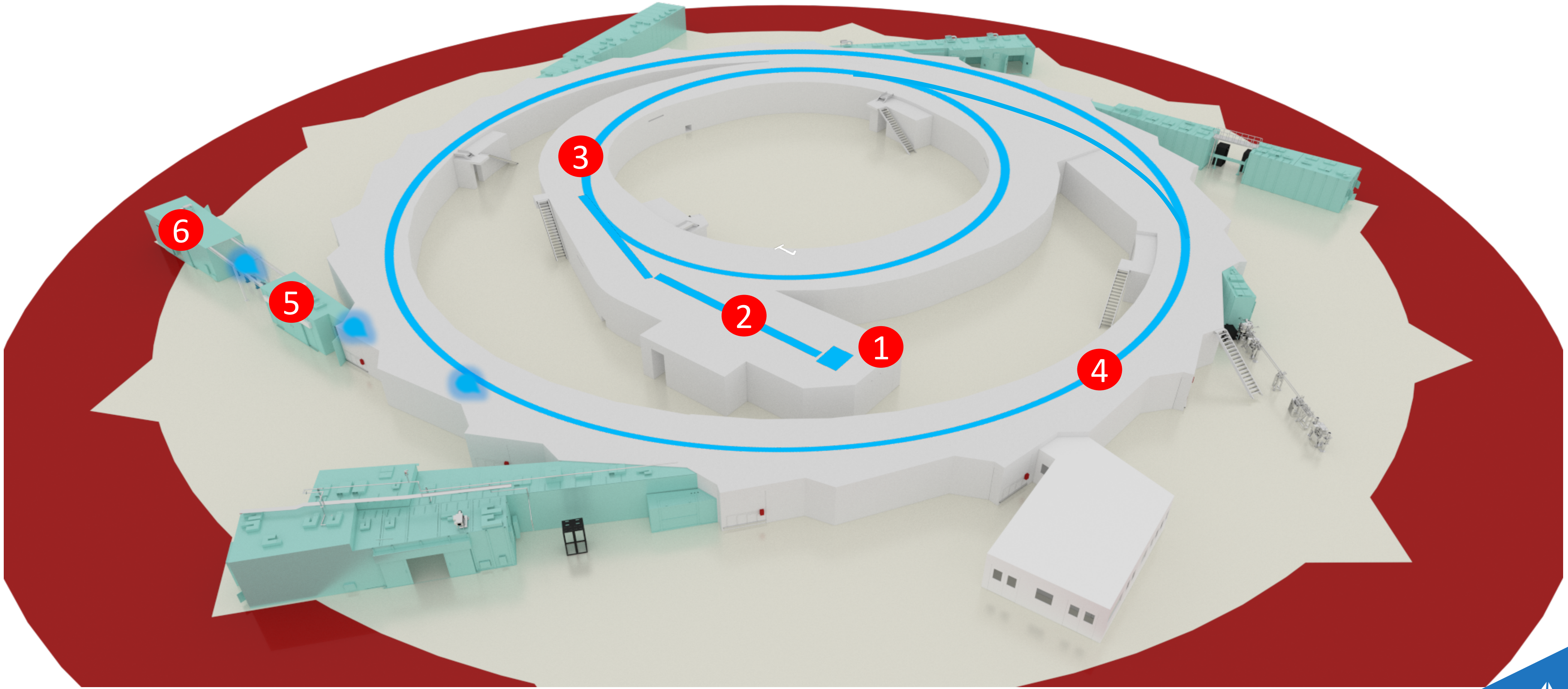
Beamline Scientist

SAXS / WAXS Beamline

Science. Ingenuity. Sustainability.

Australian Synchrotron – light source

1. Electron Gun
2. Linear Accelerator
3. Booster ring
4. Storage ring
5. Optics
6. Beamline



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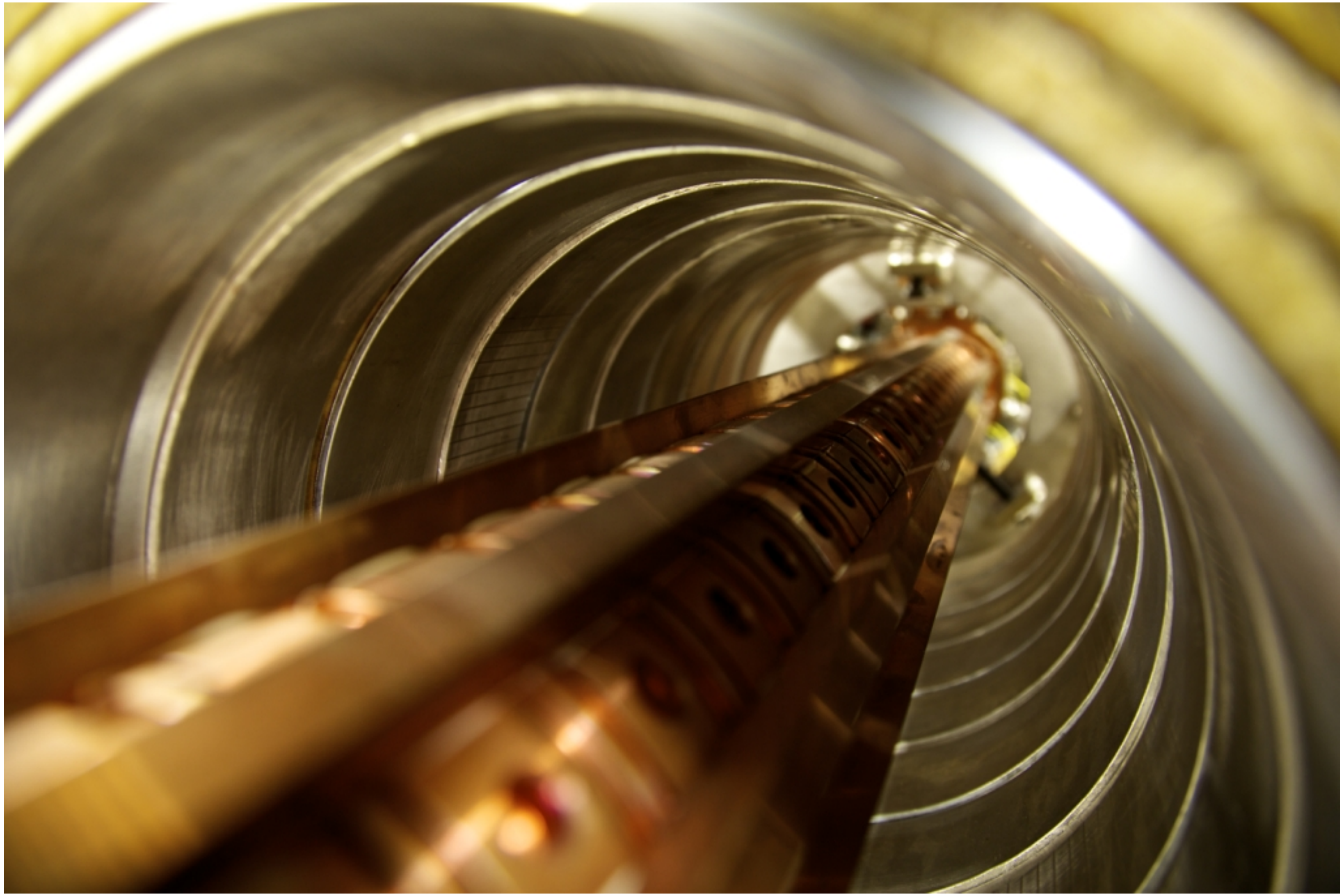
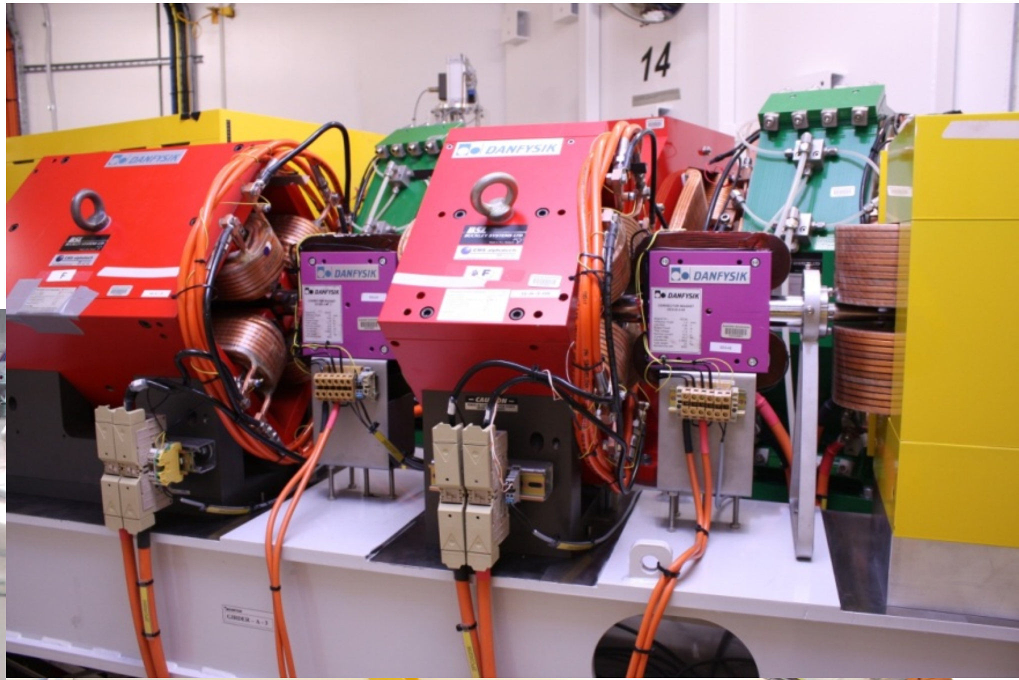
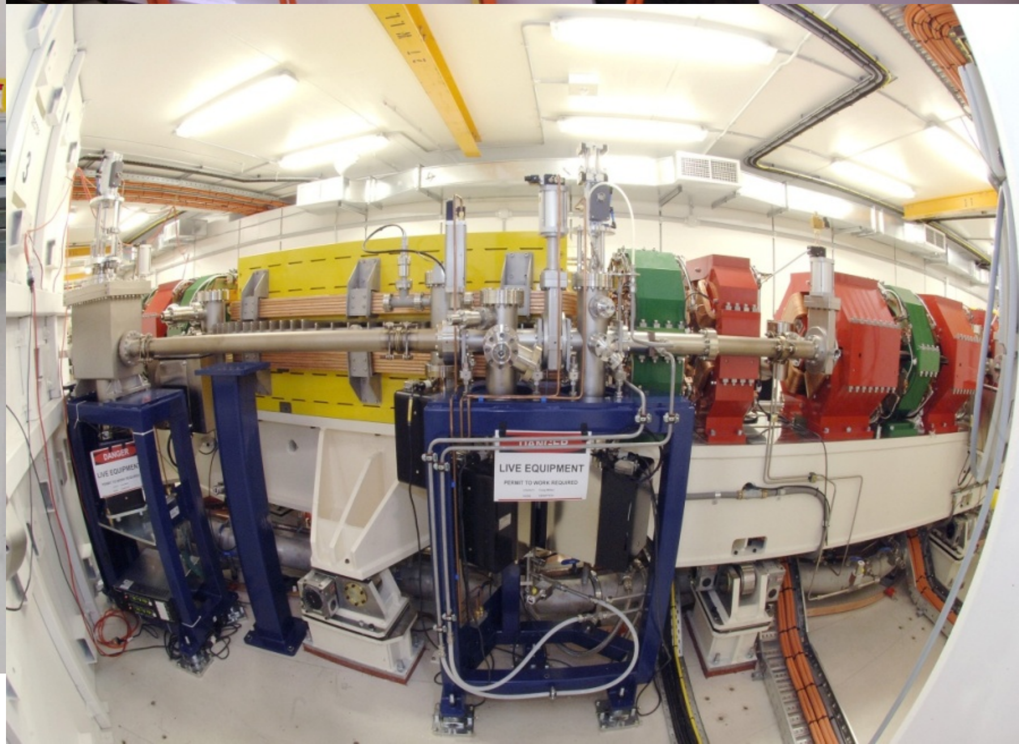
The eye of the … Synchrotron

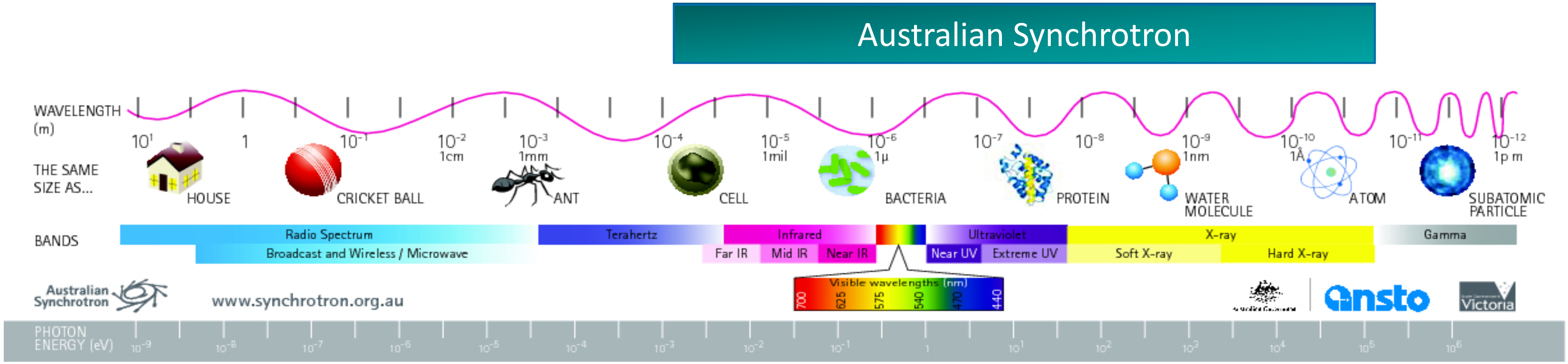


Storage and Booster Ring





What type of light do we make?



Longer Wavelengths

Big things

Shorter Wavelengths

Small things

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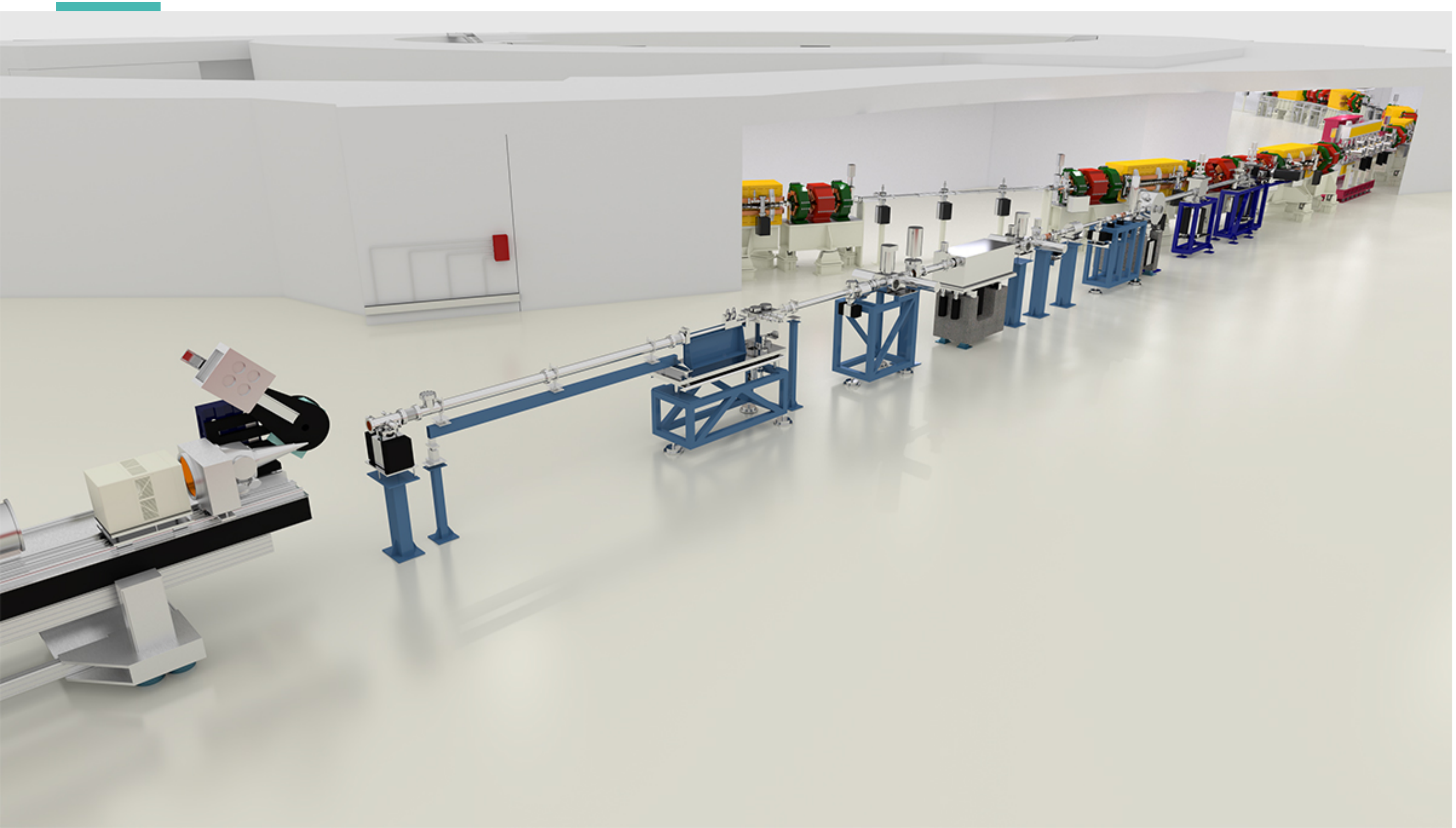
From the ring to the beamline



Beamlines at the Synchrotron

Current Beamlines

X-Ray Fluorescence Microscope

Macromolecular crystallography
Micro crystallography

Powder Diffraction

Small and Wide Angle Scattering

X-Ray Absorption Spectroscopy

Soft X-ray Beamline

Imaging and Medical Beamline

Infrared Microscope
Far Infrared

New Beamlines on the way!

Medium Energy X-Ray Absorption Spectroscopy

Micro Computed Tomography

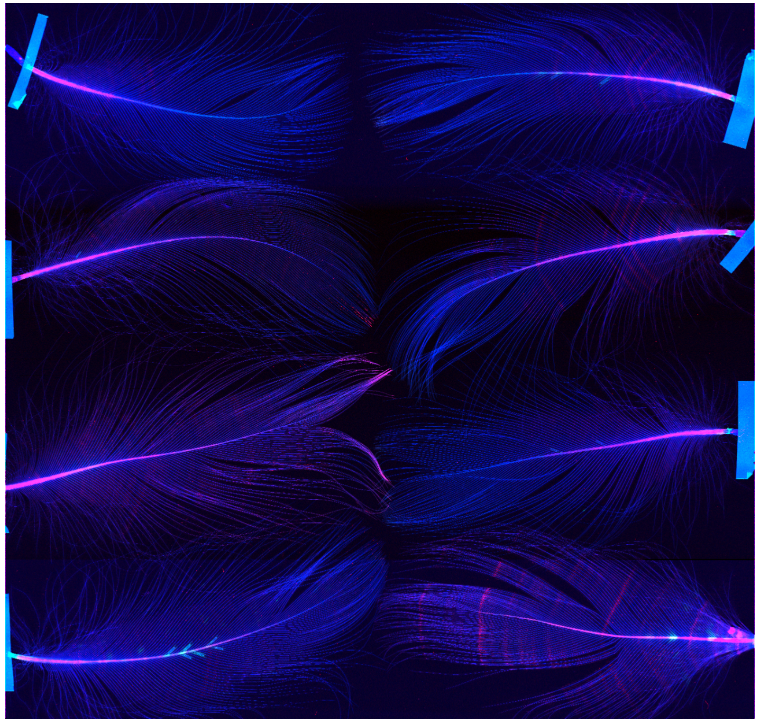
Advanced Diffraction and Scattering

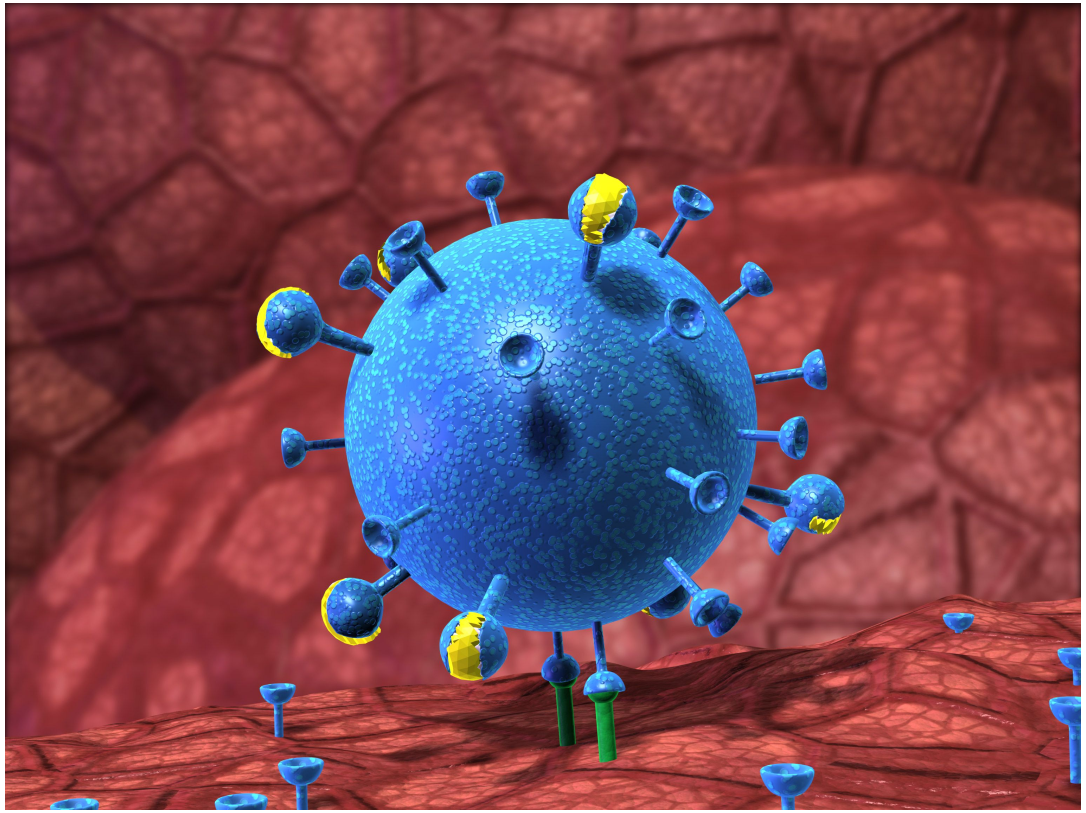
Biological Small Angle Scattering

X-Ray Fluorescence Nanoprobe

Micro Materials Characterisation

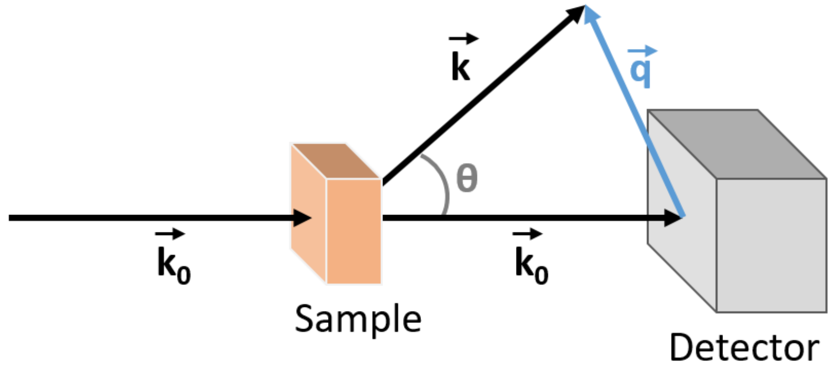
What can we do with it?

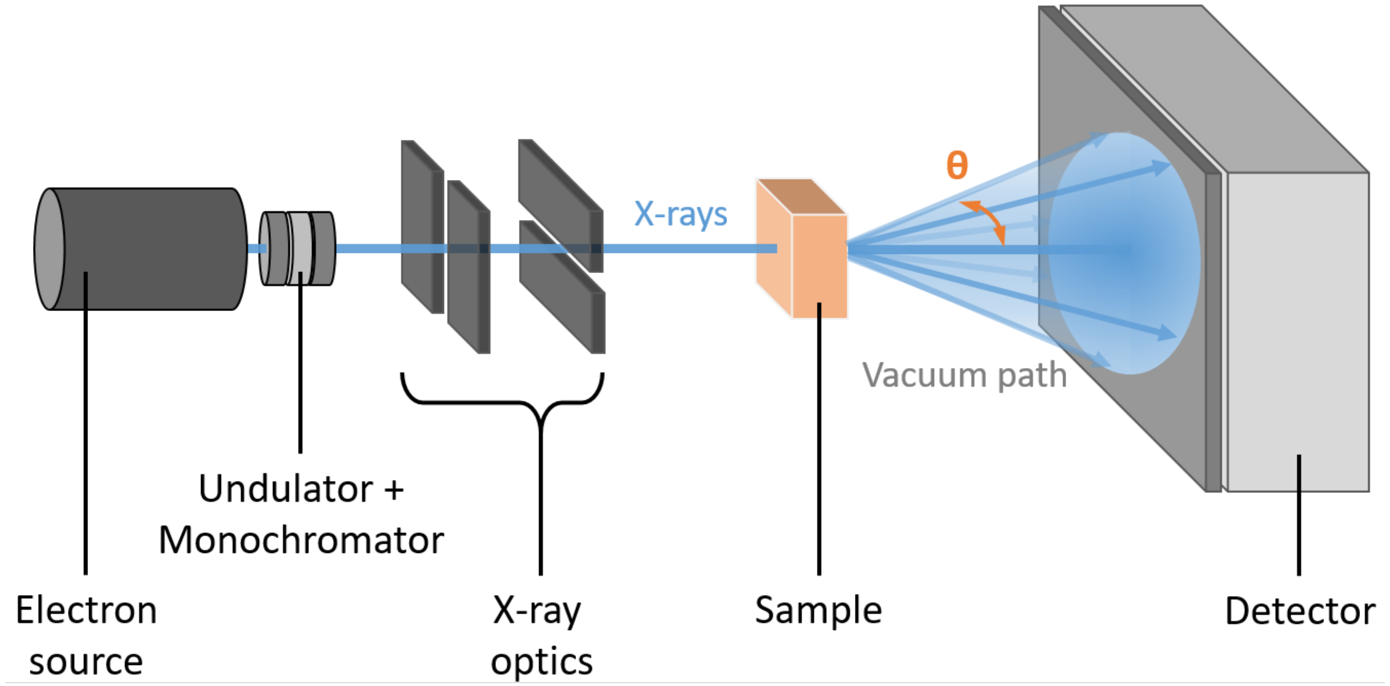


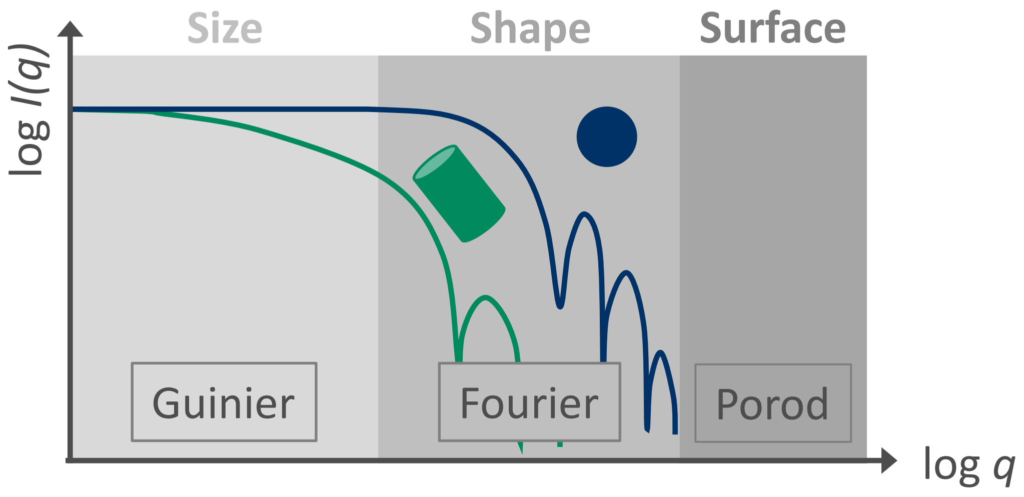


SAXS / WAXS

Small- and Wide-Angle X-ray Scattering







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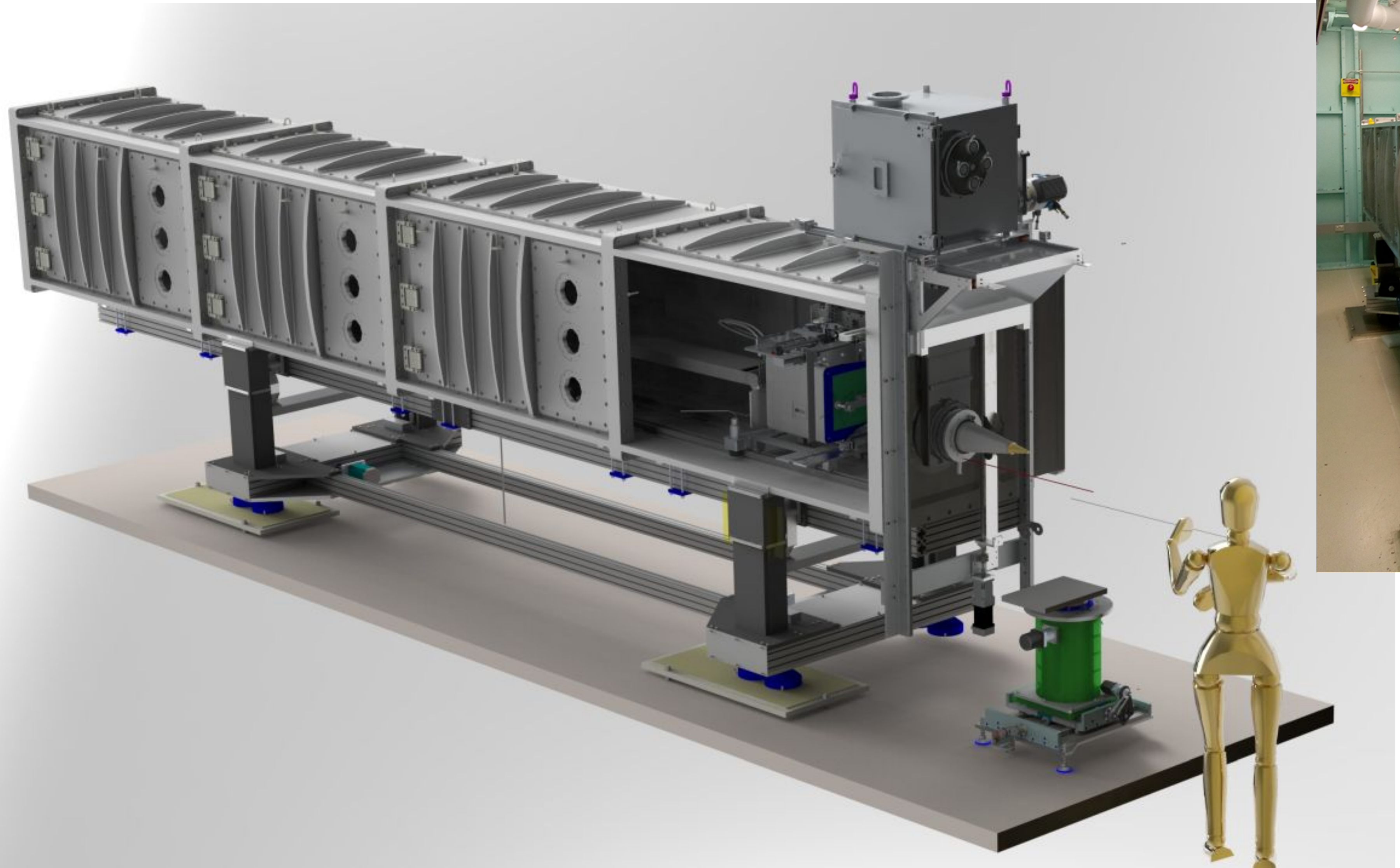
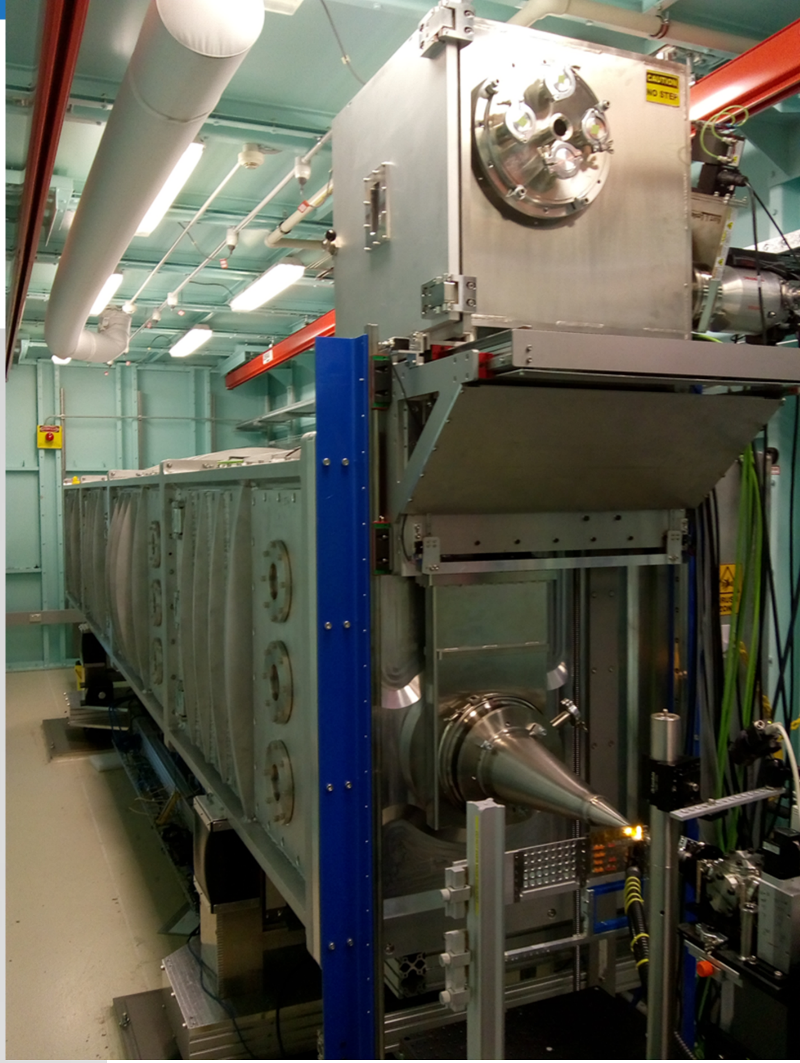
The instrument





Why use Synchrotron SAXS?

Stealing the Olympic Motto: “Citius, Altius, Fortius” & “Minus”

Faster
- Much faster than lab-based SAXS
- 1 second per data set
- Enables time-resolved studies
- High-throughput
- Versatile

Higher
- Higher X-ray flux
  - 500,000 x
- Higher scattering angle range (q-range)
- Higher energy

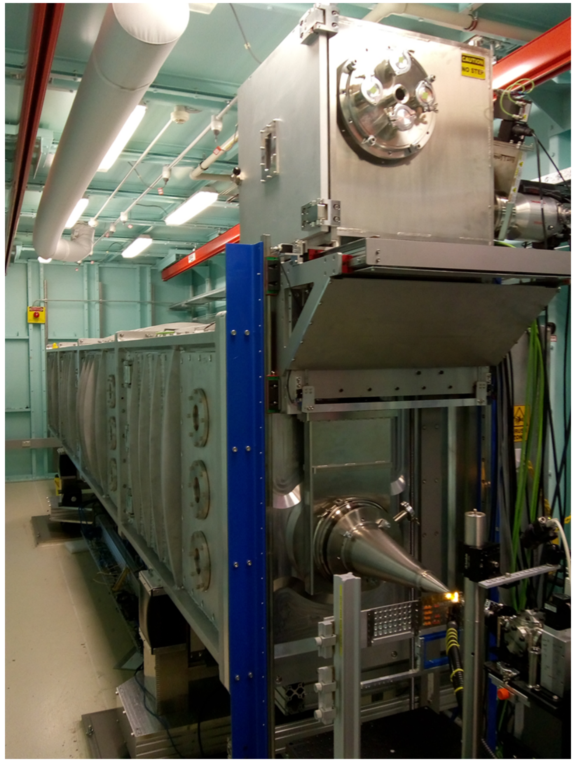
Stronger
- Stronger signal to noise
- Enables dilute or weakly scattering samples

Smaller
- Small beam size
- Enables small, thin samples, e.g. single fibres
- Enables fine spatial resolution scanning

Beamline Capabilities

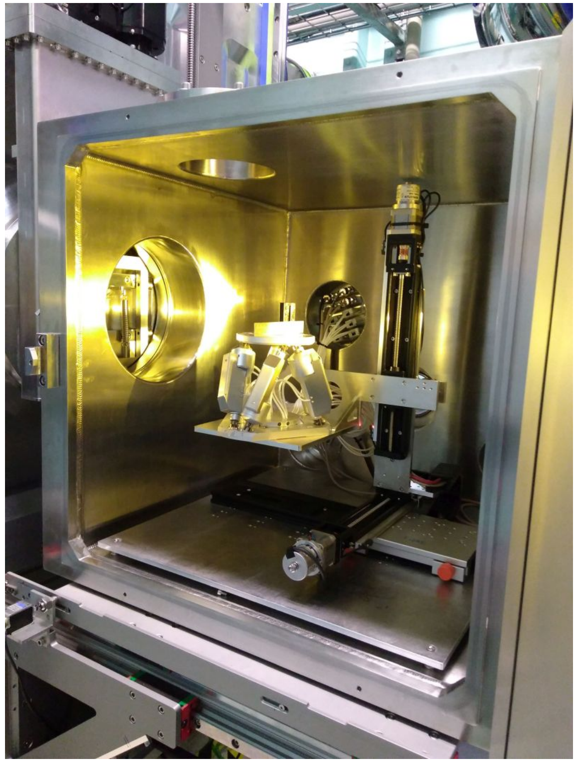
In air

Transmission



Grazing angle

Transmission



Grazing angle

In vacuum

SAXS provides information about structure in the size range of **0.5 – 500 nm**.

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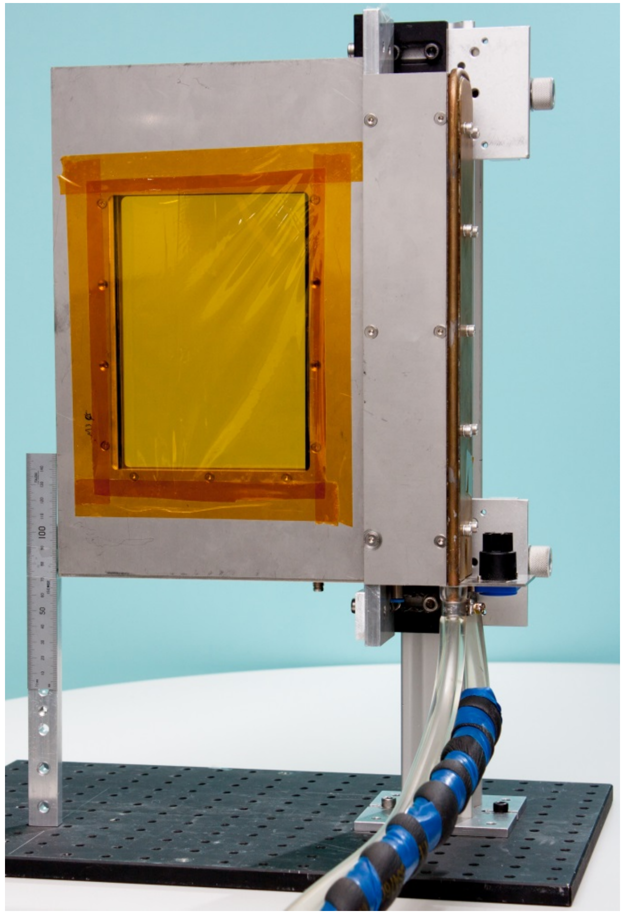
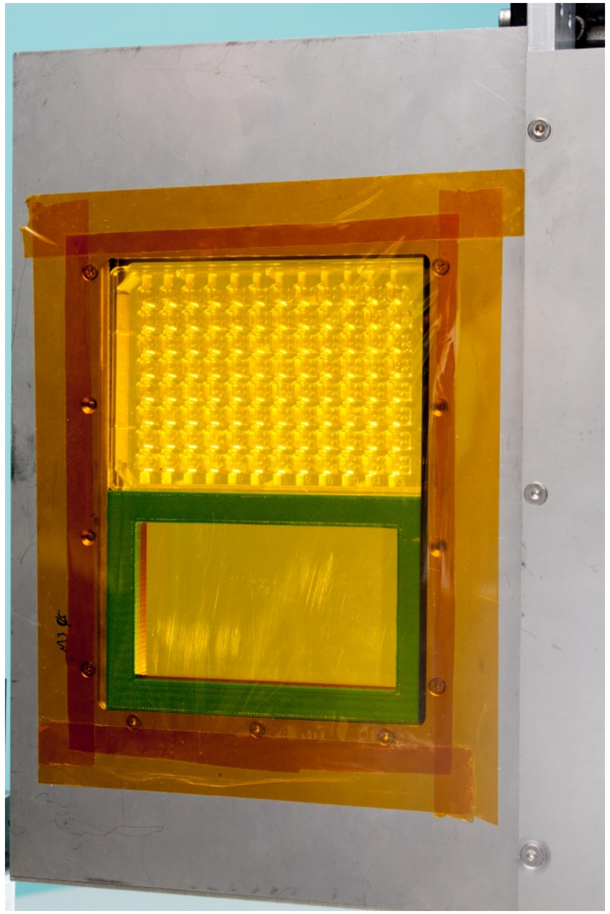
Solution setups – 96 wellplates





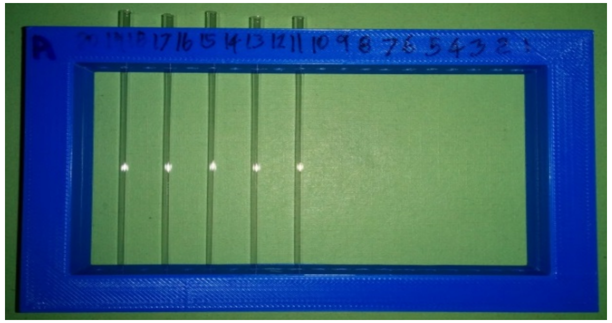
- Up to 2 well plates mounted at same time
- Temperature control 4 – 85°C
- Can fit steel plates and capillary holders

BUT Kapton windows add considerable background scattering!

Solution setups – capillaries

- for fluid, gel and sometimes powder samples
- analysed in batch
- temperature can be varied
- repeated measurements possible

- 35 capillaries per holder
- 1.5 mm outer diameter
- Temperature control approx. 4 – 85°C (water bath)
- Usually mounted vertically



- 3D printed holder (20 capillaries)
- To access entire capillary for scan

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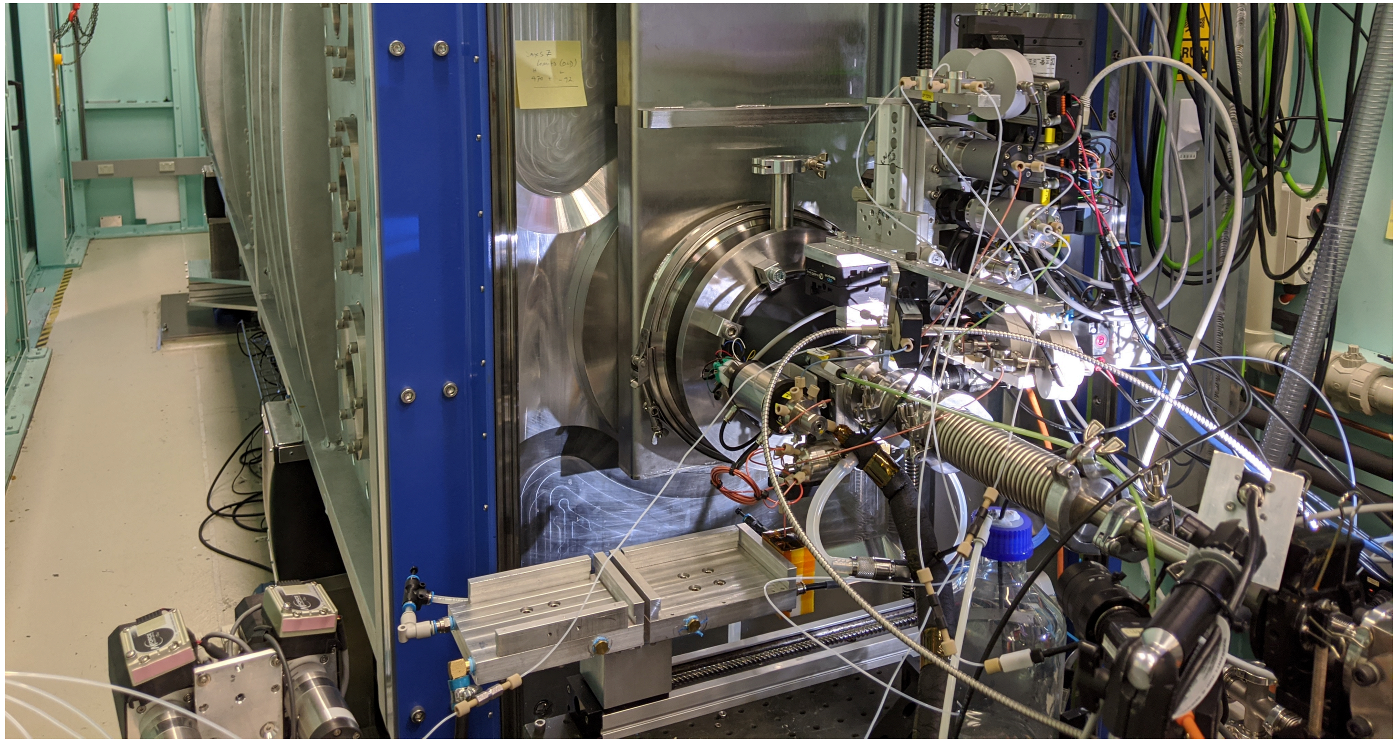
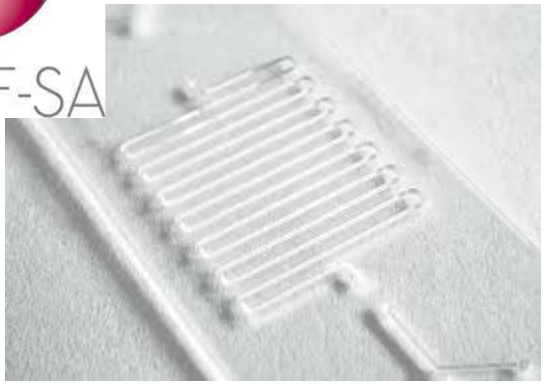
Proteins

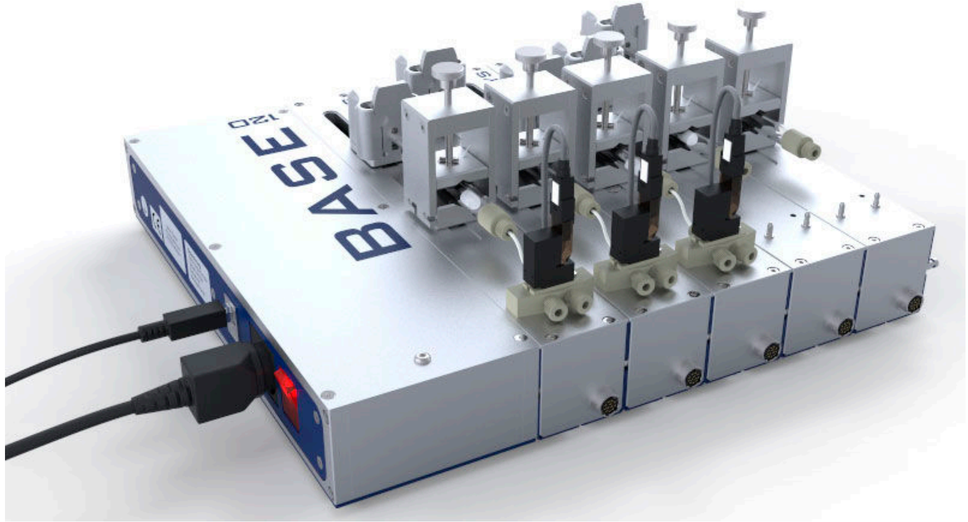


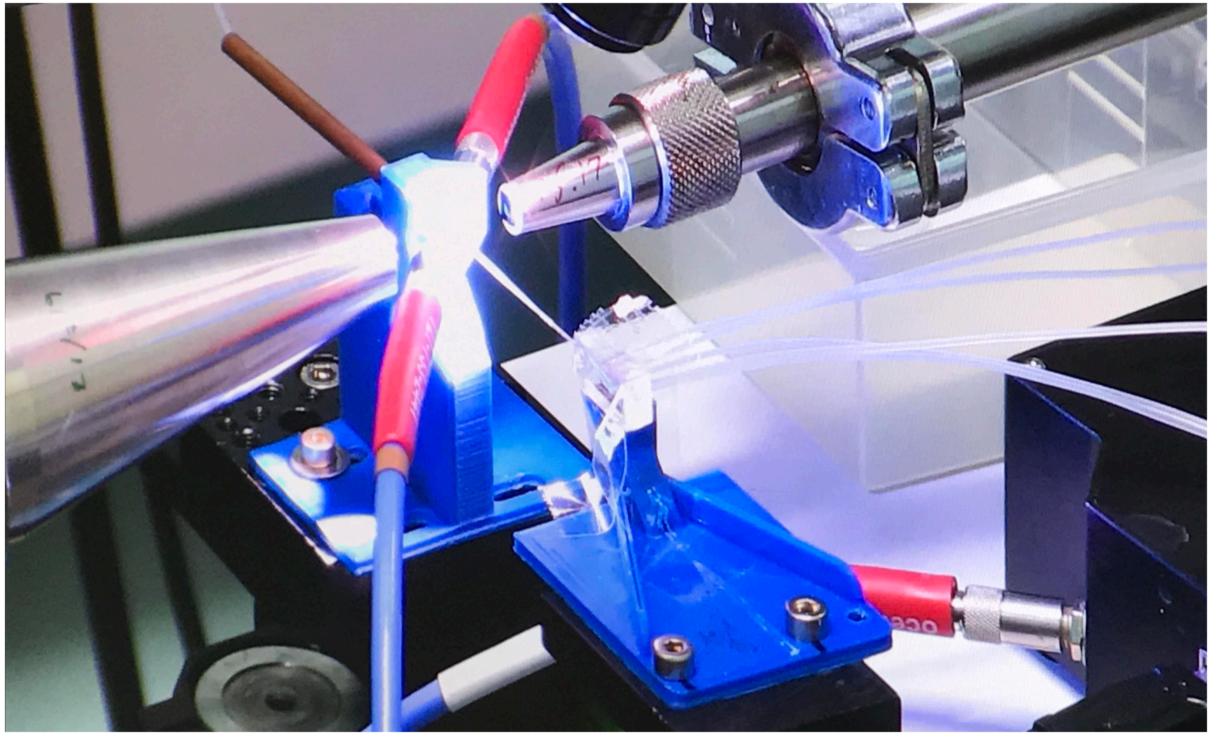
- Agilent HPLC
- Size Exclusion SAXS **in Coflow mode**
  - Mixed proteins, aggregates, etc
- Autoloader (multiple formats, potentially >>192 samples)
- Column Oven
- UV detector
- Low dilution (~3x)
- Customised mount, highly integrated ancillary setup
- <u>Batches of multiple sample (alpha version)</u>

Microfluidics

ANFF-SA







Advantages:

- Small sample volumes
- Laminar flow
- Diffusion control in 3D focussing devices
- Kinetic studies possible in situ
- Continuously fresh sample

Internal funding:

- Range of glass devices
- From mixers to tapering channels
- “Library” of standard in-house devices to use for a broad range of experiments.

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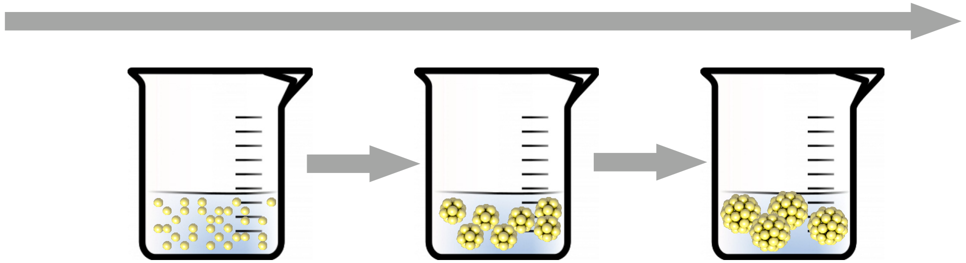
Why Microfluidics?

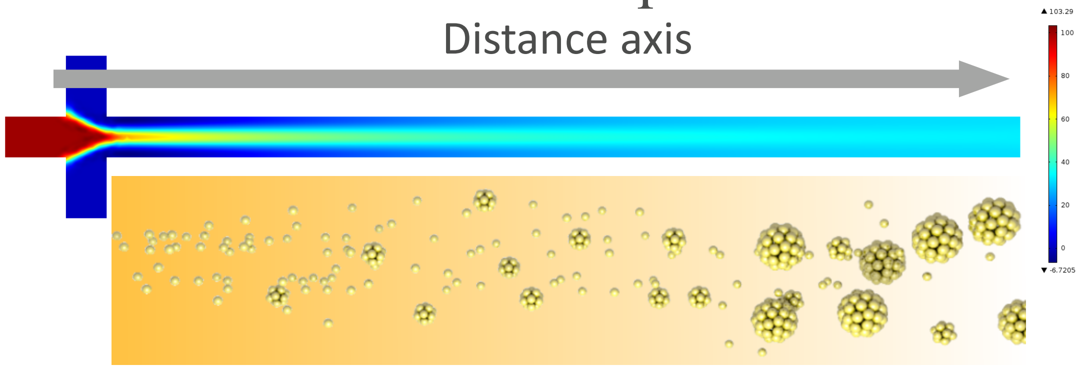
Batch experiment

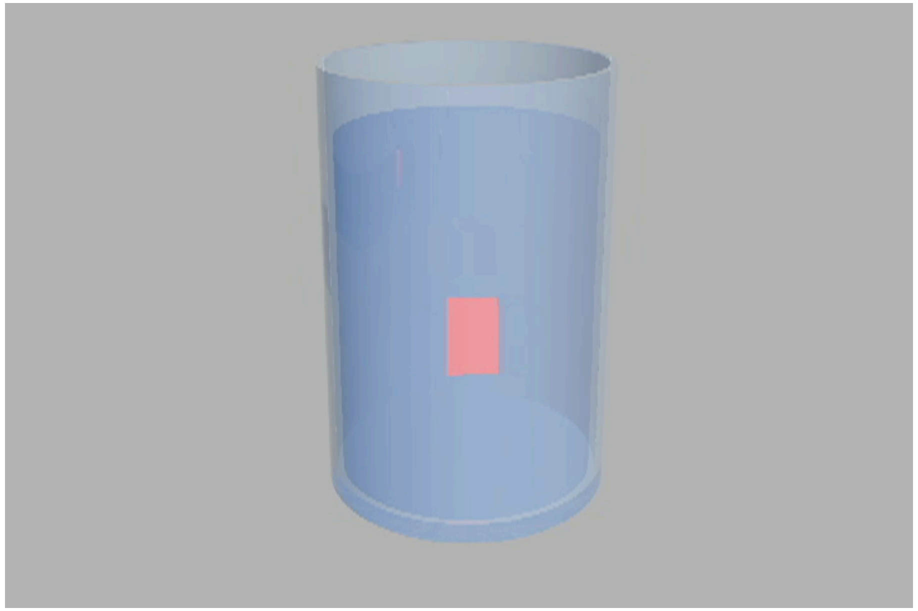
Time axis

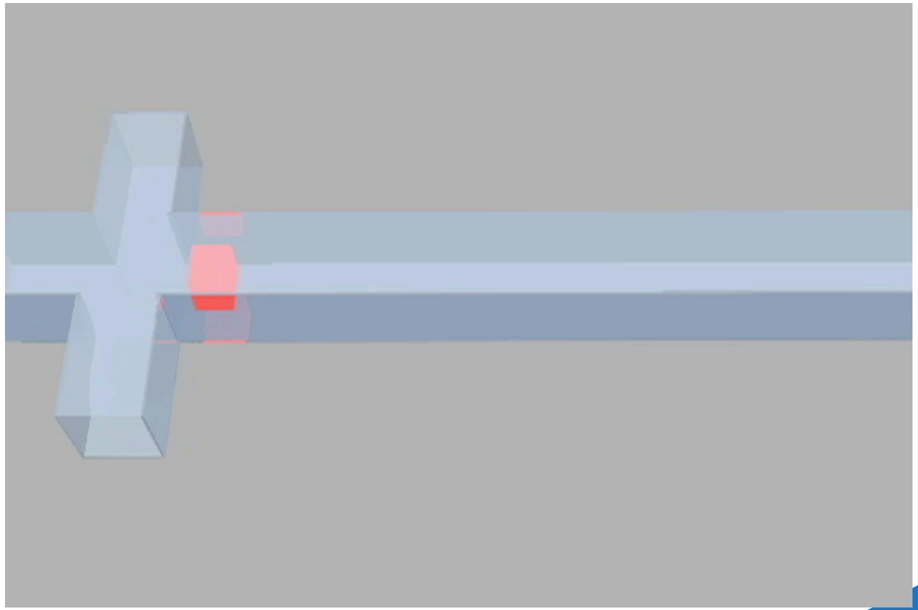


Microfluidic experiment

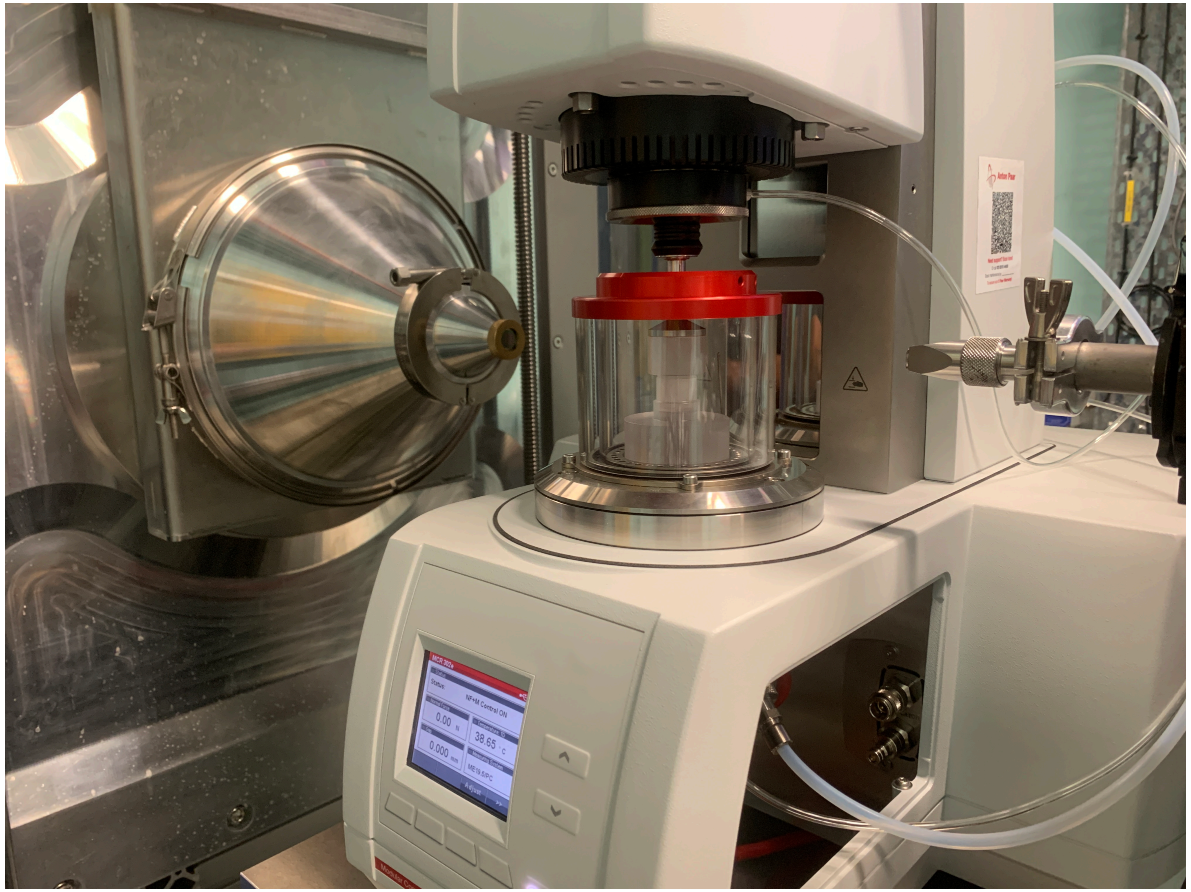
Distance axis







Rheometer



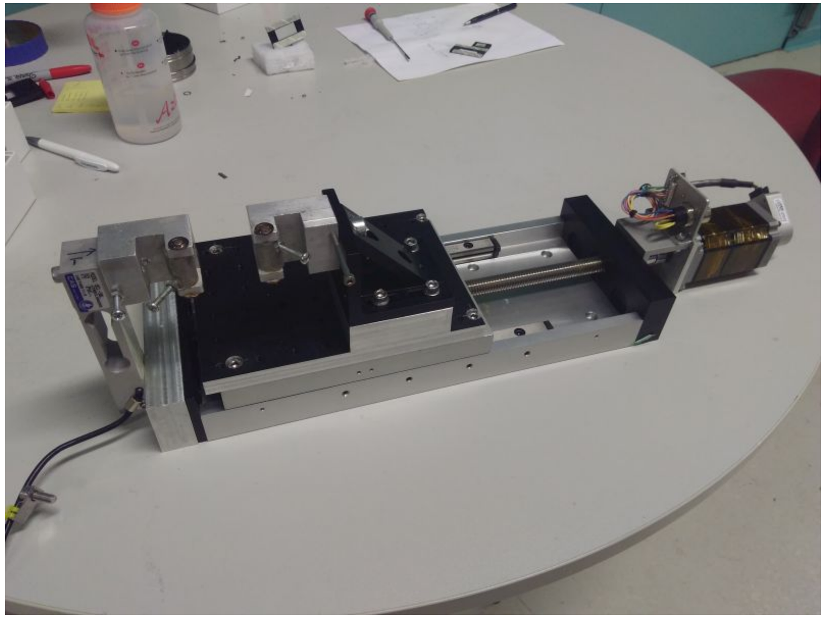
NEW: Anton Paar Rheometer MCR302e

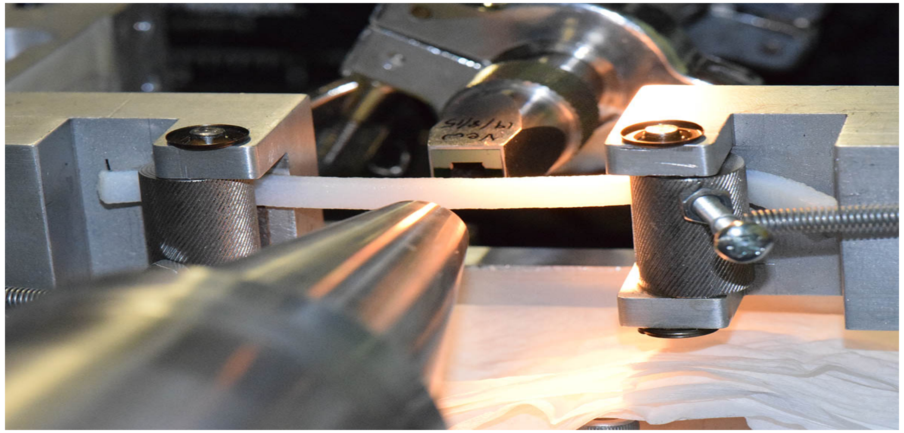
- Triggering modes: trigger SAXS acquisition on Rheometer datapoint – trigger Rheometer datapoint on SAXS acquisition.
- Temperature controlled sample environment
- Range of different geometries.
- *Upcoming:* temperature enclosure for reduced background scattering.

Measurement geometries:

- Parallel plates 
- PC cup and bob (slightly cone shaped) 
- PC bob (cone) and plate 
- Glass cup and bob 

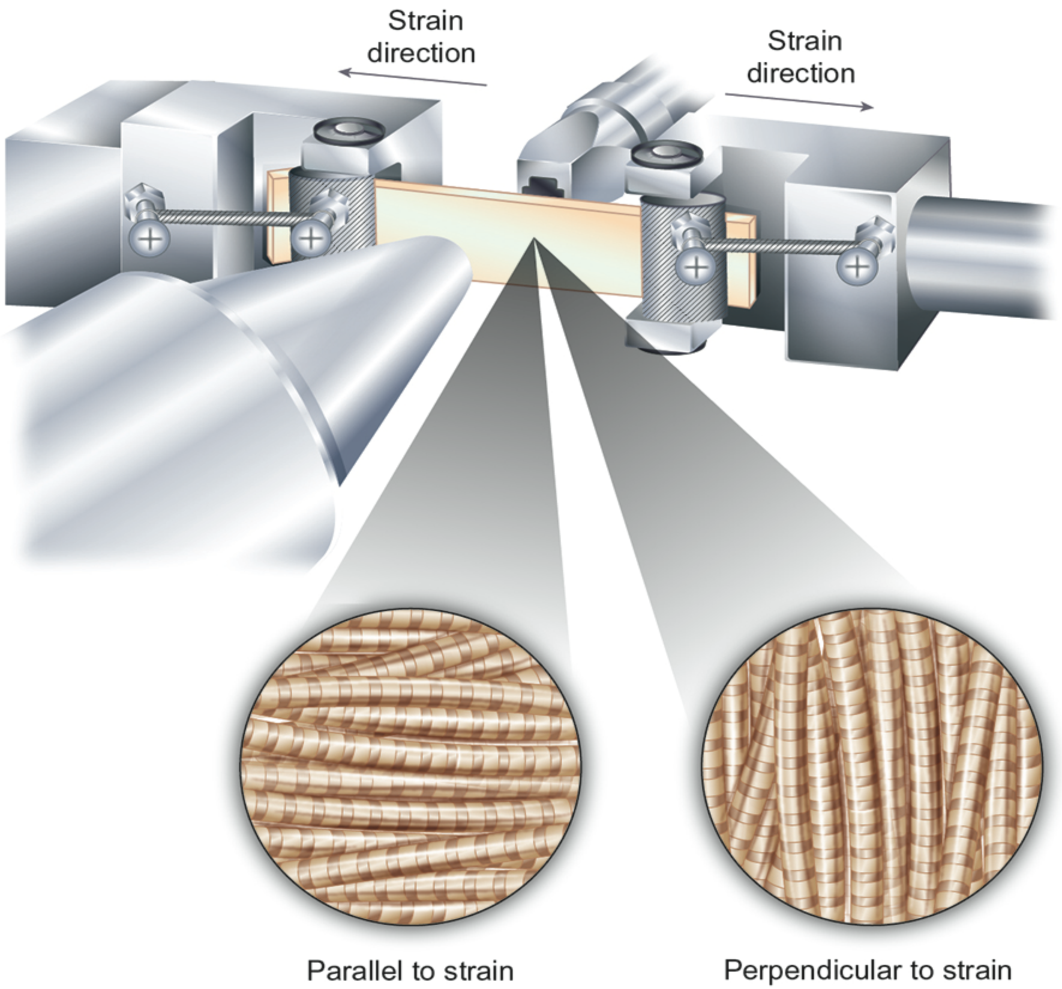
Mechanical testing

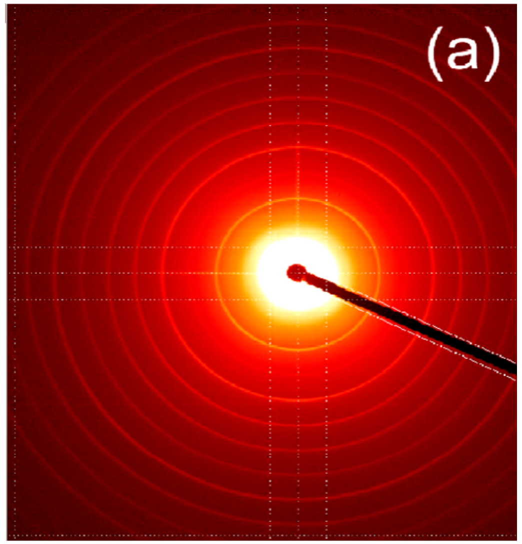




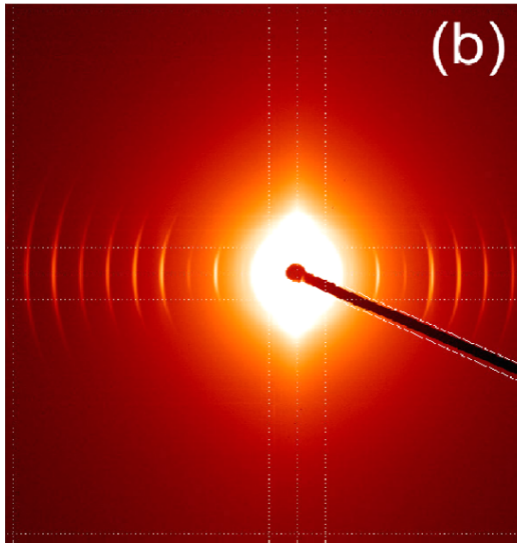
- In situ measurement under tensile or compressive strain
- High-capacity stepper motor driven linear stage
- Fitted loadcell and mounts for grippers of various types
- Loadcell capacity of 300N

Mechanical testing: Collagen





Adult pericardium collagen
(Isotropic)



Neonatal pericardium
collagen
(Anisotropic)

Sizeland, K. et al. *ACS Biomater. Sci. Eng.* **2017**, 3, 10, 2550–2558

Microfluidics

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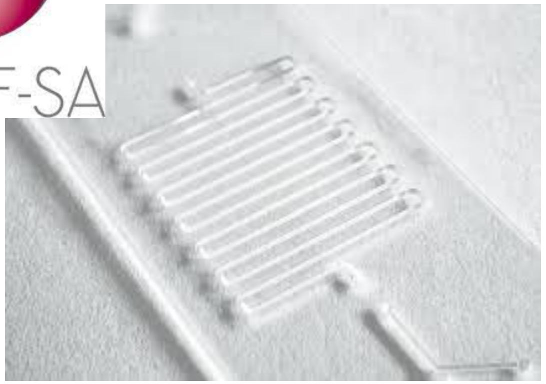
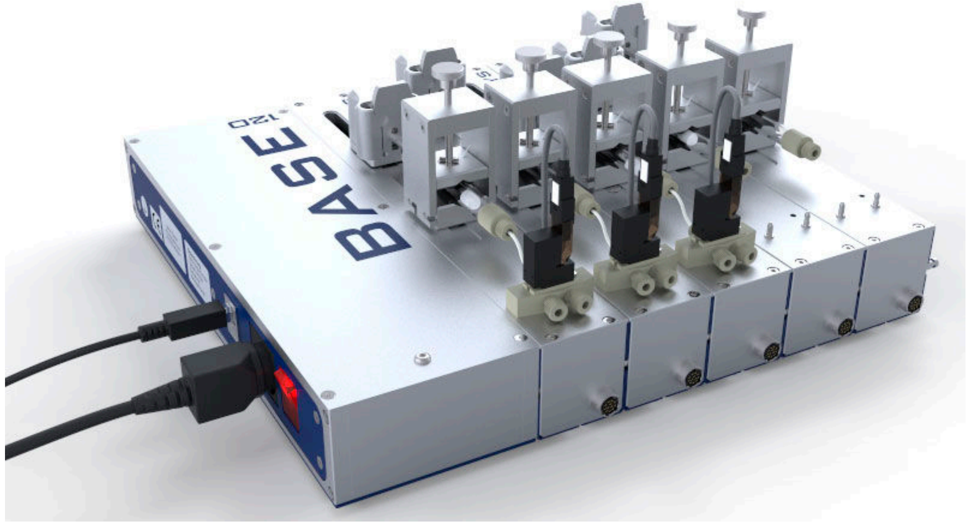
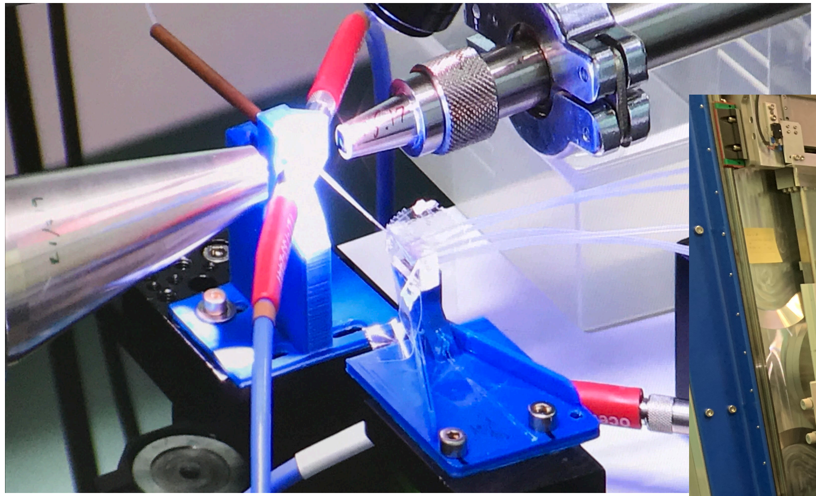
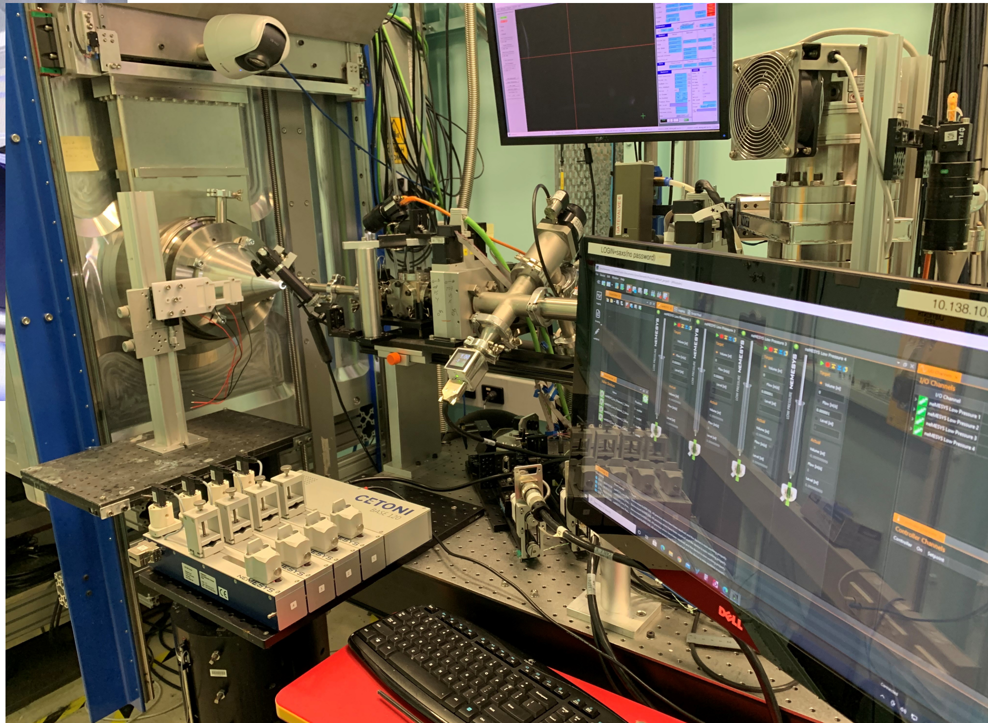


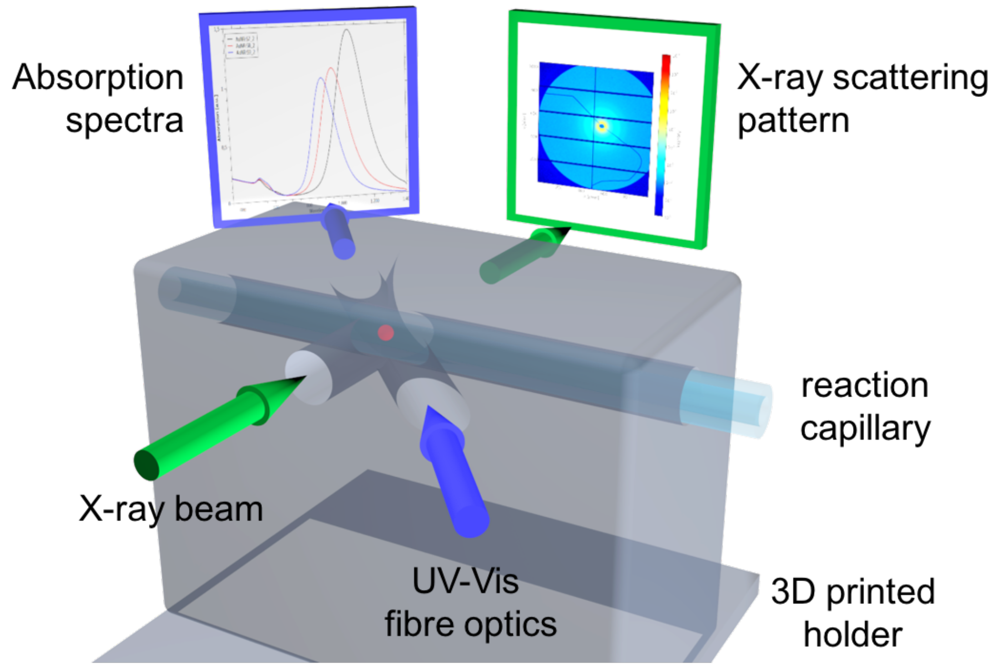


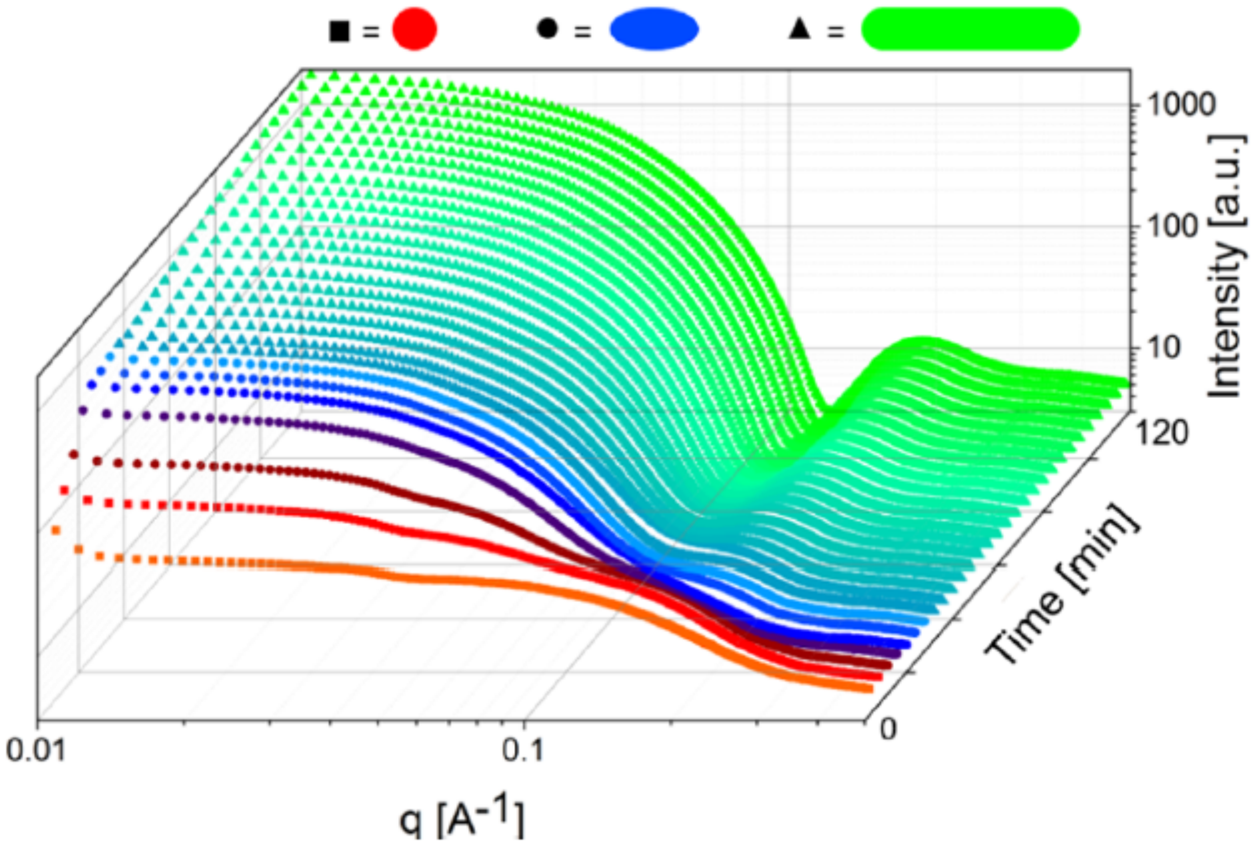




Growth Kinetics of Nanoparticles

Growth of gold nanoparticles in solution by reduction of gold(III) acid by hydroquinone, the final geometry of the particles is rod-like





Time-resolved SAXS showed the transition from spherical to ellipsoidal to rod-like (cylindrical) particles during particle growth

Seibt, S. et al. *J. Phys. Chem. C.* **2021**, 125, 19947-19960

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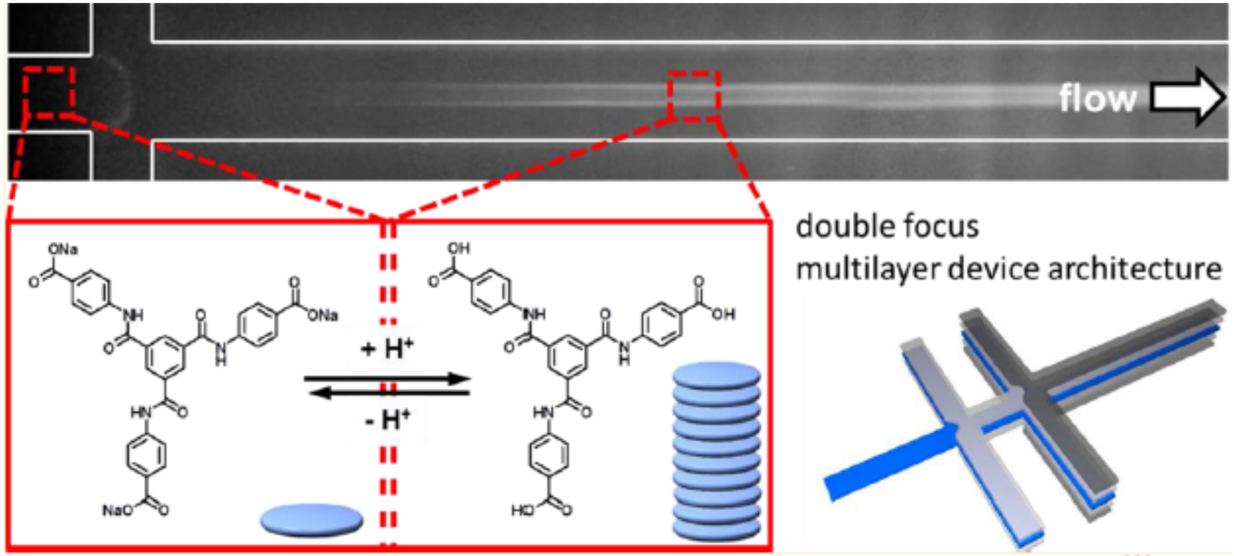
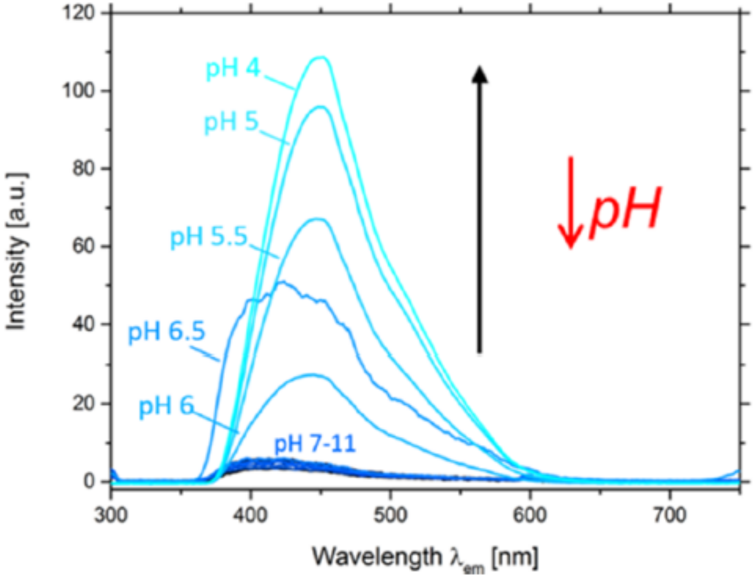
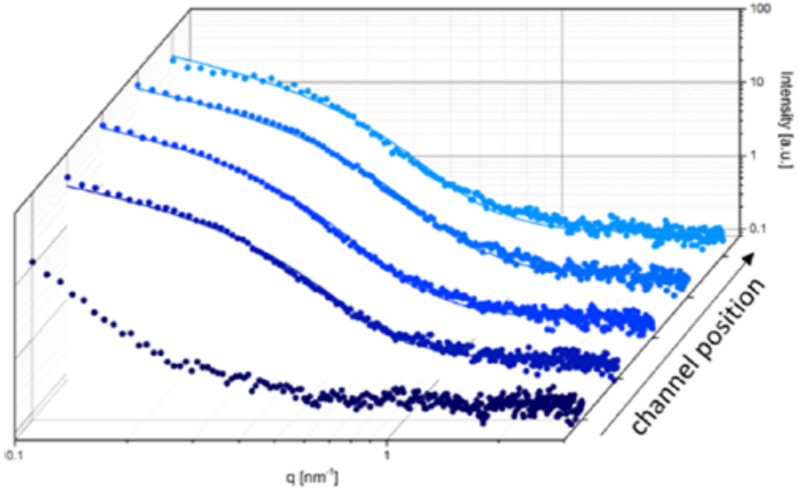
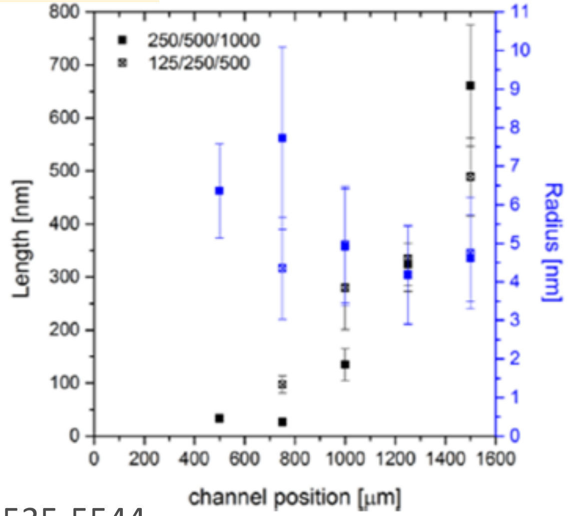
Particle assembly Kinetics





pH-drop in microchannel induces aggregation-induced fluorescence





In situ SAXS allows for the shape of the growing particles to be determined

Seibt, S. et al. *Langmuir* **2018**, 34, 5535-5544

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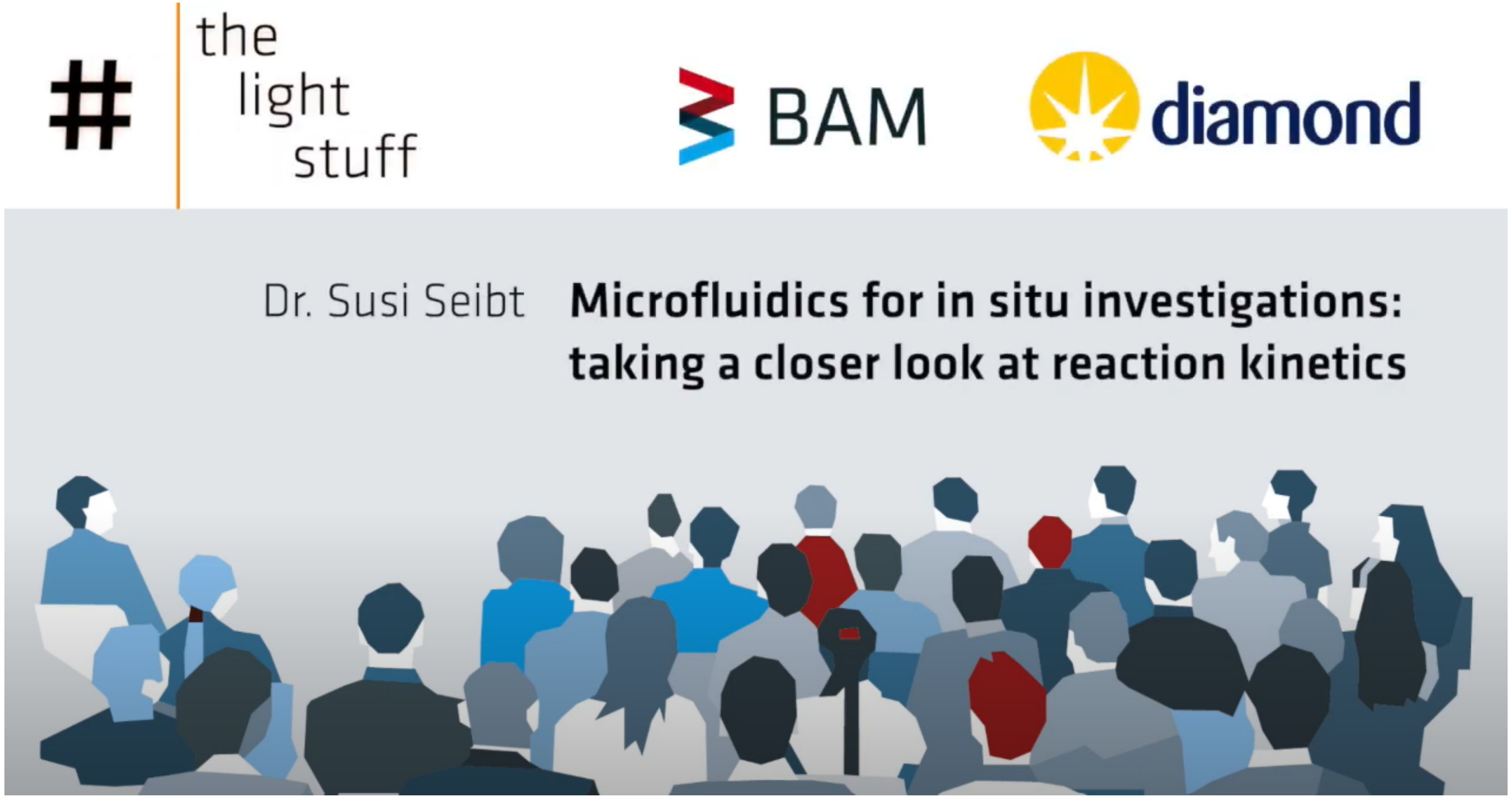
The Science behind (some of) my PhD



SCAN ME!

https://www.youtube.com/watch?v=LL6KSuehuc0

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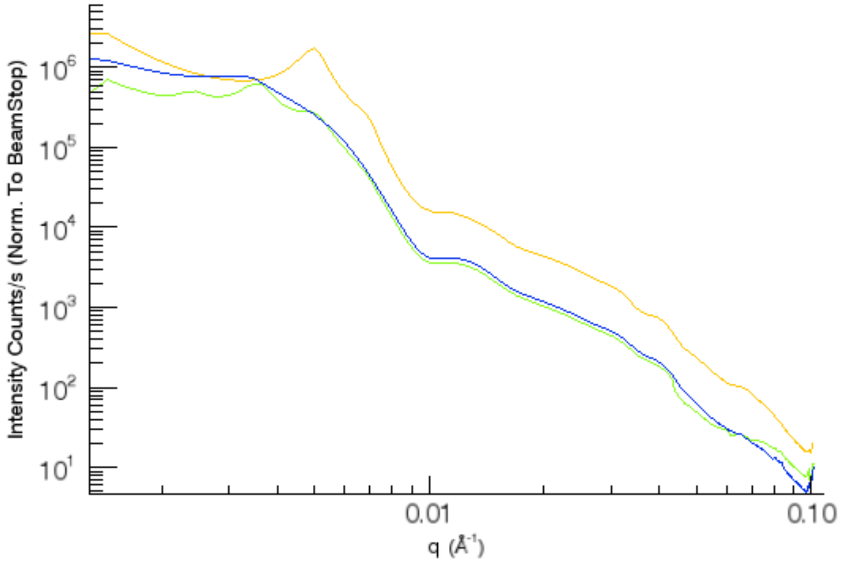
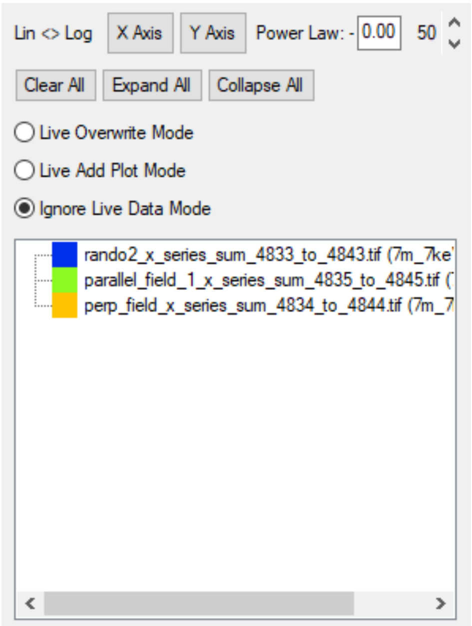
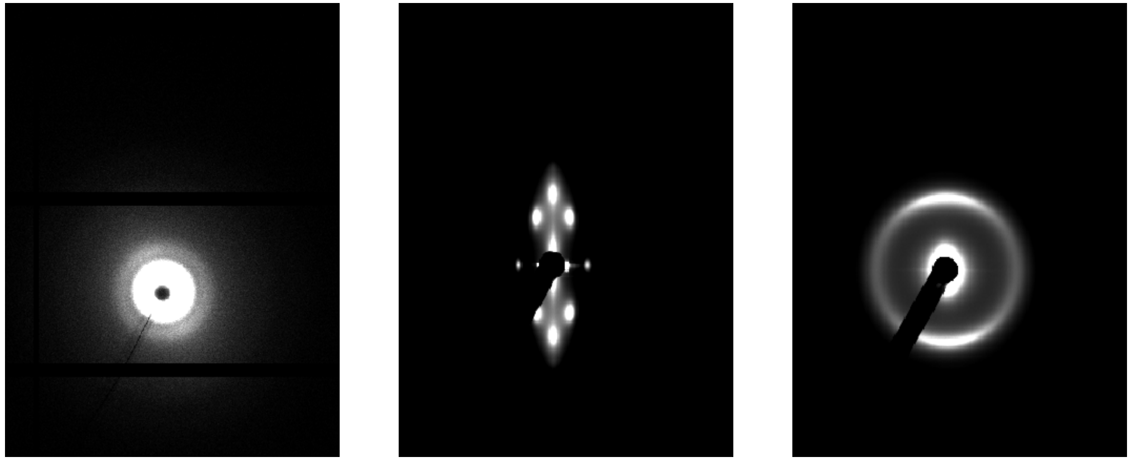
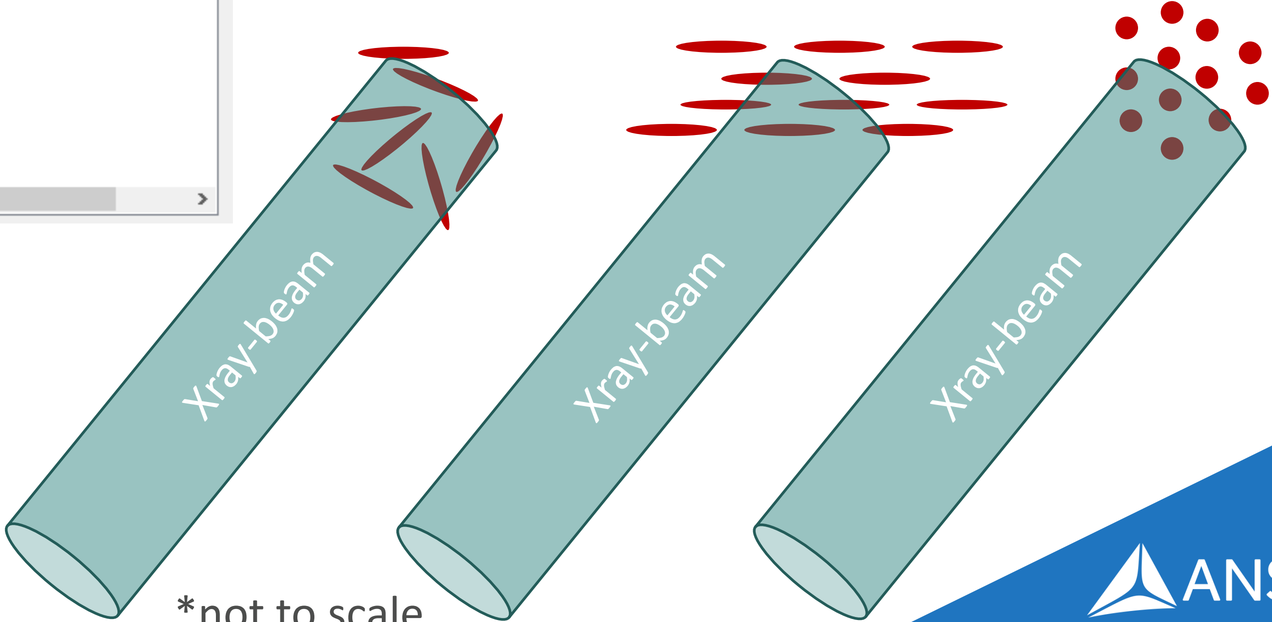
Particle Alignment





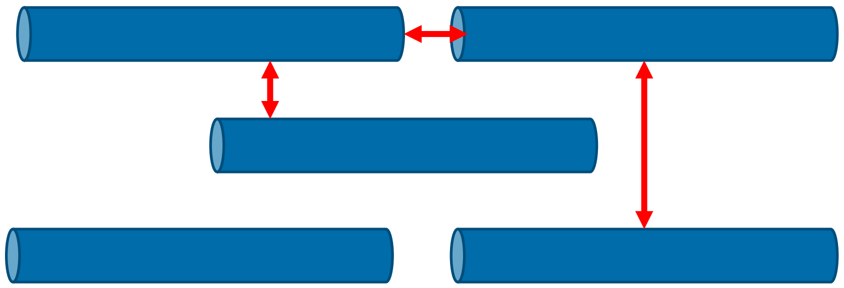


Anisotropic particles align under external influence in solution

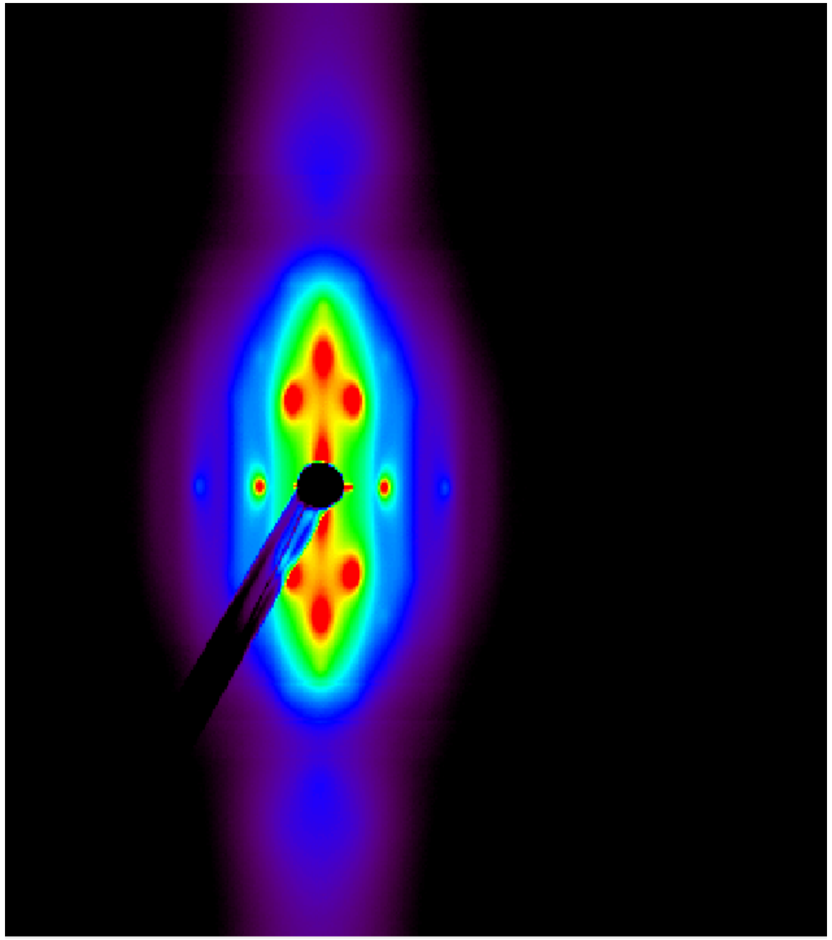


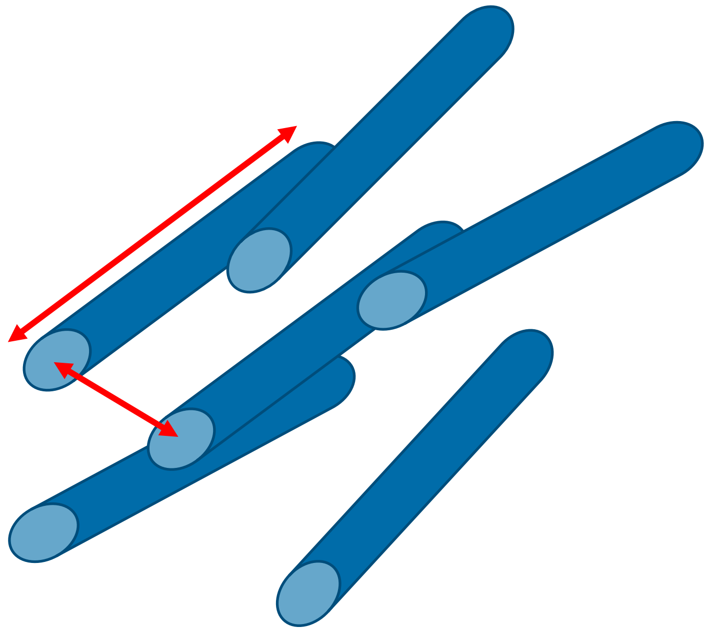
*not to scale

Particle Aligment



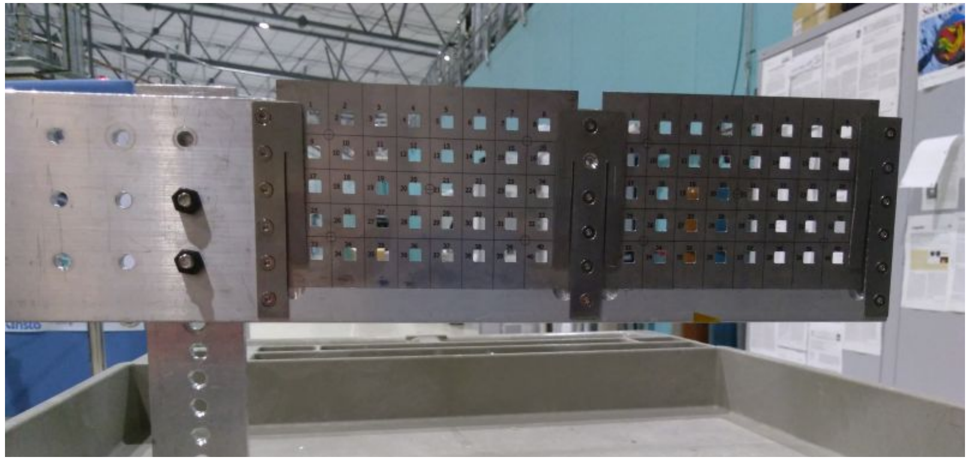
Information about particle alignment in crystal lattices and spacing as well as size and preferential direction of orientation is available.





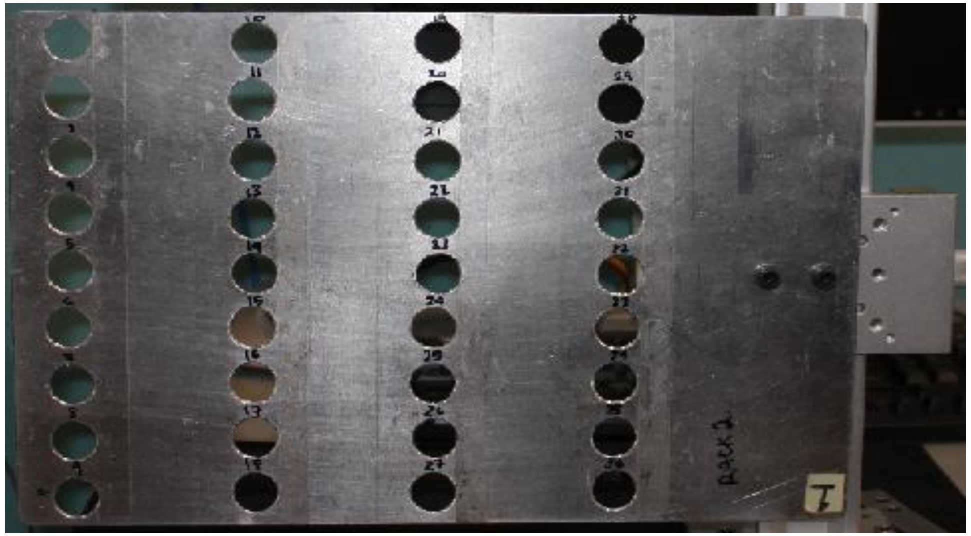
In air - Steelplates

- for solids, powders, fibres, gel samples
- mounted over holes in metal plates
- measured in transmission
- measured at room temperature (mostly)



Outer geometry of 96 well plates:

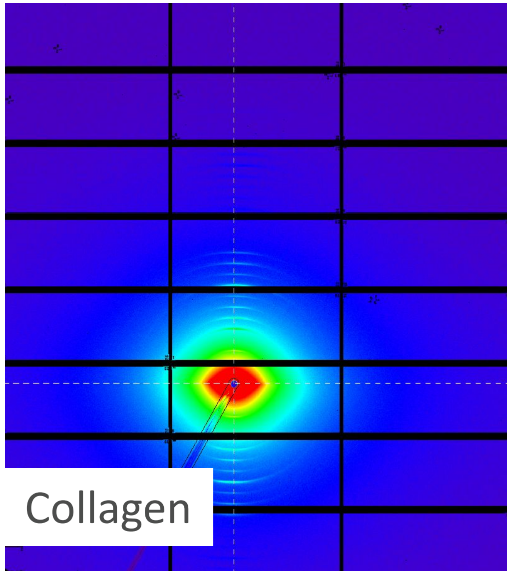
- Laser-cut steel plate
- Contains 40 square holes
- Each 5 x 5 mm
- Plate thickness either 0.7 mm or 2.0 mm
- Can be mounted in temperature holder
- Multiple plates available.

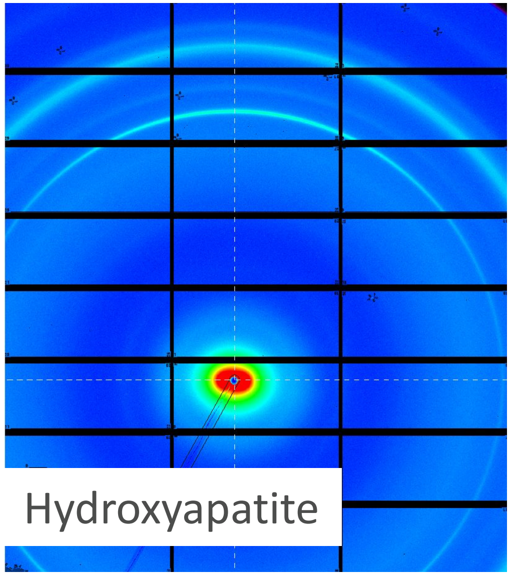


For larger samples:

- laser-cut steel plates with round holes
- 10 mm or 20 mm diameter
- Only room temperature measurements!
- Only few plates available.

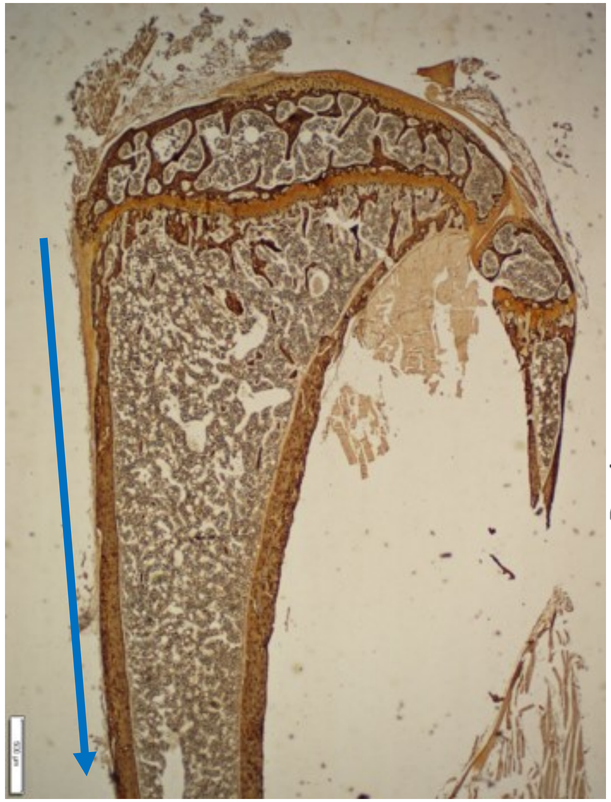
Mice bone studies against osteoporosis

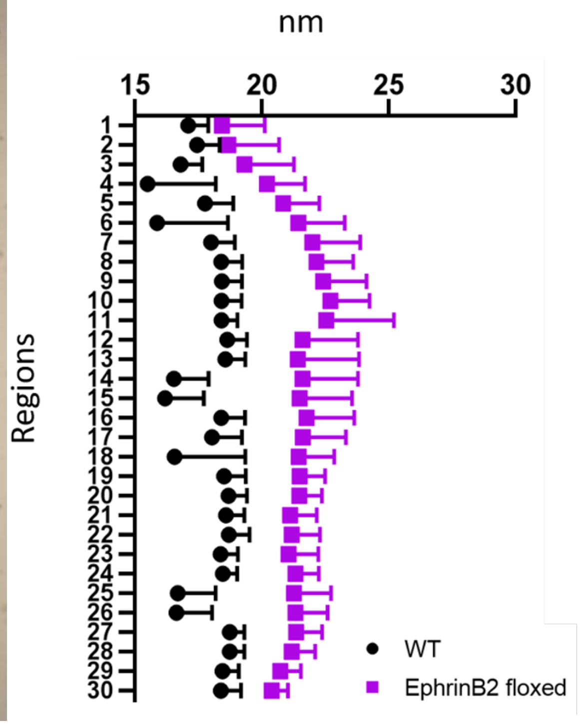


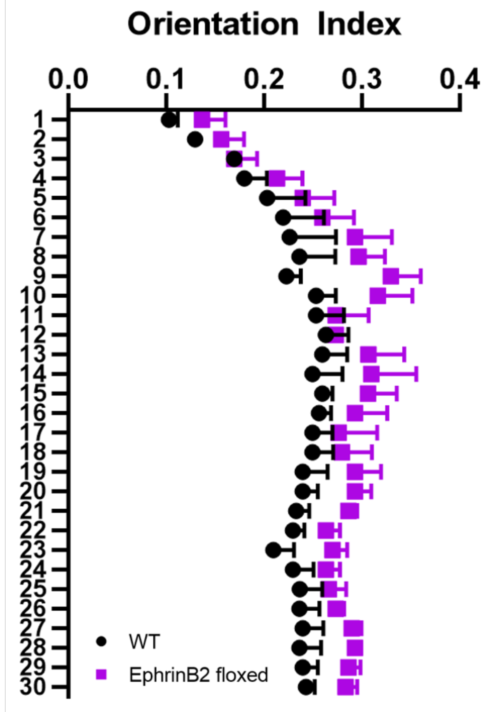


Osteoporosis through bone fractures.
Bone fragility can be caused by low bone mass or poor quality of bone.

2 main components of bone tissue:
Collagen and carbonated hydroxyapatite

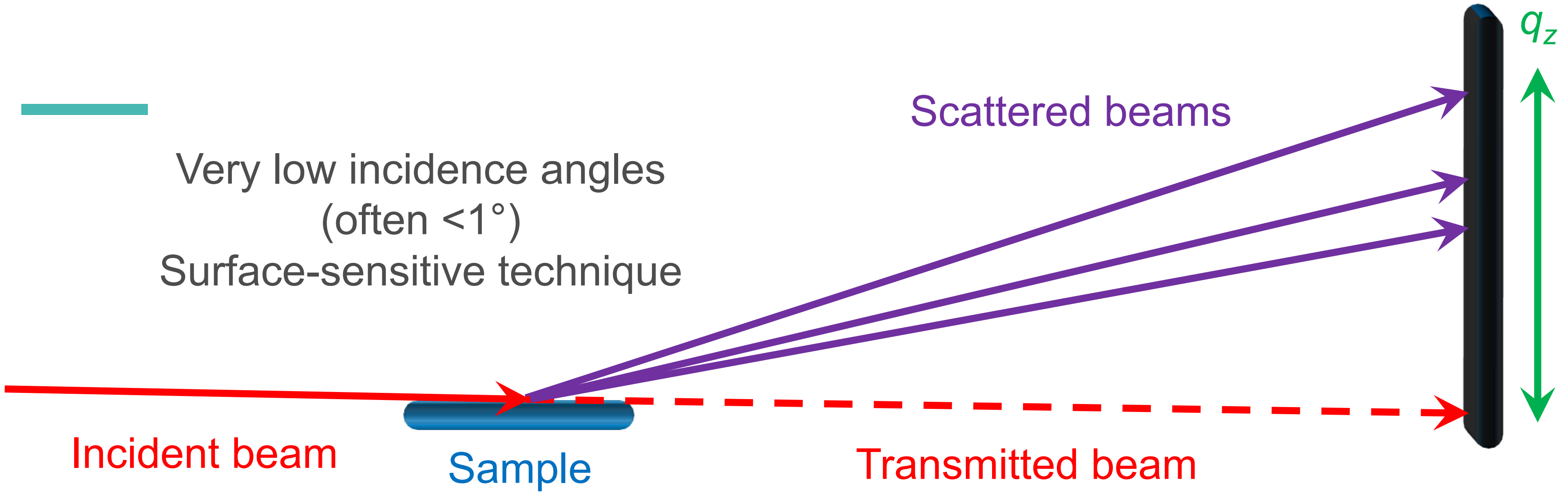


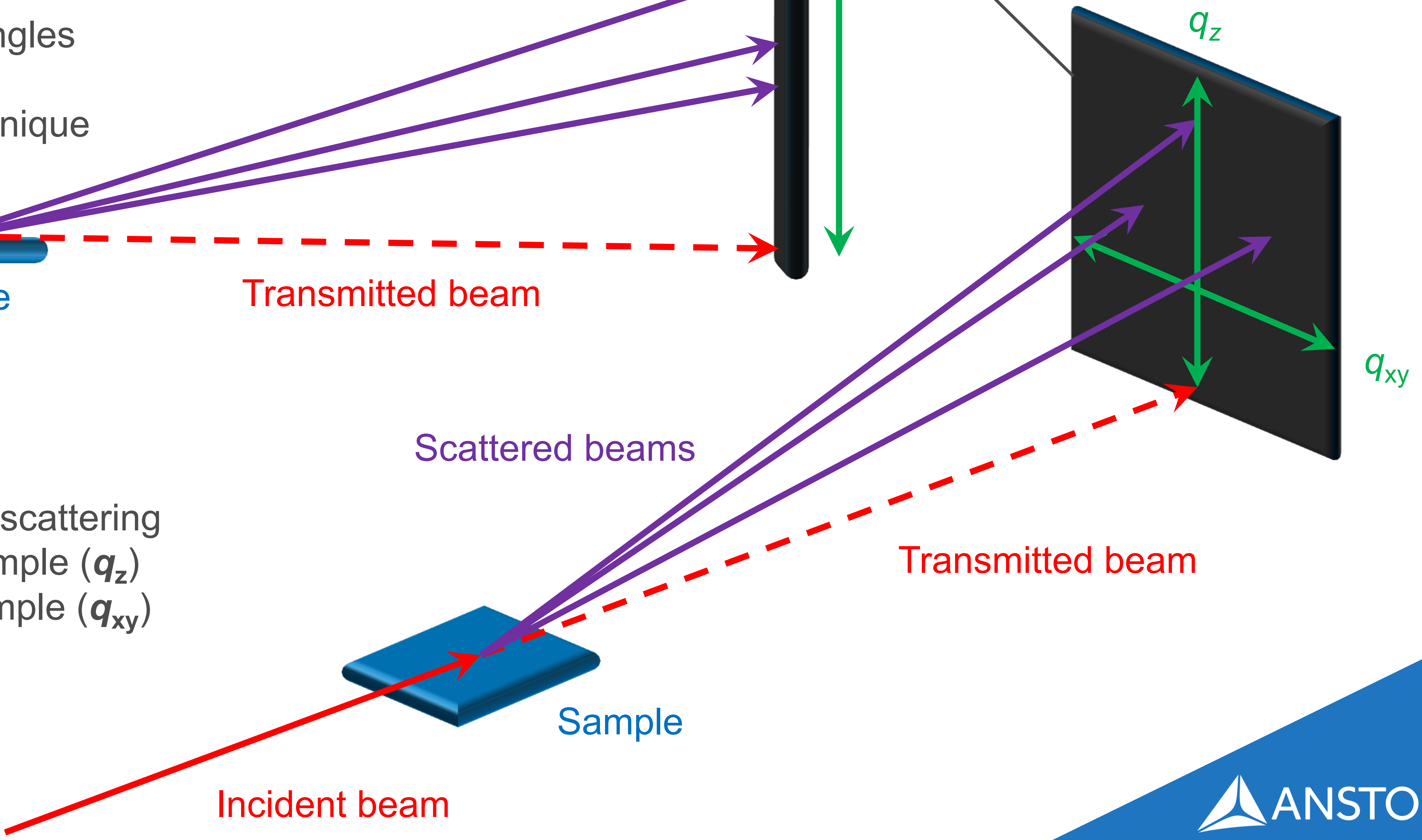




Grazing Incidence Scattering

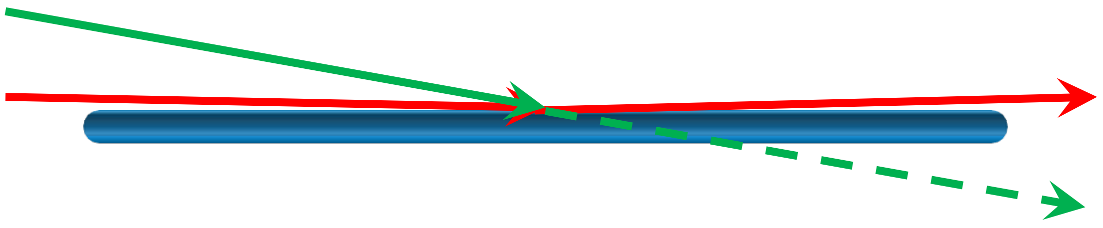
Very low incidence angles
(often <1°)
Surface-sensitive technique





Provides information about scattering out of the plane of the sample ($\boldsymbol{q_z}$) and in the plane of the sample ($\boldsymbol{q_{xy}}$)

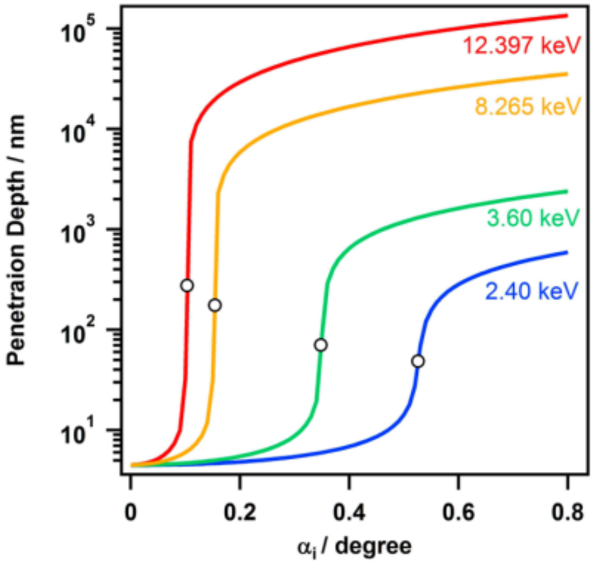
Surface Versus Bulk Sensitivity



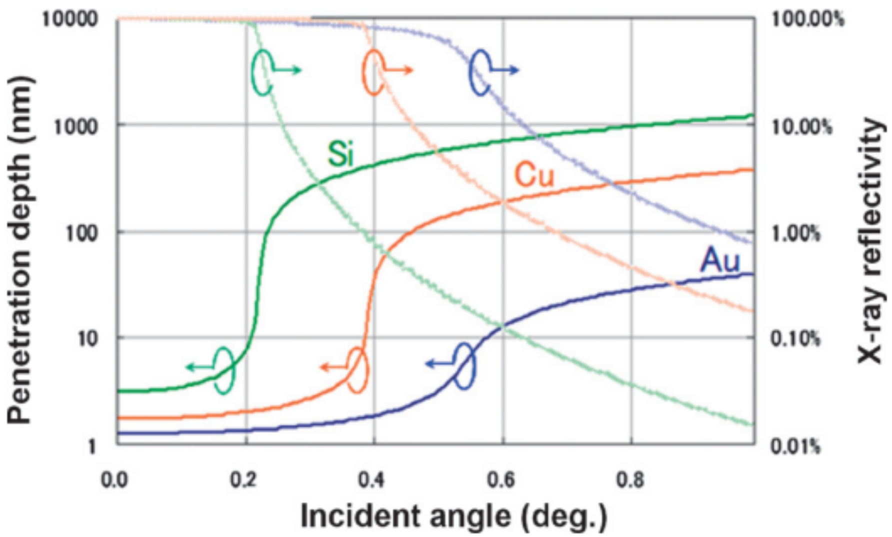
Beam below critical incidence angle
is totally reflected

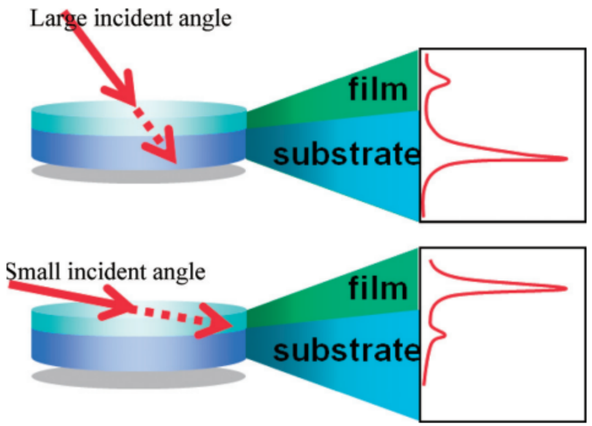
Beam above critical incidence angle
is transmitted/absorbed

Penetration depth is X-ray energy dependent



Penetration depth is material dependent

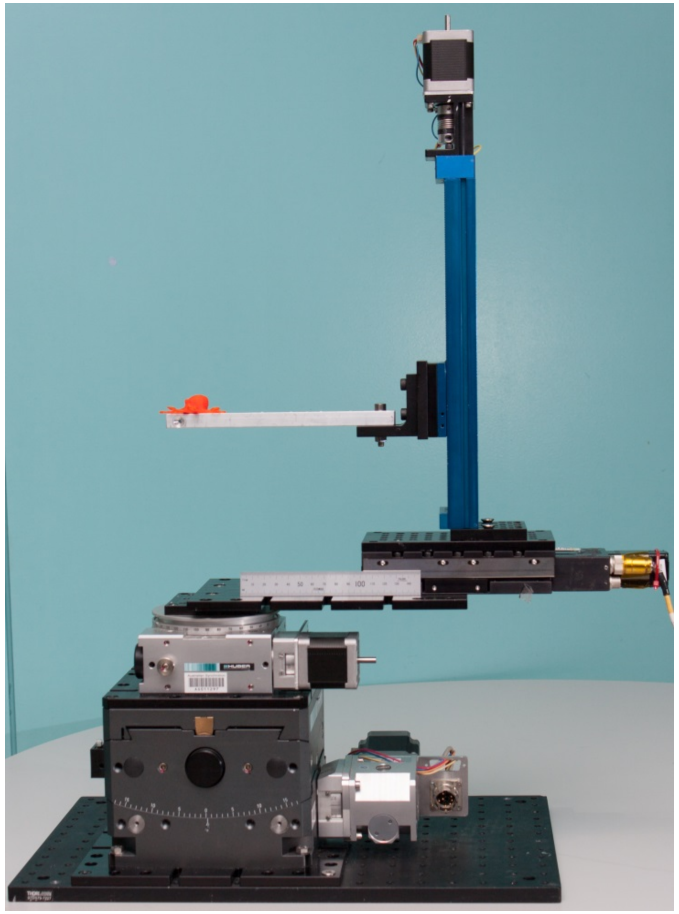


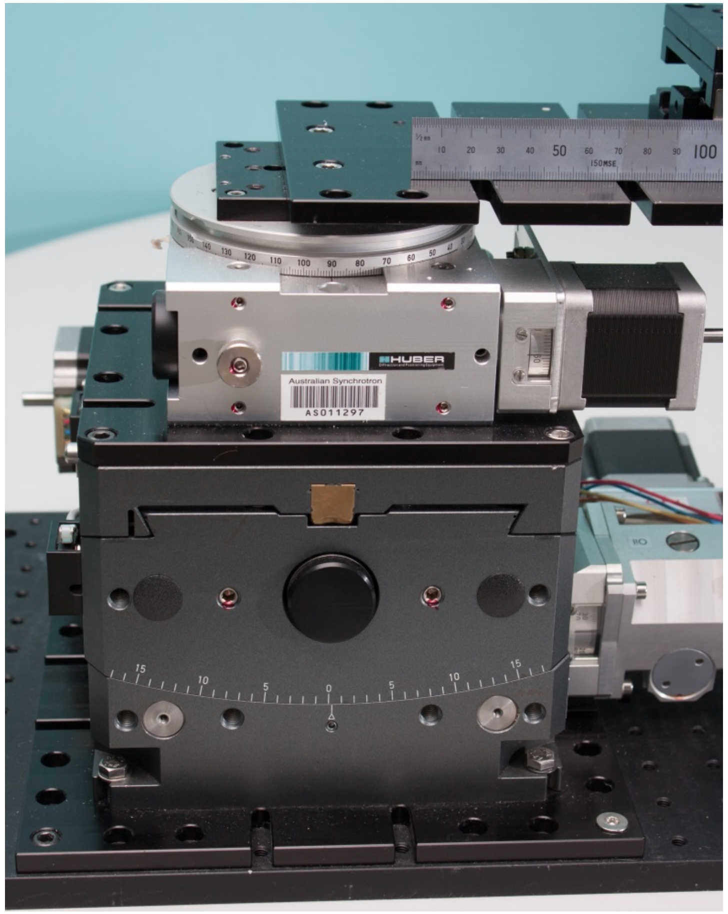


Saito et al. *Macromolecules* **2015**, *48,* 8190-8196.

Kobayashi S. *Rigaku J.* **Winter 2010**, *26* (1) 03-11.

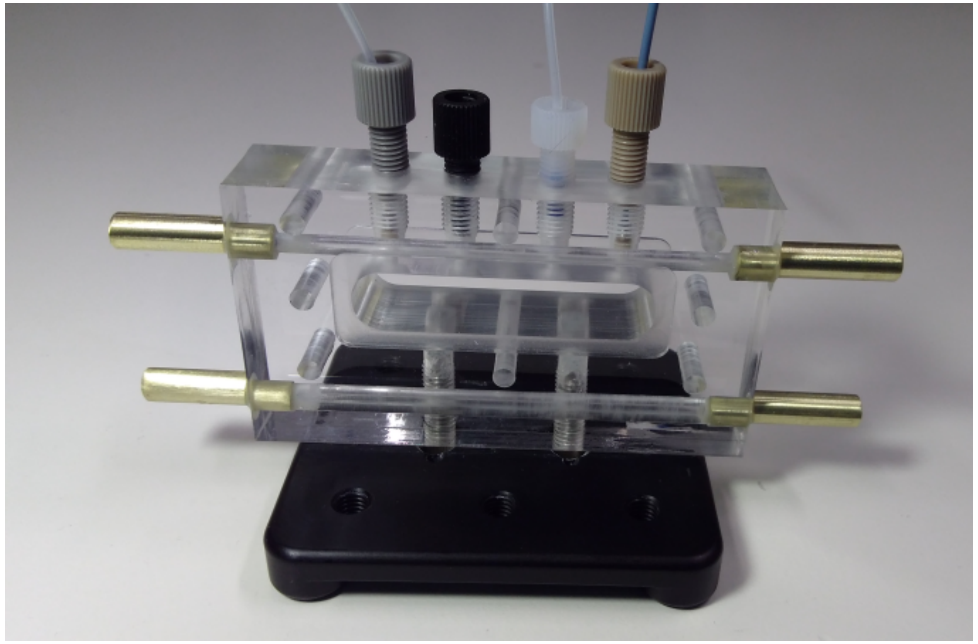
In air – Rotation stacks





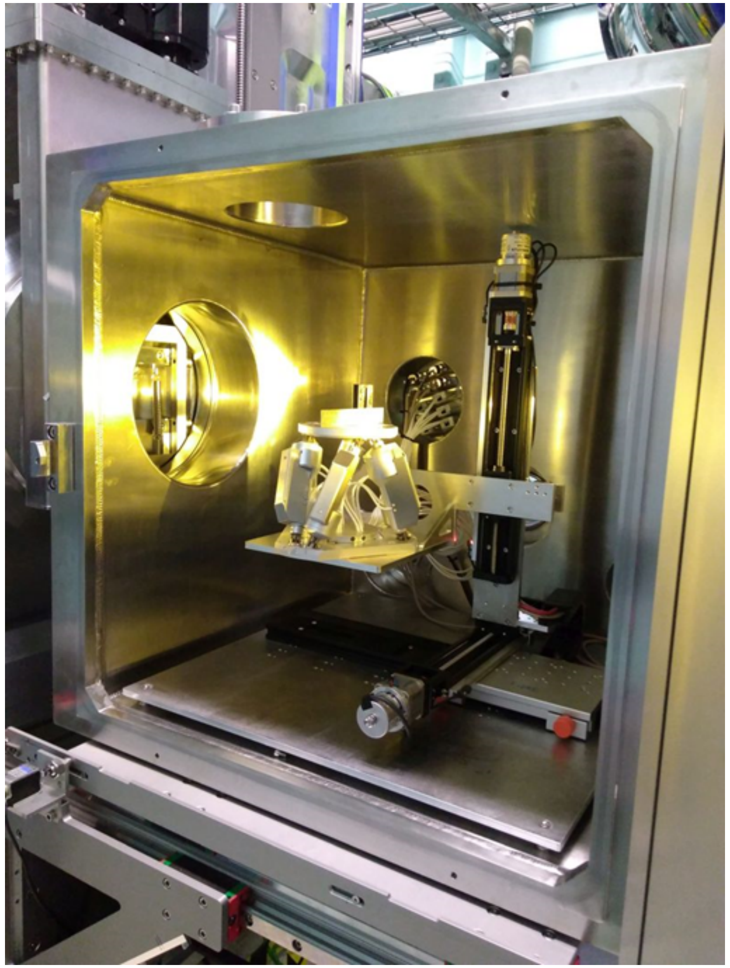
- To present samples at different angles to the beam
- Double cradle (± ~18° in each axis)
- 360° phi (yaw) stage
- XY translation inside centre of rotation
- GISAXS / GIWAXS, reflectivity
- Alignment sensitive transmission SAXS

In air – GISAXS at solid-liquid interface



- PMMA cell with Kapton windows
- Aluminium cell with Kapton windows (not shown)
- Temperature control 10 to 70°C
- Standard HPLC fittings to insert fluid in cell
- Ability to run several samples side by side

In vacuum – GISAXS hexapod



- Full translational and rotational motion
- Preferred stage for dry solid interfaces
- GISAXS / GIWAXS and reflectivity measurements
- Very low background intensity
- Linkam temperature stage

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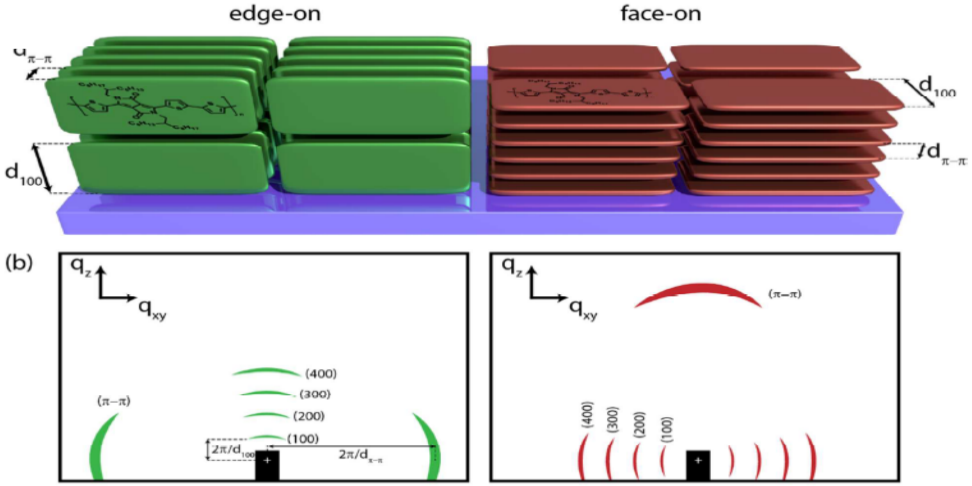
Polymer-based organic PVs

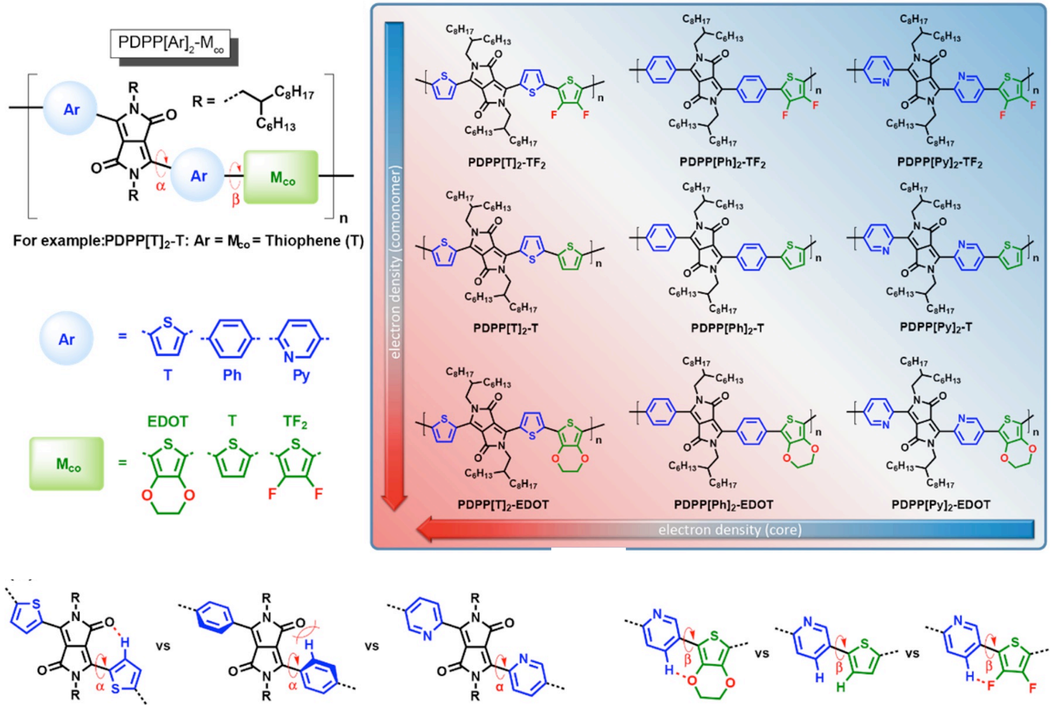
GIWAXS to determine **molecular orientation** in semiconducting polymer devices
→ high brightness and polarised X-rays

Thin films of semiconductor polymers → distinct orientation with respect to substrate

Favourable for transport in **thin film transistors**

Improve bulk transport in vertical direction for **solar cells**

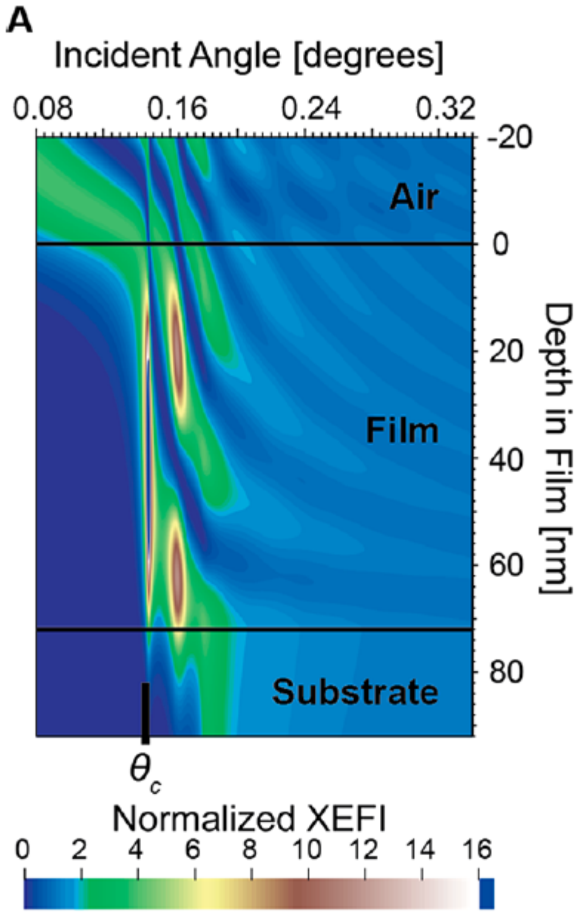


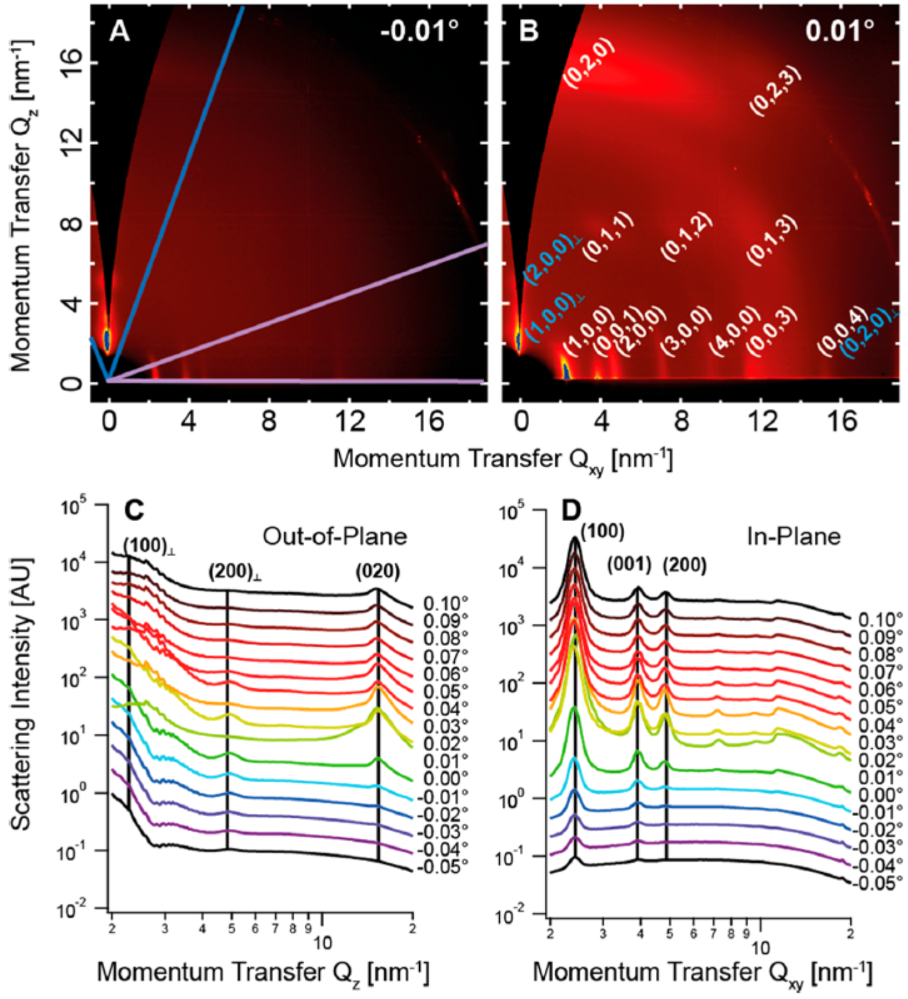


Mueller, C. et al., *Chem. Mater.* **2016**, 28, 7088-7097.

Crystalline Stratification

Very accurately controlling and varying the incident beam angle, and modelling dataset in detail to extract depth-dependent diffraction structure in polymer thin films.





Gann, E. et al. *Macromolecules* **2018**, 51, 2979-2987.

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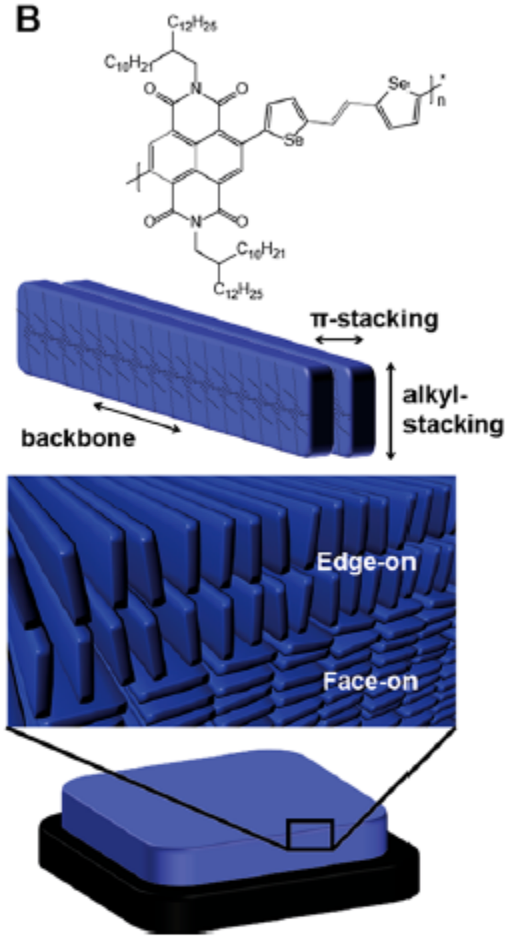
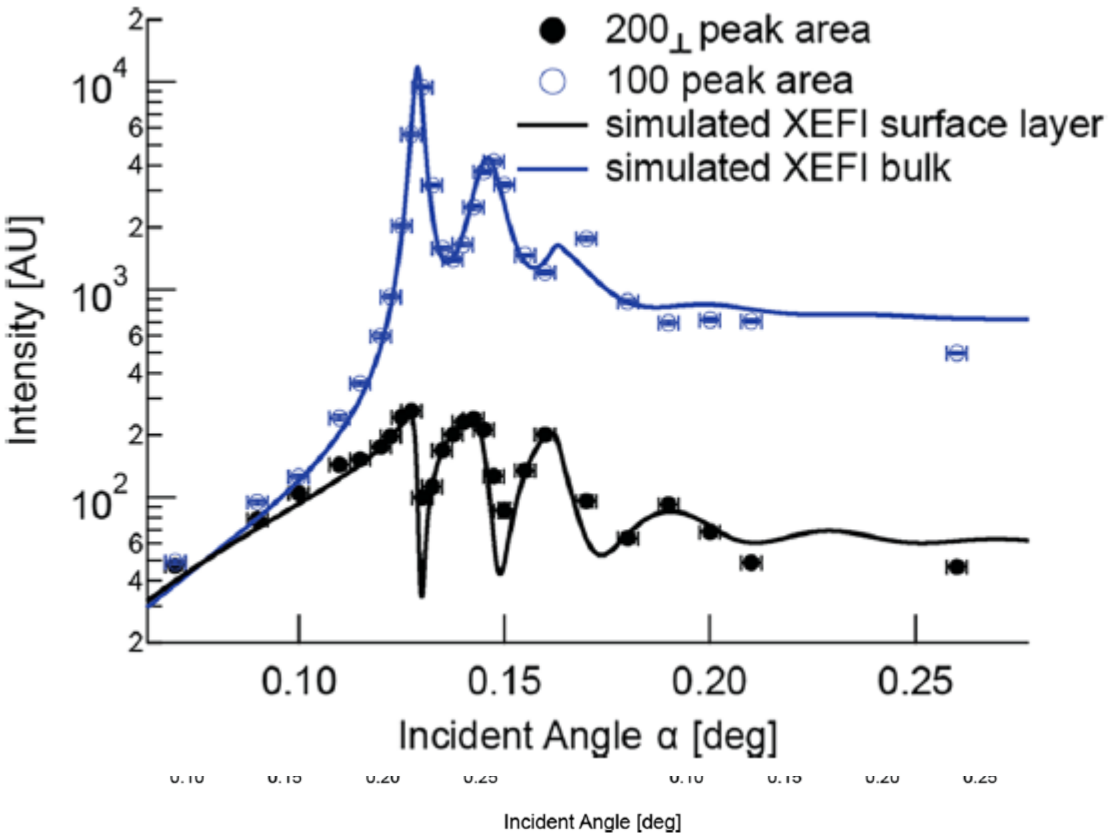
Crystalline Stratification





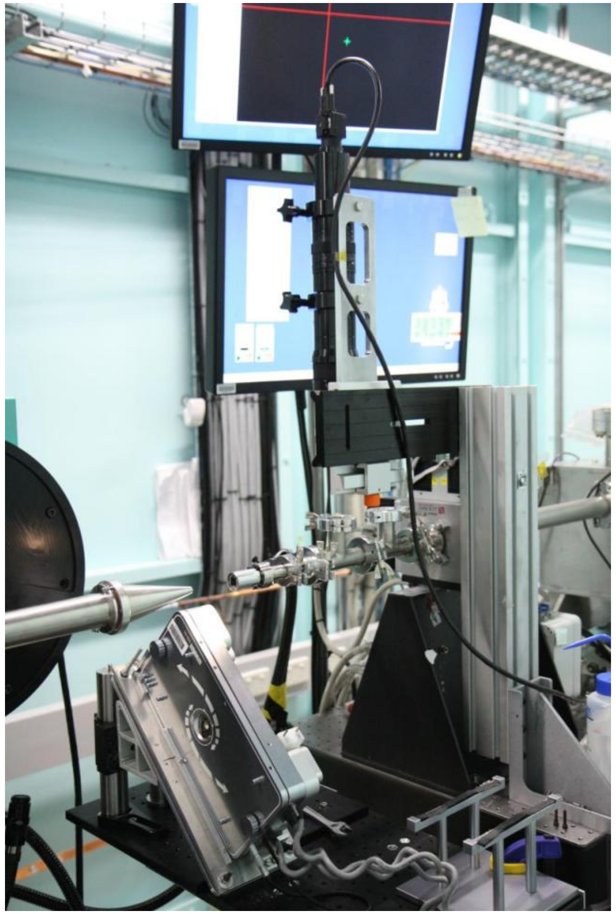
poly[(E)- 2,7-bis(2-decyltetradecyl)-4-methyl-9-(5-(2-(5-methylselenophen-2-yl)vinyl)selenophen-2-yl)benzo[lmn][3,8]-phenanthroline-1,3,6,8(2H,7H)-tetraone] (PNDI-SVS), 168 kg/mol, PDI = 2.1

Total film 70nm thick, 2nm roughness
Edge on layer, 5nm thick
Face on layer 65 nm thick
5nm transition

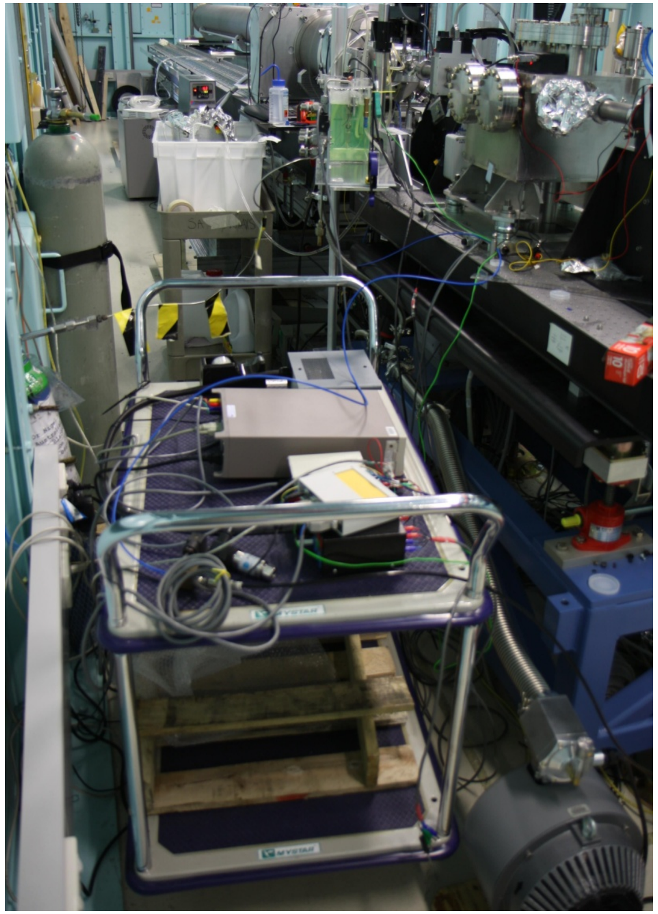
Gann, E. et al. *Macromolecules* **2018**, 51, 2979-2987.

Complex setups

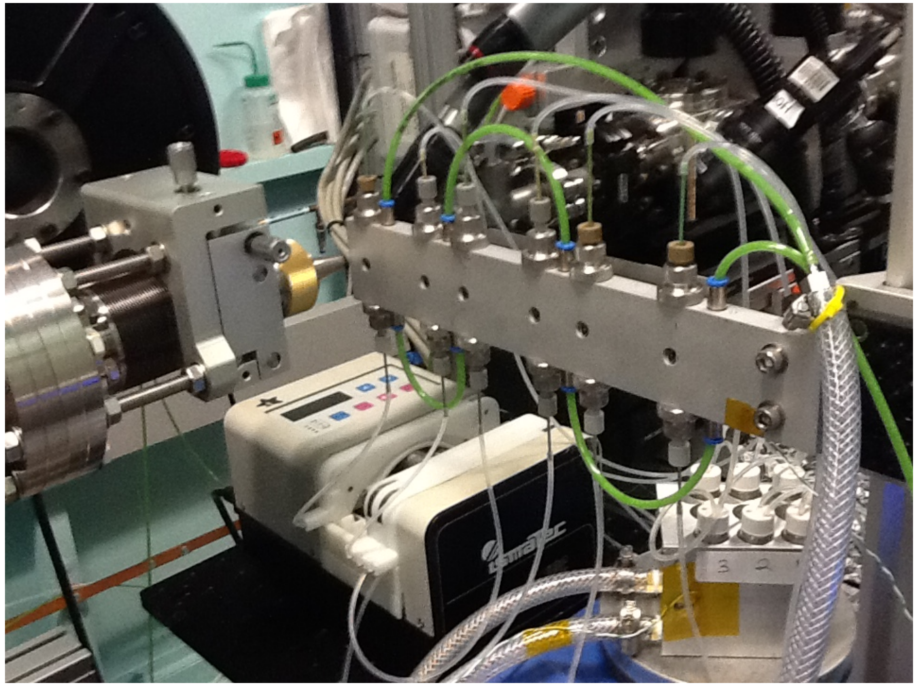
- In situ reactions, digestions, dissolution, precipitation
- self-assembled bulk phases, dispersions
- Microfluidics
- Rheology



Linkam shear stage (User-owned)

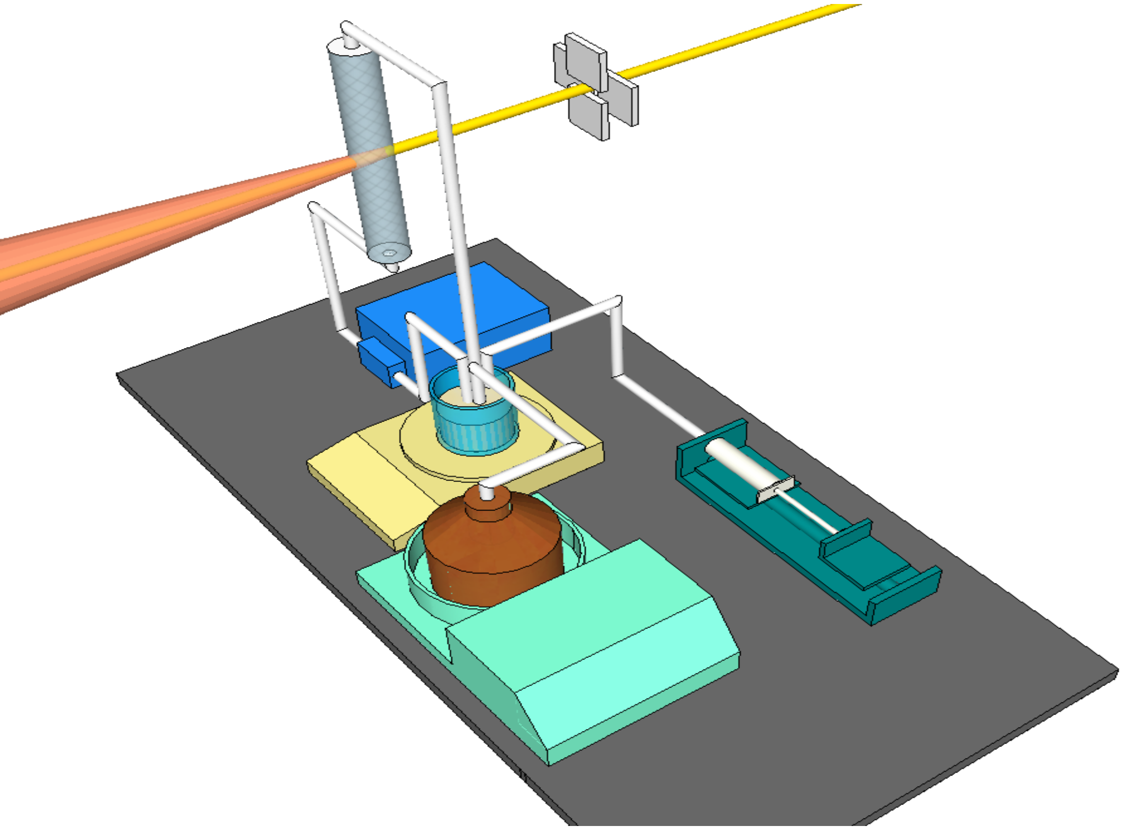


Lab in the hutch
(“everything at once”)



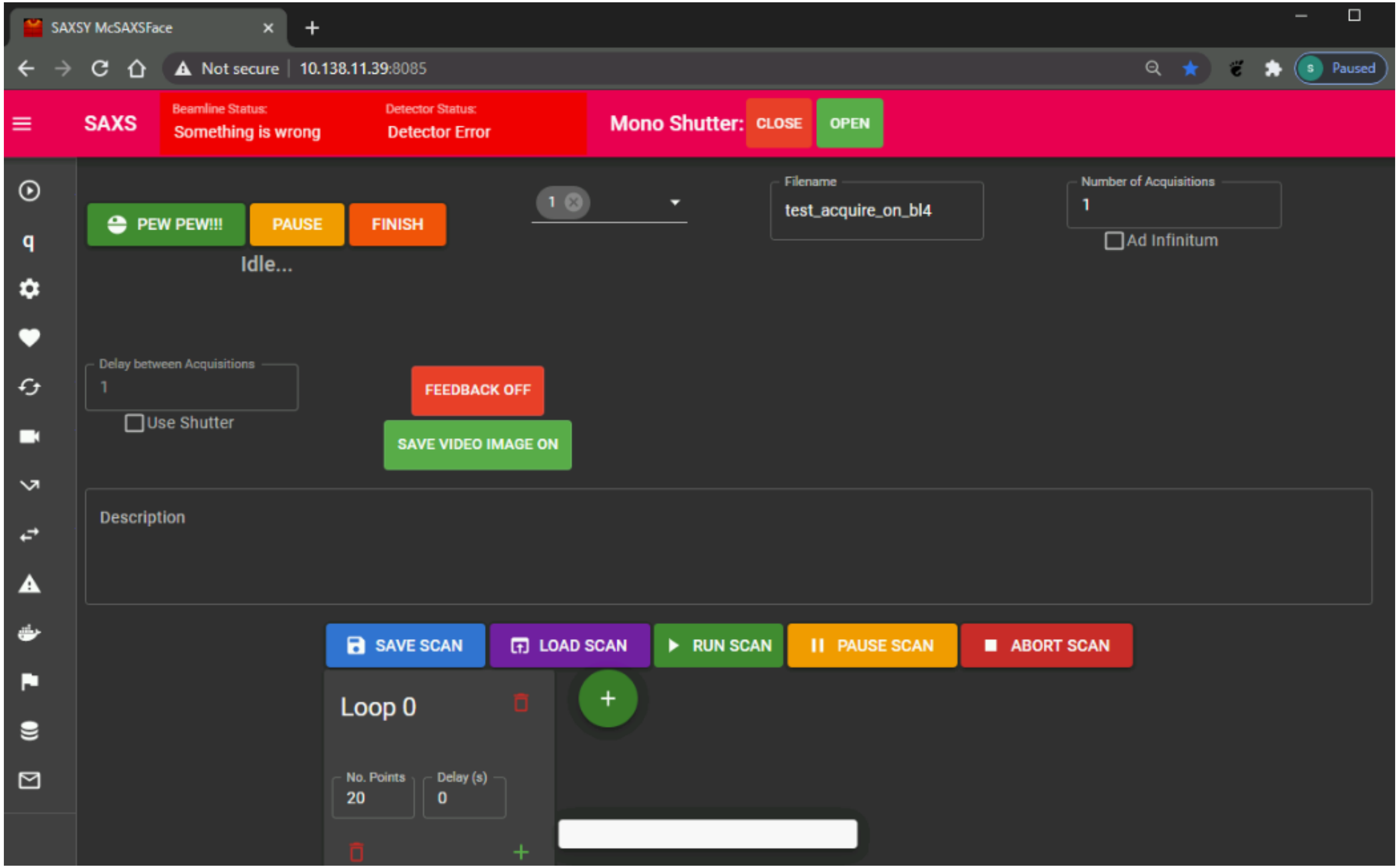
Multiple temperature controlled recirculation

Bring your own equipment? Sure, but…

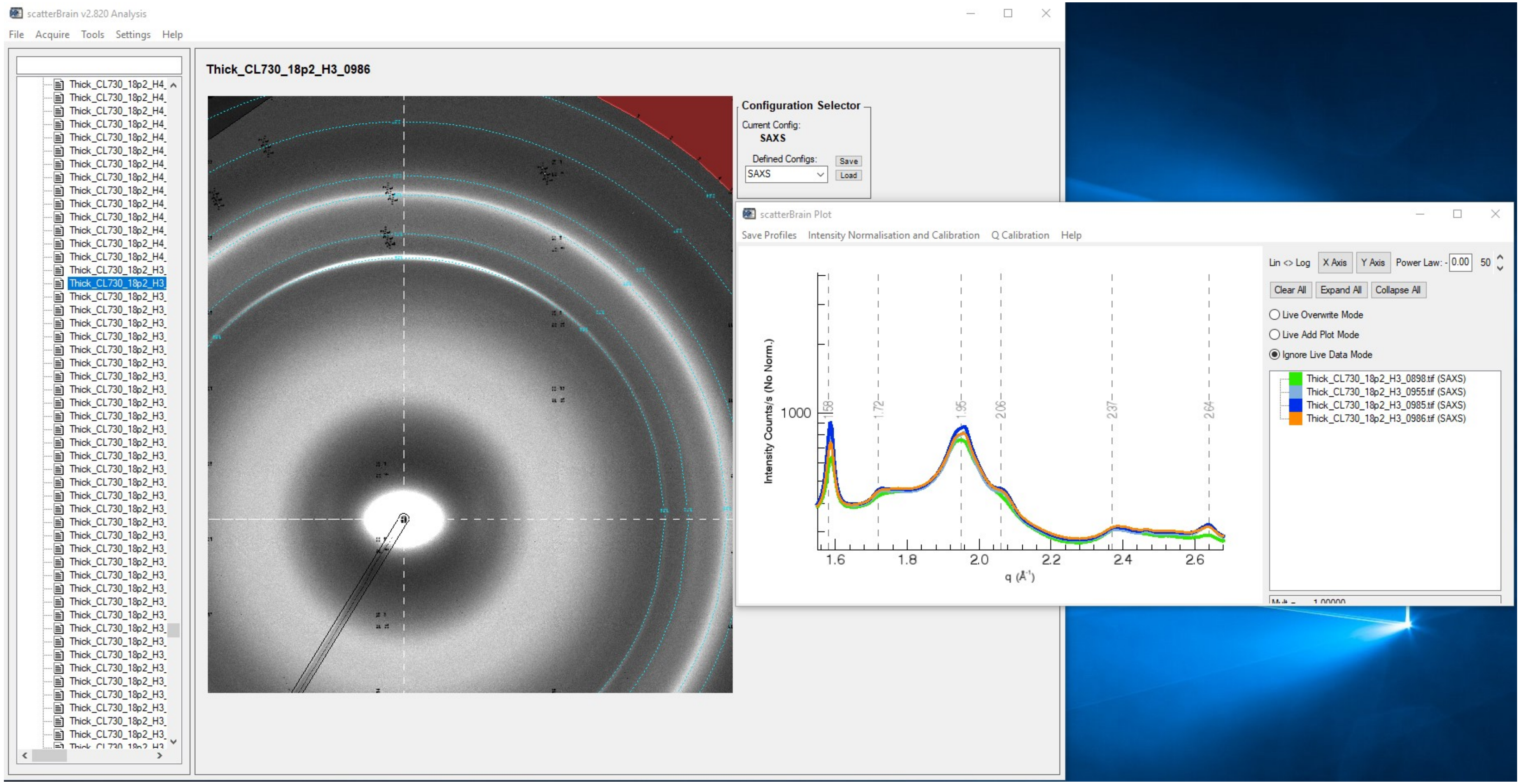


- Plan ahead
  - put it in your proposal
  - give details in proposal, esp. showing you've tested it
- must have valid electrical testing and tagging to be brought on site
- Need help?
  - let BL staff know if you need help
  - Mounting? M6 holes at 25 mm spacing is our standard hole pattern
  - Does it need interfacing with beamline controls ?
- Come at least the day before and set up

Collecting your data



Data view and analysis

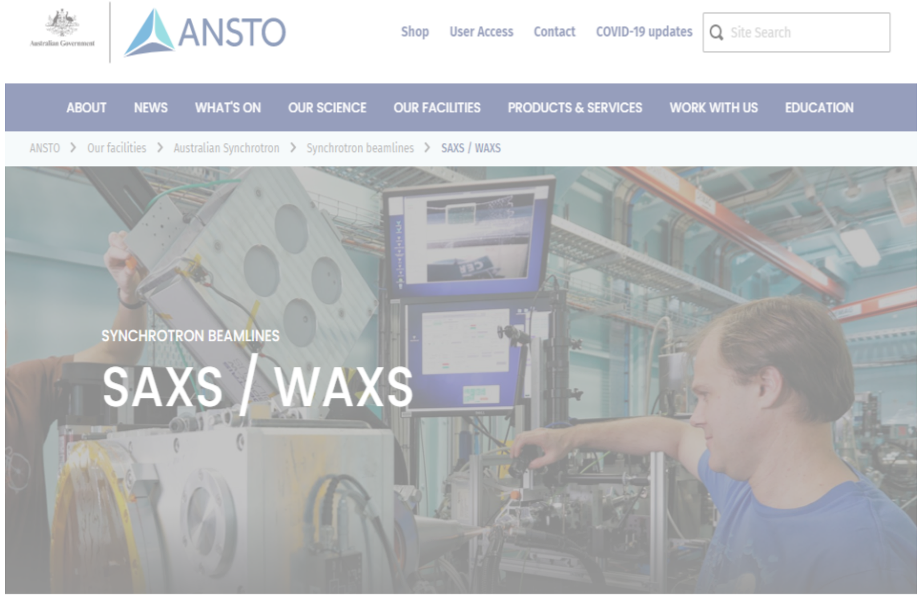


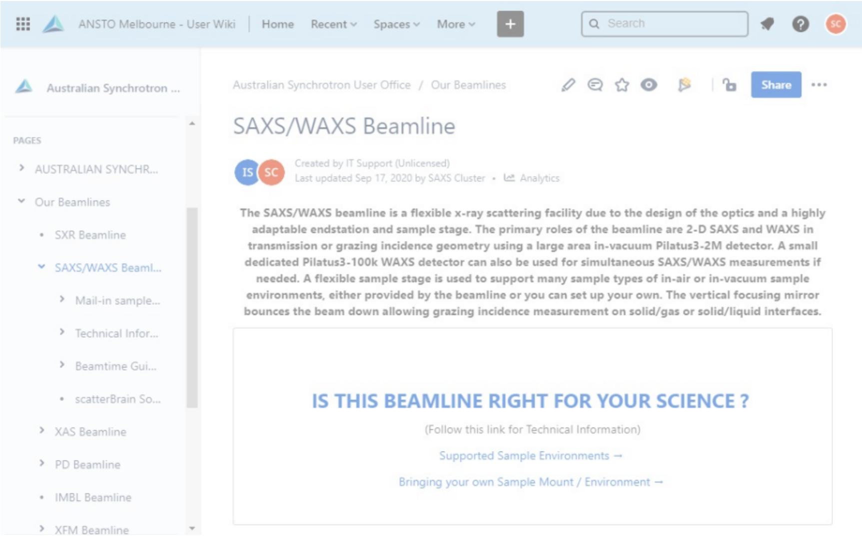
Want to know more?

Find us online!

SAXS / WAXS Website:

https://www.ansto.gov.au/user-access/instruments/australian-synchrotron-beamlines/saxs-waxs





SAXS / WAXS User Wiki:

https://asuserwiki.atlassian.net/wiki/spaces/UO/pages/22544387/SAXS+WAXS+Beamline

Thank you for your attention!

seibts@ansto.gov.au

Australian Government

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Thank you for your attention!

seibts@ansto.gov.au

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Writing a proposal

- Access to beamtime is competitive
  - Worth writing quality proposal
  - Make use of available space:
    - References
    - figures
  - Some beamlines provide specific guidance. Can be very helpful, aspects an be essential.
- Describe the scientific problem
  - What are you trying to solve?
  - How does it relate to national research priorities? Does it have a direct application?
- how will synchrotron experiment will enable you to solve the problem?
  - data analysis methodology ?
- Show relevant supporting information
  - Previous synchrotron experiments
    - Have you progressed the analysis? Published?
  - data showing you are prepared for the synchrotron experiment
    - Sample quality, complementary data, lab-SAXS data, ex-situ data
    - Planning calculations

Writing a proposal

- Establish why you require Synchrotron Light
  - Is the technique only available at a synchrotron?
  - Lack of access to an equivalent laboratory instrument is seen as poor justification
  - inadequate Signal to Noise ?
    - Do you have evidence (calculation or laboratory data)
  - Time resolved
    - What is the time scale, do you have evidence that changes happen on expected time scale?
  - In-situ experiments
    - Why does it need to be in-situ?
    - Explain relevant ex-situ work
  - For simultaneous measurements (combining synchrotron measurement with another technique)
    - Does it REALLY need to be simultaneous?

Writing a proposal

- Experiment plan
  - What equipment are you using, especially sample environments.
    - Your equipment, and/or ours?
  - Be detailed about experimental conditions
    - Explain why have chosen particular conditions, consistent with the scientific problem.
- Estimate the amount of beamtime needed
  - Be realistic, don’t pad out the time
  - allow for setup, contingency
  - show how you calculated the time requested (detail !)
- Data analysis
  - What piece of information are you trying to determine, and how will you determine it.
  - New Users – describe your data analysis approach, which might be by collaboration.

Writing a proposal

- Track record
  - If you are new, great ! Show non-SR track record. List publications that show you can progress experiment through to publication
  - List student(s). Completion timeframe(s).
  - What have you done with your last experiment(s) ?
    - It didn’t work? Fine, describe it, and explain what has changed to enable it to work this time.
    - Still working on data
      - Show progress. Include some processed data
    - List Publications
- Ask for advice/assistance
  - PAC members
  - Beamline staff
  - in advance (or after unsuccessful proposal)

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What do you need to do when you want to use our beamline?

Answer:

Apply for beamtime!

Applying for beamtime

For open-research Users:

1. Beamtime costs you nothing
2. Travel and accommodation is provided
   - Day before/after experiment
   - Up to 3 personnel

SAXS/WAXS experiments

- Typically 1-2 days
- You will need to do
  - Data collection
  - Data processing
  - Data analysis/interpretation

3 proposal rounds per year

1) Deadline: Early September
End January to May

2) Deadline: Early February
End May to August

3) Deadline: late May
September to December

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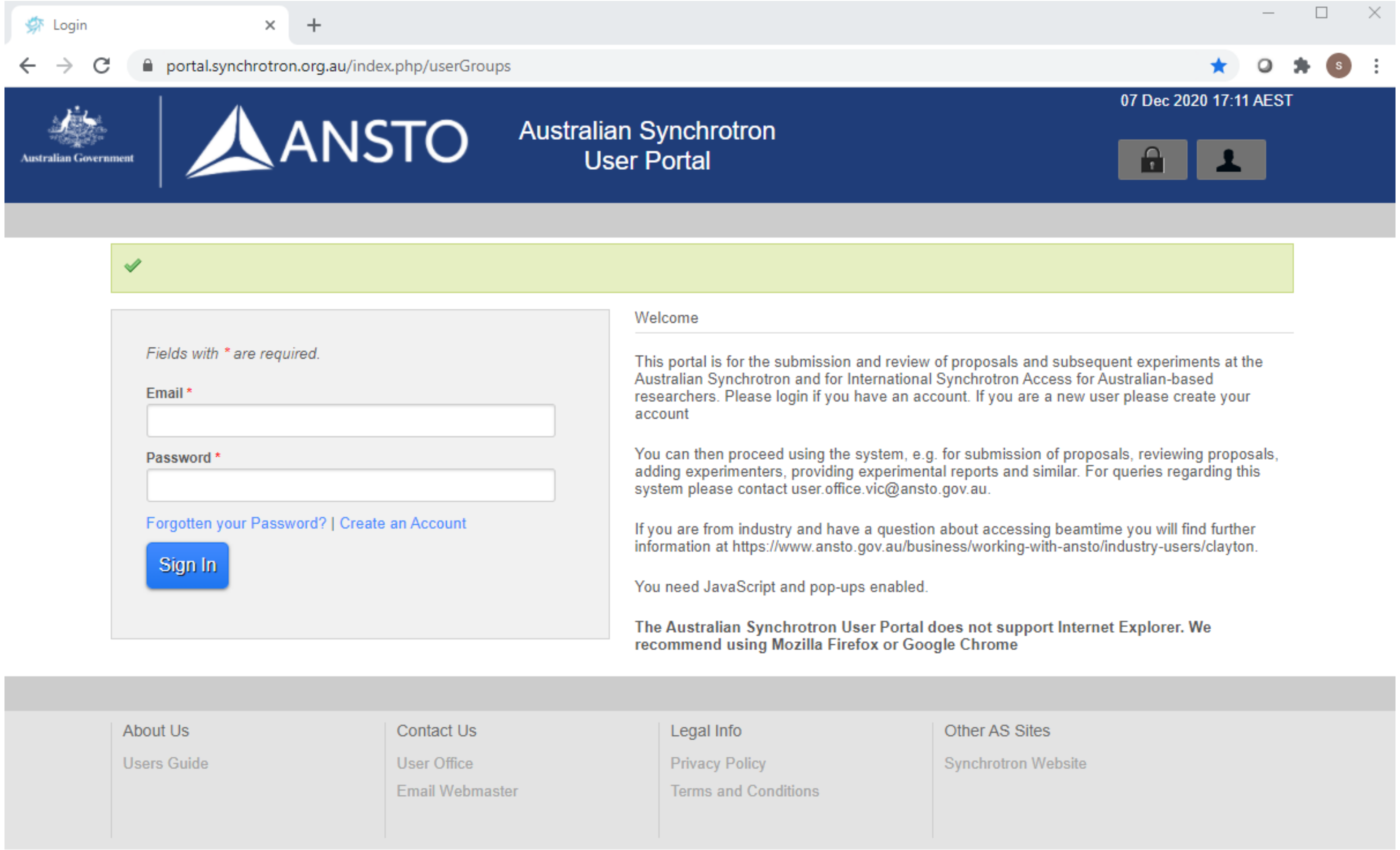
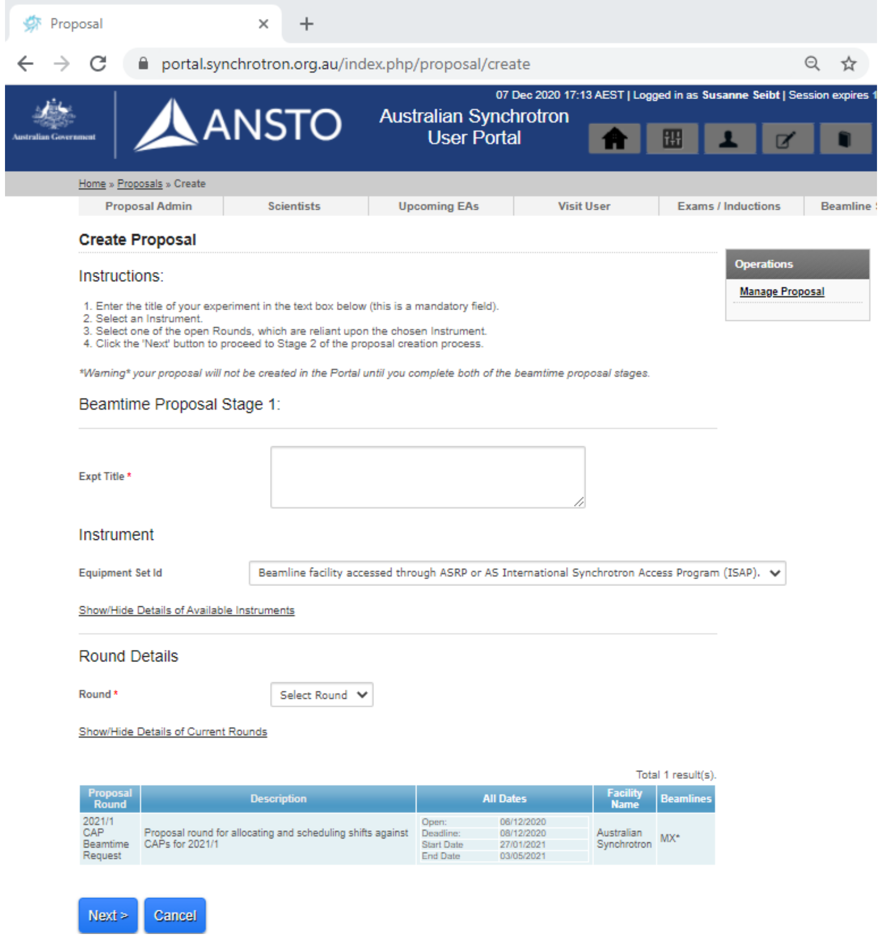
Applying for beamtime

https://www.ansto.gov.au/our-facilities/australian-synchrotron/melbourne-access





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Proposals

- Guidelines for Applicants
- Externally reviewed according to the following criteria:

Criteria	Percent of total Score
Quality of the Scientific Proposal	40%
National Benefit & Applications of the Proposed Research	30%
Track Record	30%
Need for Synchrotron Light	“Yes” or “No” decision

- Proposal Advisory Committee (PAC) reviews the referee scores and comments and adjusts the scoring (if required) to a common scale. The PAC also recommends the amount of beamtime that should be awarded to each proposal if it is successful.

The team

SAXS / WAXS beamline

Australian Synchrotron

Careers

https://www.ansto.gov.au/careers