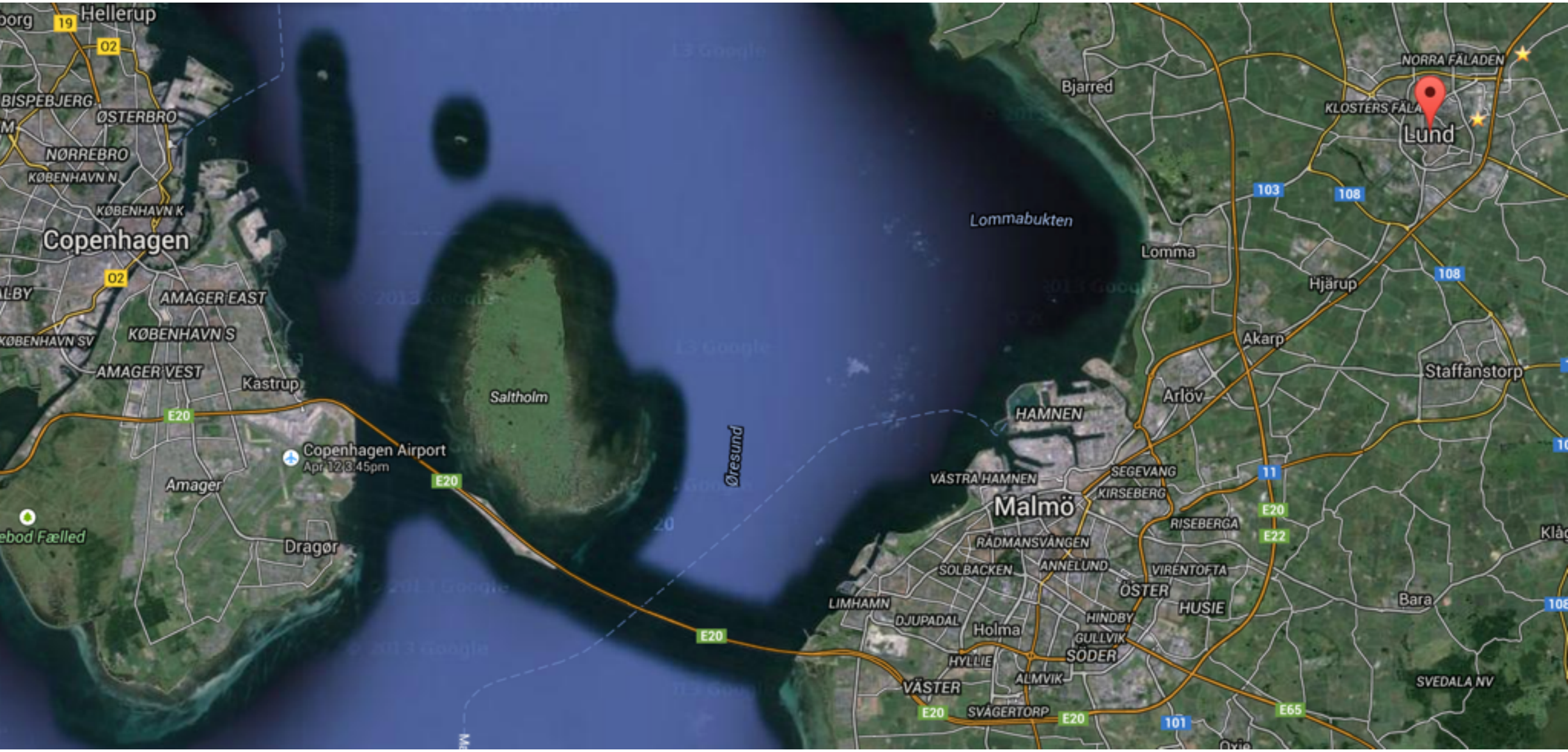
MAX-IV visit report: a forward-looking storage ring

Chris Jacobsen
Associate Division Director, X-ray Science Division, Advanced Photon Source
Professor, Physics & Astronomy, Northwestern University; Chemistry of Life Processes Institute

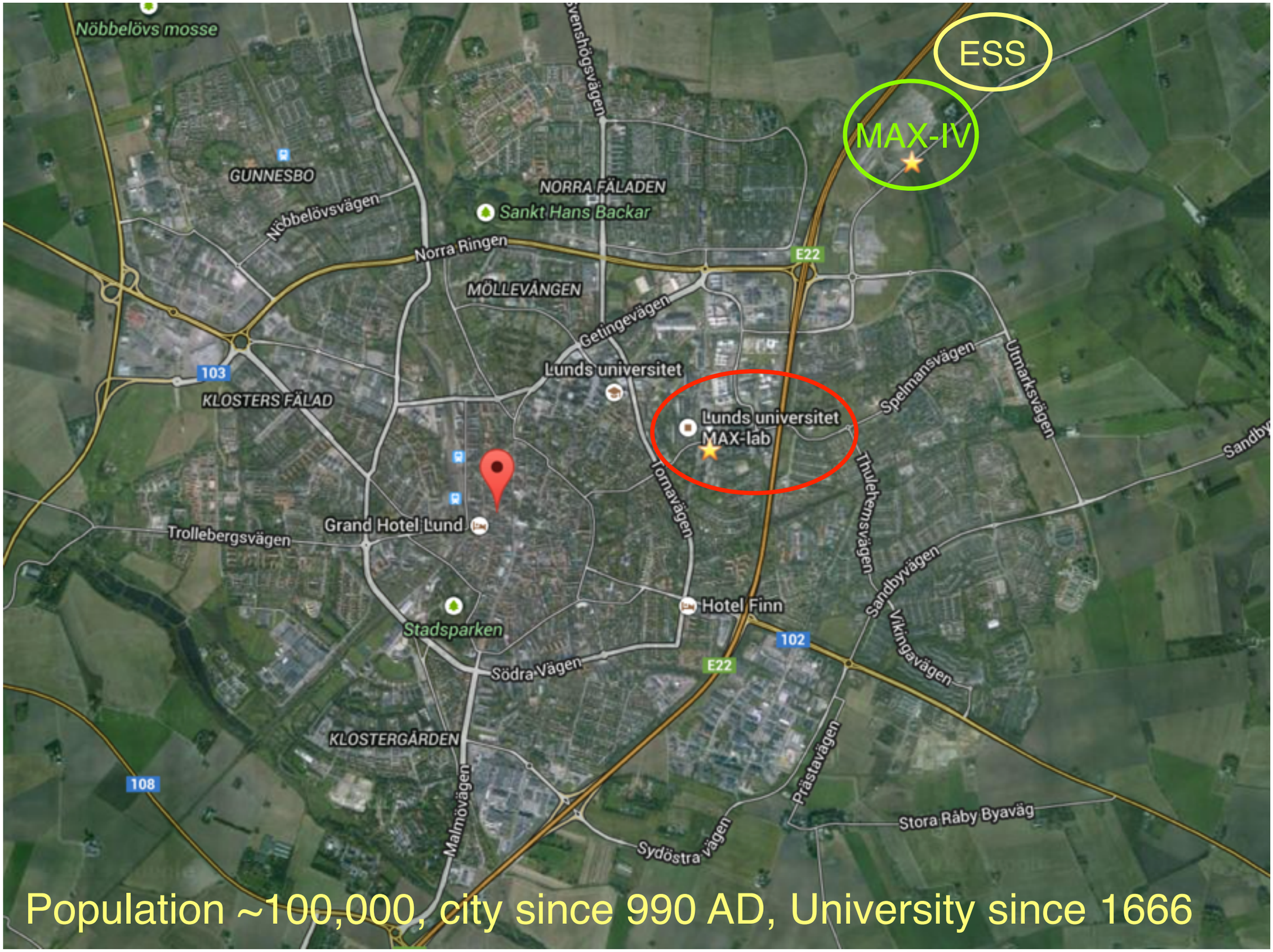
Max Baker (Denver Colorado)

Getting to Lund: fly to Copenhagen, train from airport



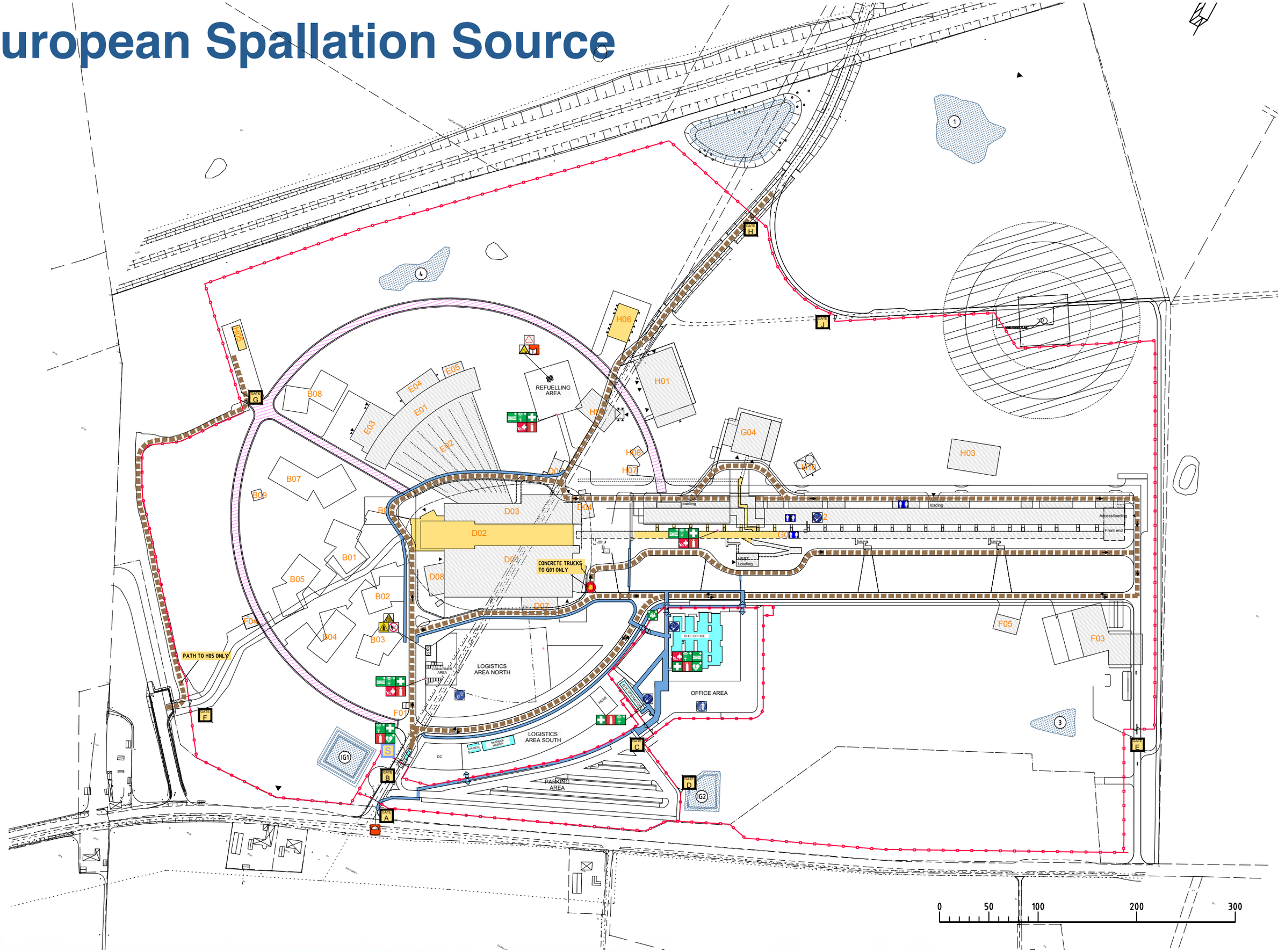
- Train stops right in airport terminal; takes about 40 minutes to get to Lund. Frequent bus service to Hotel Finn, and even a new bus line from the Lund train station out to MAX-IV!

Lund, Sweden: MAX-IV and ESS (European Spallation)



Population ~100,000, city since 990 AD, University since 1666

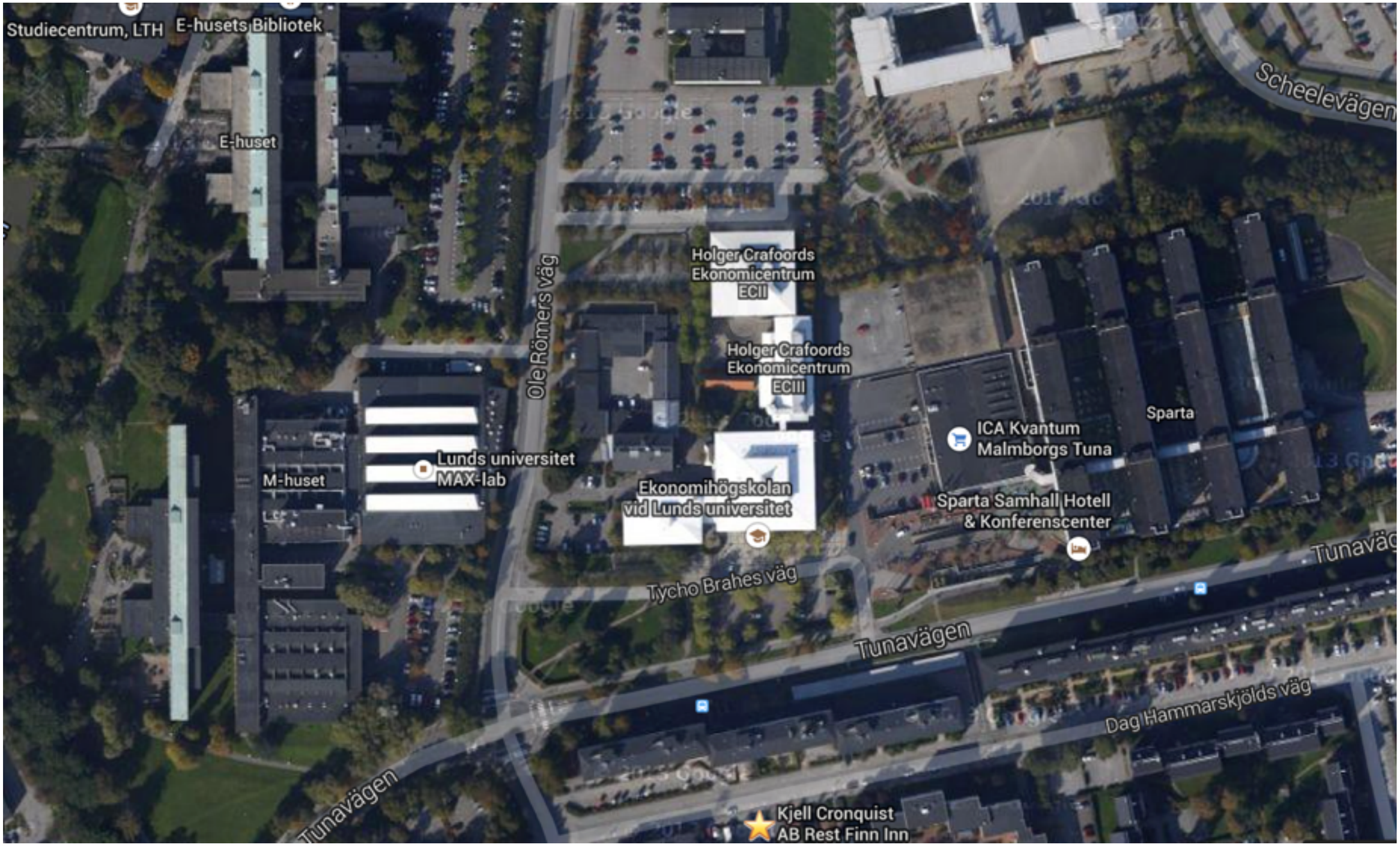
European Spallation Source



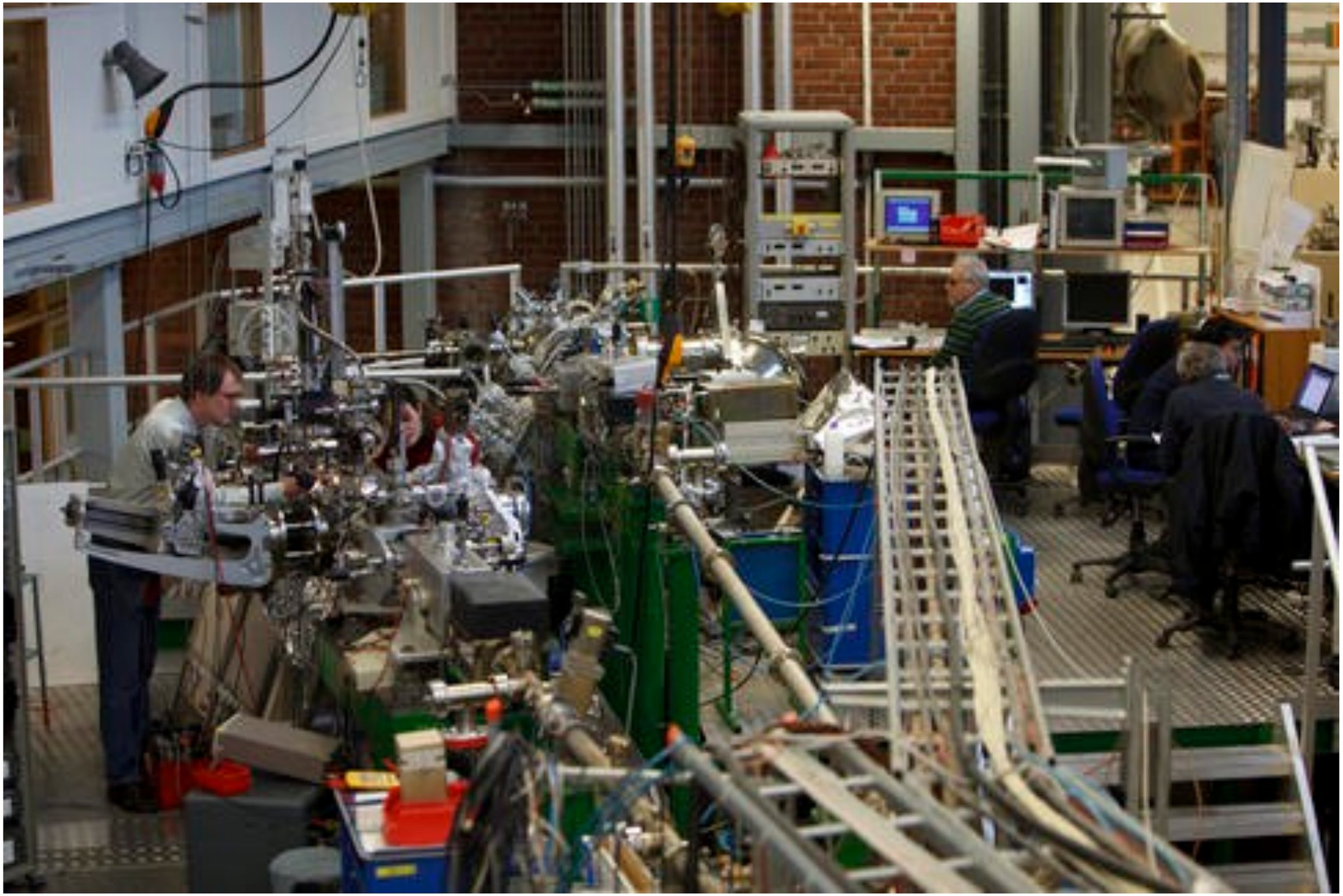
Lund: get a bike

MAX-Lab

- MAX-II: 90 m circumference, 1.5 GeV, 300 mA, 9 nm-rad horizontal emittance, 10 straight sections. Plywood “hutches”!

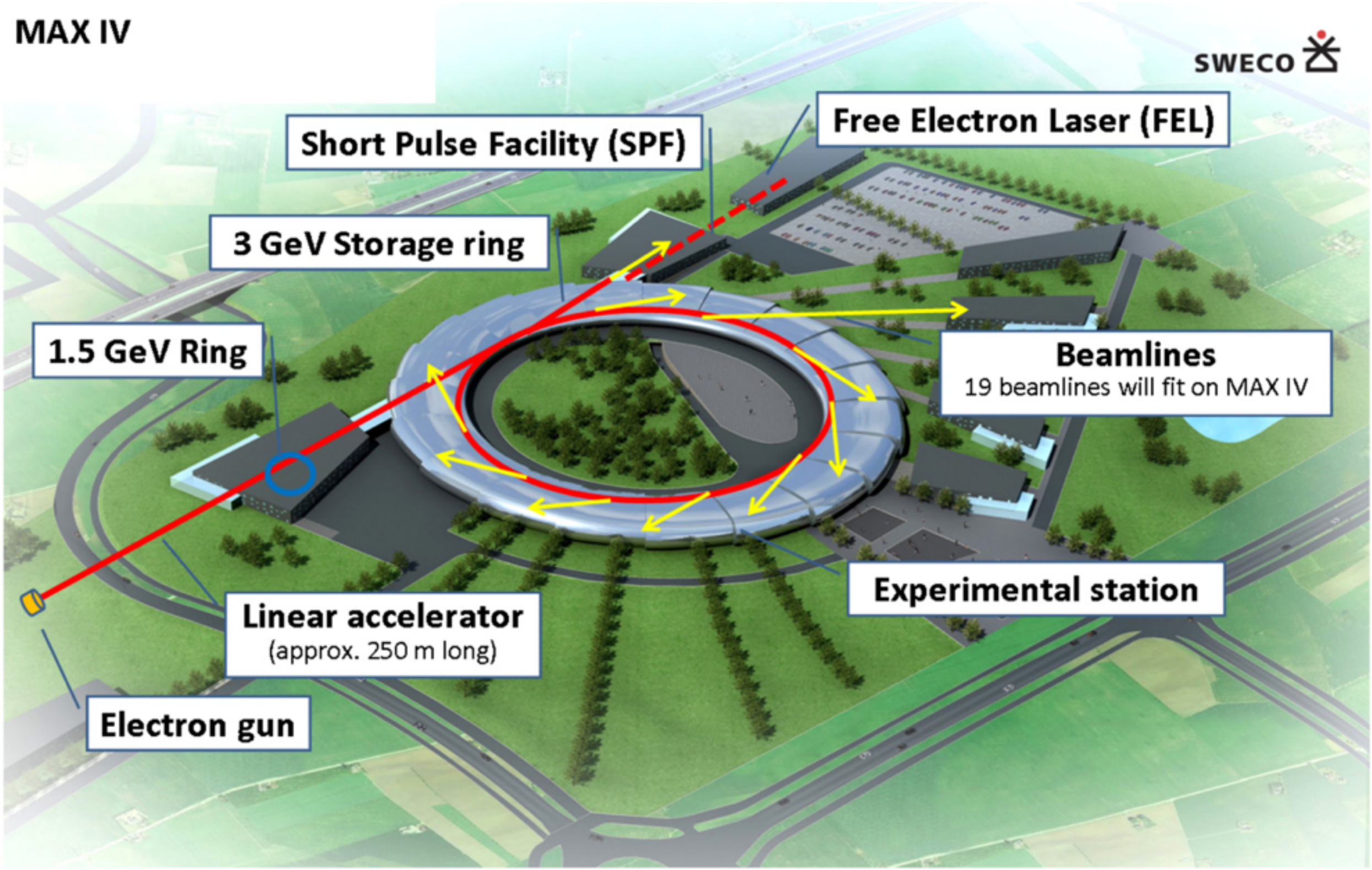


MAX-II

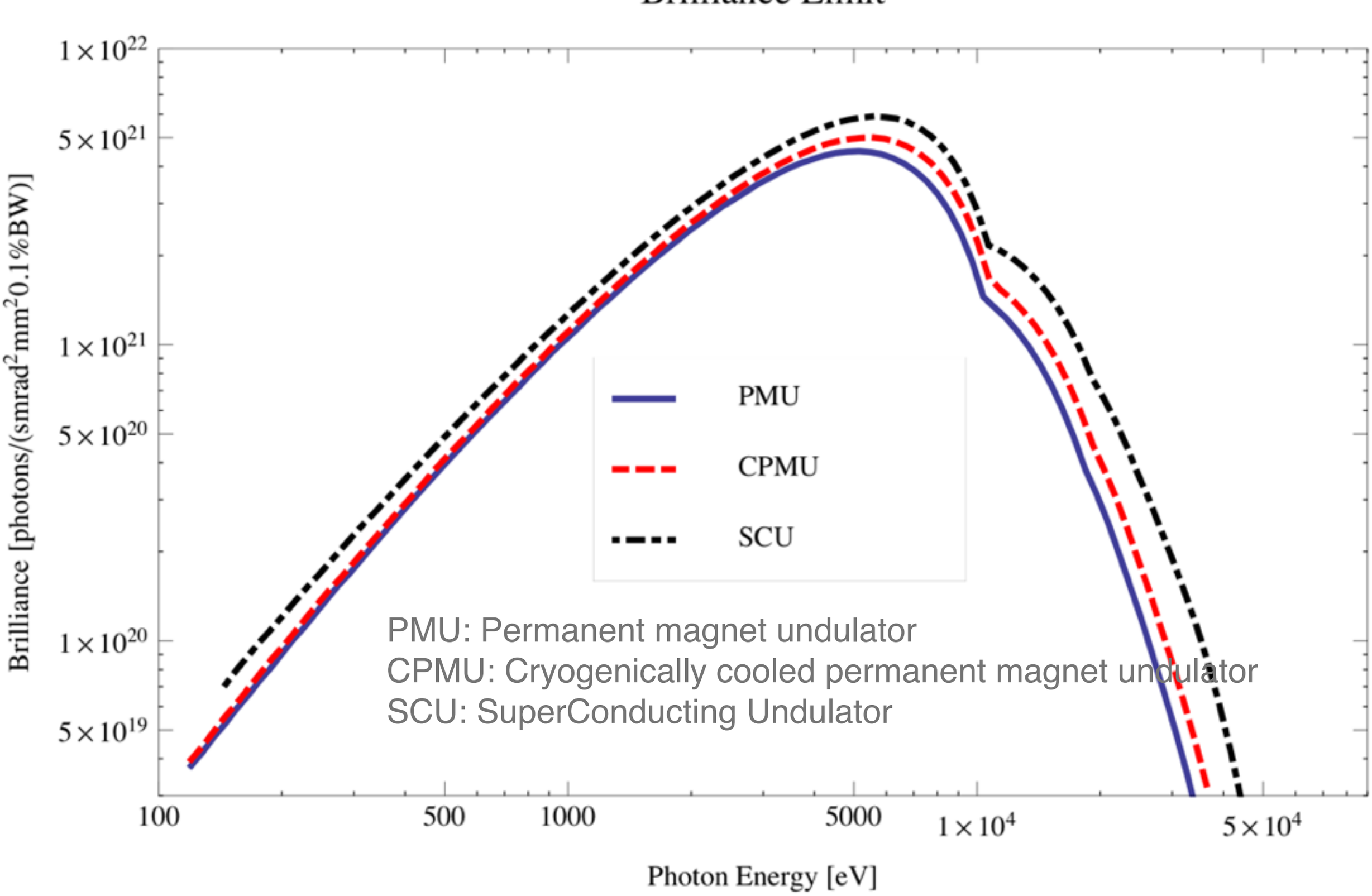


MAX-IV: the first MultiBend Achromat (MBA) lattice light source facility

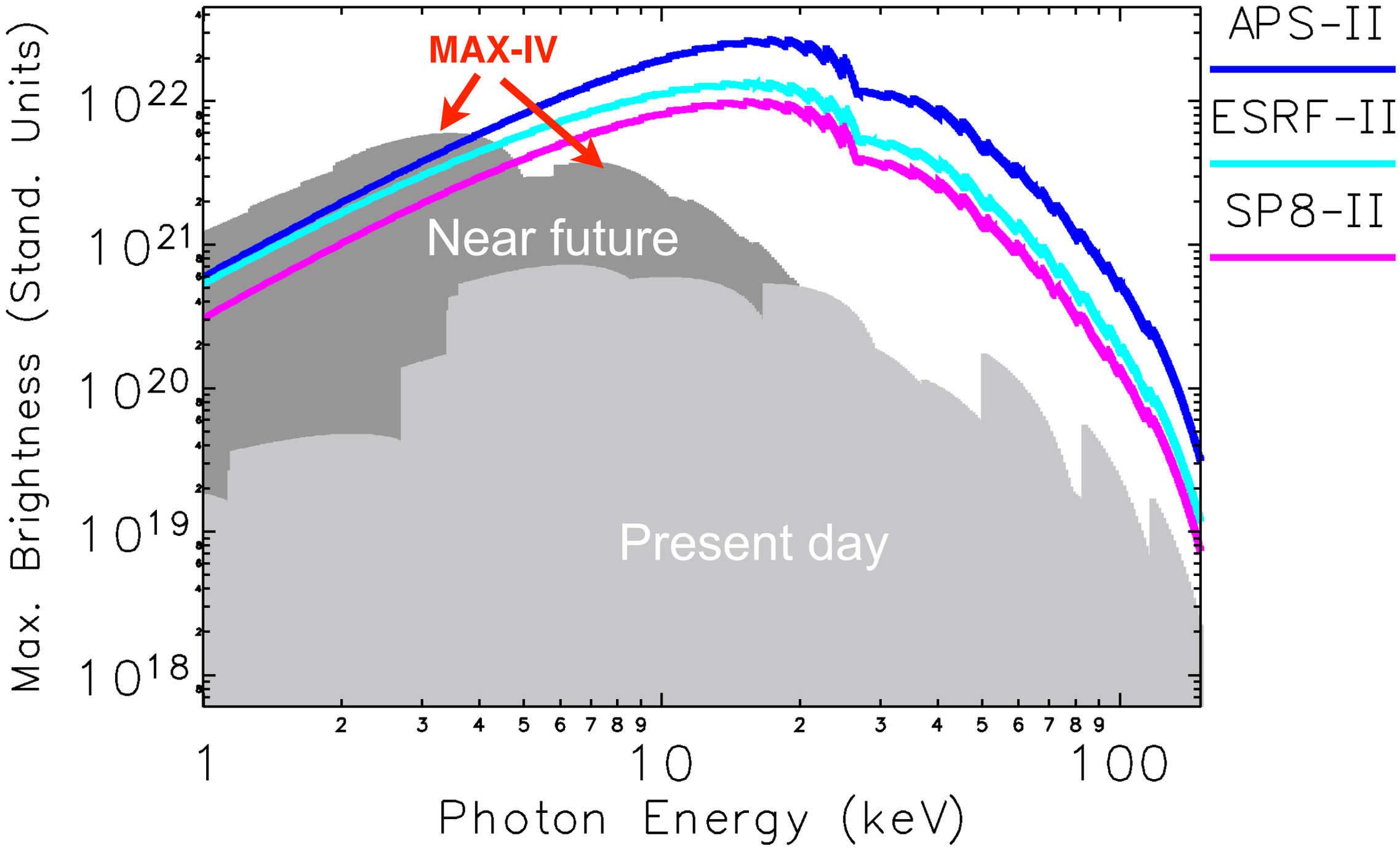
MAX-IV: 3 GeV, 500 mA, 0.26 (H) by 0.008 (V) nm-rad



MAX-IV


Brilliance Limit
Brilliance [photons/(smrad²mm²0.1%BW)]
1×10²²
5×10²¹
1×10²¹
5×10²⁰
1×10²⁰
5×10¹⁹
100
500
1000
5000
1×10⁴
5×10⁴
Photon Energy [eV]
PMU
CPMU
SCU
PMU: Permanent magnet undulator
CPMU: Cryogenically cooled permanent magnet undulator
SCU: SuperConducting Undulator

MAX-IV and the APS Upgrade

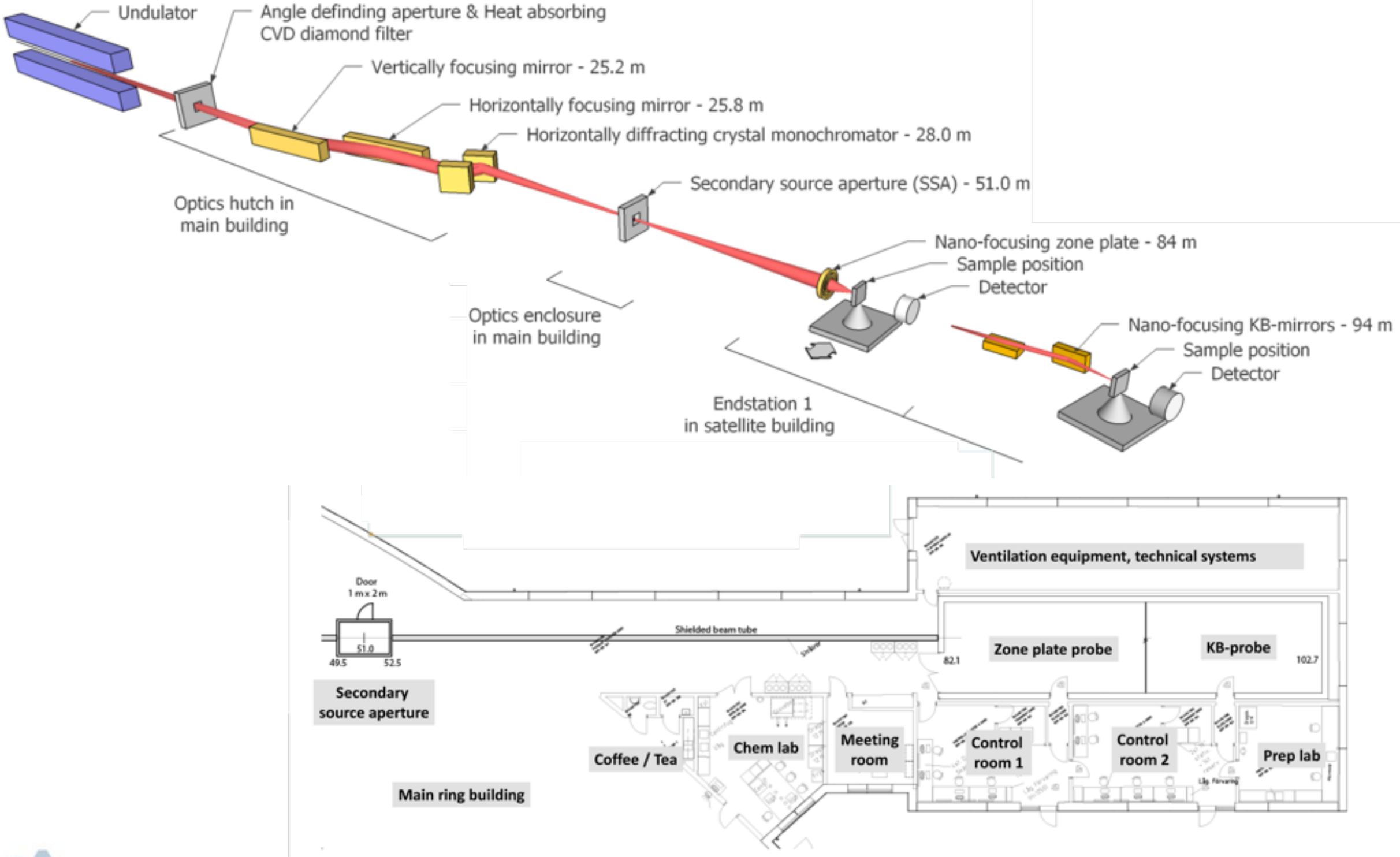


Two beamlines: NanoMAX and SoftiMAX

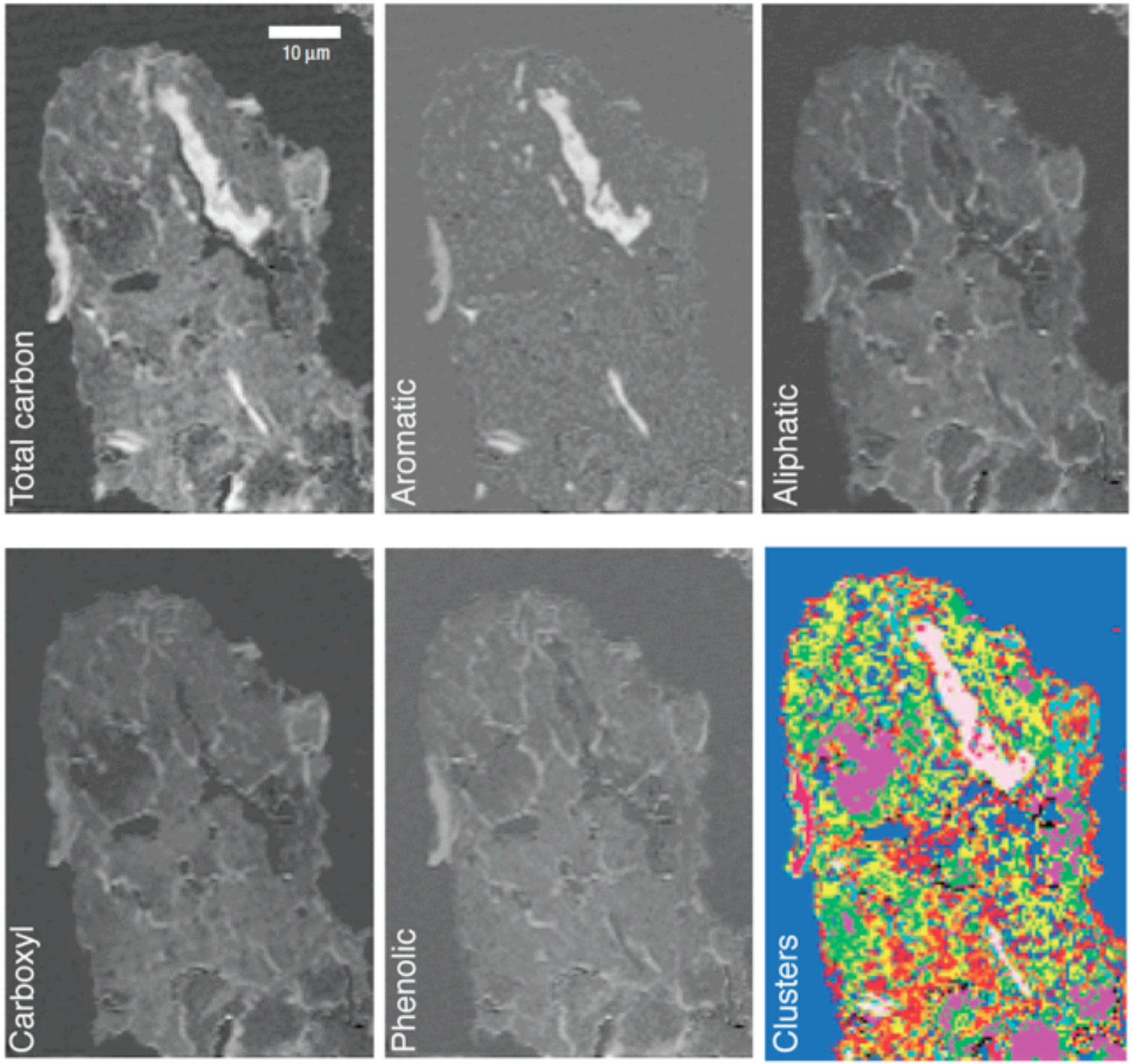
- NanoMax: Ulf Johannson, Dina Carbone
  - 5-15 keV in station 1: scanning microscope with zone plate optics
  - 5-22 keV in station 2: microdiffraction with KB mirror optics
  - Cooperation with Soleil on station 1 scanning microscope
- SoftiMAX: Karina Thånell (née Schulte)
  - 0.25-2.5 keV in station 1: scanning microscope with zone plate optics
  - 0.25-2.5 keV in station 2: flexible endstation including Fourier transform holography (Eisebitt et al.)
- Cooperation with group of Hans Hertz at the Royal Institute of Technology (KTH) in Stockholm on zone plates

NanoMAX

[1] U. Johansson, U. Vogt, A. Mikkelsen, Proc. SPIE 8851, 88510L (2013).



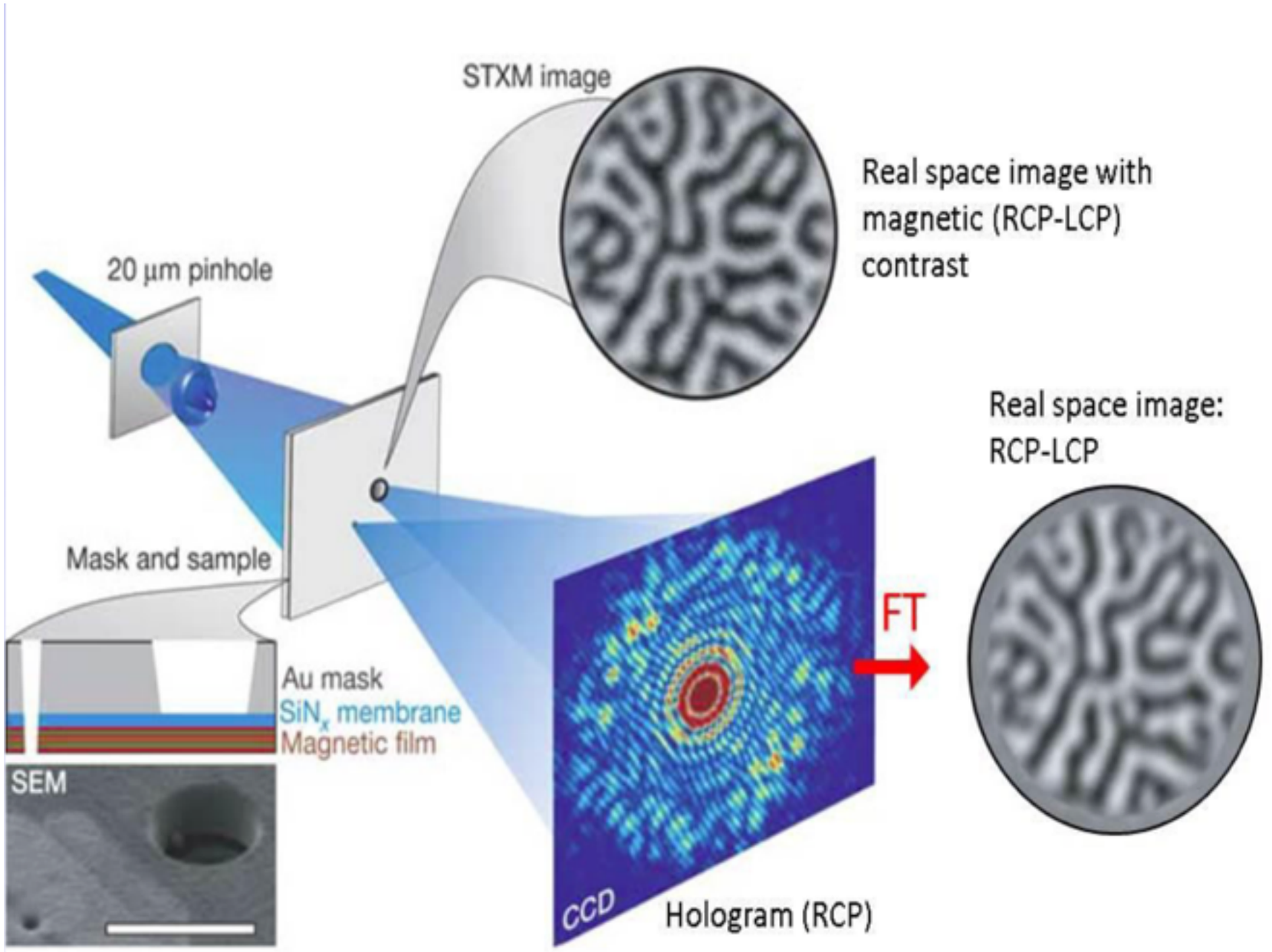
SoftiMAX: soft x-ray spectromicroscopy



Organic speciation in soils: J. Lehmann, D. Solomon, J. Kinyangi, L. Dathe, S. Wirick, and C. Jacobsen, *Nature Geoscience* **1**, 238 (2008)

SoftiMAX: Fourier transform holography

- Magnetic structure in thin films [Eisebitt *et al.*, *Nature* **432**, 885 (2004)]



Why am I visiting MAX-lab?

- NanoMAX has two endstations.
- SoftiMAX endstation beginning design.
- Respective teams considering commonalities in design.
- Can we help them, and learn from their experience?

Scanning speeds with diffraction-limited storage rings

- Vine, Preissner *et al.* “Velociprobe” LDRD project!
- Flyscan ptychography and fluorescence! See e.g., Deng *et al.*, *PNAS* **112**, 2314 (2015); Deng *et al.*, *Optics Express* **23**, 5348 (2015); Nashed *et al.*, *Optics Express* **22**, 32082 (2014).
- de Jonge, Ryan, and Jacobsen, *J. Sync. Rad.* **21**, 1031 (2014):

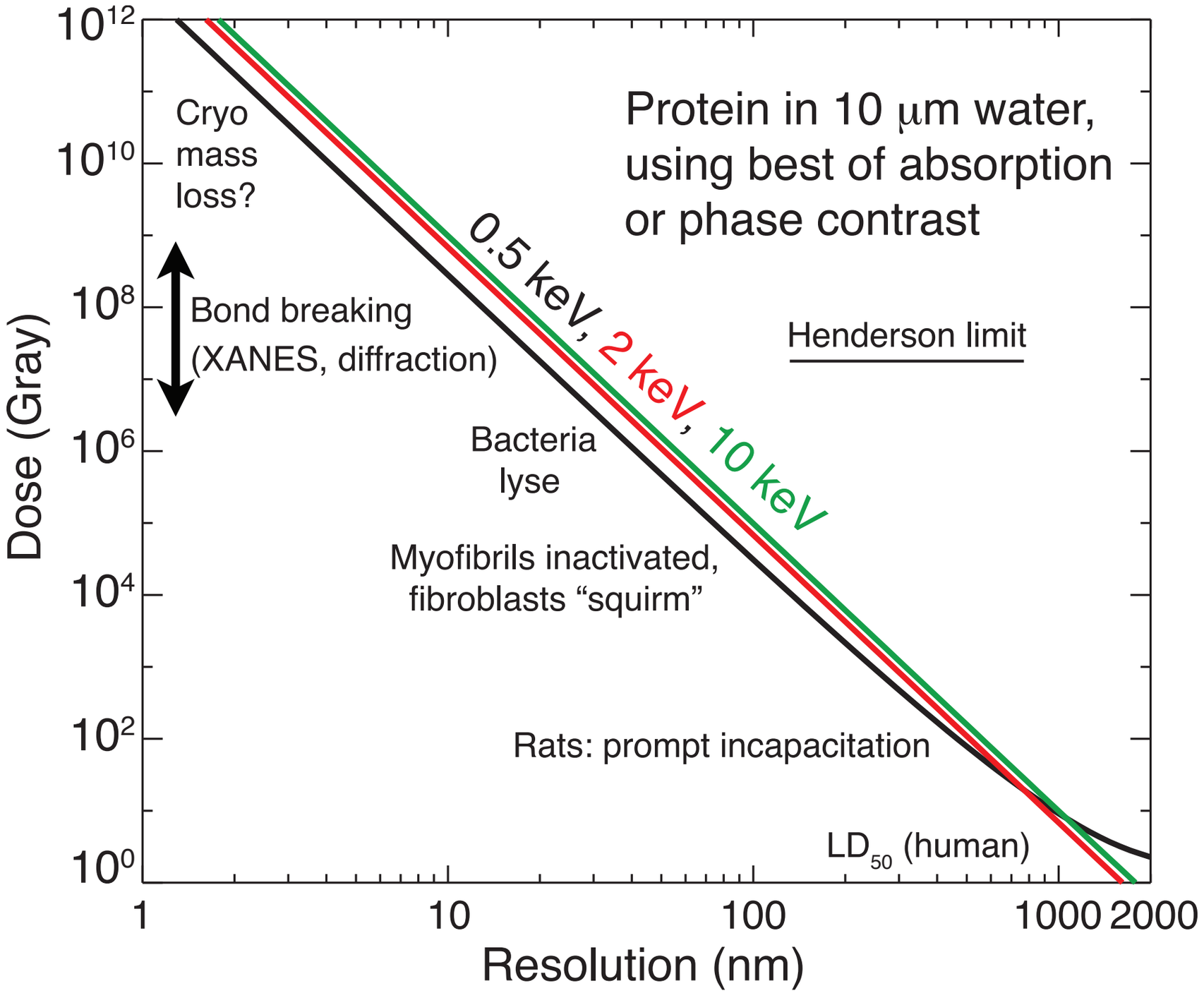
Table 2
Potential performance gain factors in X-ray fluorescence nanoprobe analysis that could be expected from a diffraction-limited storage ring along with improvements in spectral bandpass (such as the use of a multilayer monochromator instead of a crystal monochromator), improved nanofocusing optics (such as stacked, high-aspect-ratio zone plates), and improved detector solid angle (such as is offered by the Maia detector).

The total detected flux includes elastic and Compton scattered photons, while the elemental flux represents the signal in a typical elemental fluorescence line (such as from Fe or Mn) from representative cells. The baseline for these estimates is the operating experience of the Bionanoprobe at the Advanced Photon Source (Chen *et al.*, 2014), assuming pixel transit times in that instrument of 100 ms for detecting about 100 photons in typical elemental fluorescence lines. It may be difficult to realise the exact gain factors listed here, either separately or especially in aggregate, but it is still instructive for considering future possibilities.

Improvement	Potential improvement factor	Focused flux (10^9 s^{-1})	Total detected flux (10^3 s^{-1})	Elemental flux (10^3 s^{-1})	Pixel time (μs)
Bionanoprobe today	1	3.5	30	1.0	100000
Diffraction-limited storage ring	150	525	4500	150	667
Increased spectral bandpass	10	5250	45000	1500	67
Improved nanofocusing optics	3	21000	180000	6000	22
Improved detector solid angle	1.8	37800	324000	10800	12

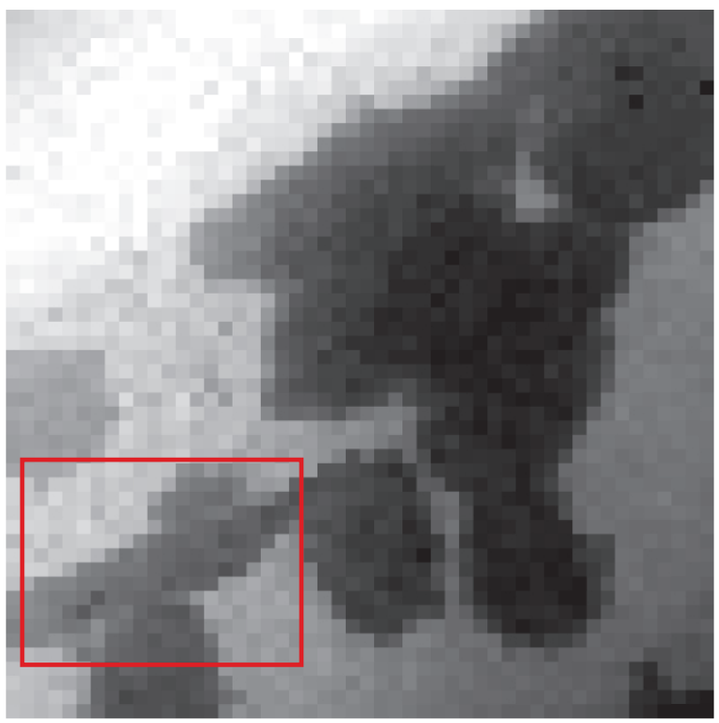
Dose versus resolution for wet soft materials

- Calculation of radiation dose using best of phase, absorption contrast and 100% efficient imaging.
- In a 3D world, high resolution structures are also thin, with lower contrast.
- Things that can be done wet at room temperature:
  - bacteria at 50 nm resolution
  - small animals at micrometer resolution (followed by sacrifice)
  - At LD_{50}, half die!



Initially living cells at room temperature

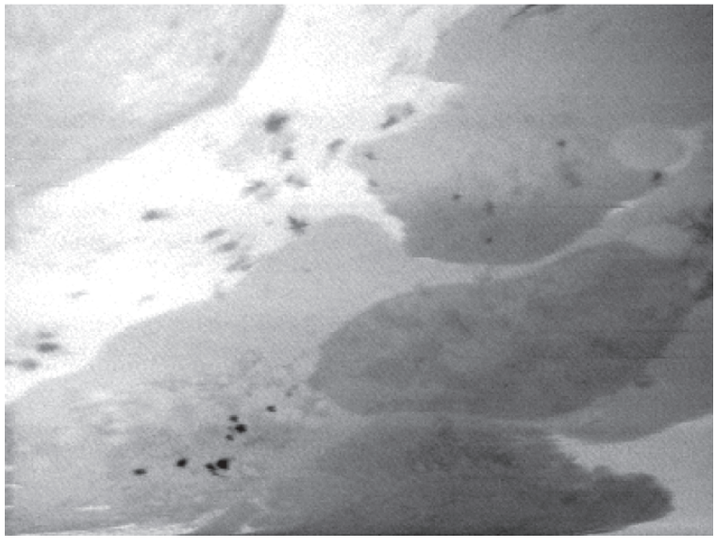
- Chinese hamster ovarian (CHO) fibroblasts in culture medium with periodic reflow.
- “Red for dead” fluorescent dyes used to confirm viability over hours with no x-ray exposure.
- Imaged in a soft x-ray scanning transmission microscope (NSLS X1A)



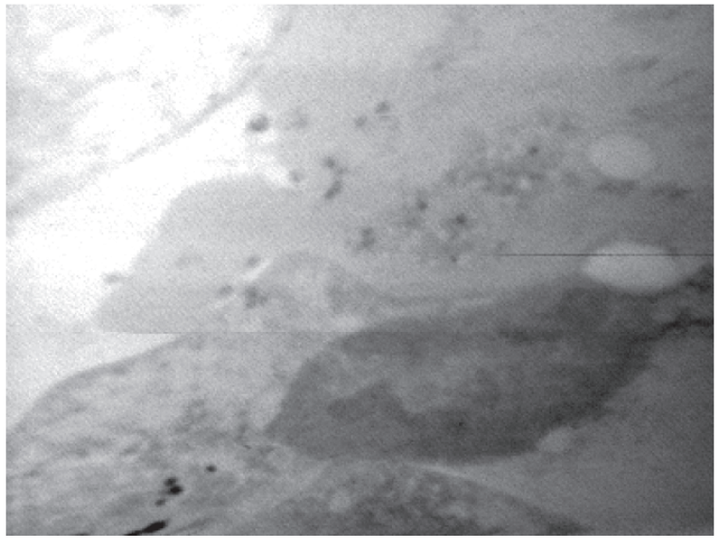
10 μm
6.0×10^2 Gray, ET=2 minutes

Experiment by V. Oehler, J. Fu, S. Williams, and C. Jacobsen, Stony Brook using specimen holder developed by Jerry Pine and John Gilbert, CalTech. In Kirz, Jacobsen, and Howells, *Quarterly Reviews of Biophysics* **28**, 33 (1995).

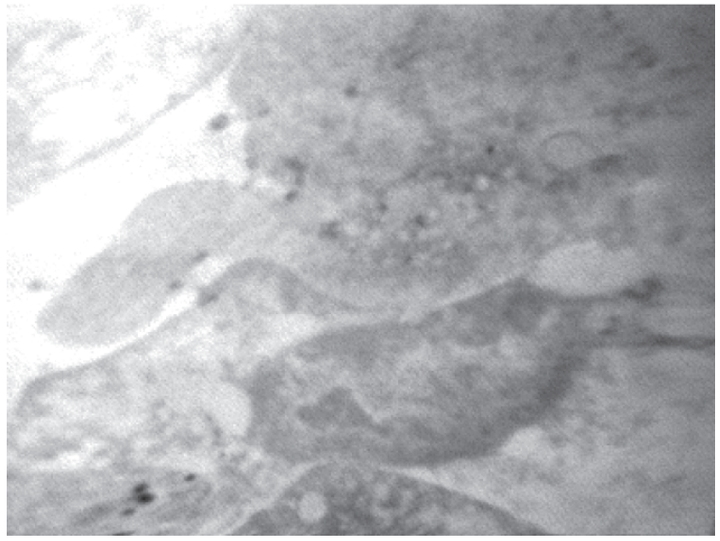
1 Gray=1 Joule/kg absorbed



5 μm
1.2×10^5 Gray, ET=9.5 min



5 μm
2.4×10^5 Gray, ET=17 min



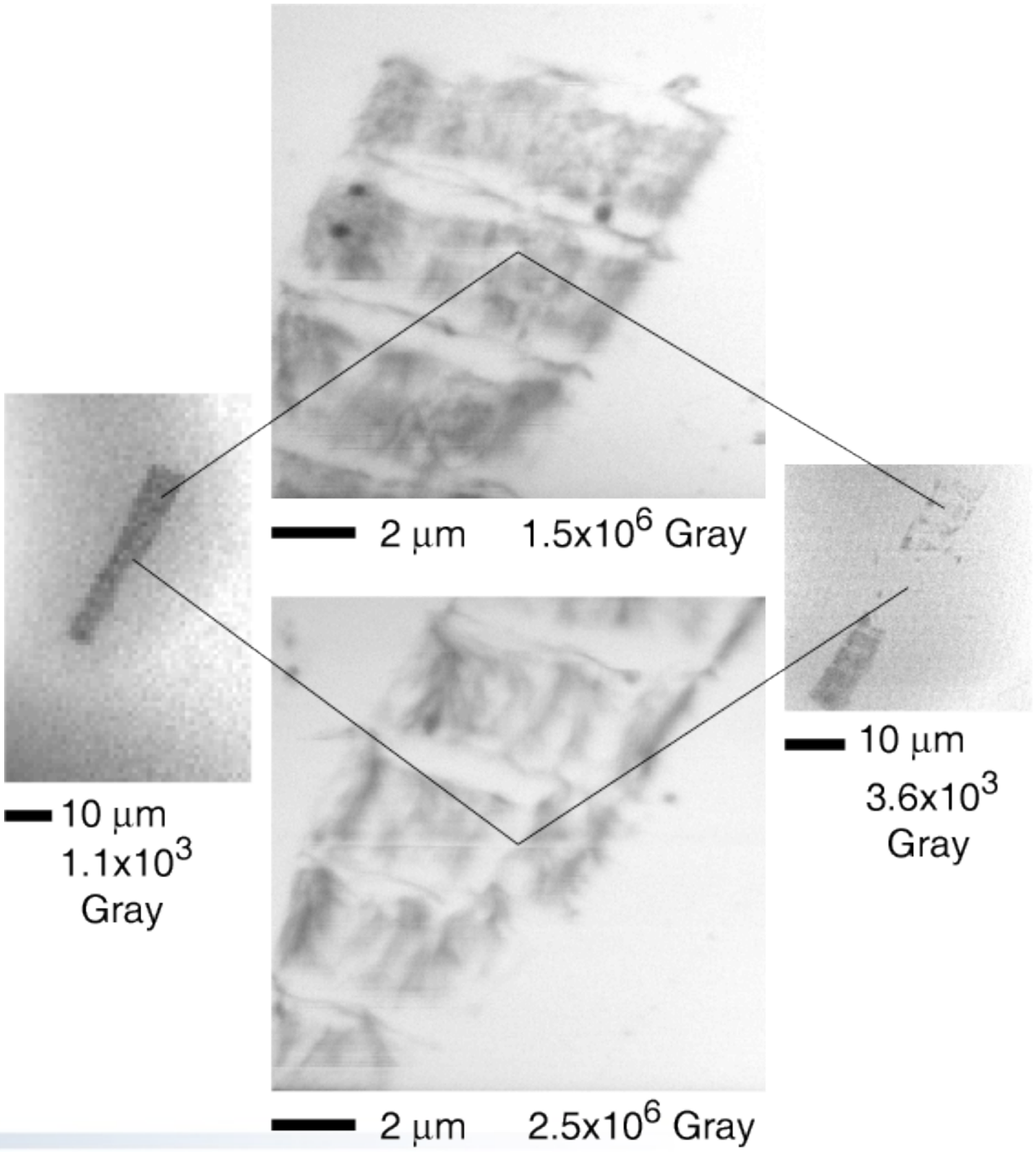
5 μm
3.7×10^5 Gray, ET=24.5 min

X-rays: damaging when wet!

- Study: polyacrilimide-induced flocculation of clays in water (irrigation of loamy soils)
- Mass loss is visible after acquiring spectra with focused beam (wet sample at room temperature)
- U. Neuhaeusler, PhD Thesis (Stony Brook/Göttingen)

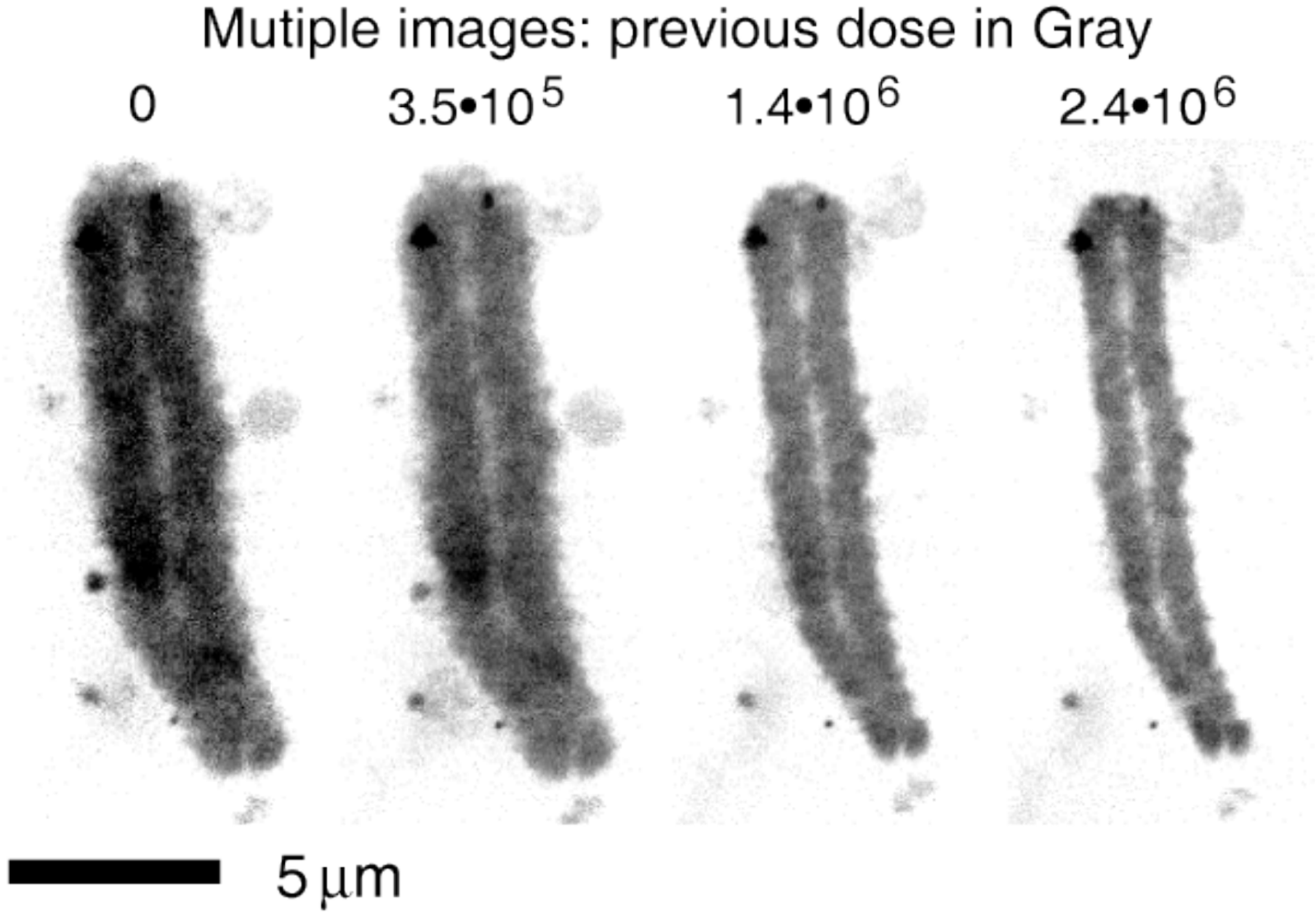
Muscle damage

- Images: dragonfly flight muscle, with Clara Franzini-Armstrong
- At 10^4 Gray, myofibrils stop contracting in the presence of ATP. Bennett *et al*., *J. Micros.* **172**, 109 (1993)

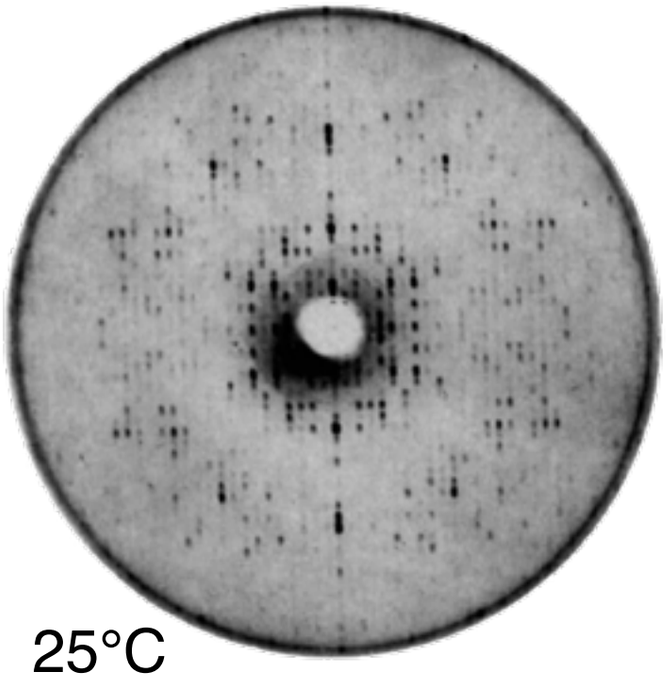


Wet, fixed samples: one image is OK

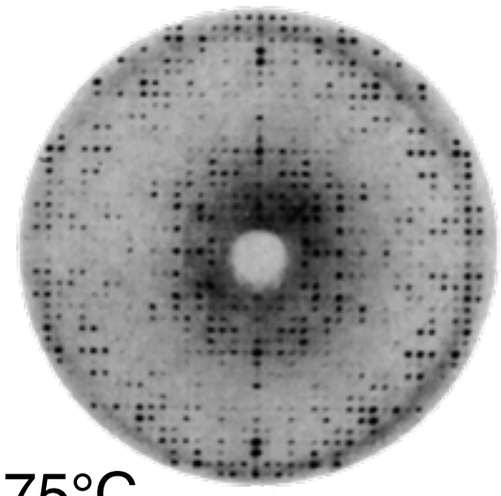
- Chromosomes are among the most sensitive specimens.
- *V. faba* chromosomes fixed in 2% glutaraldehyde. S. Williams *et al*., *J. Microscopy* **170**, 155 (1993)
- Repeated imaging of one chromosome shows mass loss, shrinkage



Cryo crystallography



25°C



-75°C

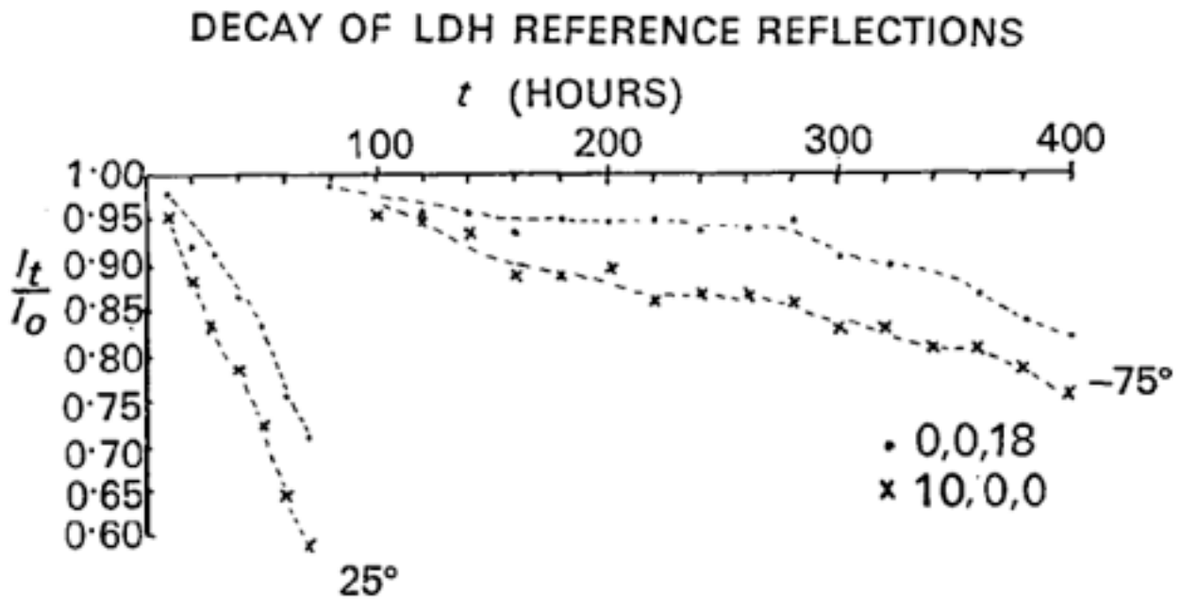


Fig. 5. The ratio I_t/I_0 for two reference reflections plotted as a function of exposure time for a typical native and frozen crystal. I_t represents the intensity at time t. Results for 0,0,18 and 10,0,0 are shown with dots and crosses respectively.

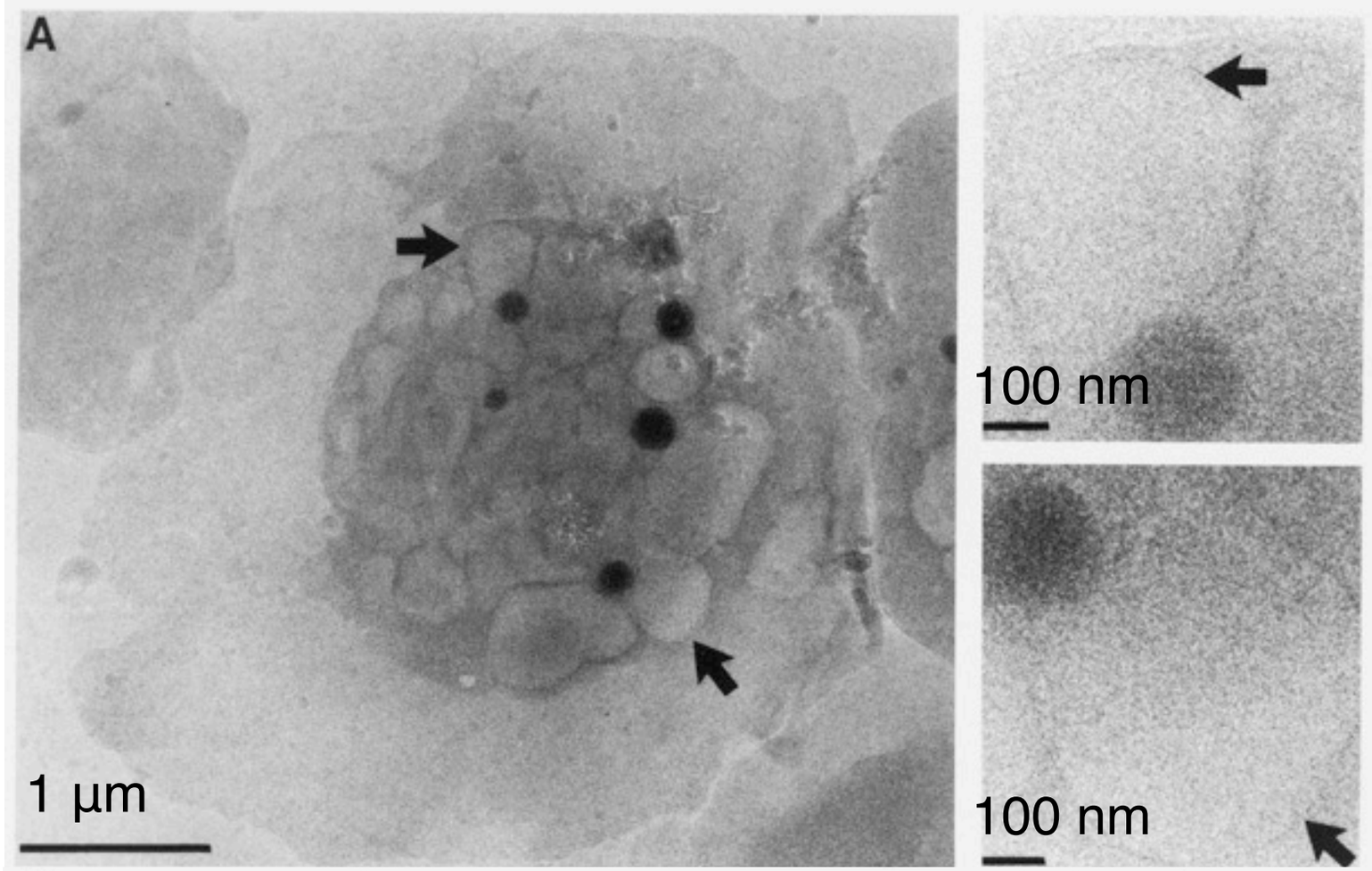
Acta Cryst. (1970). B**26**, 998

Crystallographic Studies on Lactate Dehydrogenase at -75°C

BY DAVID J. HAAS* AND MICHAEL G. ROSSMANN

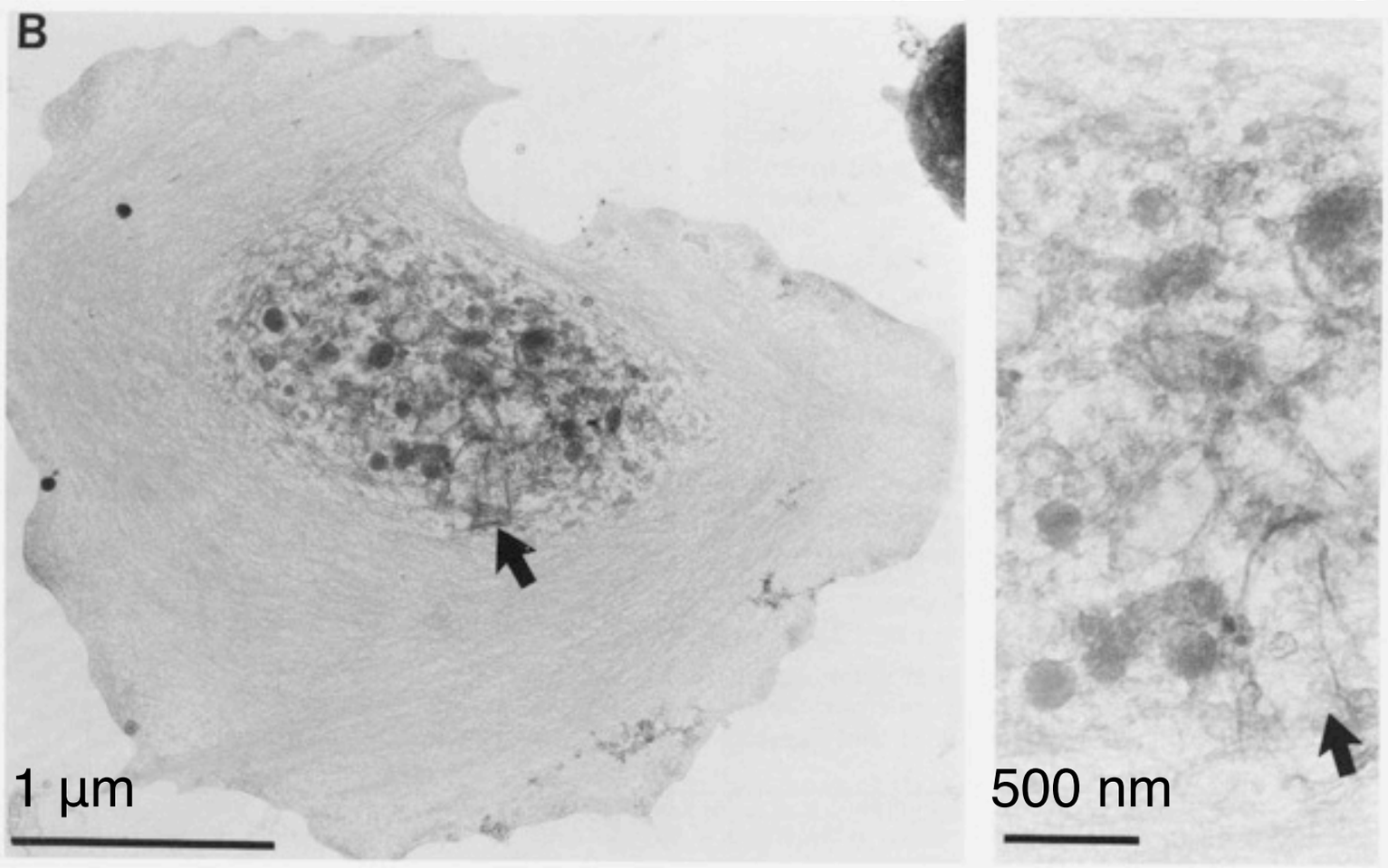
Crystals of lactate dehydrogenase (LDH) were frozen by equilibration in a sucrose–ammonium sulfate solution, and then dipping into liquid nitrogen. The rate of radiation damage for frozen crystals was tenfold less than for crystals at room temperature. The physical properties of frozen crystals are discussed. Analysis of 3·5 Å data collected at −75°C for native LDH and two heavy atom derivatives showed that these derivatives retained their isomorphism in the frozen state.

See also Low, Chen, Berger, Singman, and Pletcher, *PNAS* **56**, 1746 (1966)



Frozen hydrated

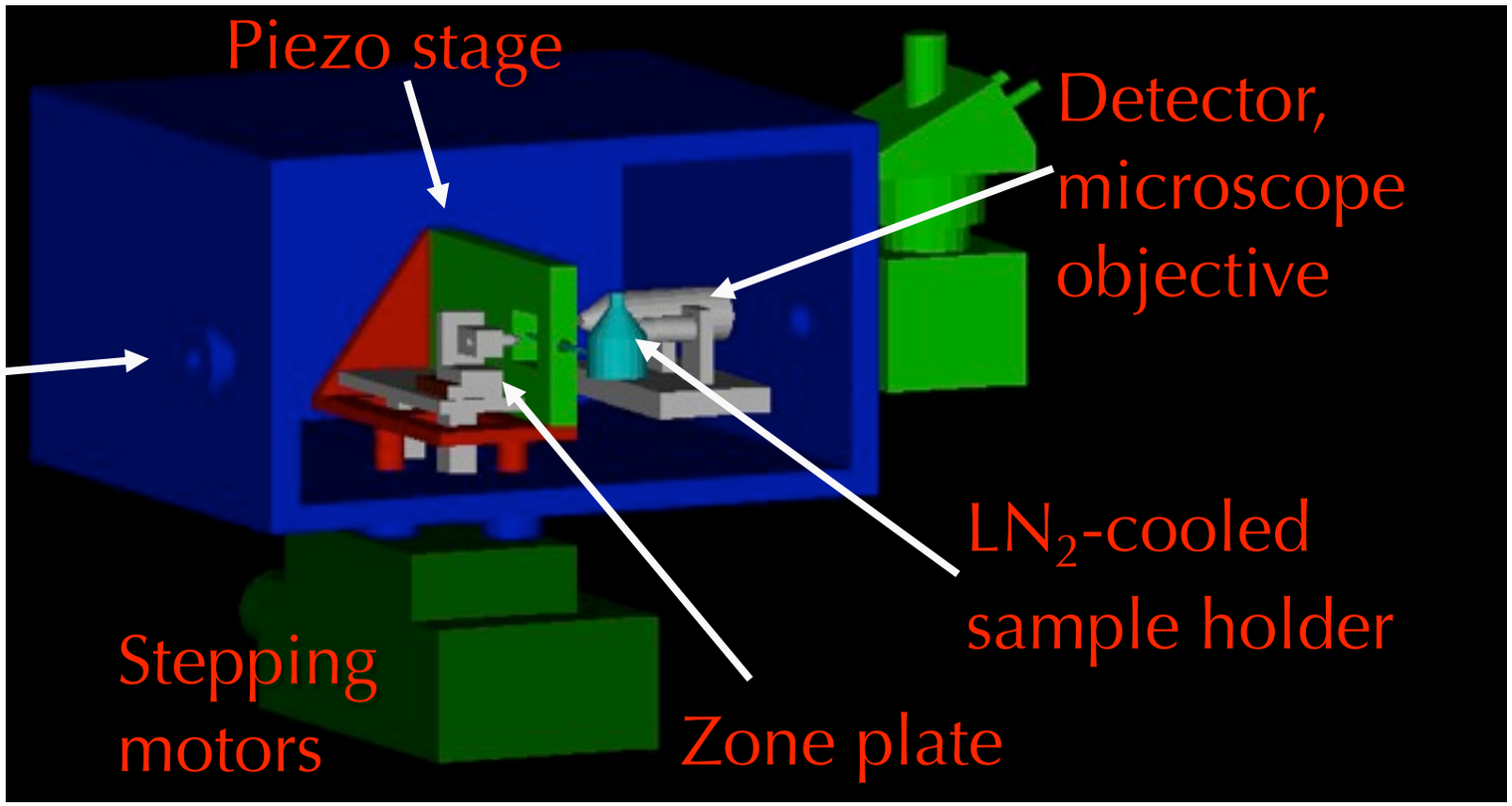
- Human blood platelets
- 1 MeV transmission electron microscope (JEOL-1000)
- O'Toole, Wray, Kremer, and McIntosh, *J. Struct. Bio.* **110**, 55 (1993)



2% glutaraldehyde fix
1% OsO_4 postfix
critical-point dry

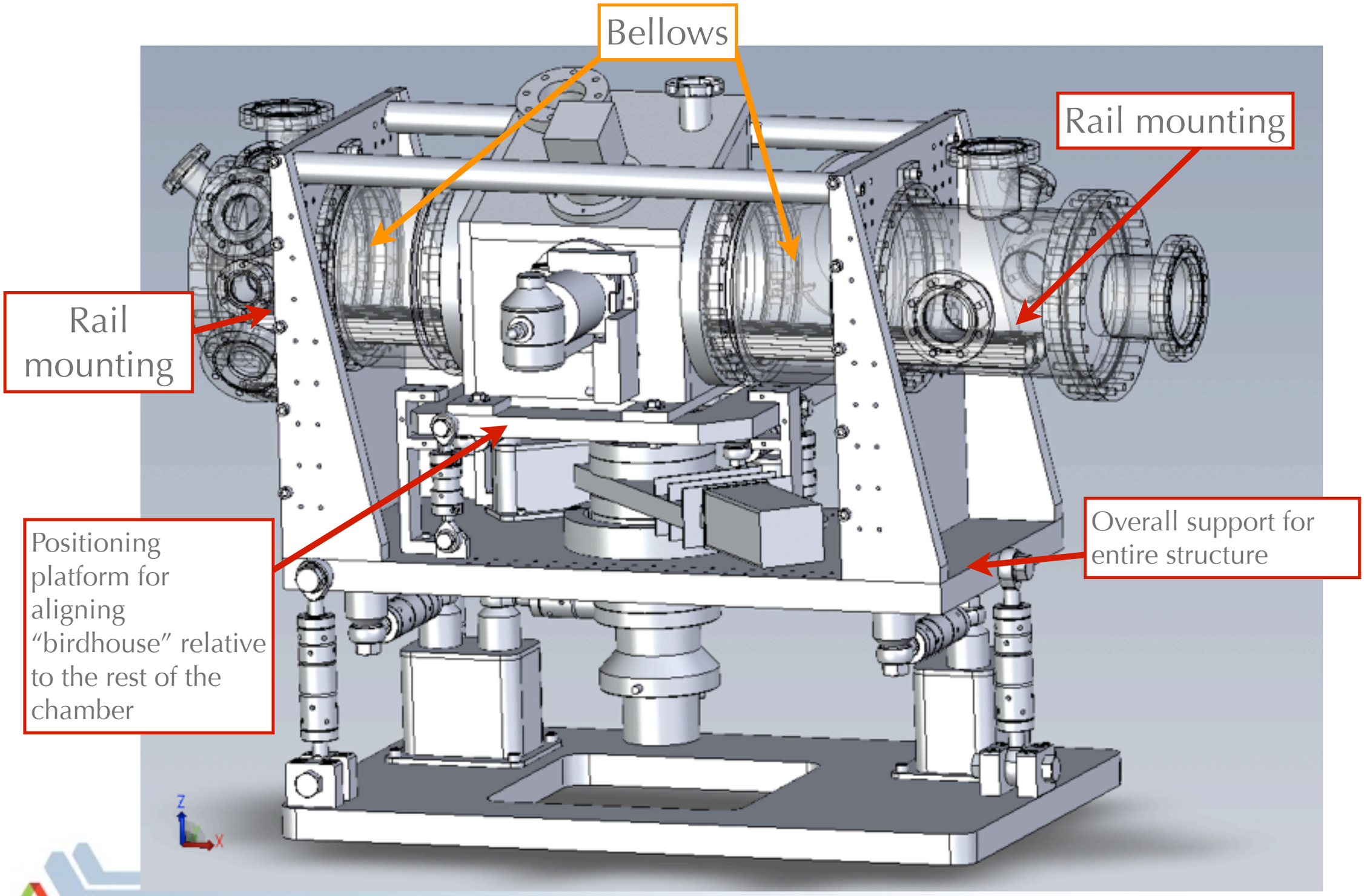
The Stony Brook cryo STXM

J. Maser *et al.*, *J. Micros*. **197**, 68 (2000).



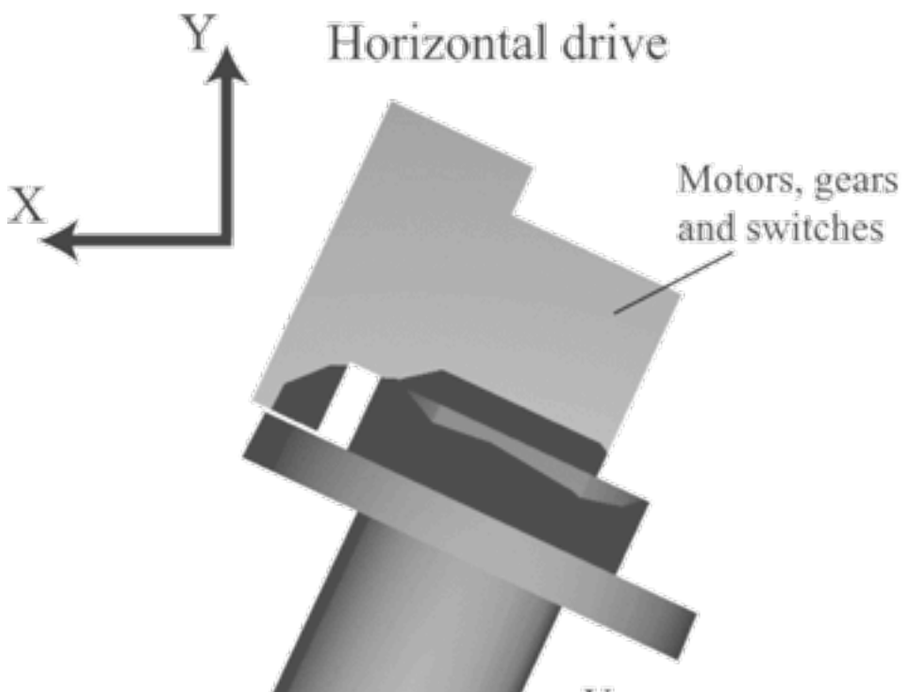
Stony Brook/ALS cryo CDI apparatus

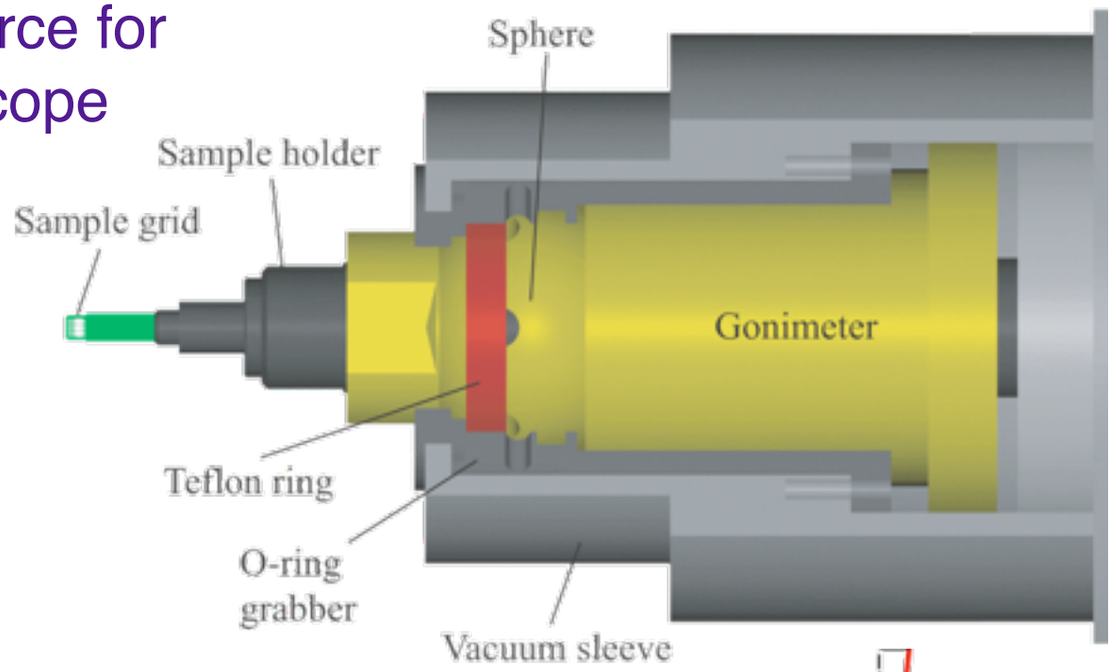
First version: Stony Brook. Beetz et al., *NIMA* **545**, 459 (2005)
Enhancements with Tony Warwick, ALS, ~2008

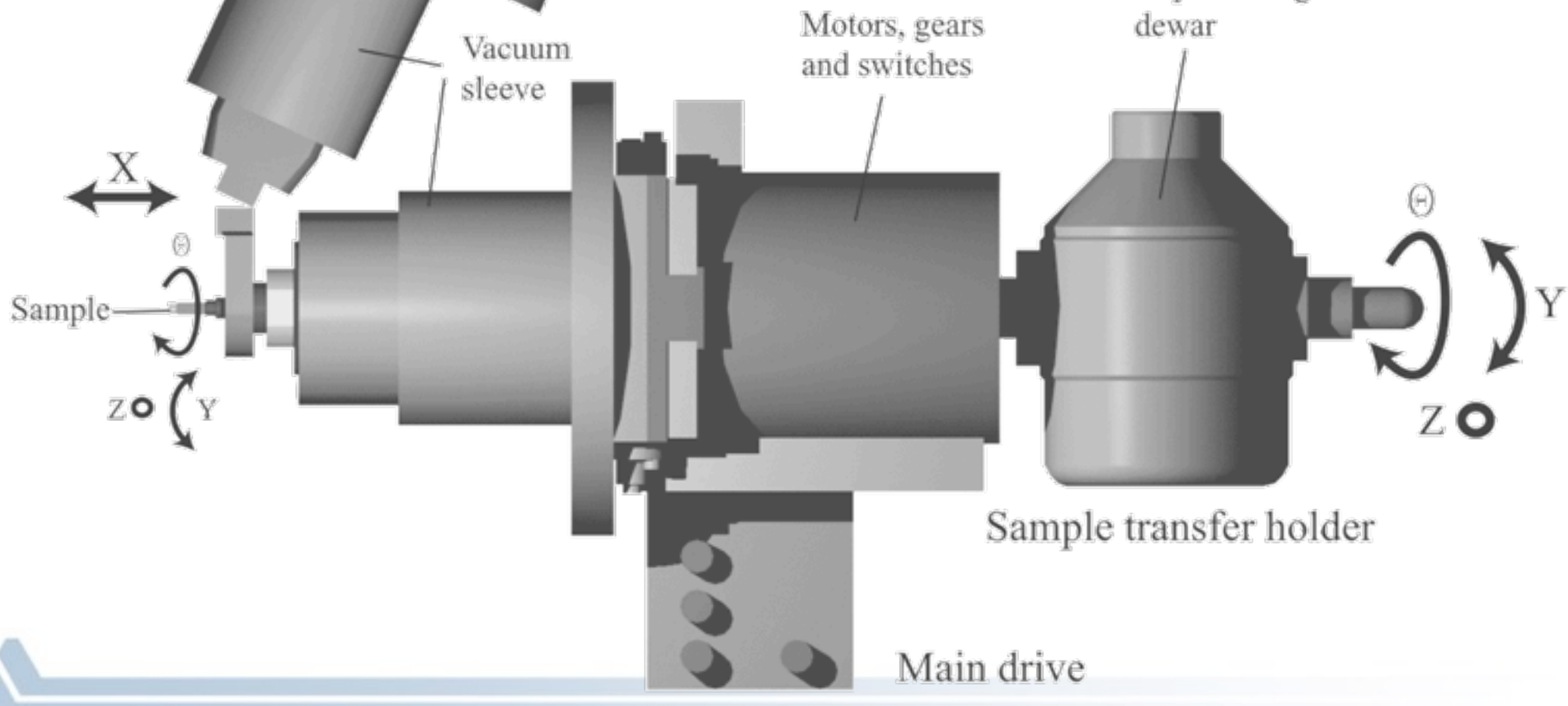


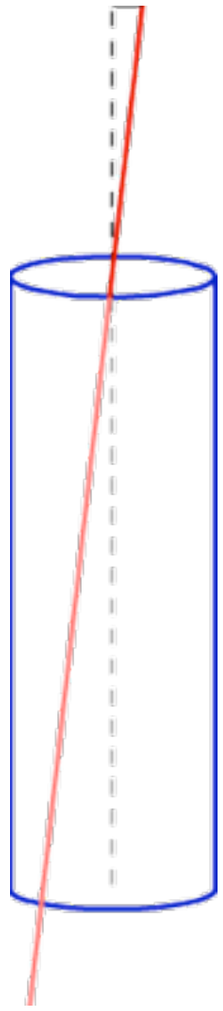
JEOL 2000-type goniometer schematic

Now on permanent loan to Canadian Light Source for a cryo scanning transmission soft x-ray microscope









Xradia, 2007/2008

- Conceptual design and engineering tests of a new cryo microscope approach. Instruments at APS, ALBA, Diamond, ANKA, Hefei

(12) **United States Patent**
Jacobsen et al.

(10) **Patent No.: US 8,602,648 B1**
(45) **Date of Patent: Dec. 10, 2013**

(54) **X-RAY MICROSCOPE SYSTEM WITH CRYOGENIC HANDLING SYSTEM AND METHOD**

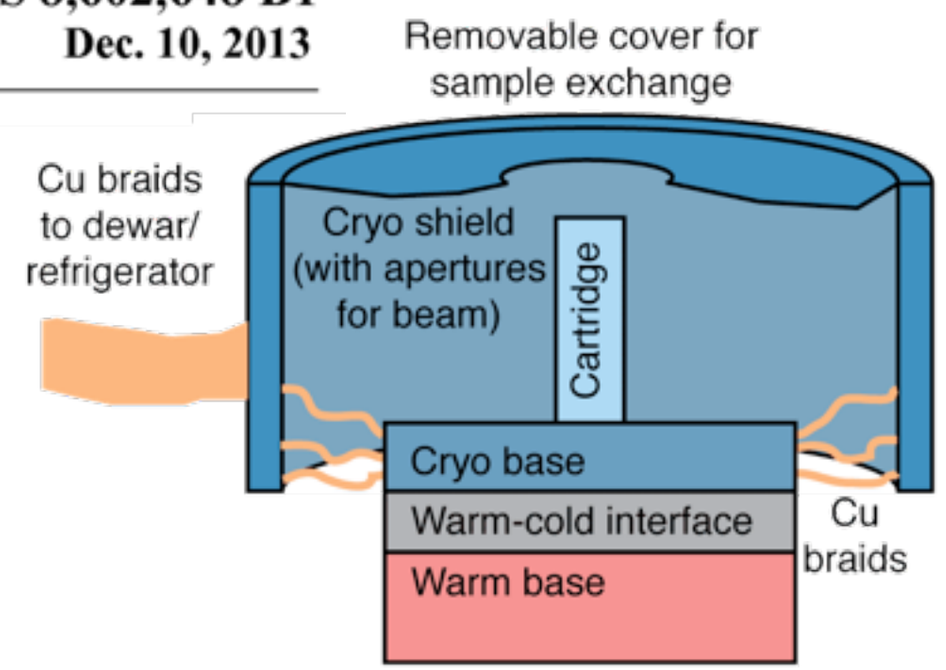
(75) Inventors: **Chris J. Jacobsen**, Sound Beach, NY (US); **Wenbing Yun**, Walnut Creek, CA (US)

(73) Assignee: **Carl Zeiss X-ray Microscopy, Inc.**, Pleasanton, CA (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 960 days.

(21) Appl. No.: **12/559,183**

(22) Filed: **Sep. 14, 2009**



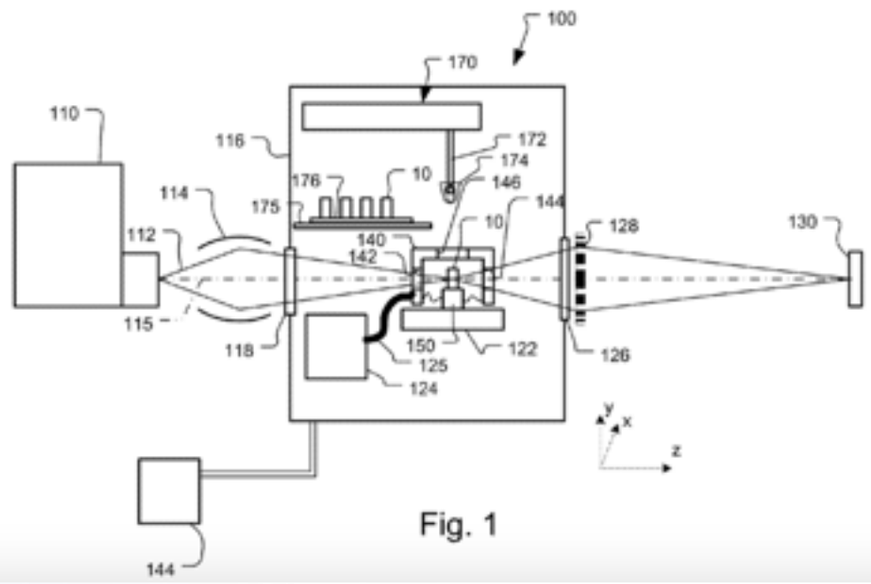
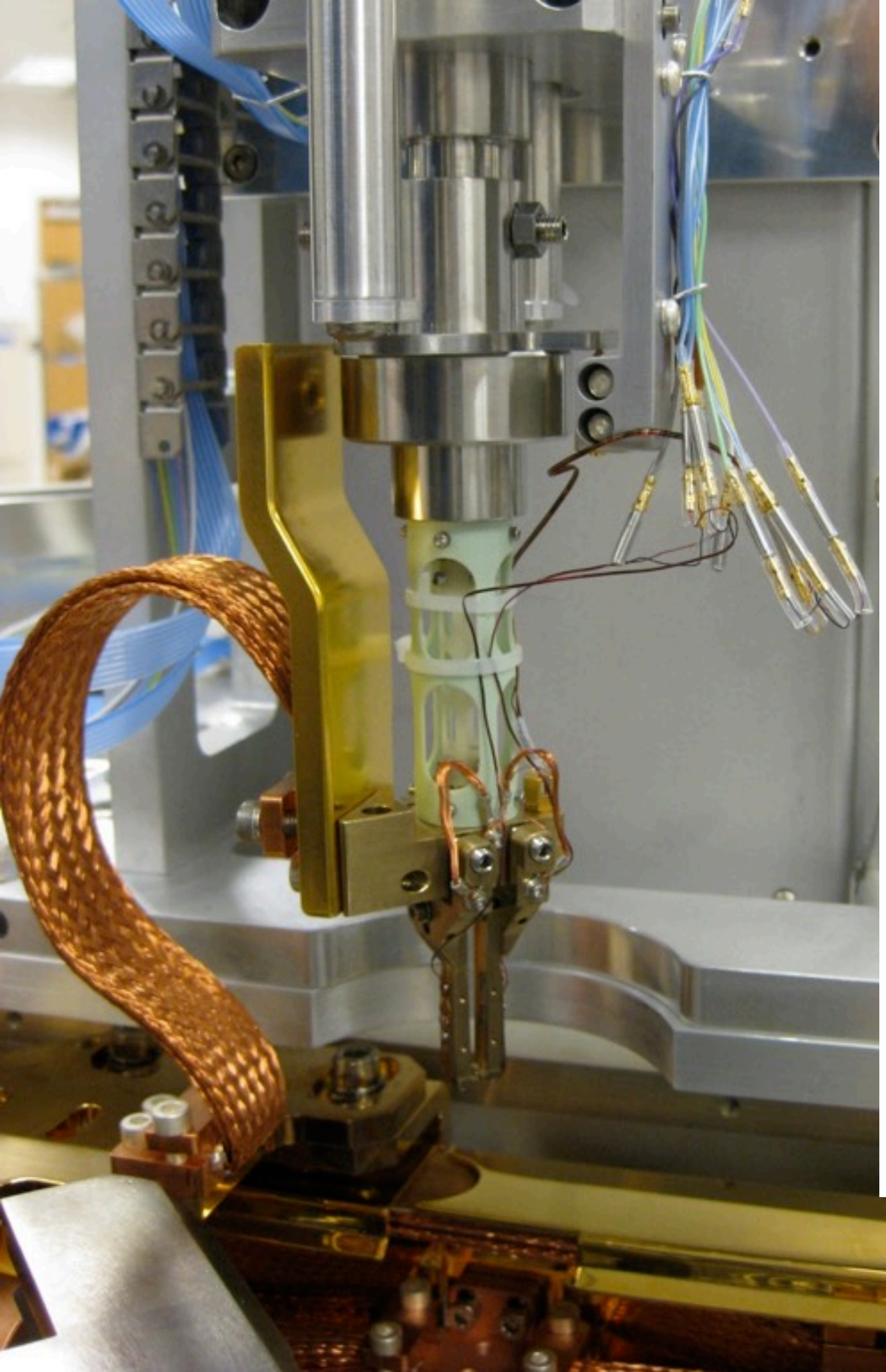
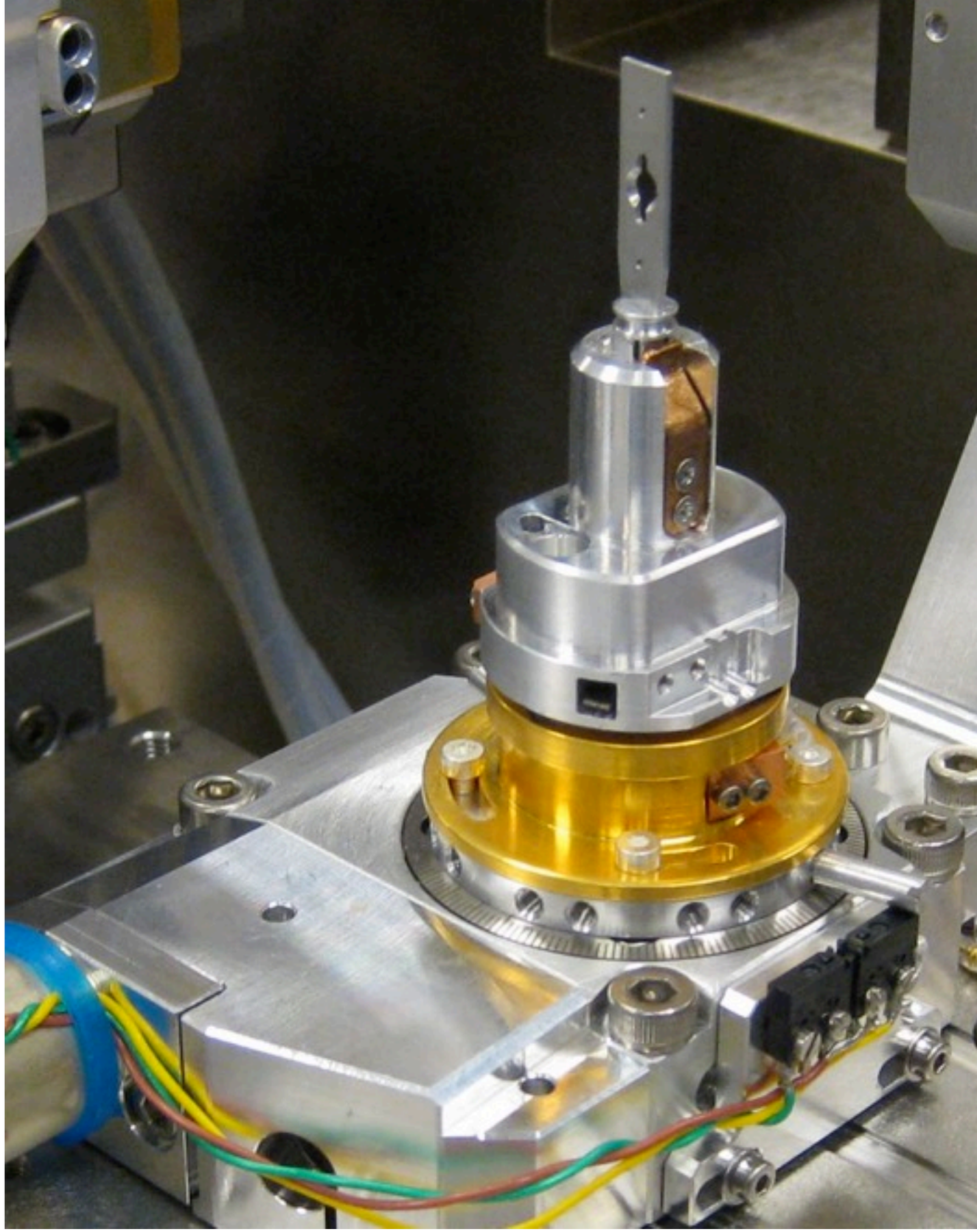


Fig. 1





J. Synchrotron Rad. (2014). **21**, 66–75

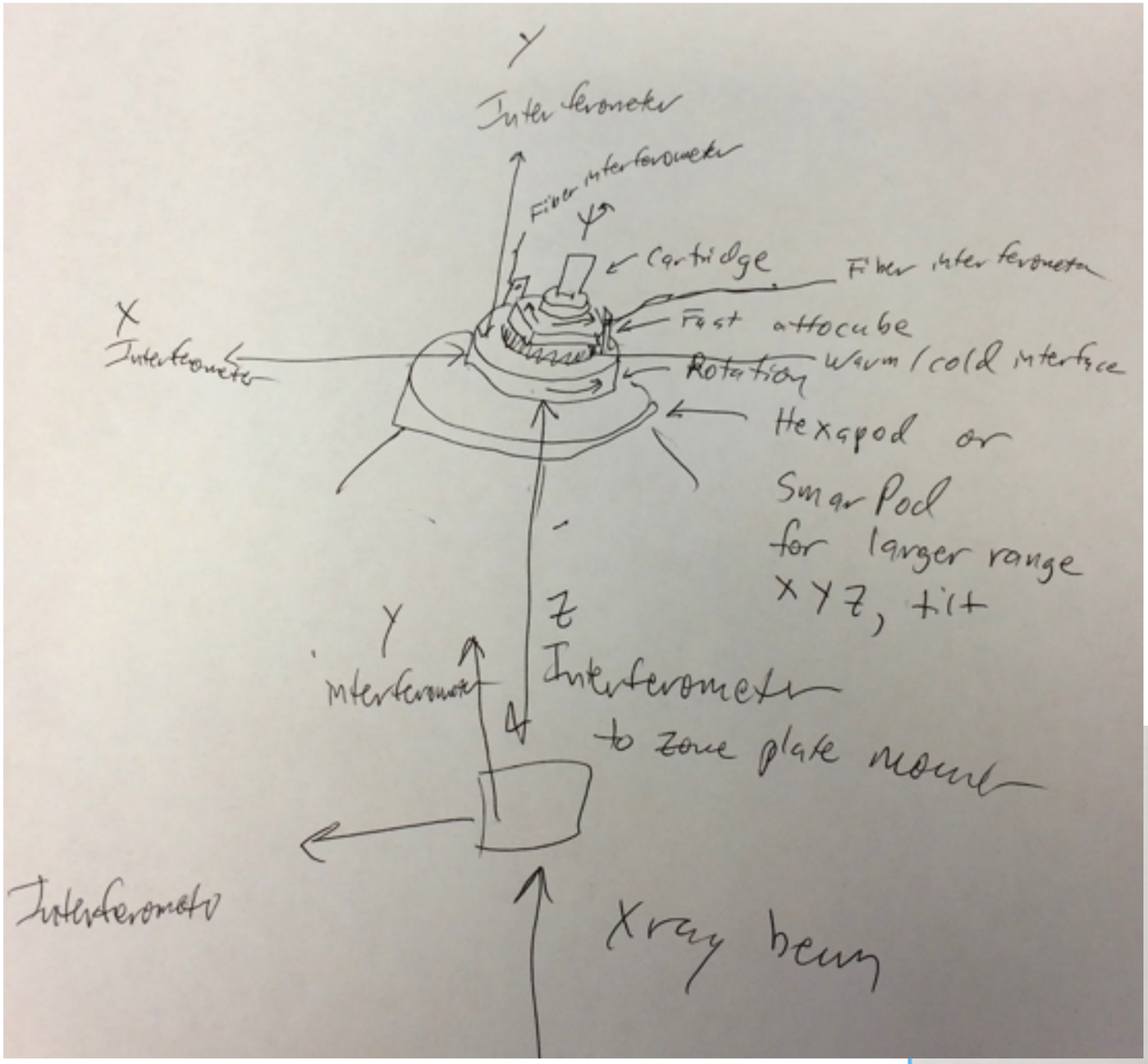
The Bionanoprobe: hard X-ray fluorescence nanoprobe with cryogenic capabilities

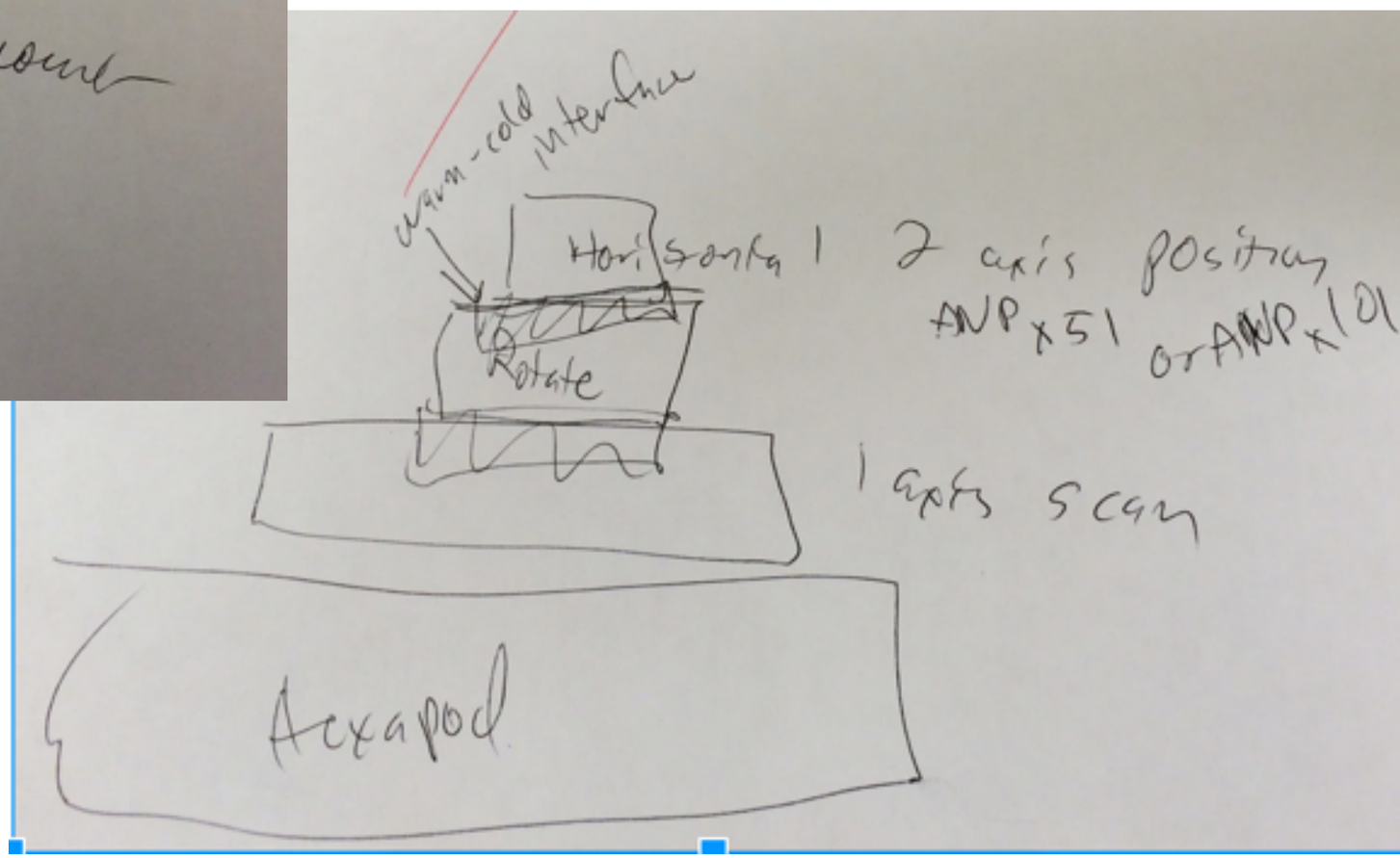
S. Chen,[a]* J. Deng,[b] Y. Yuan,[c] C. Flachenecker,[d] R. Mak,[e] B. Hornberger,[d] Q. Jin,[e] D. Shu,[a] B. Lai,[a] J. Maser,[a] C. Roehrig,[a] T. Paunesku,[c] S. C. Gleber,[a] D. J. Vine,[a] L. Finney,[a] J. VonOsinski,[f] M. Bolbat,[f] I. Spink,[d] Z. Chen,[d] J. Steele,[d] D. Trapp,[d] J. Irwin,[d] M. Feser,[d] E. Snyder,[d] K. Brister,[f] C. Jacobsen,[a,b,e,g] G. Woloschak[c] and S. Vogt[a]

[a]X-ray Science Division, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA, [b]Applied Physics, Northwestern University, Evanston, IL 60208, USA, [c]Department of Radiation Oncology, Northwestern University, Chicago, IL 60611, USA, [d]Xradia Inc., Pleasanton, CA 94588, USA, [e]Department of Physics and Astronomy, Northwestern University, Evanston, IL 60208, USA, [f]Northwestern Synchrotron Research Center, Argonne, IL 60439, USA, and [g]Chemistry of Life Processes Institute, Northwestern University, Evanston, IL 60208, USA.
*E-mail: sichen@aps.anl.gov

xradia is now ZEISS

Funded by NIH Grant to Gayle Woloschak *et al.*
Argonne technical lead: Stefan Vogt


Y
Interferometer
Fiber interferometer
Cartridge
Fiber interferometer
X
Interferometer
Fast attocube
Warm/cold interface
Rotation
Hexapod or
Smar Pod
for larger range
XYZ, tilt
Y
interferometer
Z
Interferometer
to zone plate mount
Interferometer
Xray beam


warm-cold interface
Horizontal
2 axis positioning
ANP x51 or ANP x101
Rotate
1 axis scan
Acxapod

So that’s why... so let’s look at some pictures!


A19
A20
A1
A2
A3
A4


20