Psiché

Pression, structure et imagerie par contraste à haute énergie

Psiché: Synchrotron tomography and diffraction for materials science

Andrew King, Synchrotron SOLEIL
J.-P. Itié, N. Guignot, J.-P. Deslandes, E. Boulard,
P. Zerbino, A. Delmotte, and many others at SOLEIL

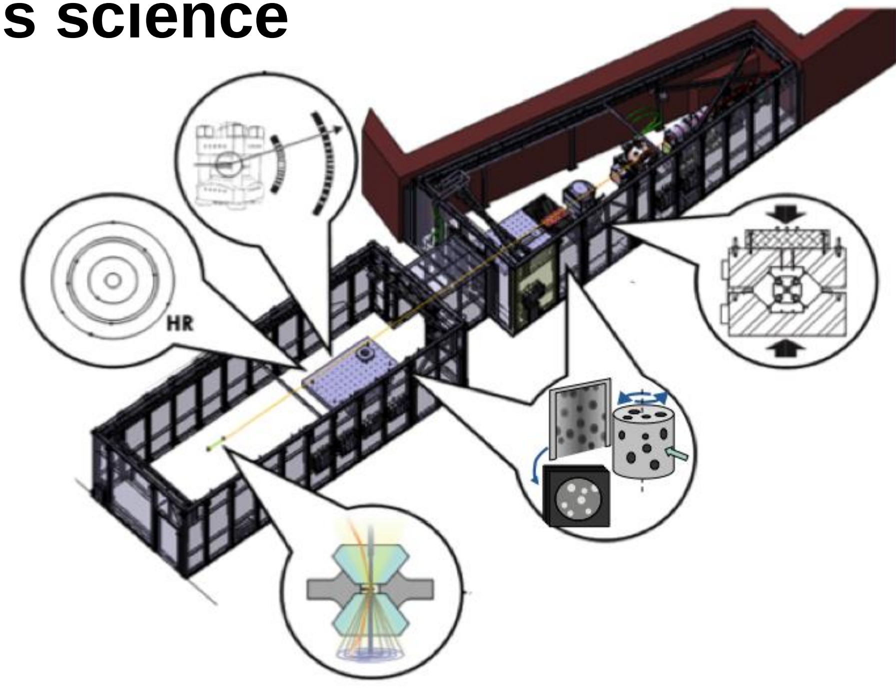
SOLEIL:

- The French national synchrotron radiation source
- Located at St Aubin, 25 km south of Paris
- First electrons accelerated 2006
- 2.75 GeV storage ring, 354 m circumference, typically ~~430~~ 450 mA top up (will go to 500 mA)
- Peer reviewed proposal system, open to all.
  - Next call 15 February 2016
- Reimbursement for French institutions of travel, accommodation, food
- For European users reimbursement via European Calipso program - finished May 2015…

Psiché beam line

Psiché
Pression, structure et imagerie par contraste à haute énergie

- Pression, Structure et Imagerie par Contraste à Haute Energie
- Highest energy beam line at SOLEIL
  - In vacuum wiggler, photons from ~20-100 keV
- Two types of experiment
  - **Diffraction at extreme pressures**
  - **Tomography for materials science**
- Polyvalent and flexible



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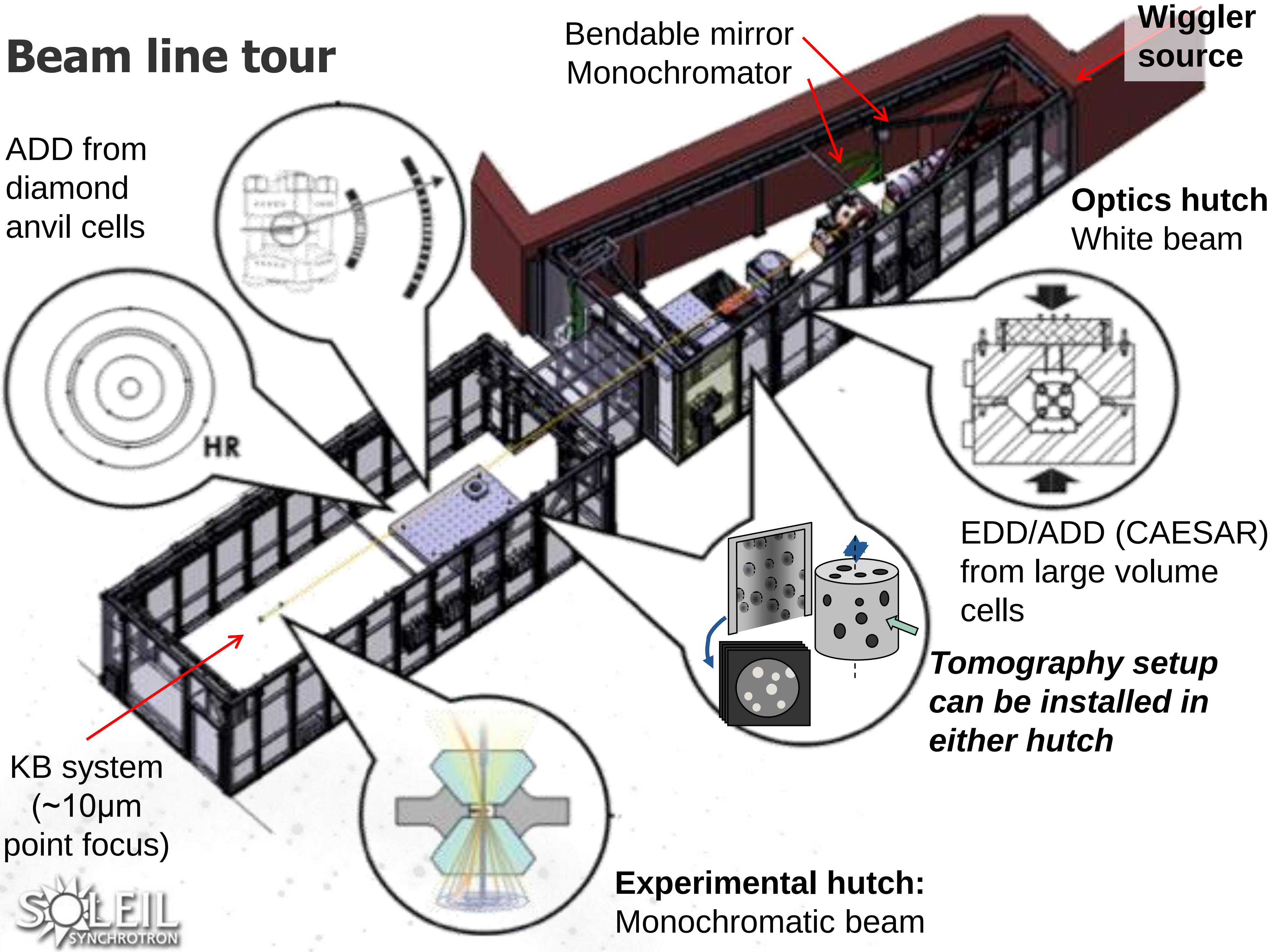
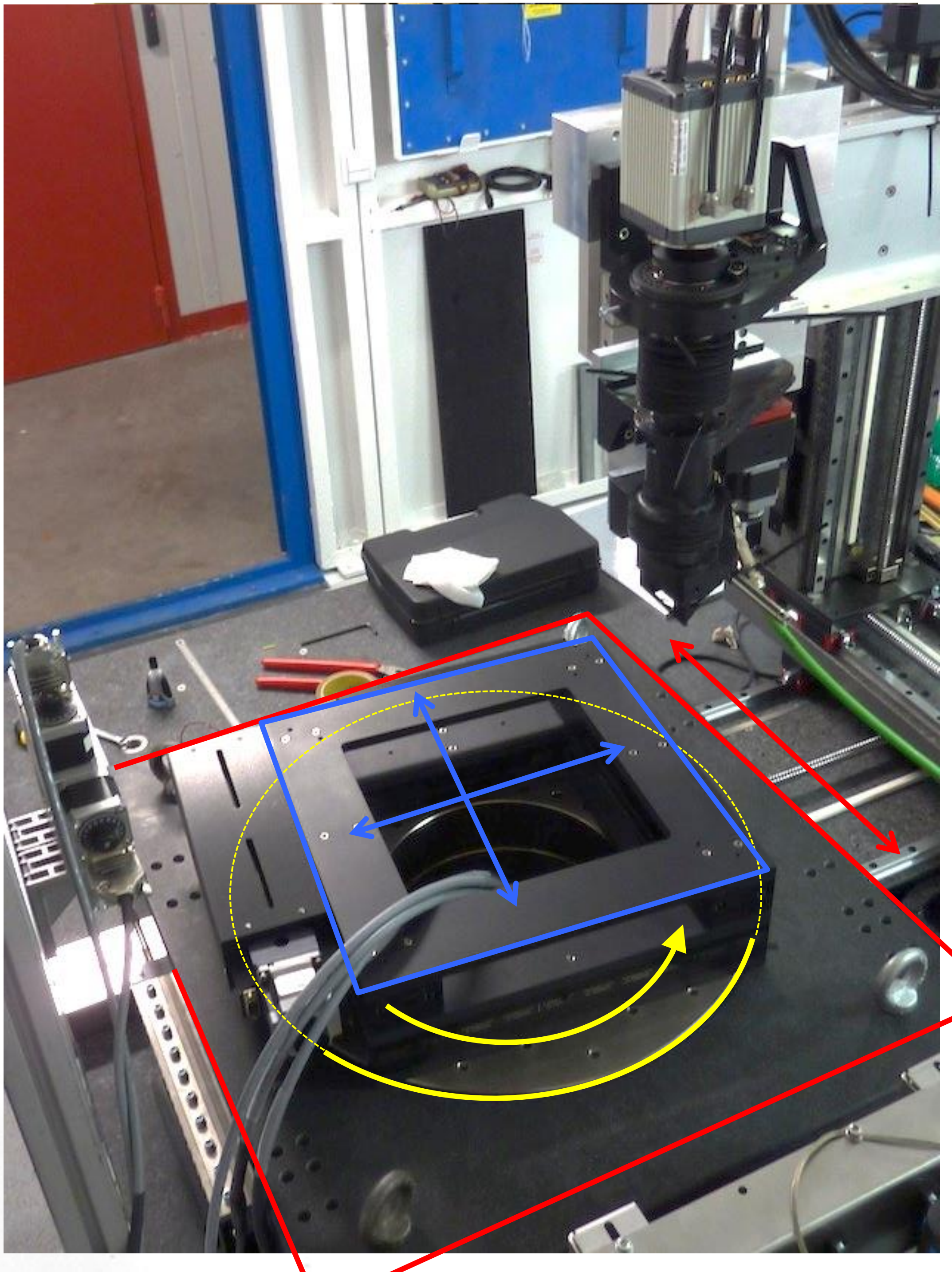
Beam line tour



The Psiché tomograph

- Optimised for *in-situ* materials science
- Built by Leuven Air Bearings
- High precision, heavy load rotation stage (>50kg, ~100nm error, 60 rpm)
- Fully open 250 mm aperture
  - Space for in-situ rigs
  - “build downwards”
- Collaboration with M. Bornert and N. Lenoir (ENPC)



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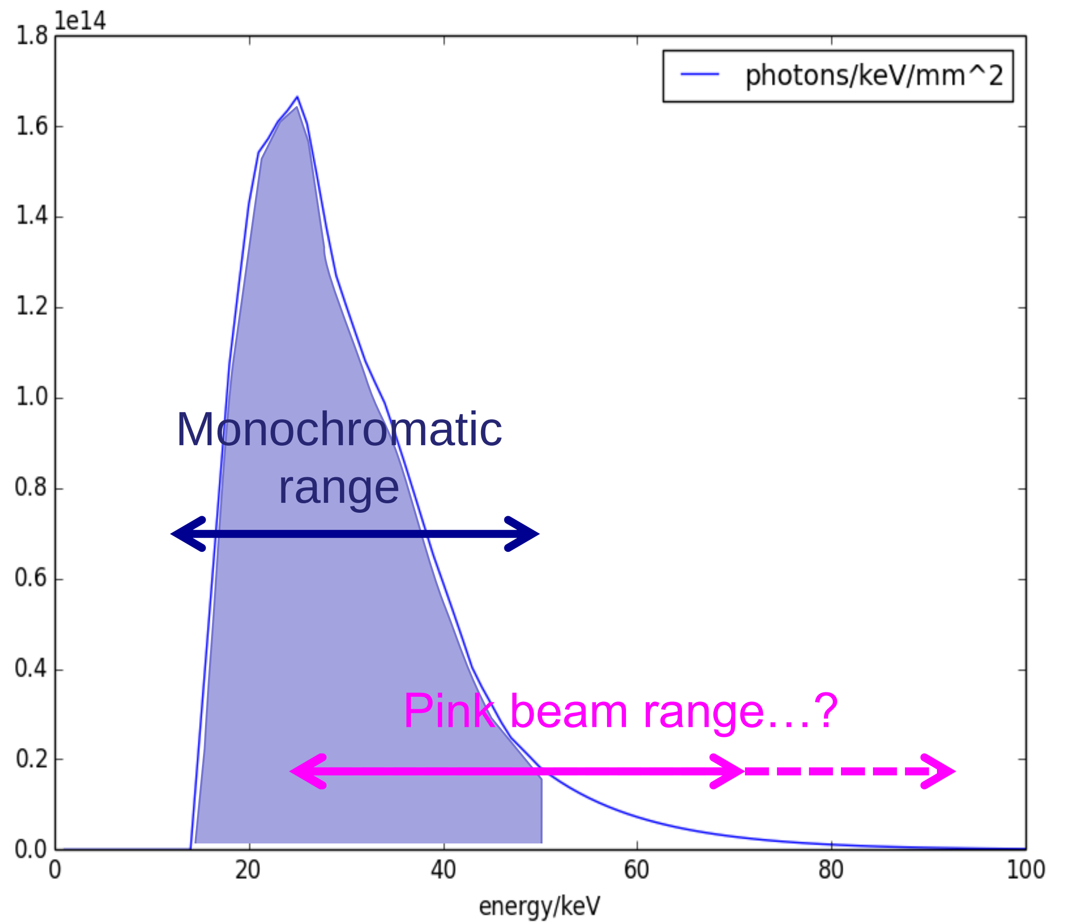
Illumination

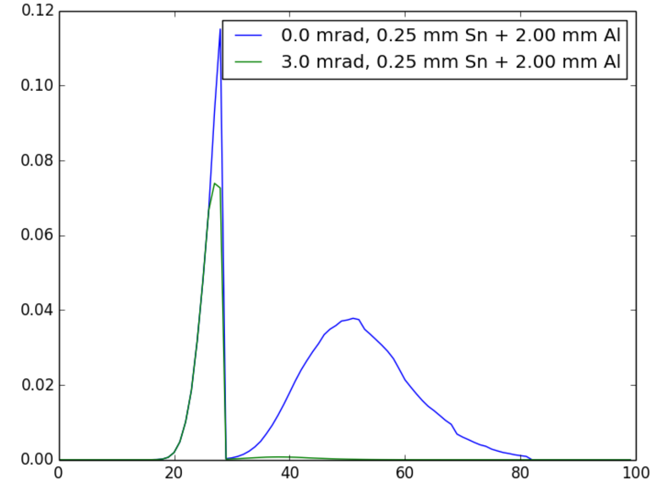
- Max beam size ~15 x 4.5 mm
  - samples to 30 mm dia. using half acquisition
  - Can focus beam vertically to increase flux on small samples
- Si 111 Bragg mono
  - ($\Delta E/E \sim 10^{-4}$, 20-50 keV)
  - Delicate samples
  - Diffraction contrast tomography
- White / Pink beam
  - Much faster
  - Use filters (absorption edges) and mirror to tune spectrum
  - exploit the highest energies

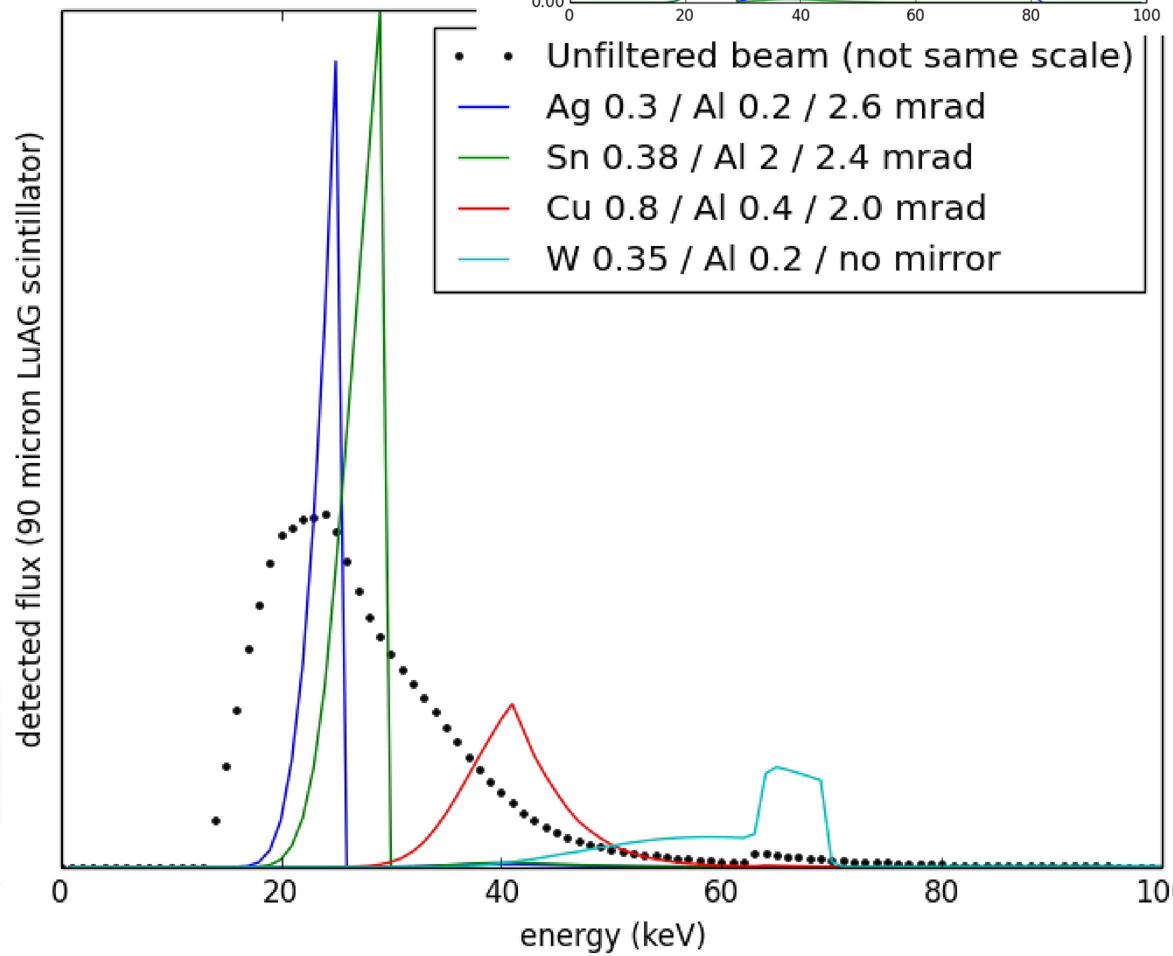


Illumination – Pink beam

- Combine mirror (as low pass filter) and filters.
- Some examples giving about 1% transmitted power

  ~25 - 65 keV
- Cooled filter support and sandwich system allows low melting point filters like tin, even at high power
- Sufficiently monochromatic for phase contrast and minimal beam hardening





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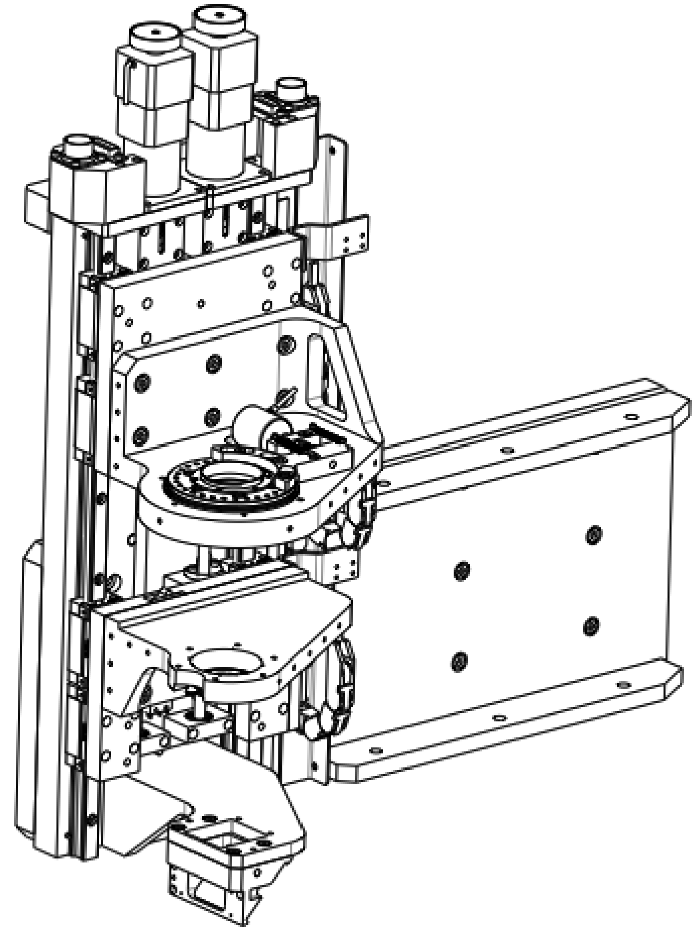
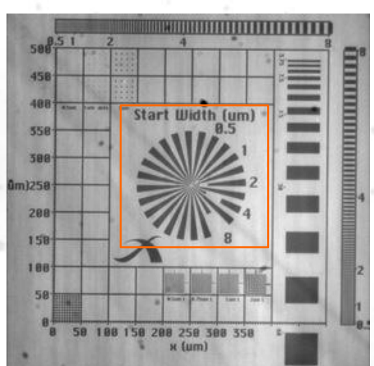
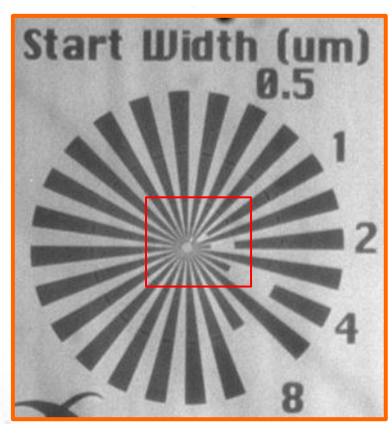
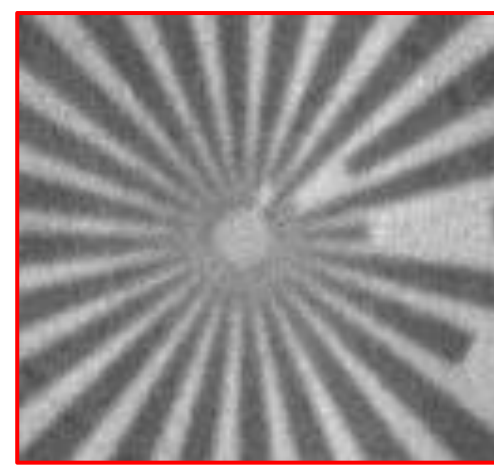
White beam compatible detector

- Designed in-house at SOLEIL
- 0.3 – 8 µm pixel size
- Hamamatsu sCMOS camera
  - Up to 100 2k^2 pixel frames per second sustained (20 mins)
  - Cooled for low noise
- Flexible design:
  - Motorised camera and objective
  - X-ray transparent, compact head
  - Plan to go to 0.13 µm pixels, ~0.5 µm resolution
  - Or very fast camera optics
  - Zoom optics









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Instrument control and computing

- Psiché uses **Spyc** for control
  - Official SOLEIL Python based command line + scripting
- SOLEIL's **FlyScan** for fast (continuous) acquisition
- Via Spyc:
  - Focus/zoom/avoid collisions in detector
  - Align the instrument
  - Setup and launch FlyScan acquisition
  - **Can quickly and easily develop experiments**
- Local data storage and reconstruction server in place.
  - 24 + 60 TB storage, ~~2~~ 3 NVidia K40 GPUs
  - Reconstruction with PyHST2 (ESRF)

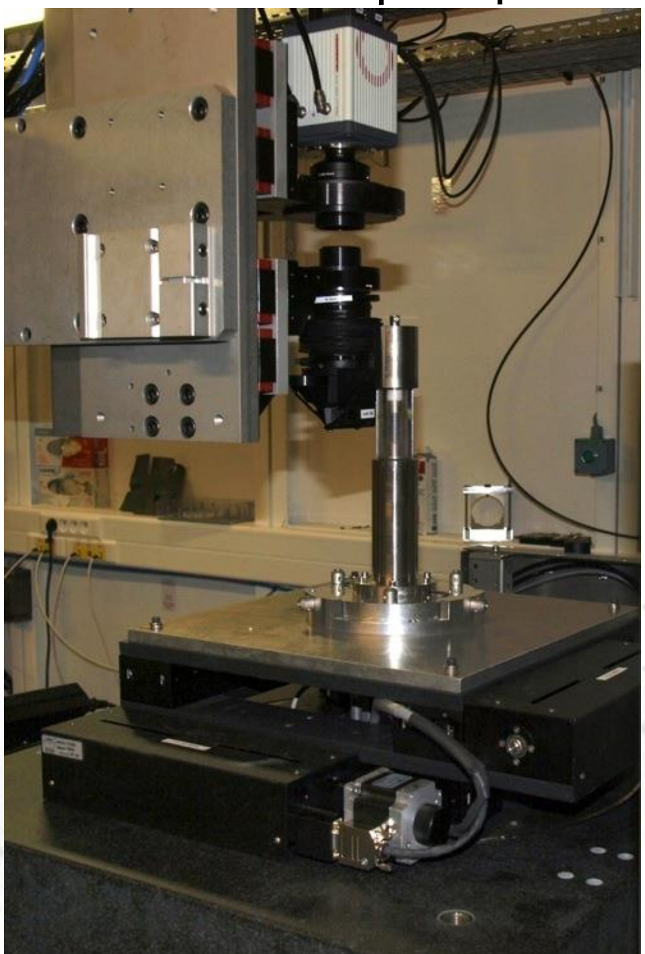
Technical summary

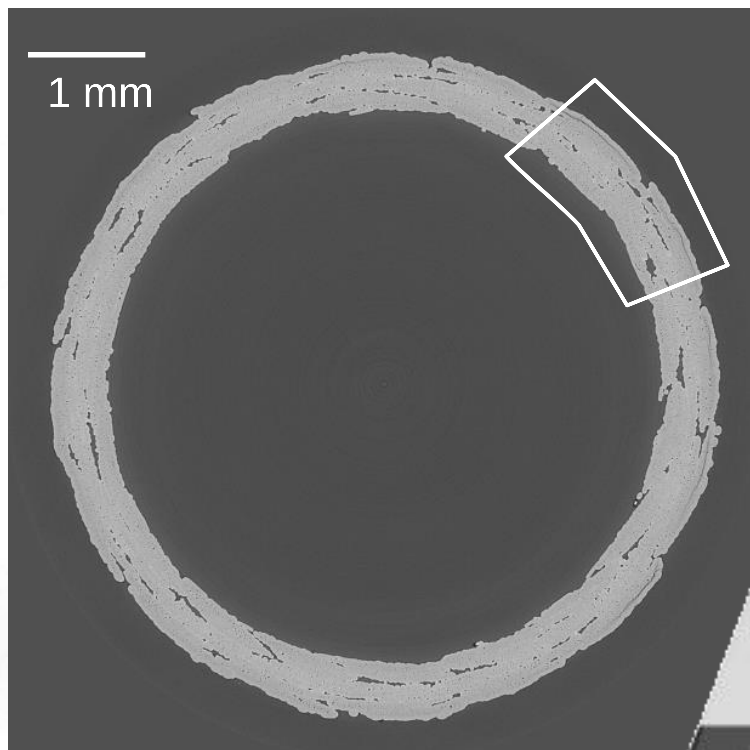
Strong points of Psiché for tomography:

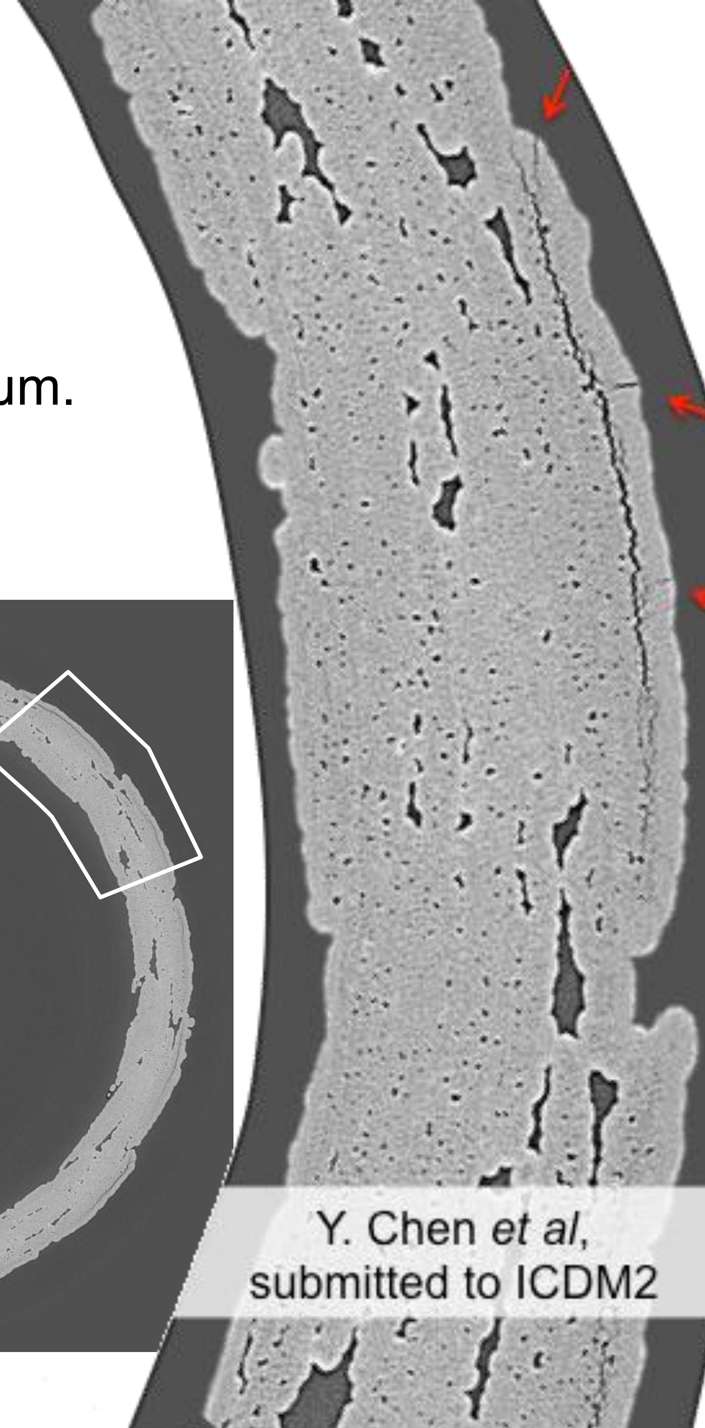
- Designed for in-situ materials experiments
- (Very) high flux (➔500 mA, in-vacuum wiggler @ 24 m)
- Flux at high energies (fast acquisition at 65 keV pink beam, good for steel)
- Good spatial resolution and image quality
- Useful phase contrast
- Fast, efficient, local data storage and processing
- Flexible control system
- Options to combine tomography and diffraction

SiC composite failure mechanisms

- L. Gelebart, *et al*…
- In-situ loading in ENPC machine
- 33keV, 5μm, 35mm
- Now repeated in pink beam - ~40 keV, 2.6μm. Next step 1.3μm x 4000 pixels

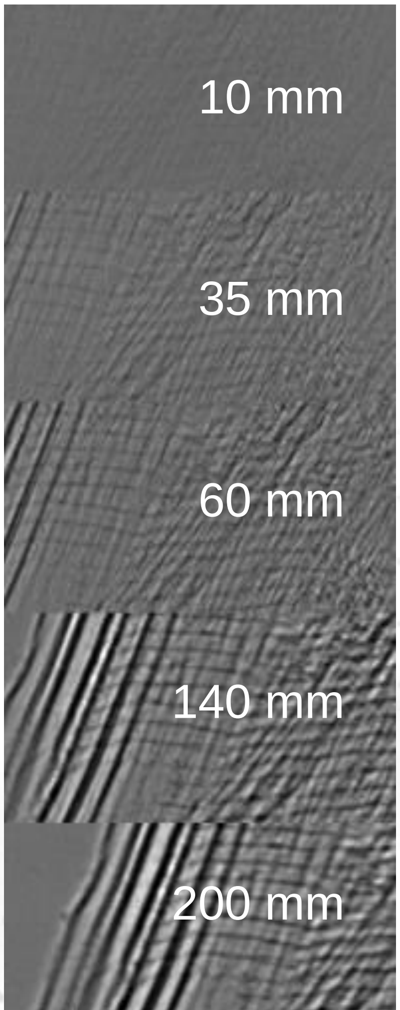


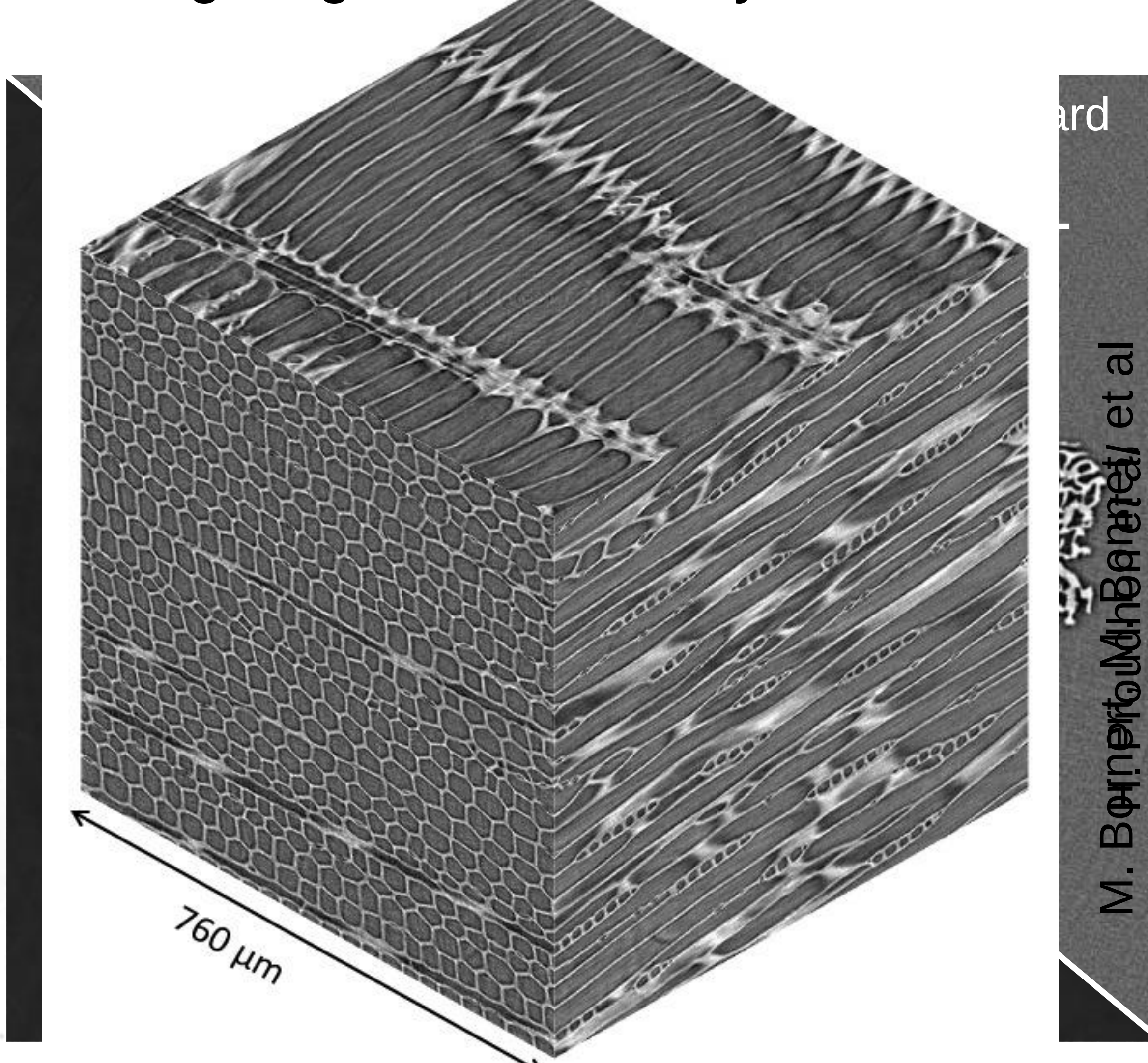




Wood: Phase contrast imaging

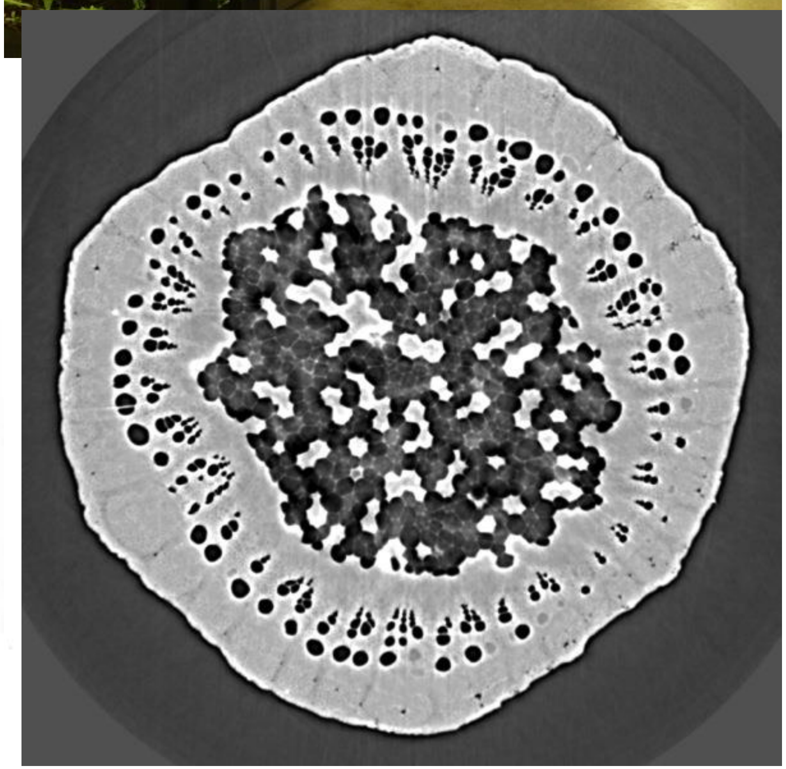
- Beam line not designed for this, but it works well
- Phase retrieval using Paganin filter in PyHST2





Plants in-situ (April 2015)

- Studying water in the xylem in response to drought
- The hole in the table allows imaging the top of tall plants – unique to SOLEIL and ENPC
- Next beamtime in May 2016



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- Good spatial resolution and image quality
- Useful phase contrast
- Fast, efficient, local data storage and processing
- Flexible control system
- ***Options to combine tomography and diffraction…***

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Diffraction

- Measure radiation scattered from periodic structures
- Tomography observes real space
- Diffraction observes reciprocal space
  - X-ray diffraction gives information on the scale of interatomic distances and crystals planes
  - Peaks in spectrum correspond to planes

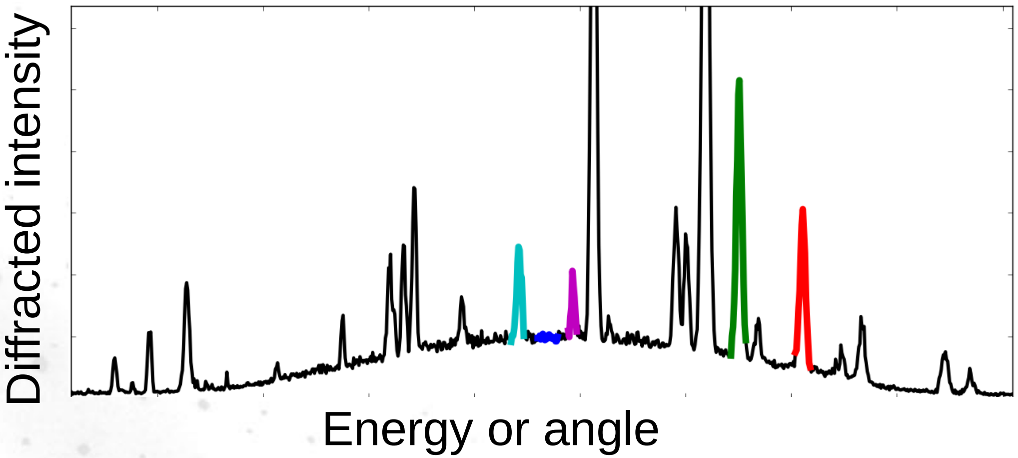
Index peaks ➔ Identify crystalline phase

Peak shifts ➔ Measure elastic deformation

etc…

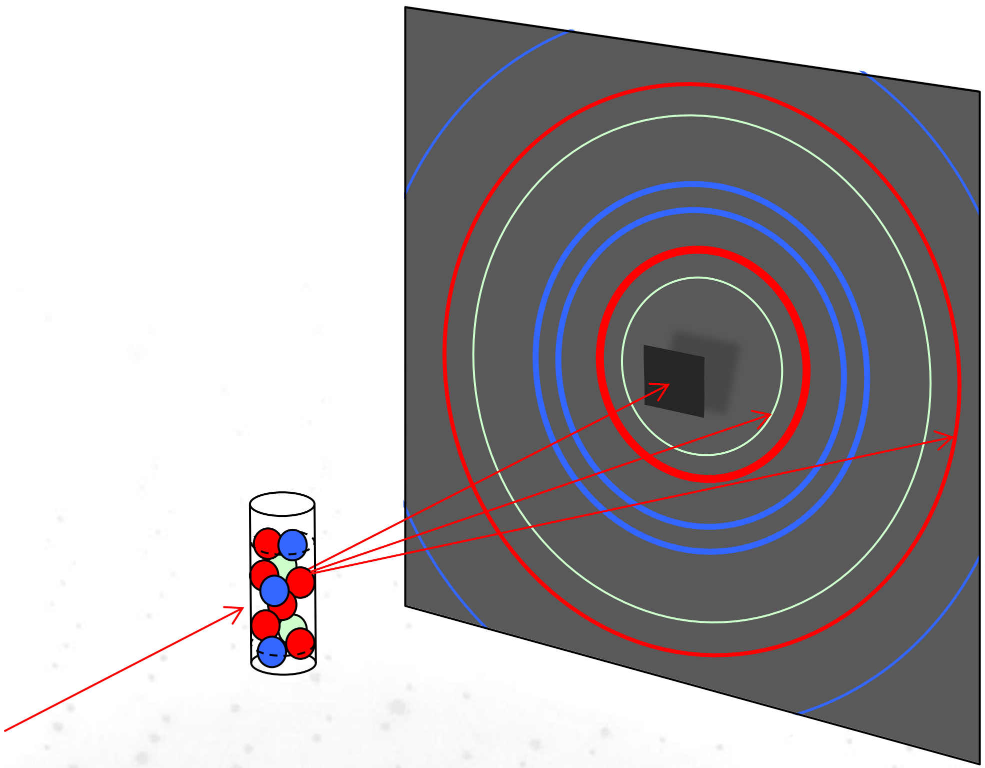
Bragg's law:

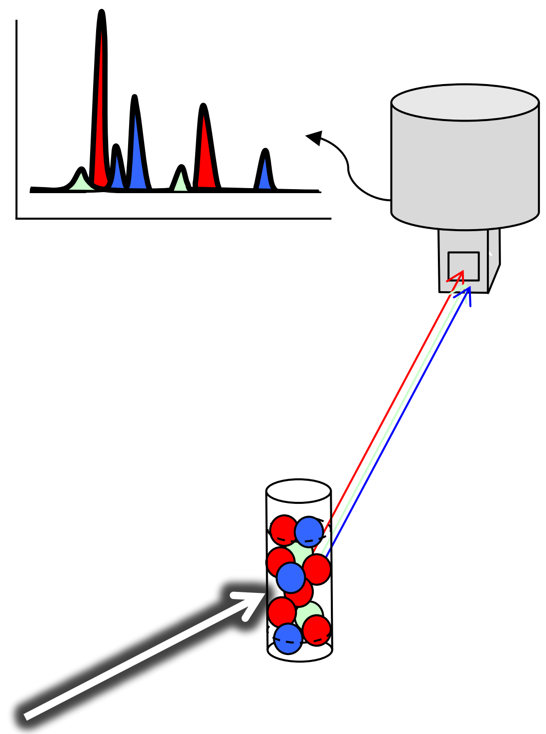
$$\lambda = 2d \sin(\theta)$$



PSICHE: Two mode of diffraction

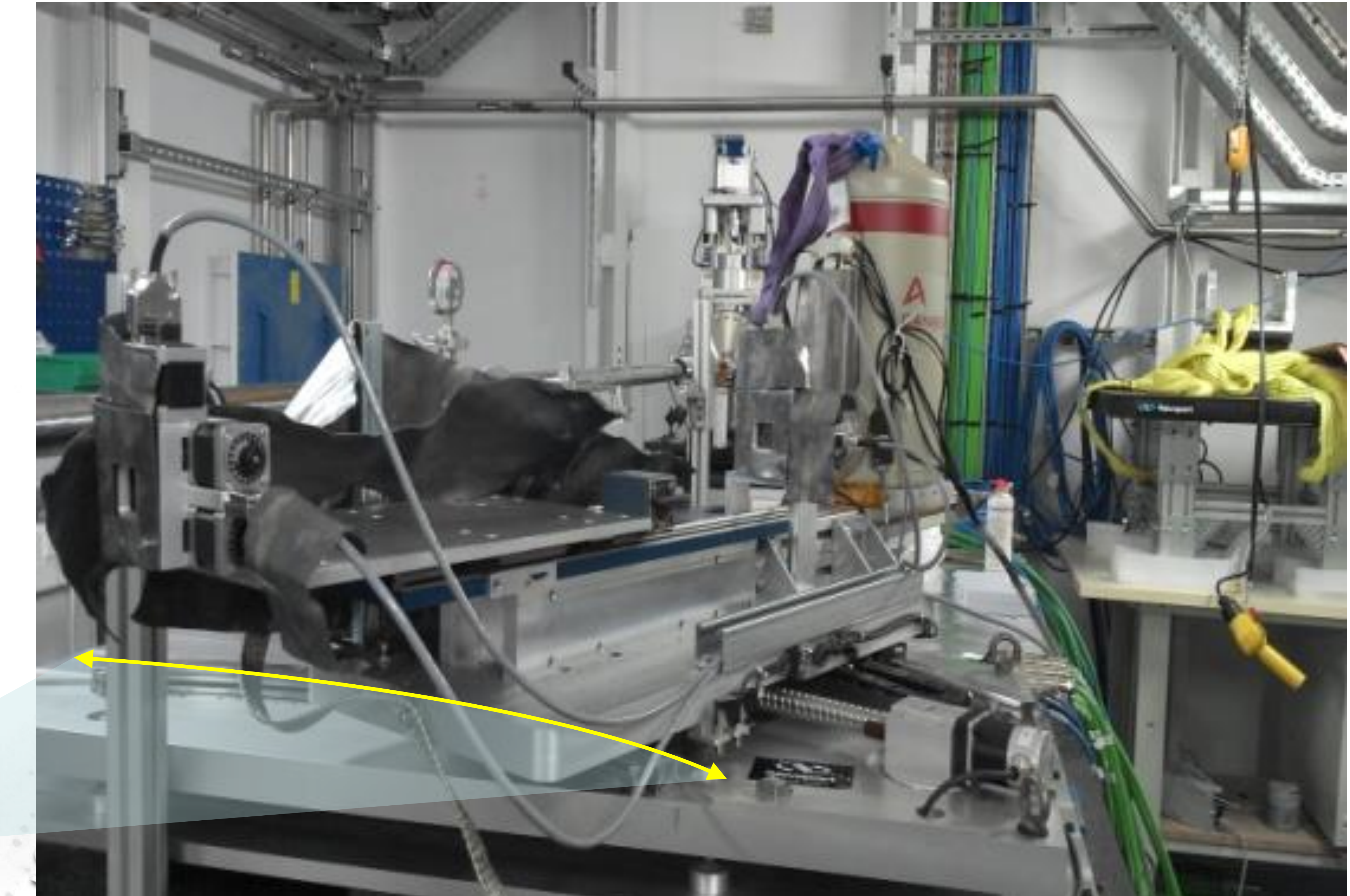
- Monochromatic beam and angular dispersion
  - Fix λ, measure θ, find $\boldsymbol{d}$
- Polychromatic (white) beam and energy dispersion
  - Fix θ, measure λ, find $\boldsymbol{d}$





What possibilities for combining with tomography?

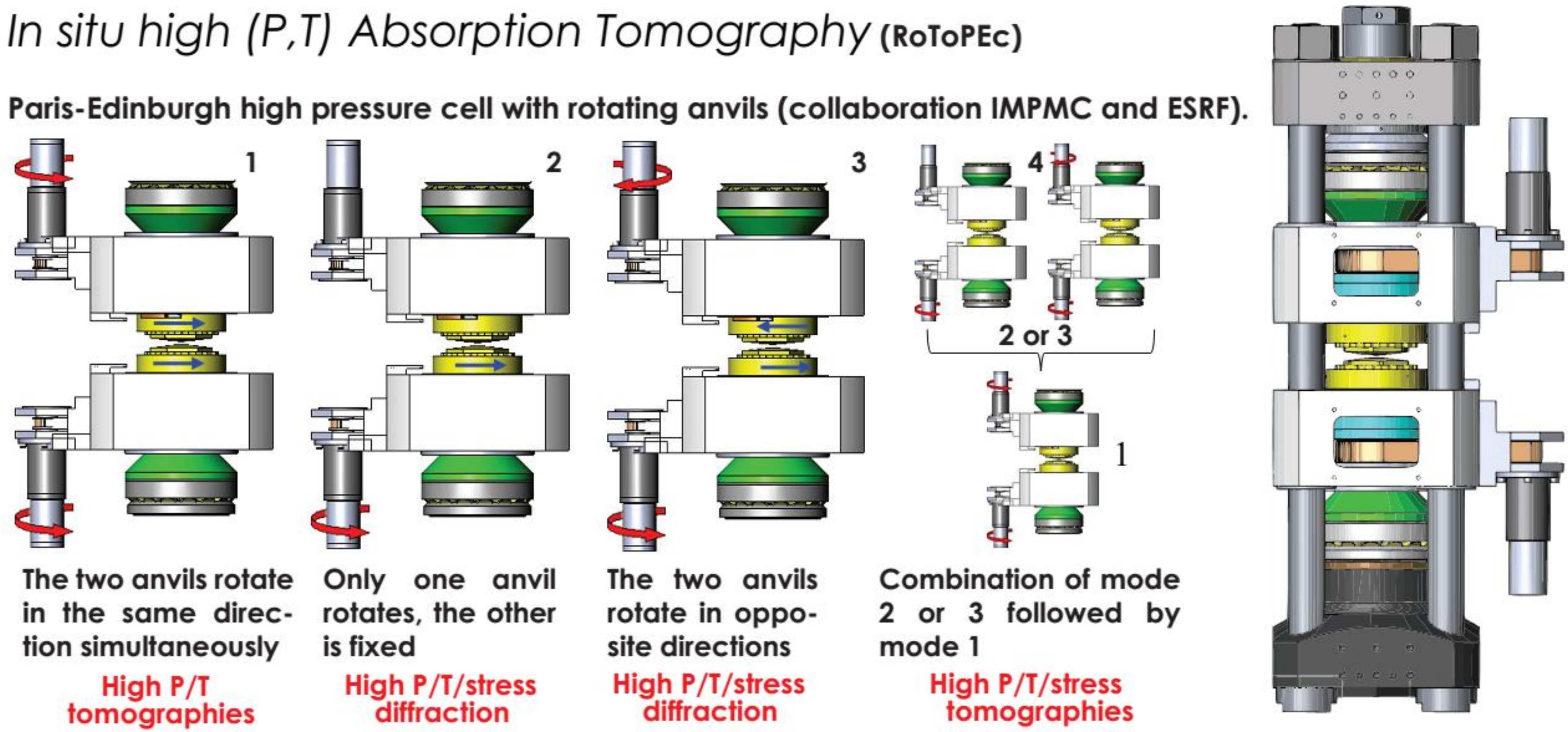
Energy dispersive setup: CAESAR



two theta / deg   two theta / deg

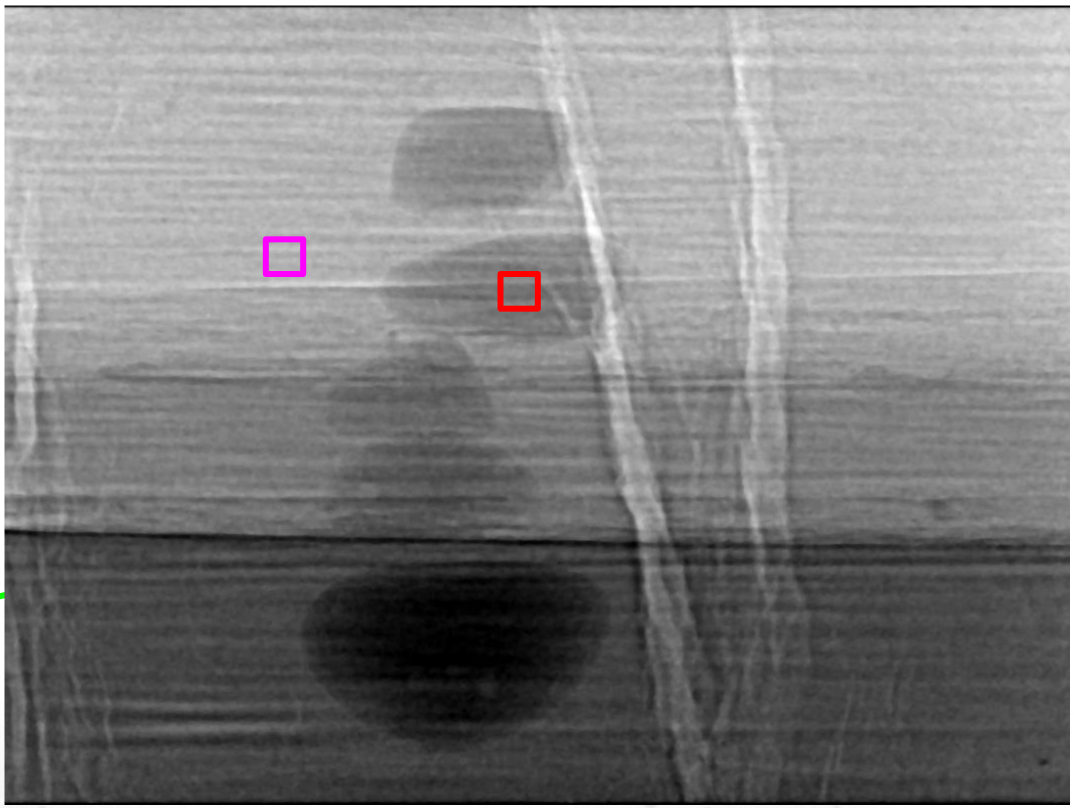
First combined experiment

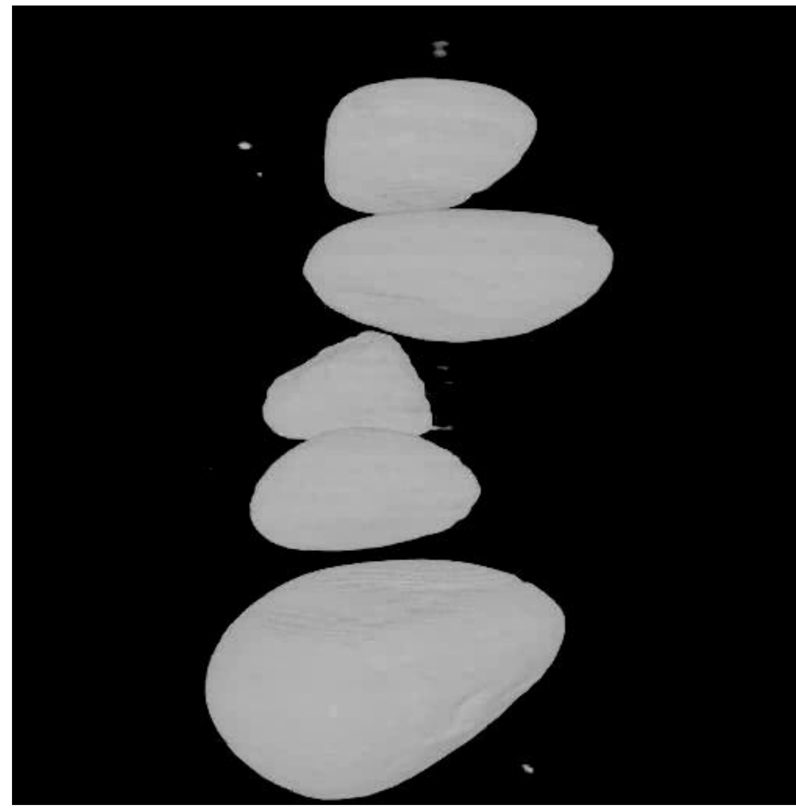
- RoToPEc. Now done three user experiments
- Simultaneous imaging and diffraction



RoToPEC

- Combined pink beam imaging and energy dispersive diffraction (Caesar system – N. Guignot)
- Measure volume as f(pressure) in amorphous materials
- Radiography greatly assists diffraction alignment

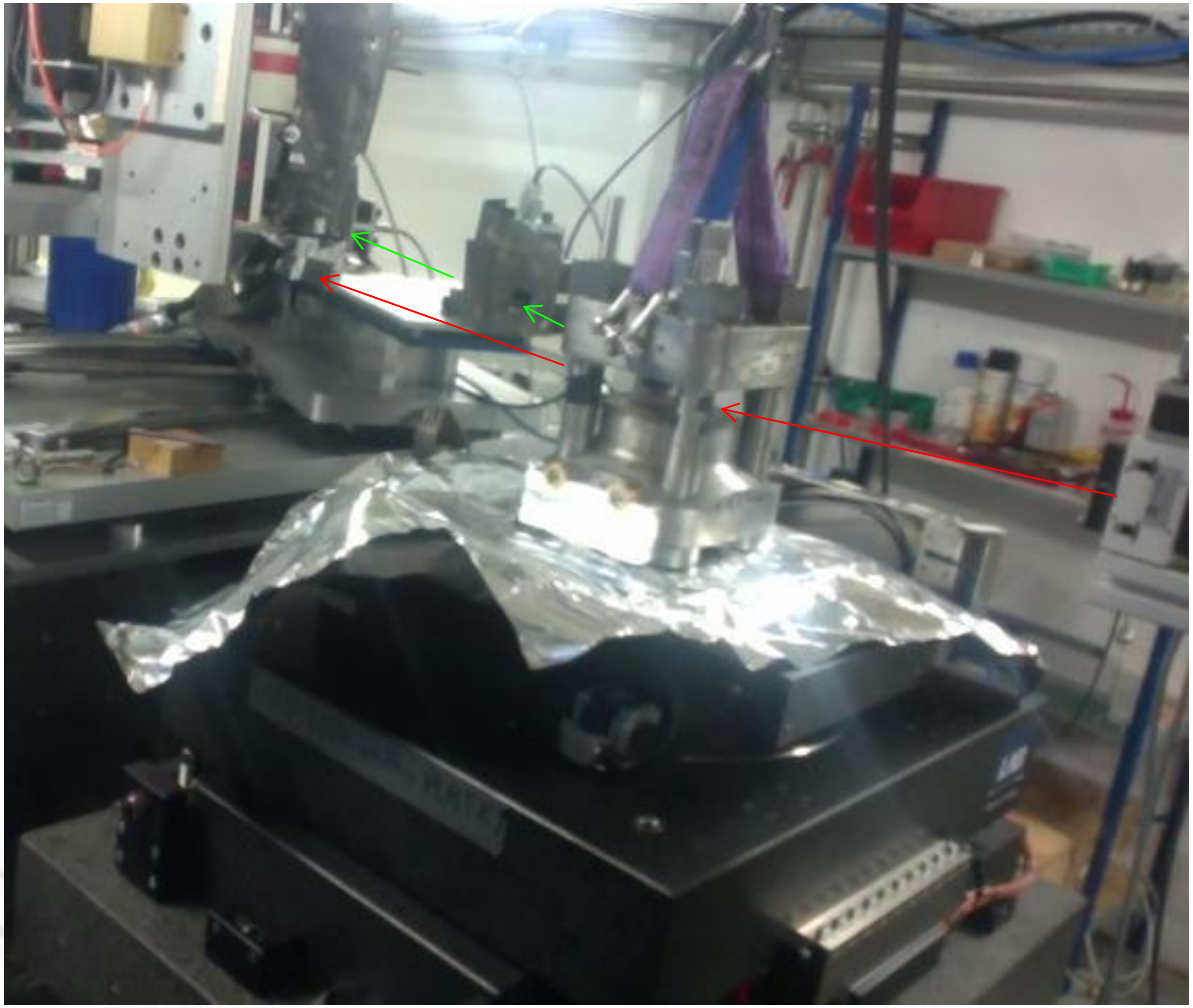




J.-P. Perrillat, natural olivine glass

Diffraction + imaging for high pressure

- Similar experiment but with tomograph
- Fast tomo (for 20 mins ➔15 s, 140° rotation)
- Currently in progress… inhouse test



E. Boulard et al…

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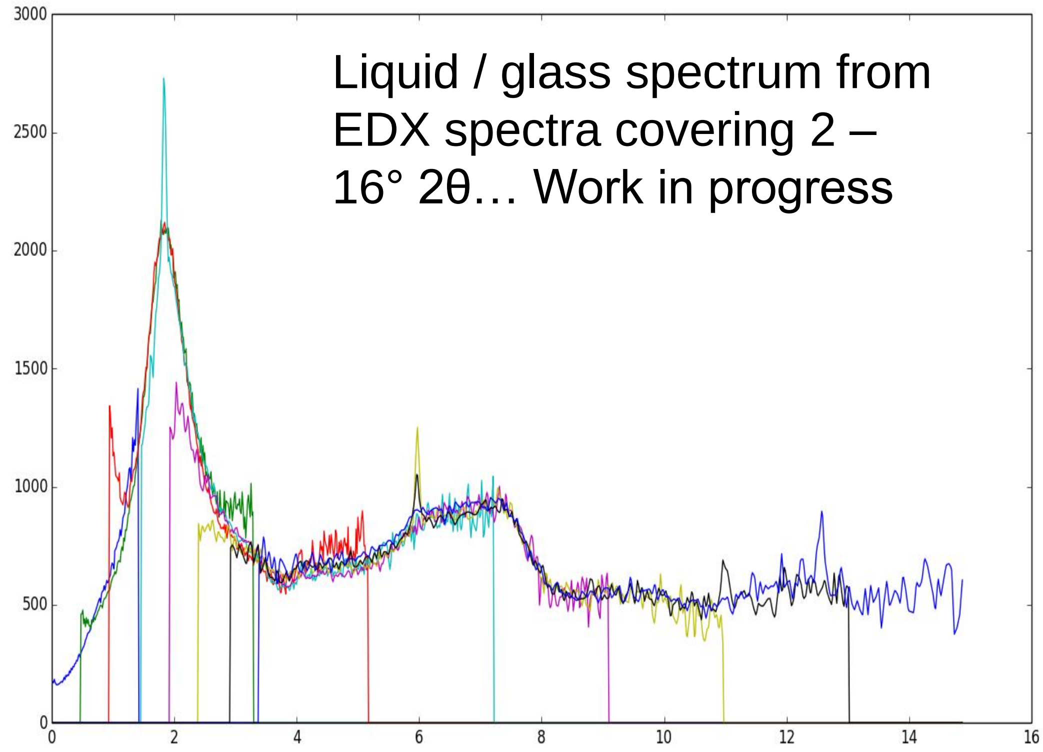
CAESAR diffraction for glasses

- Glasses: Periodic order that is interesting to study
- Tomography to observe compression under pressure
- CAESAR measurement
  - Cover extended Q range
  - Normalise and remove background



A. Clark, G. Morard, PSICHE etc…

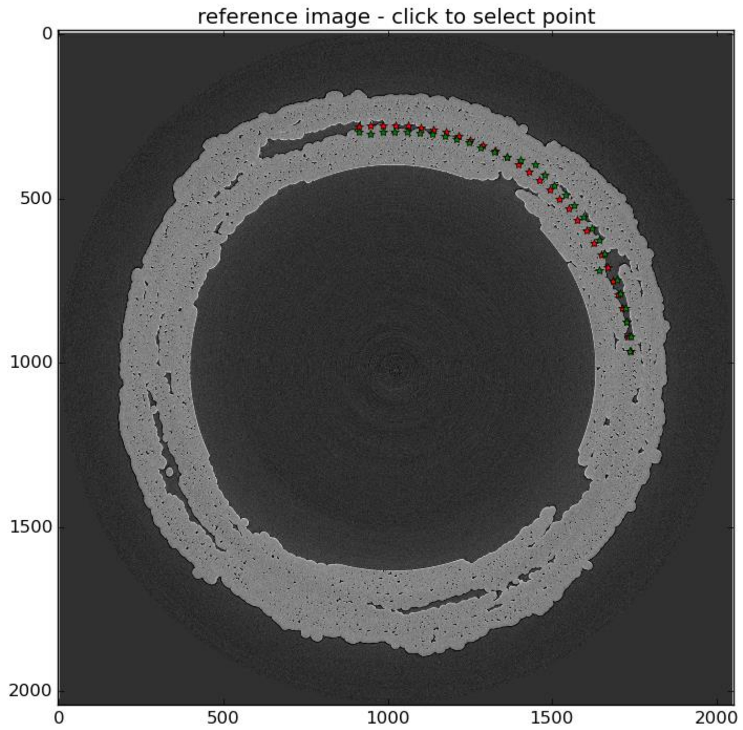
Tomography and diffraction

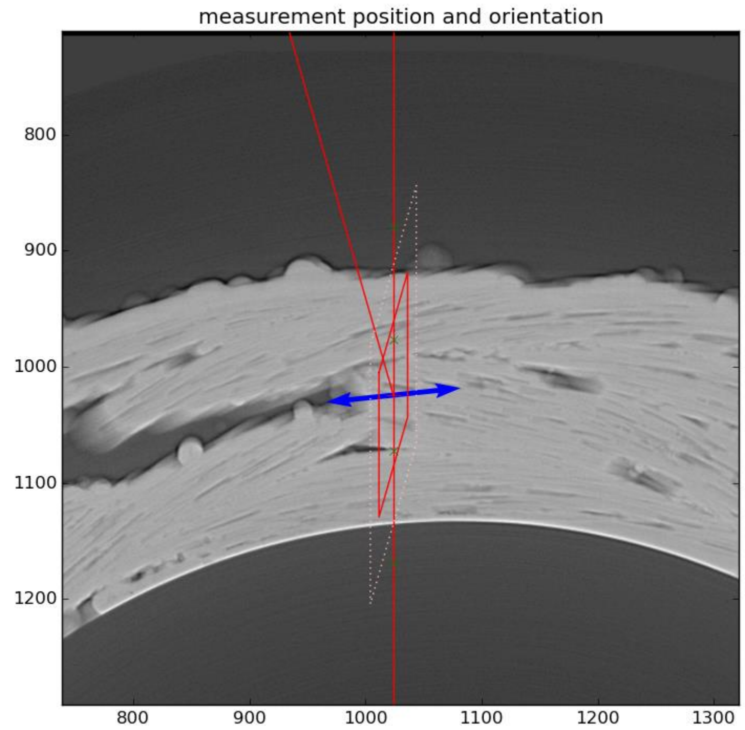
- Some existing techniques that combine both:
  - White beam diffraction + monochromatic XRD (former ID15 ESRF)
  - White beam diffraction + white beam tomo (BESSY ?)
  - Diffraction contrast tomography / 3DXRD (ID11 ESRF)
  - Diffraction tomography (ID21, ID15, RoToPEC at ID2…?, others…)

- Not much prior work on tomo + energy dispersive diffraction
  - PSICHE can do both… natural direction to develop

Combining tomography and diffraction

- Start by tomography of a sample to reveal shape and structure – need fast reconstruction
- Then diffraction to investigated in more detail
- Use tomogram to plan diffraction measurement
  - Interesting features, centering gauge volume…



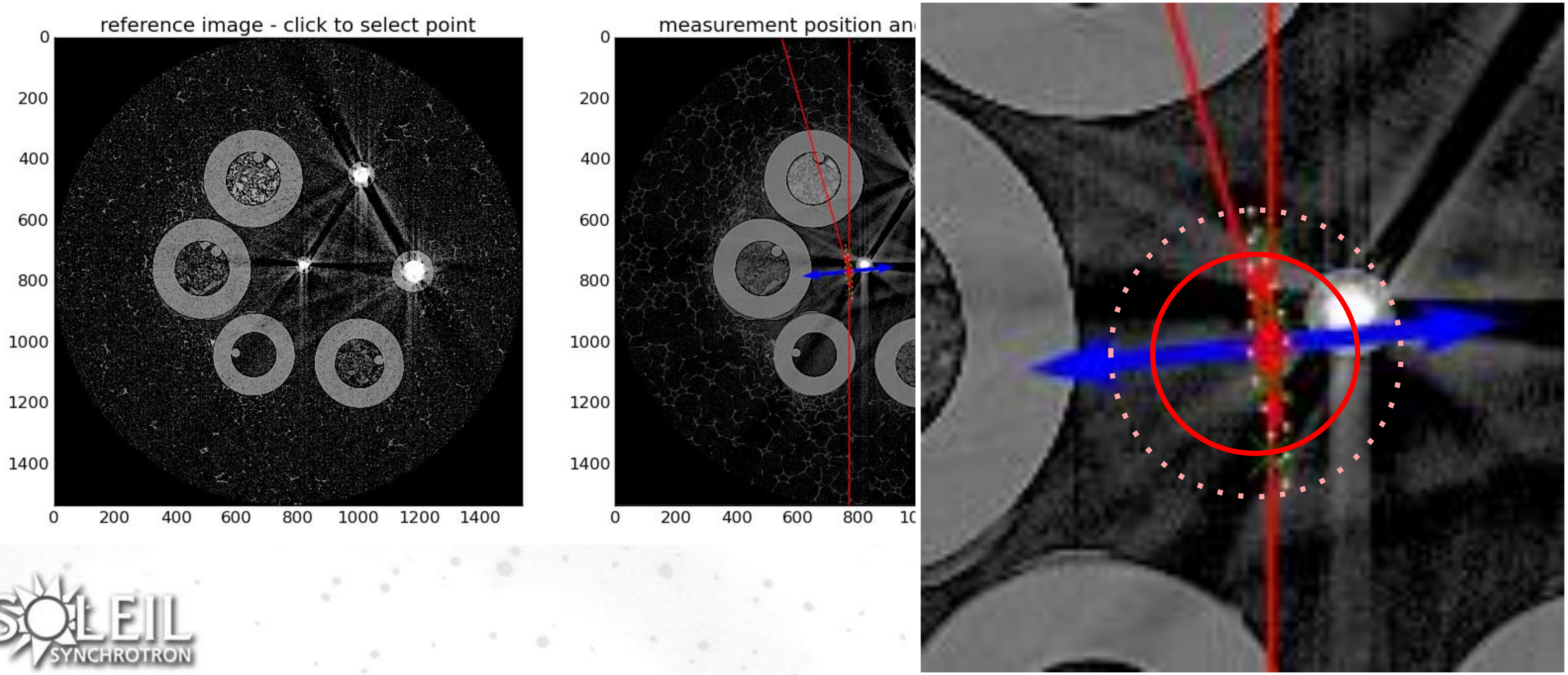


Gelebart, Chen, et al – July 2016

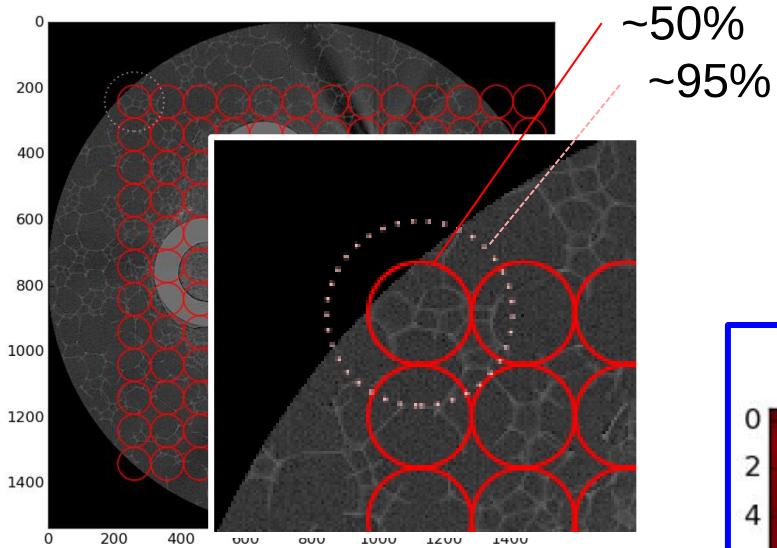
Mapping phases with diffraction

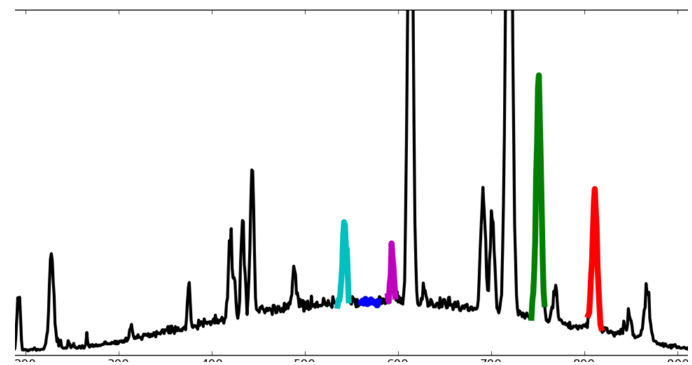
- First idea:
  - Choose a point in the sample
  - Put this point on the centre of rotation
  - Measure diffraction while rotating sample

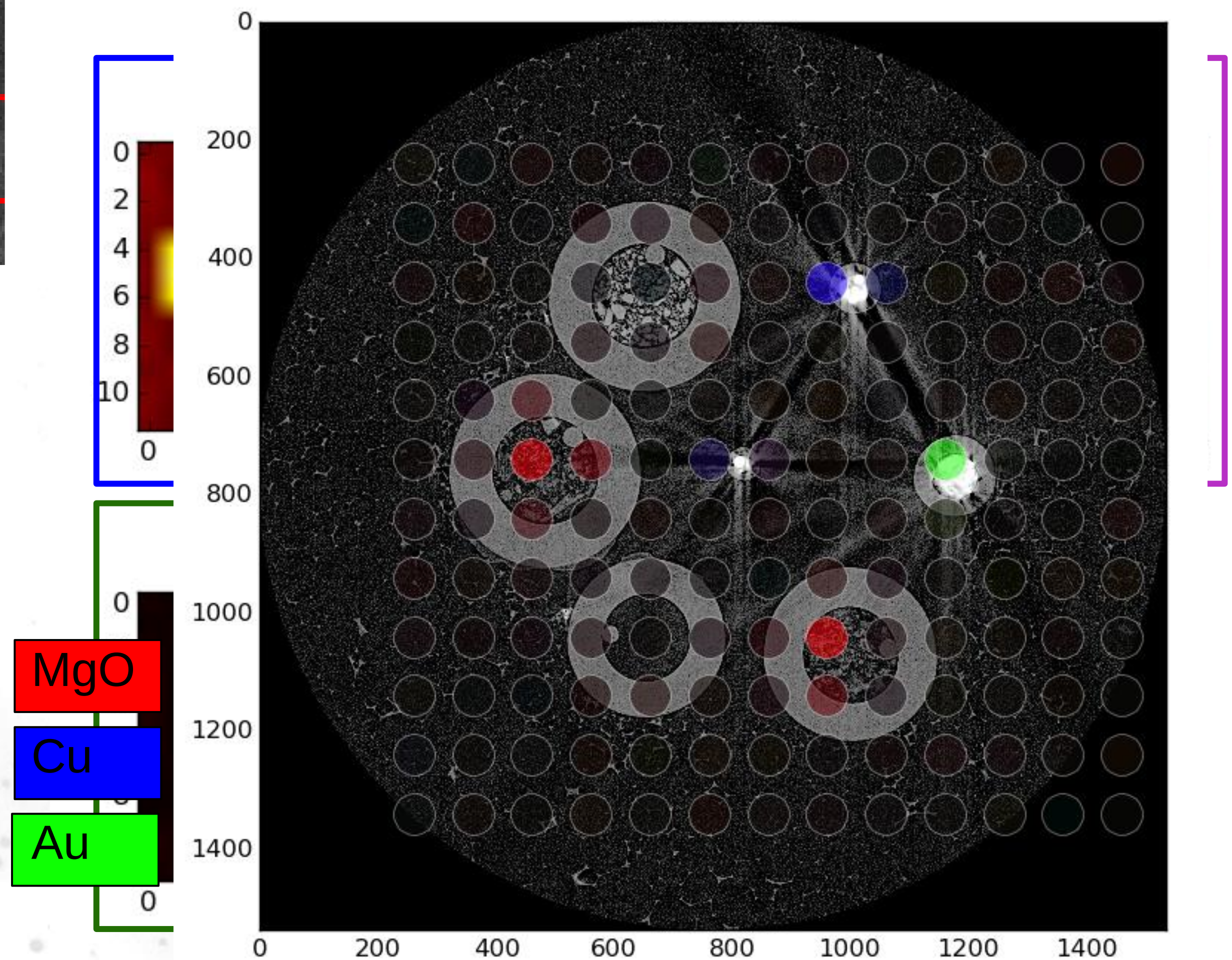
(sample more grains, make an isotropic gauge volume)



Mapping – first results

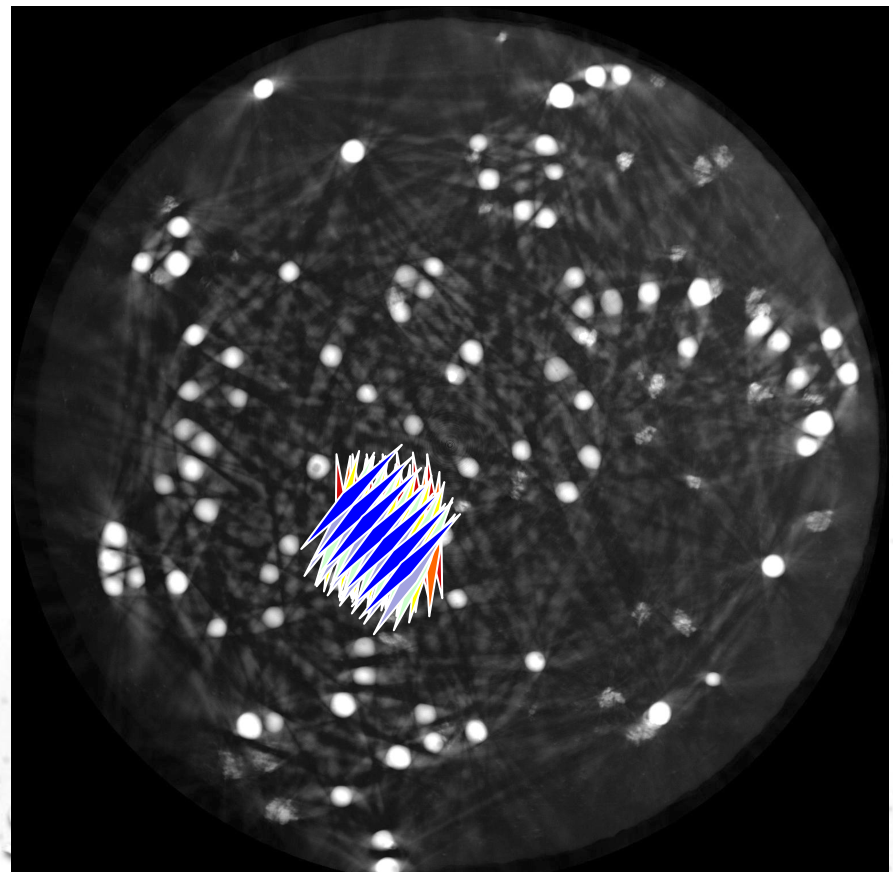






Mapping phases – one step further?

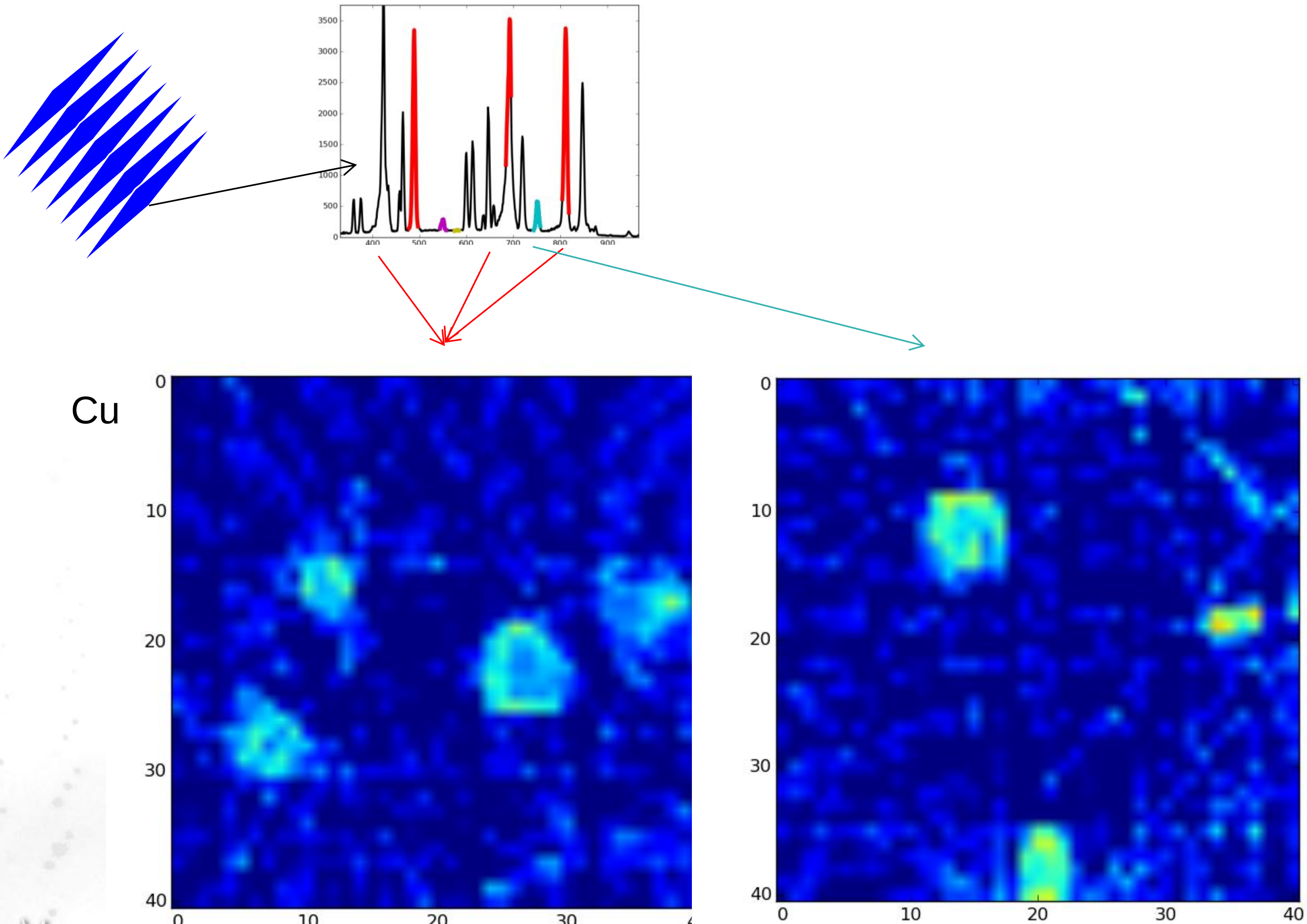
- This approach gives spatial resolution ~ longest dimension of the diffraction gauge
- Can we use tomographic approach to improve spatial resolution?



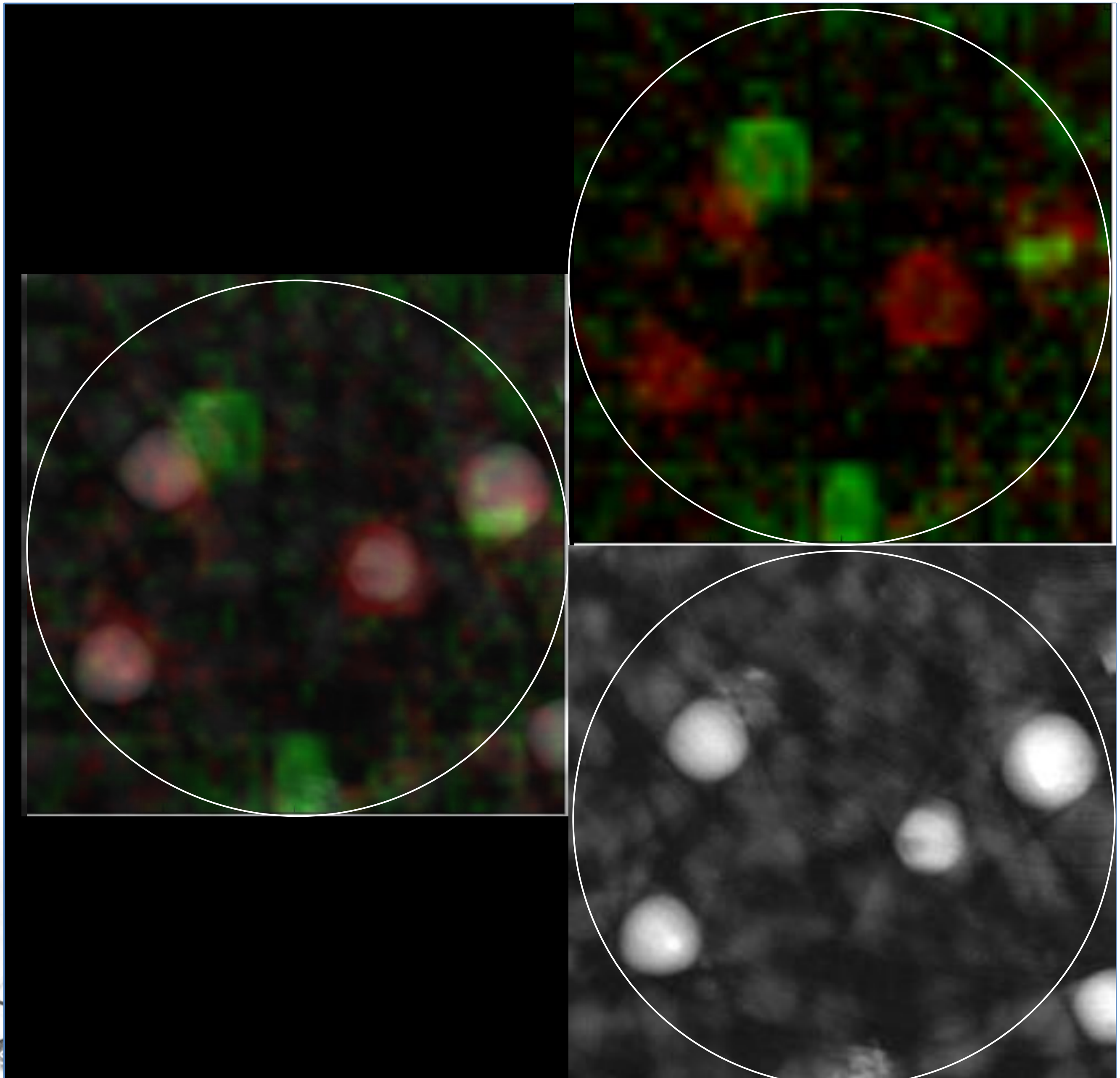
- Select an ROI
- Scan diffraction gauge across ROI to build “projection”
- Repeat at multiple angles to make sinogram
- Reconstruct…

Test sample: magnesium alloy cylinder with gold and copper powder on the top surface

EDX Diffraction tomography

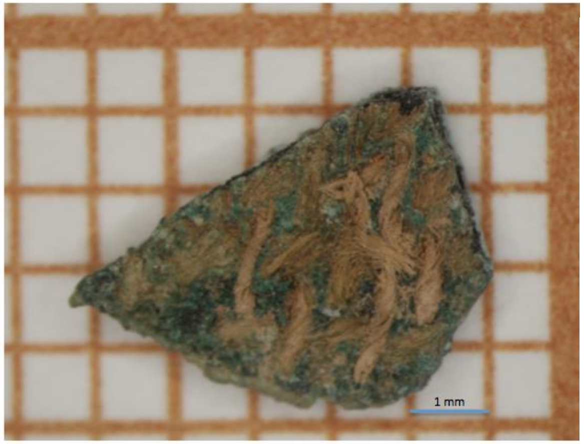


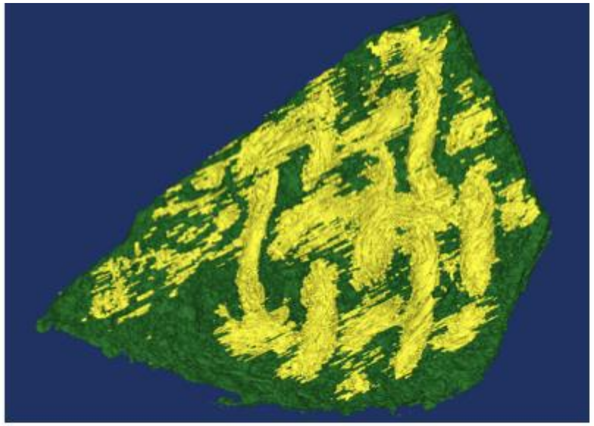
Compare to absorption contrast



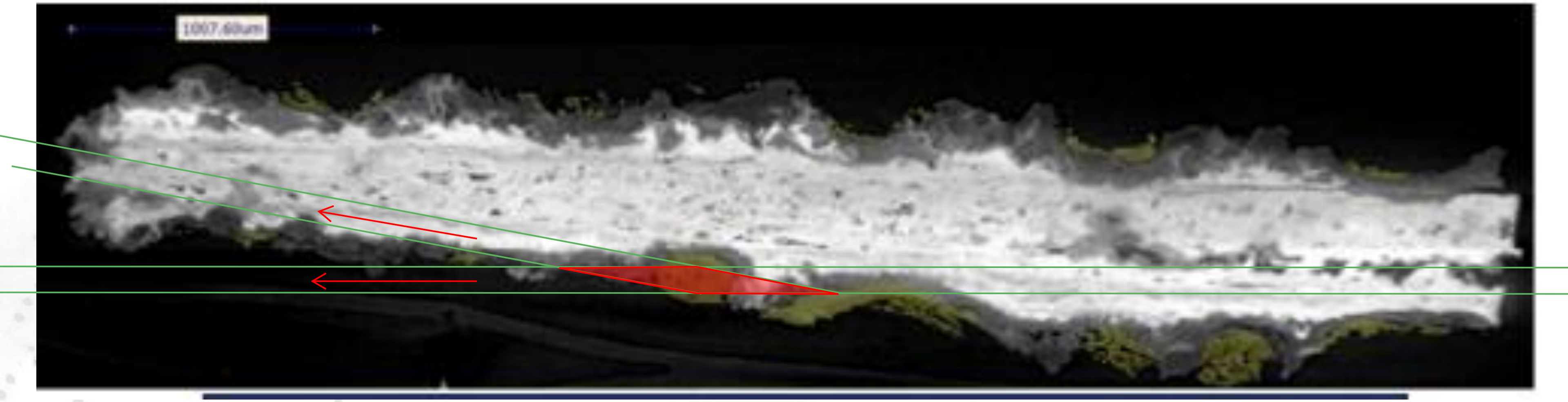
First user application

- Ancient textiles preserved by corrosion of metal artefacts
- Identify phases, understand preservation and corrosion mechanisms. Beam time November 2016…



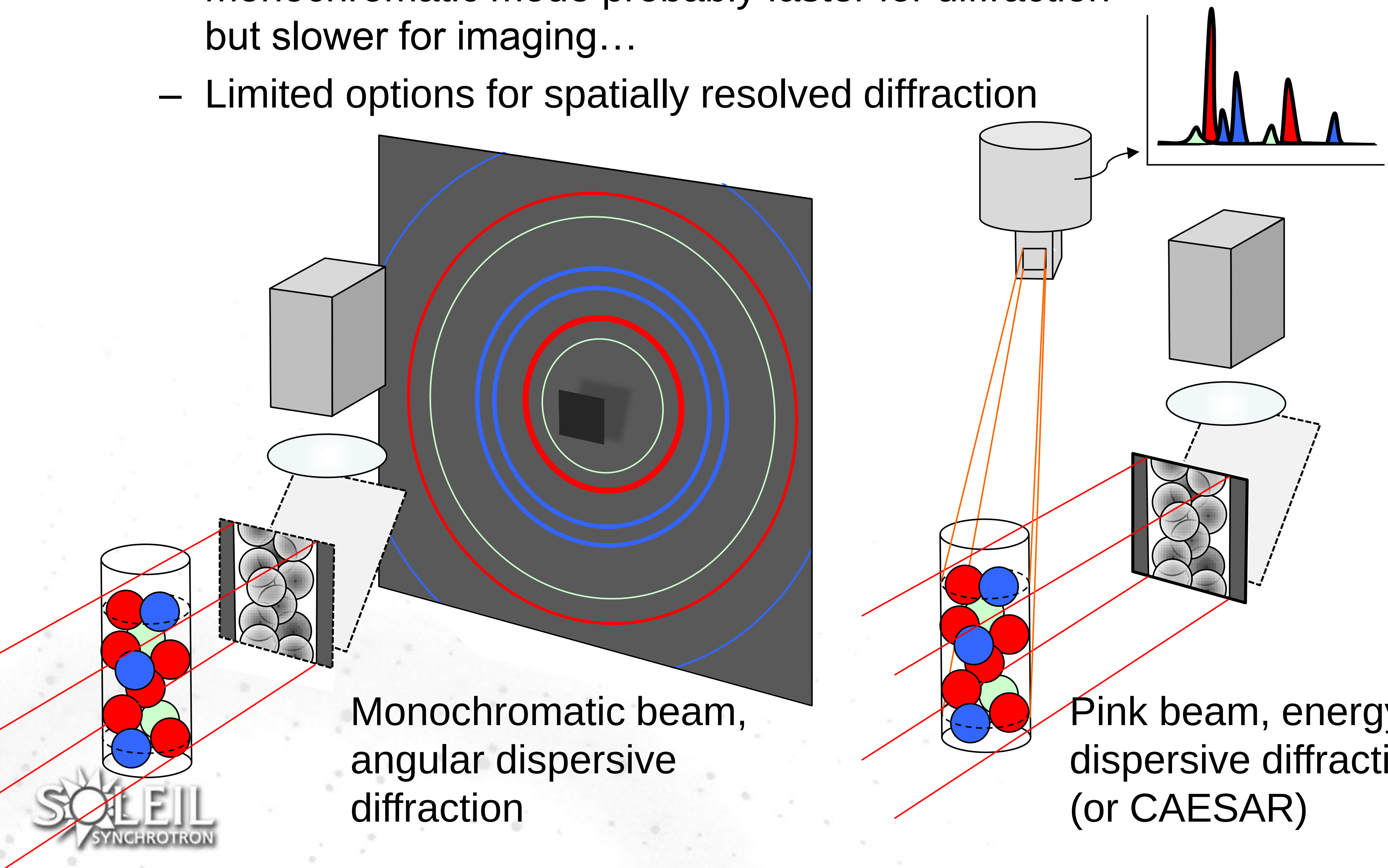


J. Li, M. Bellato, L. Bertrand IPANEMA



Simultaneous diffraction and imaging

- Semi transparent imaging detector
  - monochromatic mode probably faster for diffraction but slower for imaging…
  - Limited options for spatially resolved diffraction



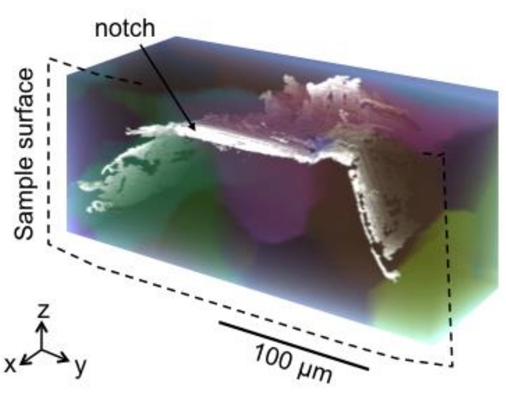
Monochromatic beam, angular dispersive diffraction

Pink beam, energy dispersive diffraction (or CAESAR)

Future directions

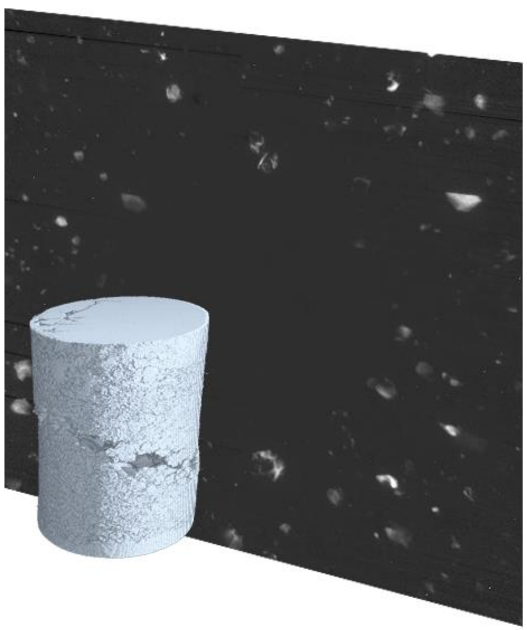
- PSICHE (and my research) : imaging and diffraction
  - logical progression is to combine these

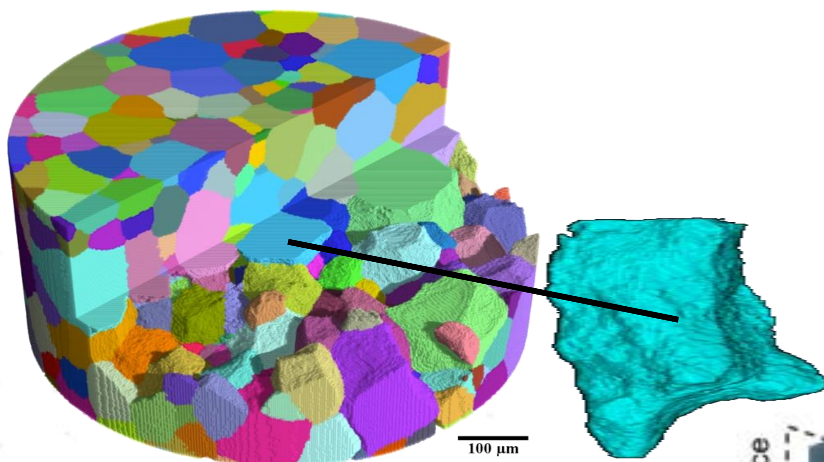
- Various levels of complexity
  - Add imaging to the large volume cell high pressure experiments
  - Combine diffraction and imaging to study amorphous materials
  - Tomography plus crystallographic information (strain, phase changes)
  - Diffraction contrast tomography (mainly a software task)
  - Diffraction tomography

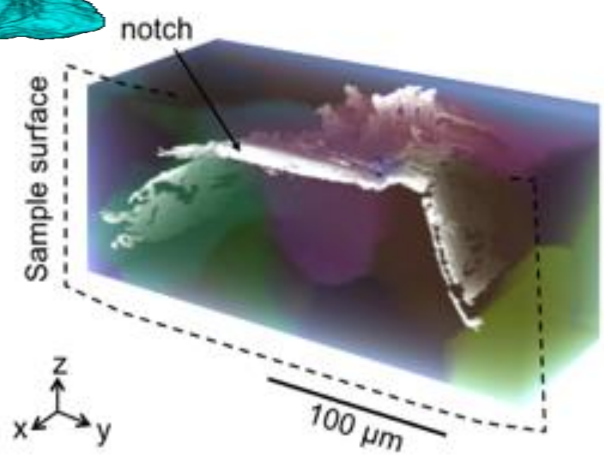


Diffraction contrast tomography (~3DXRD)

- Use the diffraction signal from grains in a polycrystal to build a grain map
- Each grain reconstructed tomographically
- Available at ID11 (ESRF), near future at Psiché.







The future - some targets

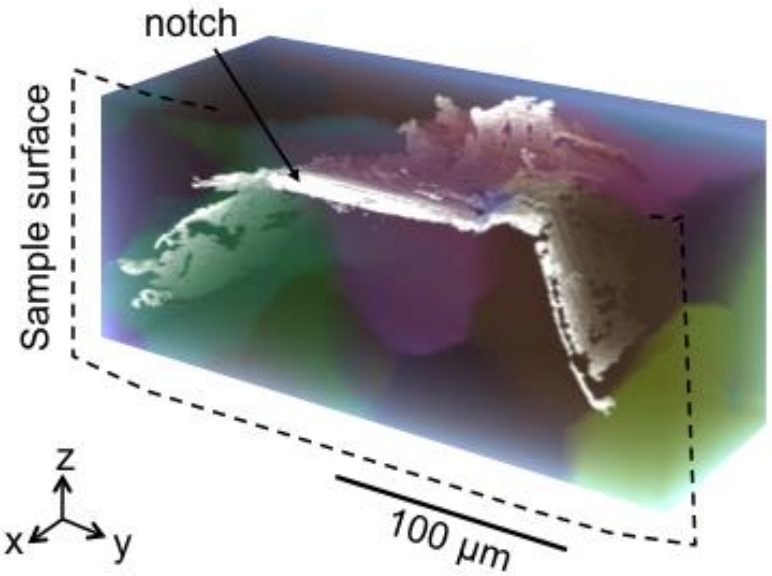
- Faster tomography
  - Full high res scan $2k^2$, 1500 images in 15 seconds
  - Fast reconstruction and local data storage
- Add imaging to high pressure diffraction experiments

More or less done

- Setup collaborations for our in-situ sample environments
- Add diffraction/fluorescence info to tomography
- Try to get a PhD student for tomography

Now!

- Implement diffraction tomography techniques
  - Diffraction contrast tomography
  - Diffraction tomography (point scanning type)
- Very fast tomography
  - full high res scan in ~0.5 seconds



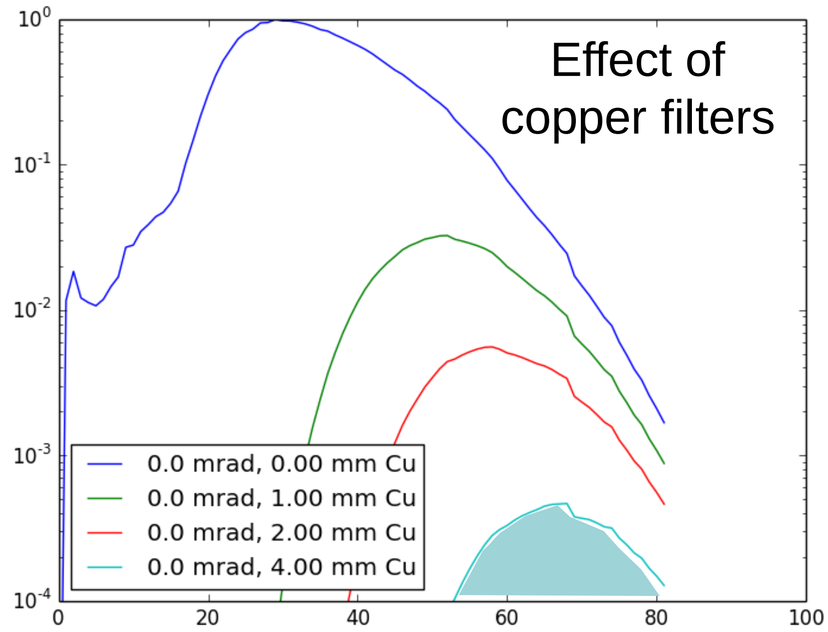
Conclusions

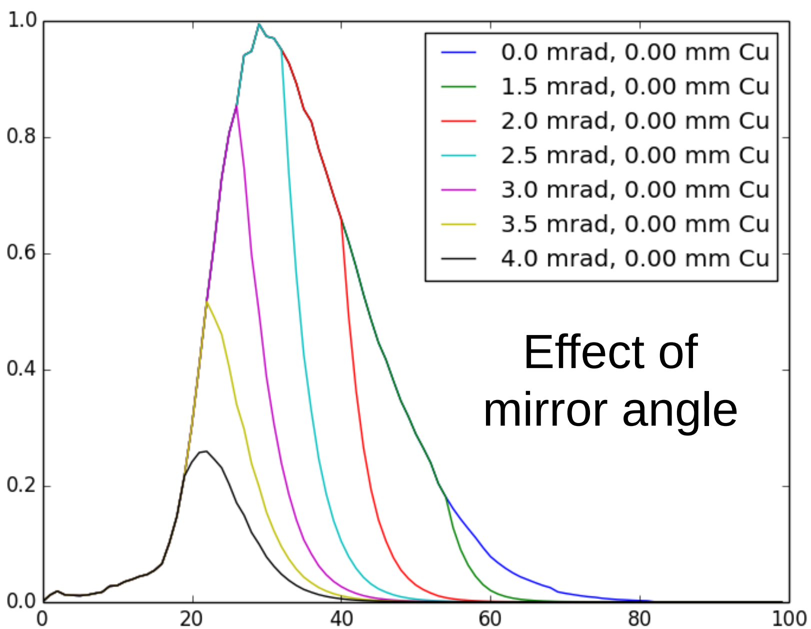
- Psiché – A new synchrotron tomography beam line at SOLEIL
- Interesting position for our community intersecting imaging, diffraction, high pressure, materials…
- Just in service, lots of future potential
- Feel free to get in touch to discuss

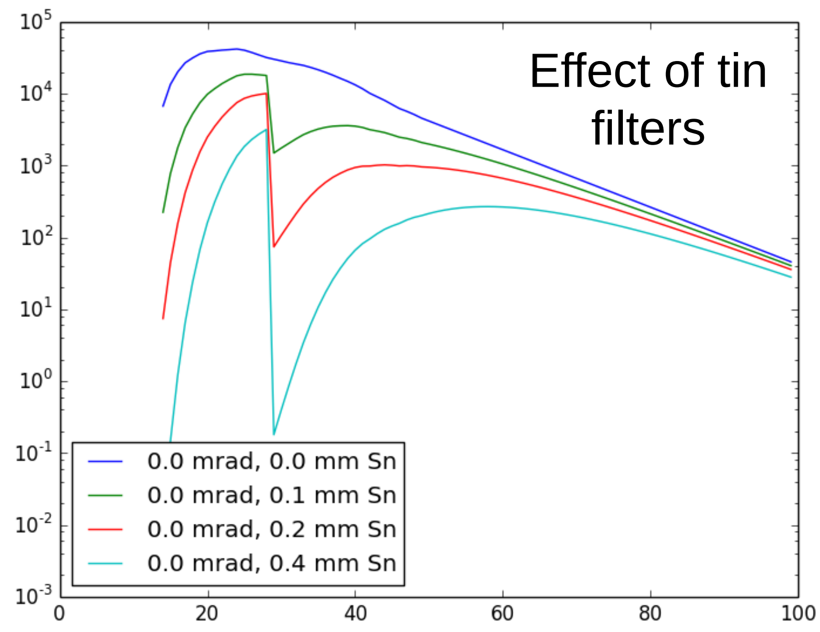
king@synchrotron-soleil.fr

Pink beam illumination

- Commissioning in progress
- Tune spectrum
  - Filters, absorption edges in filters and scintillators
  - Use mirror as low pass filter
- (Very) fast tomography
  → Limited by beam damage

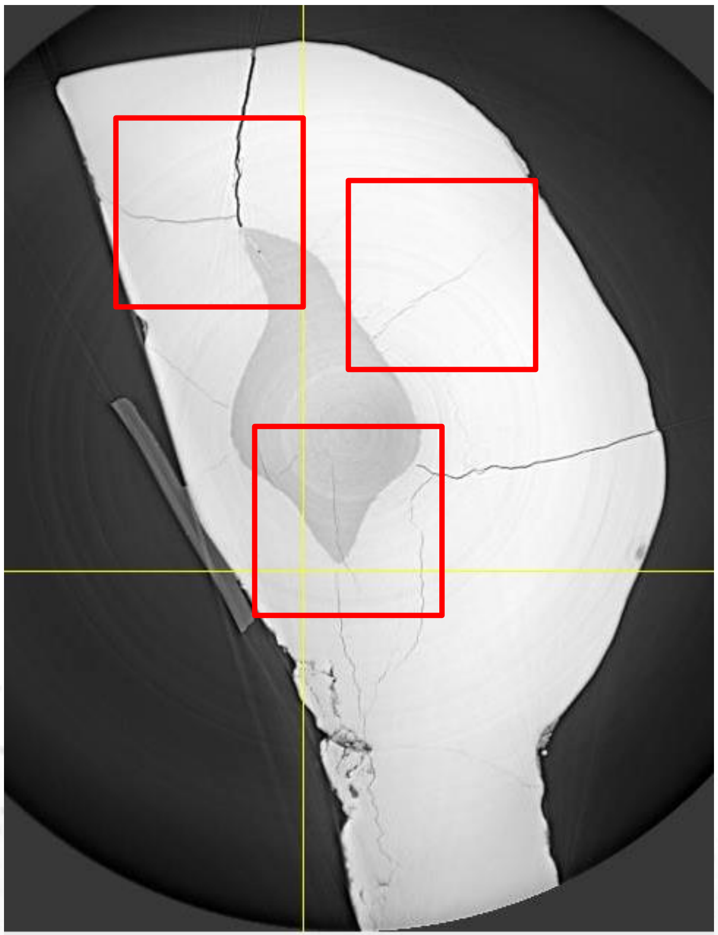




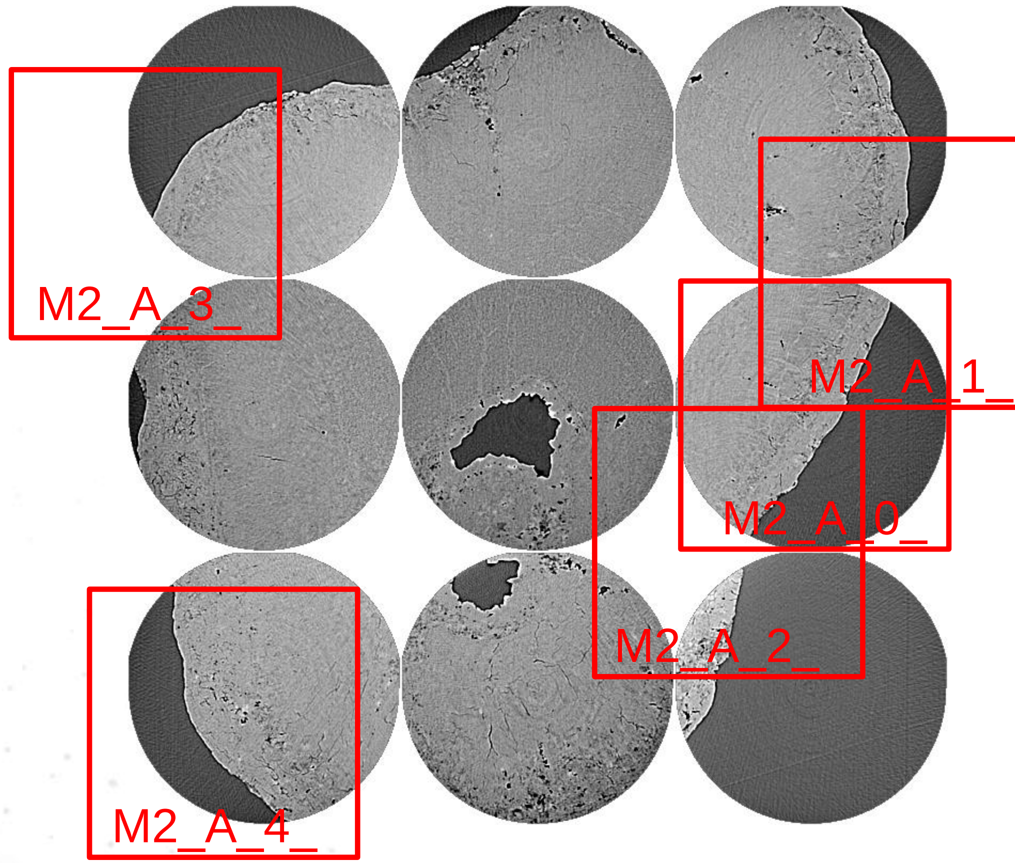


Using tomography to plan a (diffraction) measurement

- Select positions of high resolution local tomograms



Low resolution tomo



Mosaic of fast high resolution tomos – avoid changing optics

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