Accelerator Based X-ray Science: Beyond the optical microscope

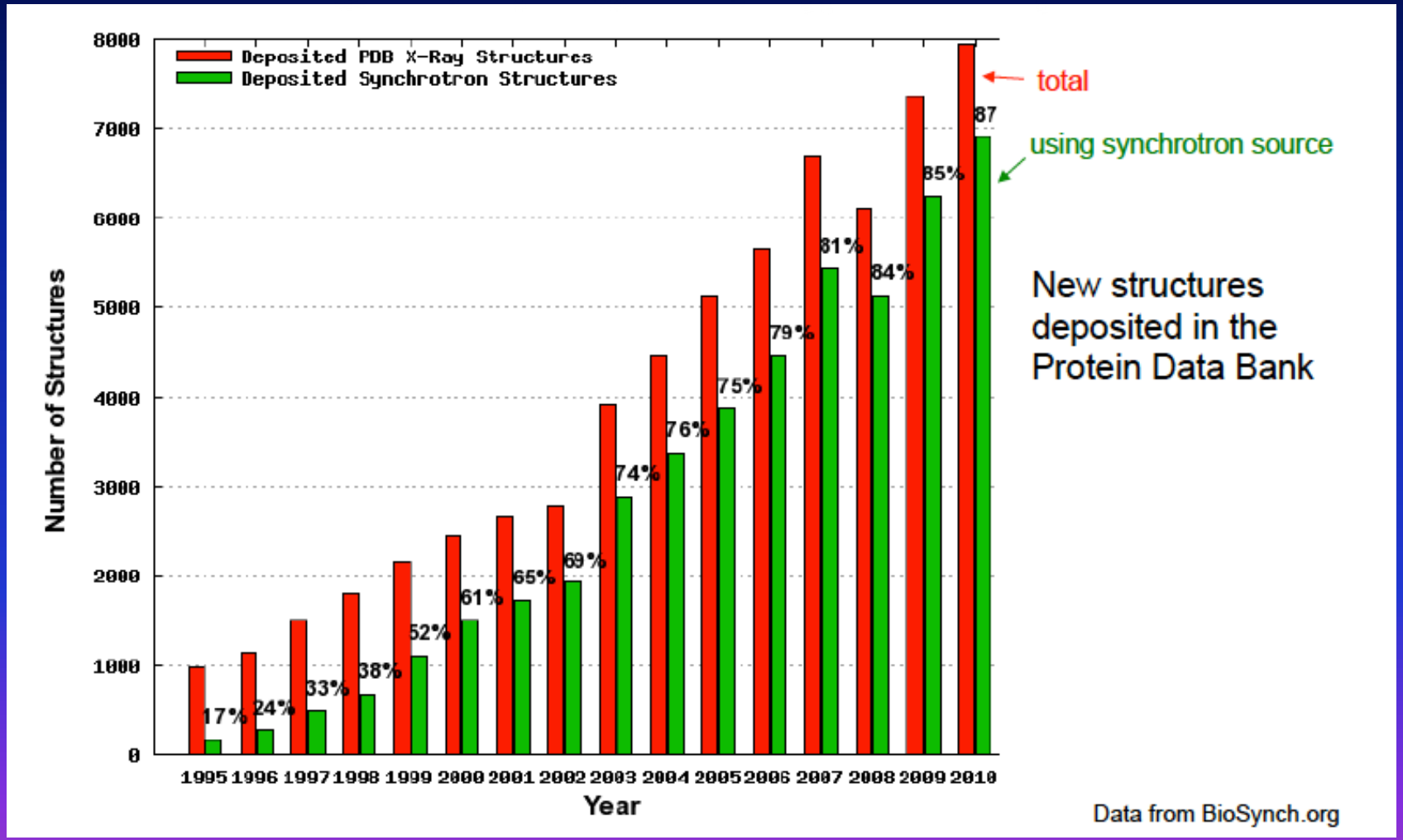
J. B. Hastings

SLAC *National Accelerator Laboratory,* **Sept. 8, 2011**

SLAC NATIONAL ACCELERATOR LABORATORY

IPAC11

Synchrotron radiation has revolutionized Protein Crystallography



PX1

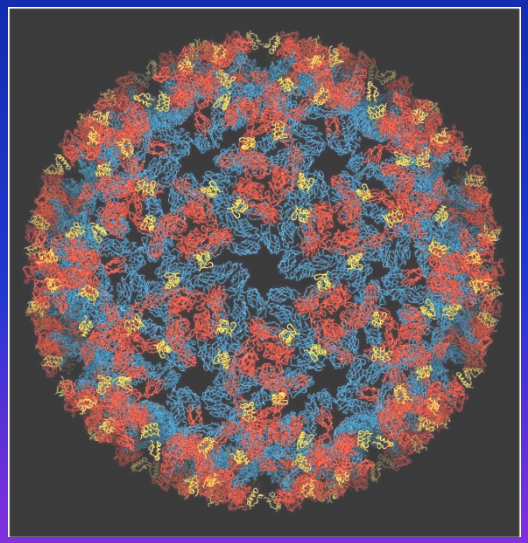
Glycoprotein organization of Chikungunya virus particles revealed by X-ray crystallography.

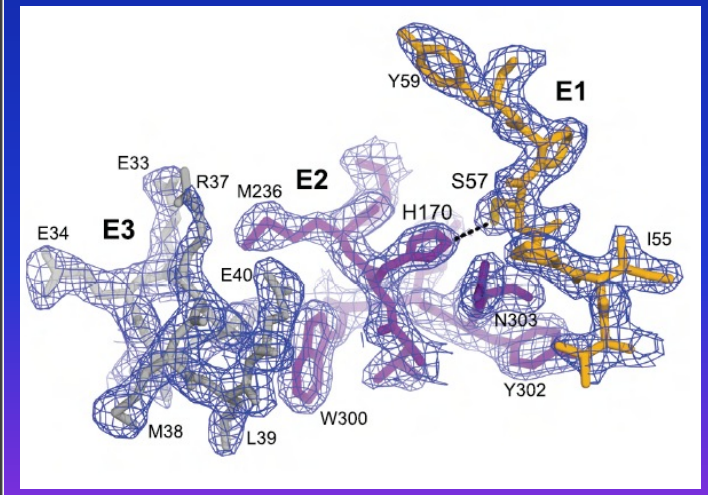
J. E. Voss, M-C Vaney, S. Duquerroy, C. Vonrhein, C. Girard-Blanc, E. Crublet, A. Thompson, G. Bricogne & F. A. Rey (2010) Nature, 2 December. Cover page

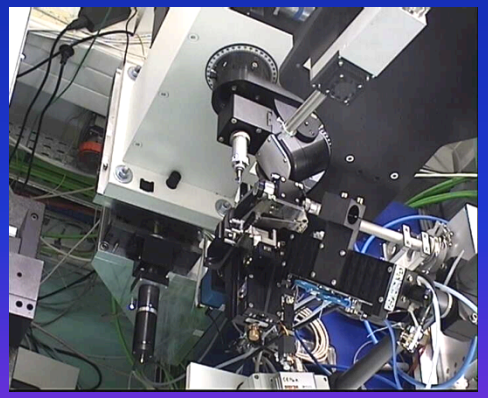
Virus endocytocis: Structural modifications of its surface protein core allows fusion with endosome

Replication: formation of p62 (E2 precursor)- E1 heterodimer
Processing and formation of mature E2 and E3
Protein core formation by heterotrimer E3-E2-E1

p62/E1 and mature E3/E2/E1 complexes.







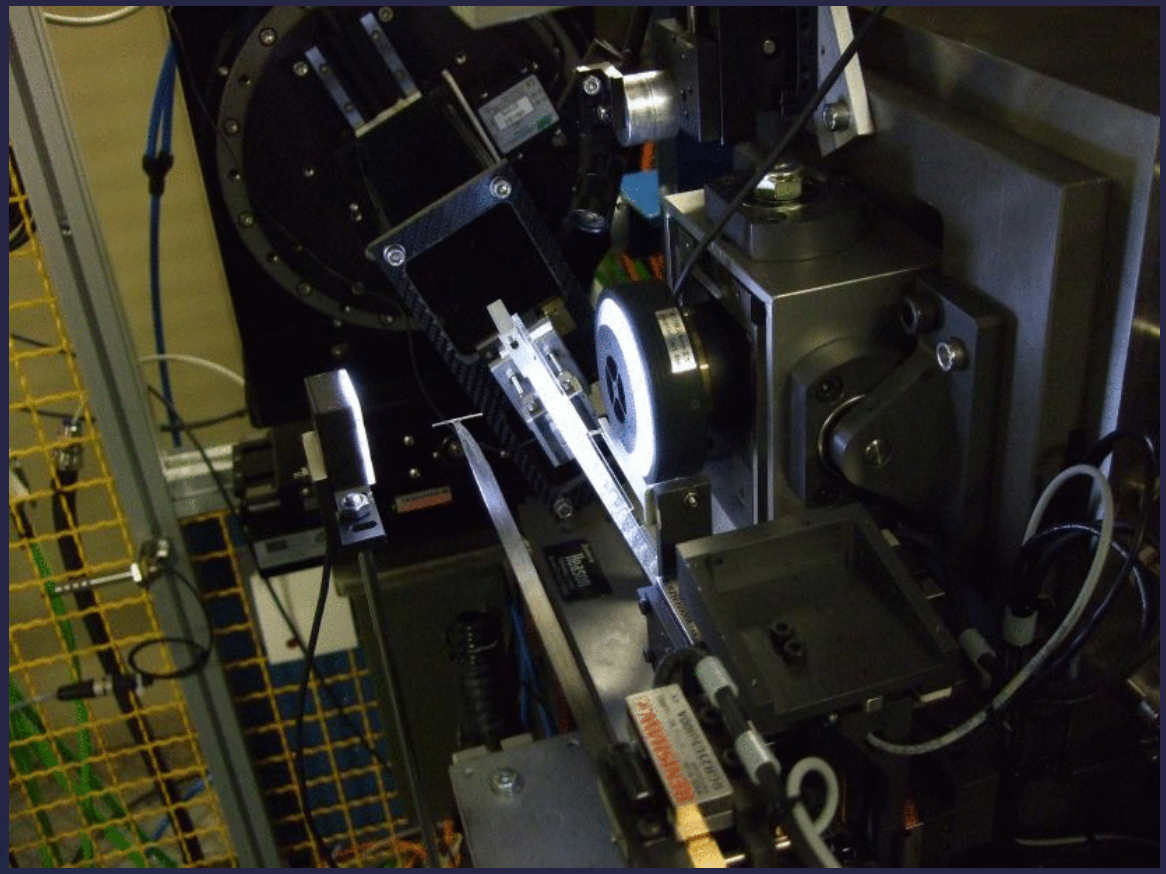
E3-E2-E1 Icosaedric arrangement coating the virus membrane

SOLEIL SYNCHROTRON

Diamond- I24: *in situ* data collection

1. Membrane protein crystal screening
2. High-throughput strucuture solution
3. Virus crystal data collection

- goniometer can handle many varieties of 96 well plate. Angular range is plate dependent but typically >30°

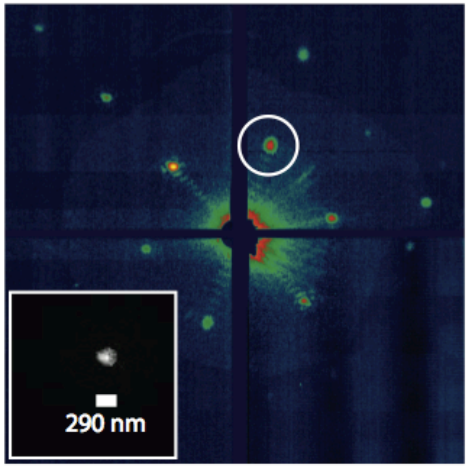


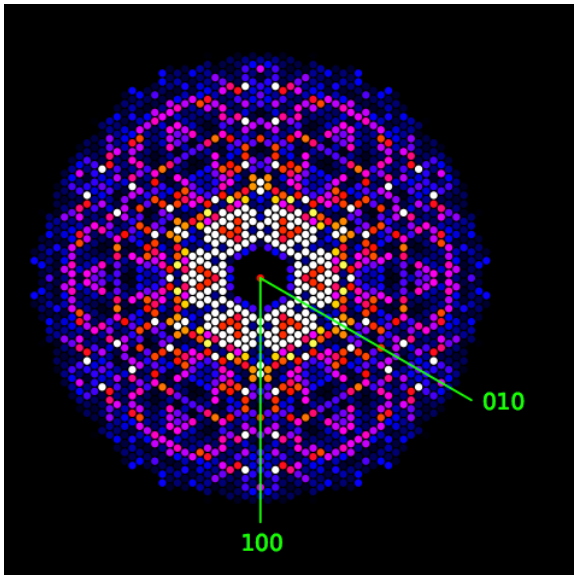
D. Axford, R. Owen, J. Aishima, G. Evans
Collaboration between Diamond Light Source
and Membrane Protein Laboratory at Diamond (Wellcome Trust)

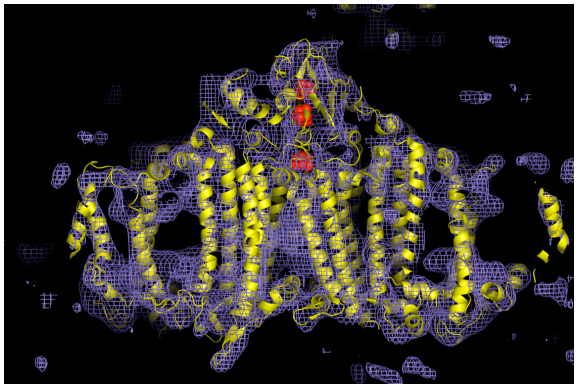
diamond

Femtosecond x-ray nanocrystallography overcomes limitations of radiation damage

A new paradigm opens up macromolecular structure determination to systems too small or radiation sensitive for synchrotron studies, and may save years of effort in crystallization trials



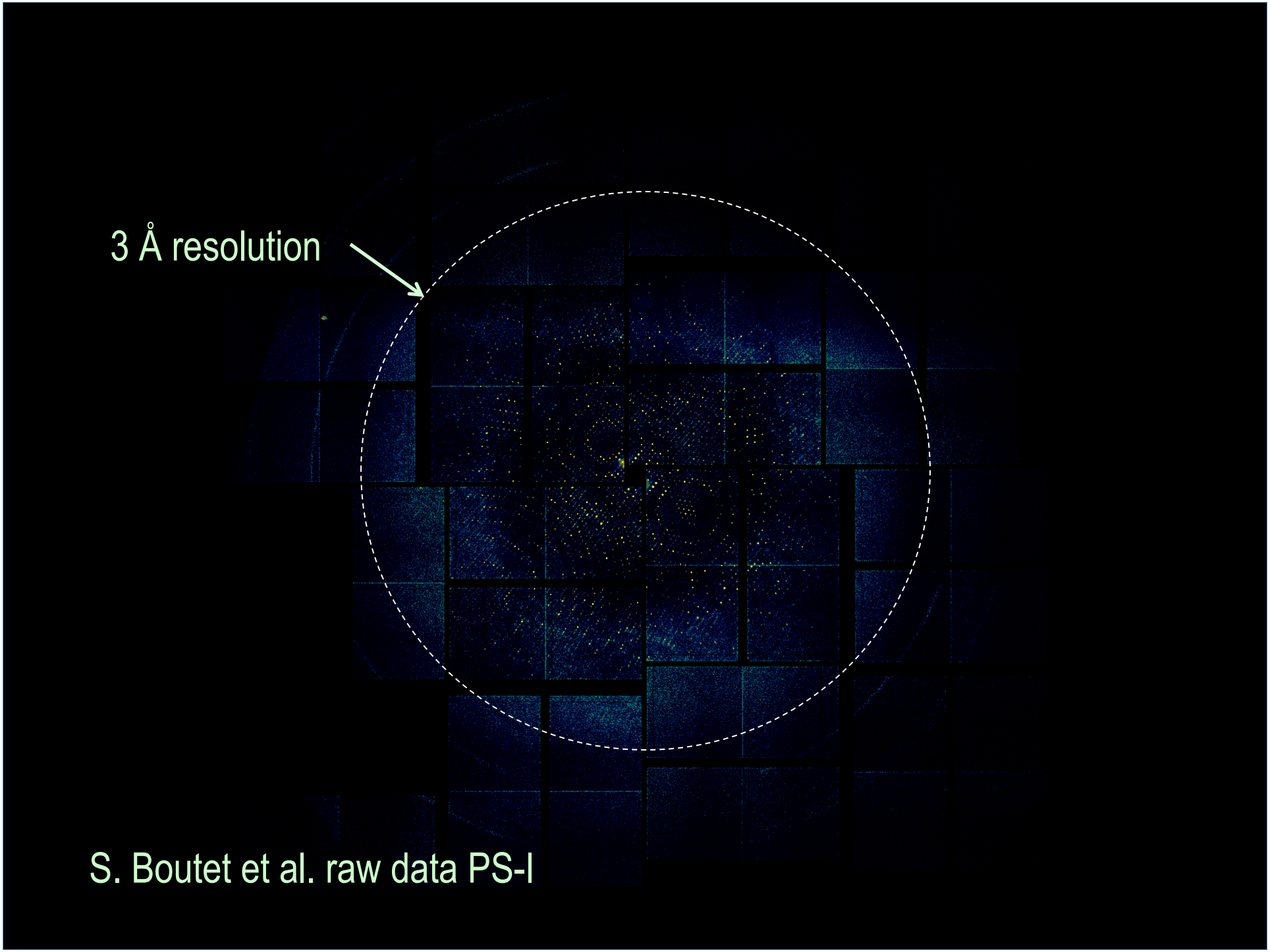




Single-shot diffraction patterns are recorded with 70 fs pulses. Coherent diffraction shows the crystal size is sub-micron (top left) and that the crystal has a perfect lattice. Individual shots are oriented in 3D and combined to build up the full information content of the underlying macromolecule (top right). This first demonstration was carried out at 2 keV photon energy, limiting the resolution to about 9 Å. (This will be improved with the dedicated CXI instrument.) The quality of the data are demonstrated by carrying out molecular replacement refinement (right). Structural details such as helices can be observed.

- The ultrafast LCLS x-ray pulses allow us to record "diffraction before destruction" where information is obtained before the onset of structural damage.
- Diffraction can be measured from sub-micron crystals containing less than a thousand molecules.
- Demonstrated using Photosystem I, a membrane protein, key to photosynthesis, that is extremely difficult to grow into large crystals.
- 30 single-crystal patterns per second were recorded from a liquid stream carrying a suspension of nanocrystals. 15,000 of these were indexed and combined into a full diffraction pattern which was analyzed with standard tools.
- Data are collected at room temperature. No cryogenic cooling or stabilization required.

H.N. Chapman et al., Nature **470,** 73 (2011)


3 Å resolution
S. Boutet et al. raw data PS-I

Hard X-Ray Nanoprobe and Scanning Microscopy

Highest resolution:

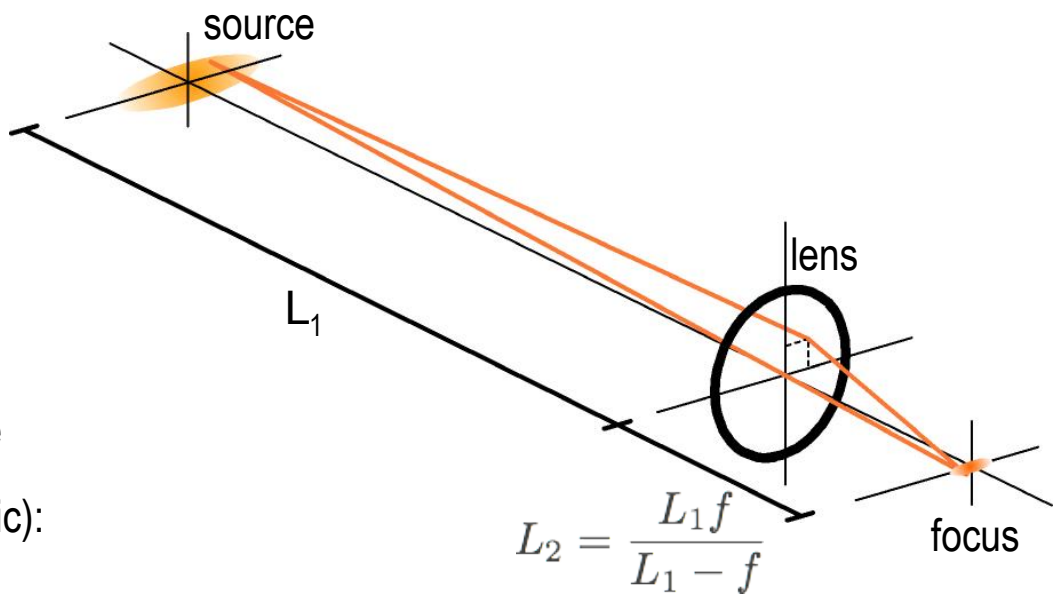
- diffraction limited imaging of source onto sample
- flux on sample given by coherent flux F_c and efficiency T of optic

Scanning microscopy:

- different contrasts:

  fluorescence
  absorption (XAS)
  diffraction (SAXS & WAXS)
  ...

  Resolution limited by focus size



Improve resolution (independent of optic):

coherent x-ray diffraction imaging
combined with nanofocusing

Institut für Strukturphysik, TU Dresden, Christian Schroer (schroer@xray-lens.de)

X-ray Optics

external total reflection

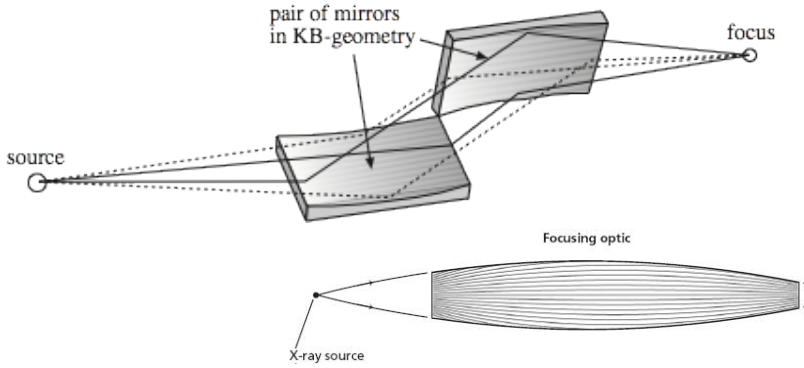
- mirrors (25nm)
- capillaries
- waveguides (25nm)

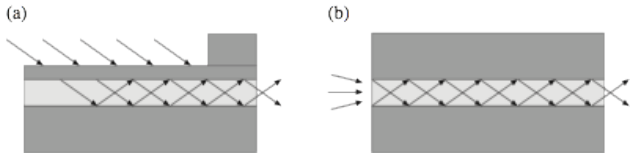
diffraction:

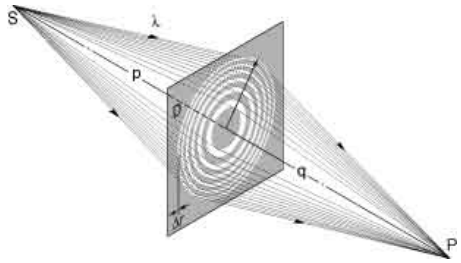
- Fresnel zone plate (30nm)
- multilayer mirror (7nm)
- bent crystals

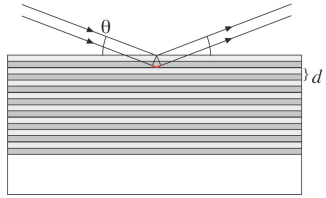
refraction:

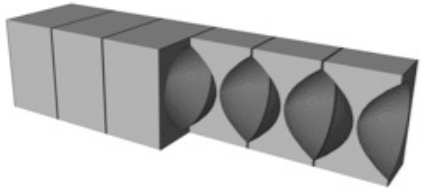
- refractive lenses (43nm)







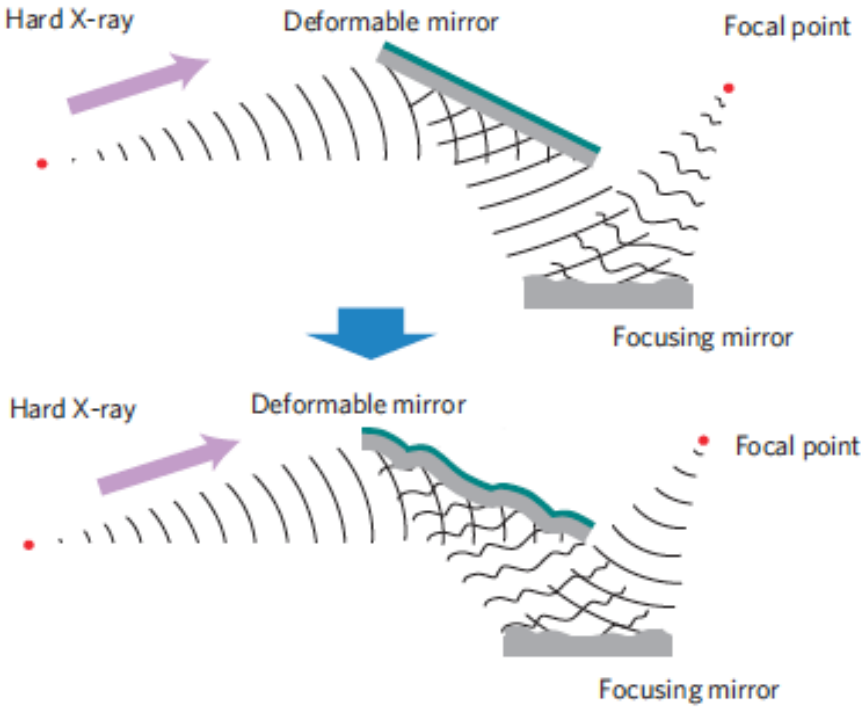


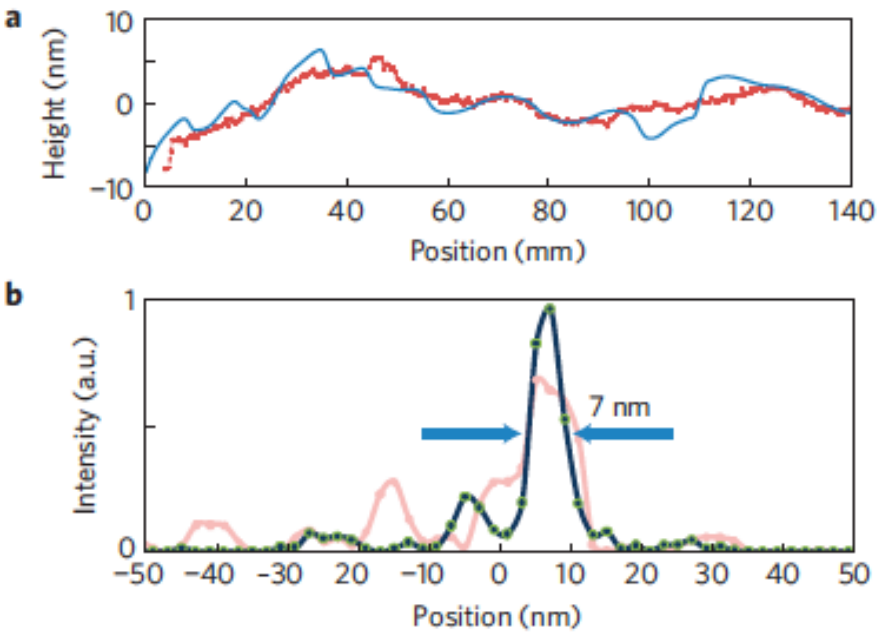


Institut für Strukturphysik, TU Dresden, Christian Schroer (schroer@xray-lens.de)

Breaking the 10 nm barrier in hard-X-ray focusing

Hidekazu Mimura[1]*, Soichiro Handa[1], Takashi Kimura[1], Hirokatsu Yumoto[2], Daisuke Yamakawa[1], Hikaru Yokoyama[1], Satoshi Matsuyama[1], Kouji Inagaki[1], Kazuya Yamamura[3], Yasuhisa Sano[1], Kenji Tamasaku[4], Yoshinori Nishino[4], Makina Yabashi[4], Tetsuya Ishikawa[4] and Kazuto Yamauchi[1,3]



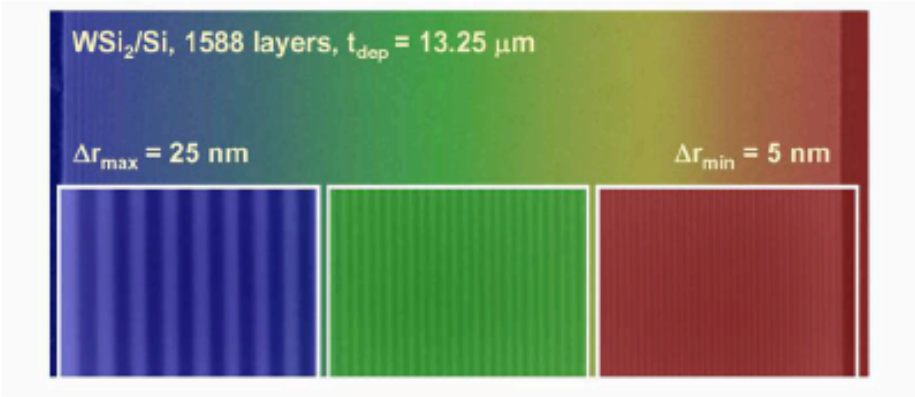


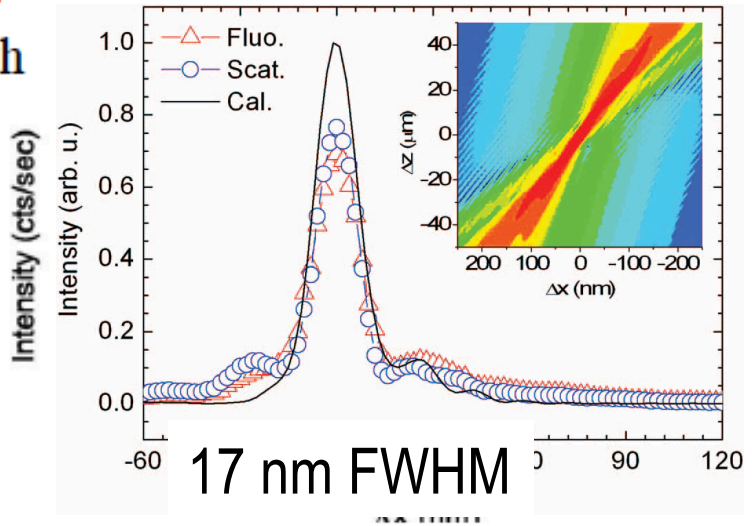
SPring-8

Nature Physics, **6**,122 (2009)

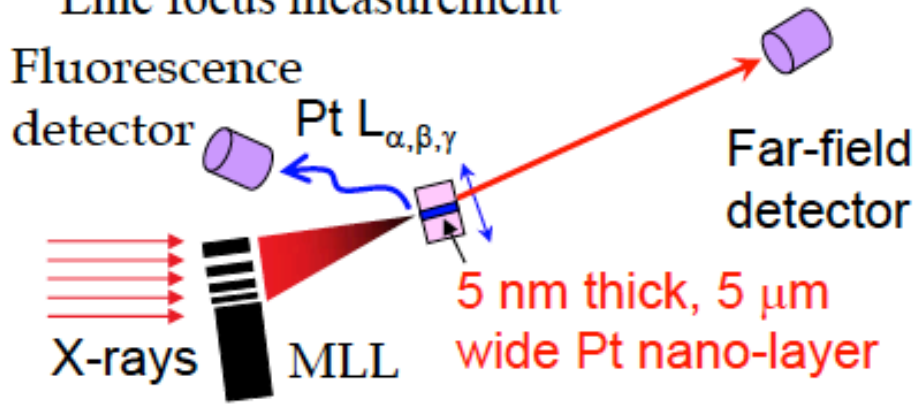
Experimental Achievement at APS

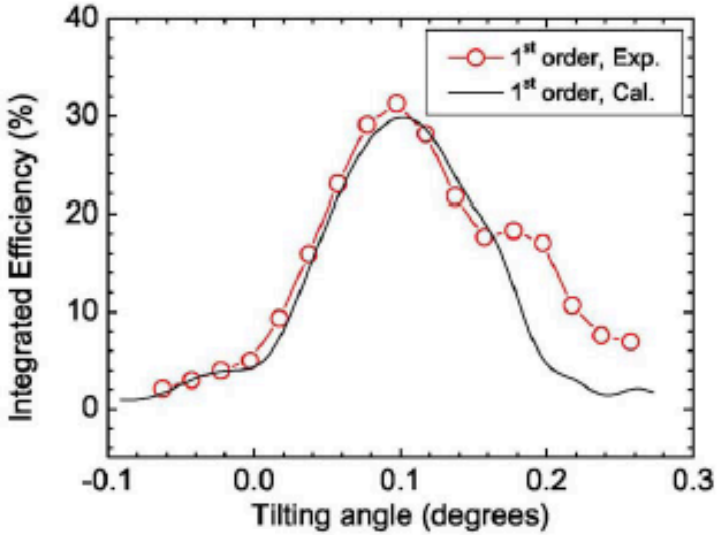
- 40% of full structure, 5 nm outmost zone width





- Line focus measurement

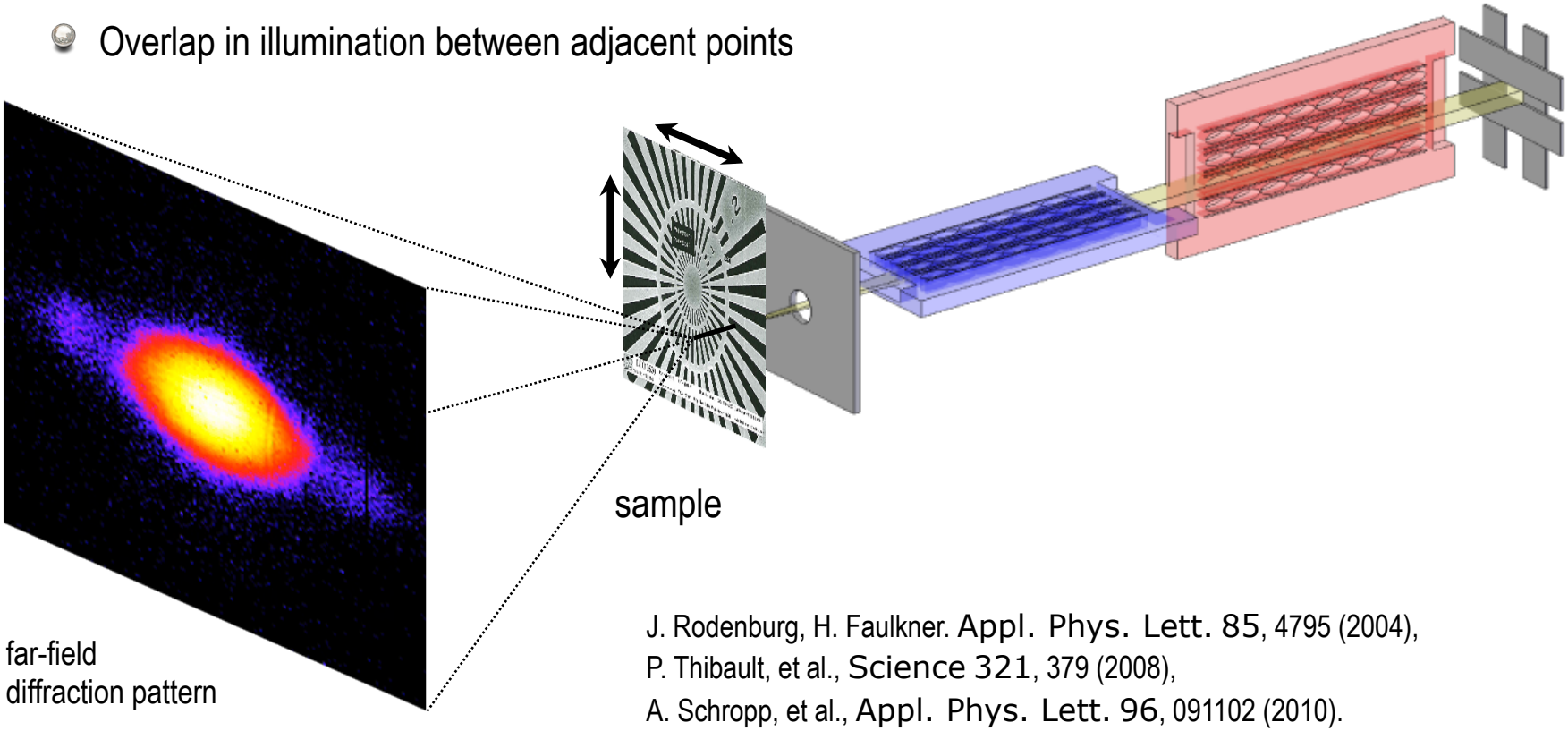




Kang et al., Appl. Phys. Lett., **92**, 221114 (2008)

Scanning Coherent X-Ray Diffraction Imaging: Ptychography

- Sample is raster scanned through confined beam
- At each position of scan: diffraction pattern is recorded
- Overlap in illumination between adjacent points



J. Rodenburg, H. Faulkner. Appl. Phys. Lett. 85, 4795 (2004),
P. Thibault, et al., Science 321, 379 (2008),
A. Schropp, et al., Appl. Phys. Lett. 96, 091102 (2010).

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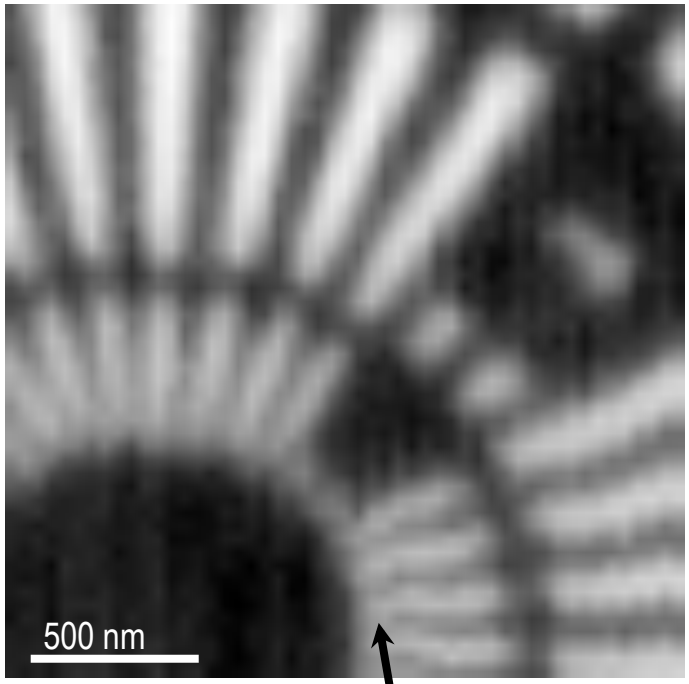
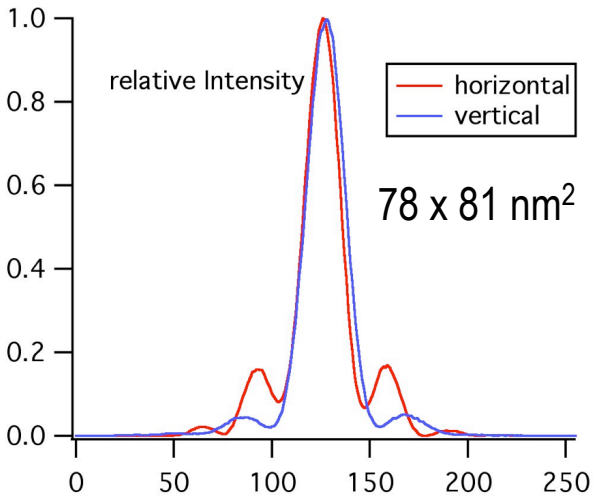
Scanning Microscopy: Fluorescence Imaging

Ta Kα fluorescence



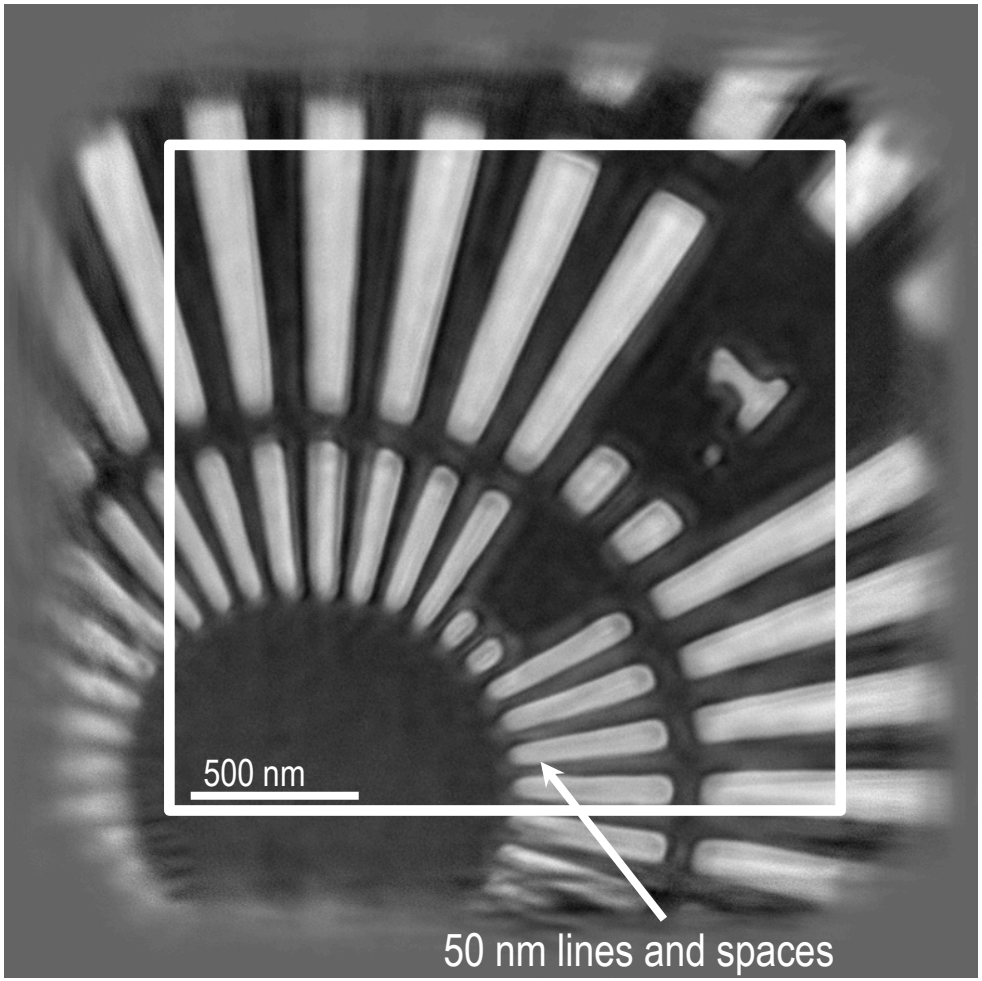
50 nm lines and spaces

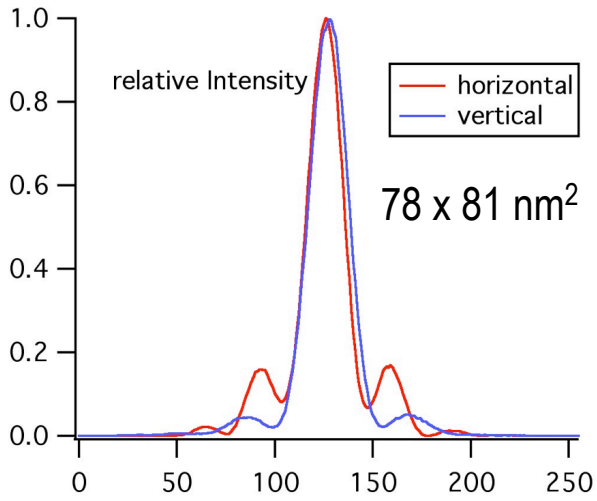


E = 15.25 keV
50 x 50 steps of 40 x 40 nm^2
2 x 2 μm^2 FOV
exposure: 1.5 s per point

Institut für Strukturphysik, TU Dresden, Christian Schroer (schroer@xray-lens.de)

Scanning Microscopy: Ptychography



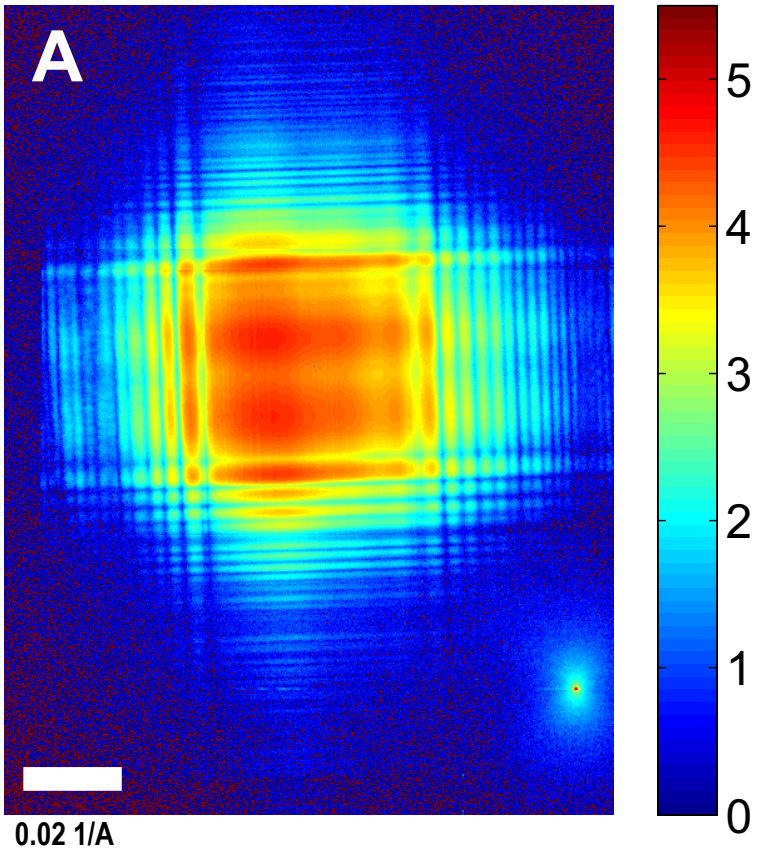


E = 15.25 keV
50 x 50 steps of 40 x 40 nm^2
2 x 2 μm^2 FOV
exposure: 1.5 s per point
dose density: 10^5 ph/nm^2

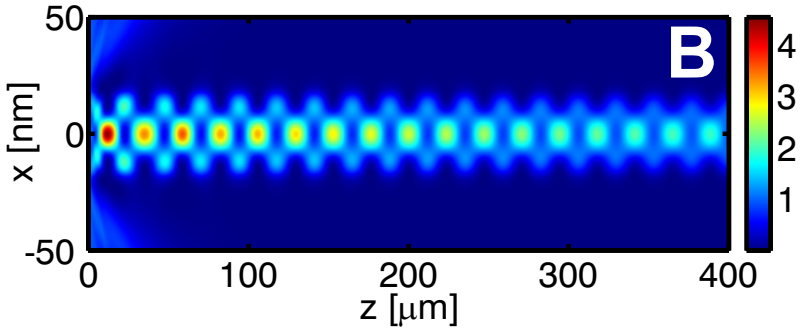
resolution: < 10 nm

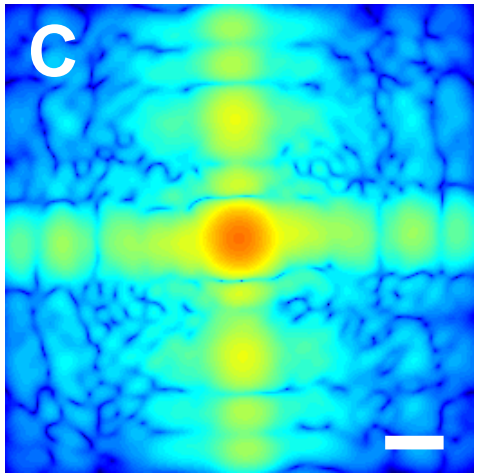
Institut für Strukturphysik, TU Dresden, Christian Schroer (schroer@xray-lens.de)

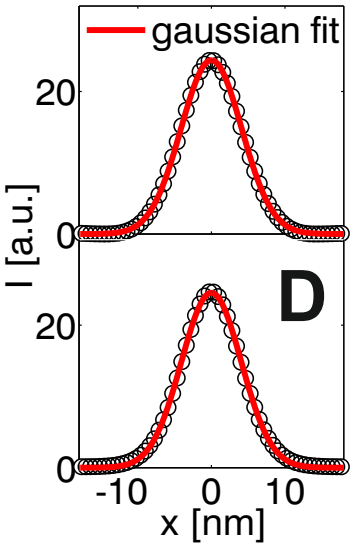
PETRA III: towards extremely small photon beams



Frauenhofer diffraction in 5m distance







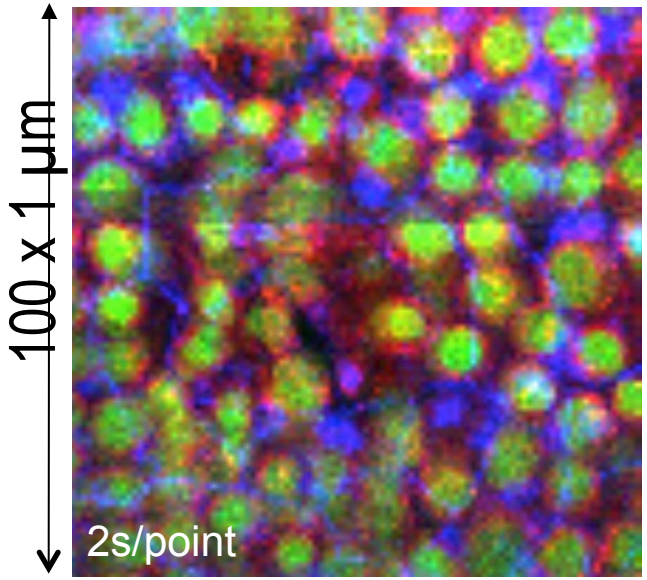
Reconstructed focus after the crossed waveguides:

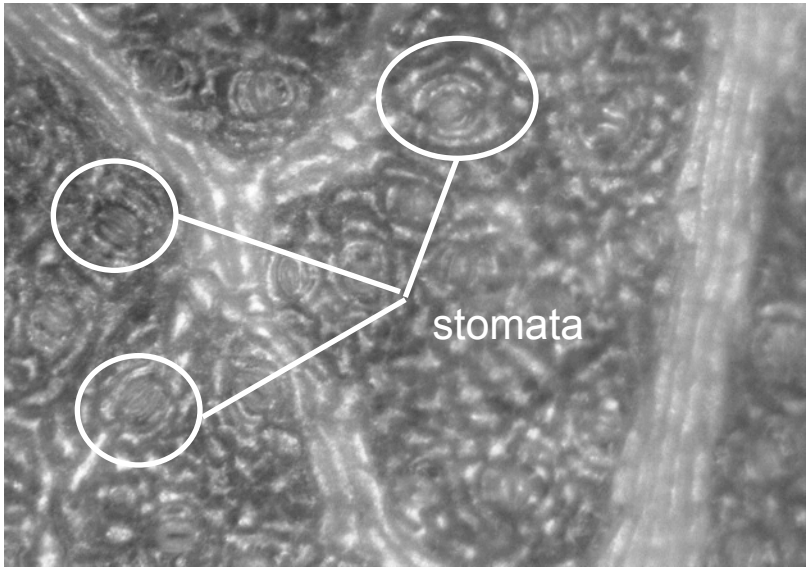
➔ 9.7 x 9.5 nm² spot size achieved at 15 keV

➔ **Spatially resolved studies in the 10nm range with hard X-rays will be possible**

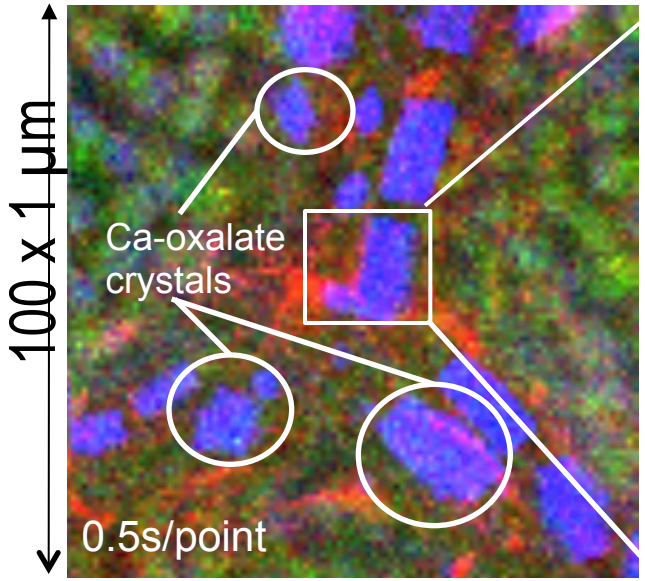
Team of University Göttingen:
S.P. Krüger, M. Bartels, R. Wilke, S. Kalbfleisch, M. Priebe, M. Osterhoff, C. Olendrowitz, T. Salditt
HASYLAB: M. Sprung

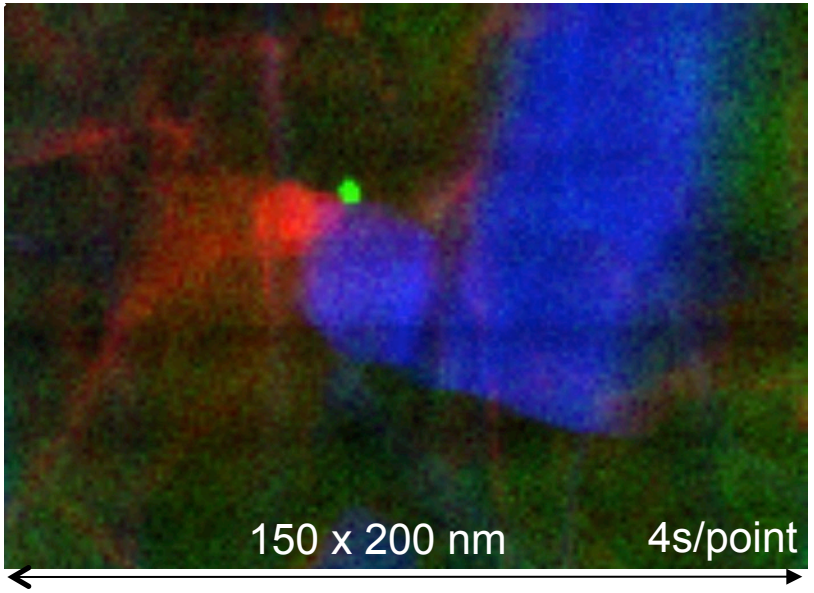
PETRA III: Nano-XRF on Poplar leaves

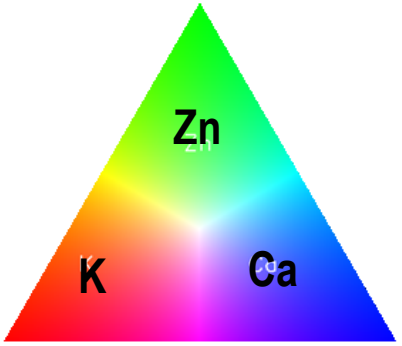




Investigation of ion transport in stomata from poplar leaves



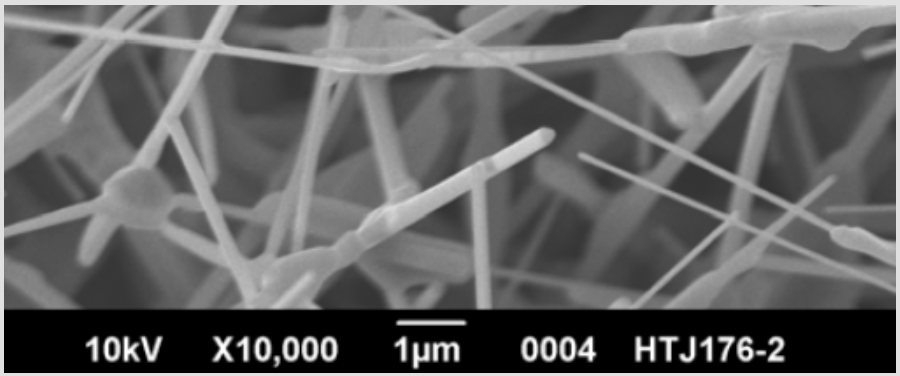


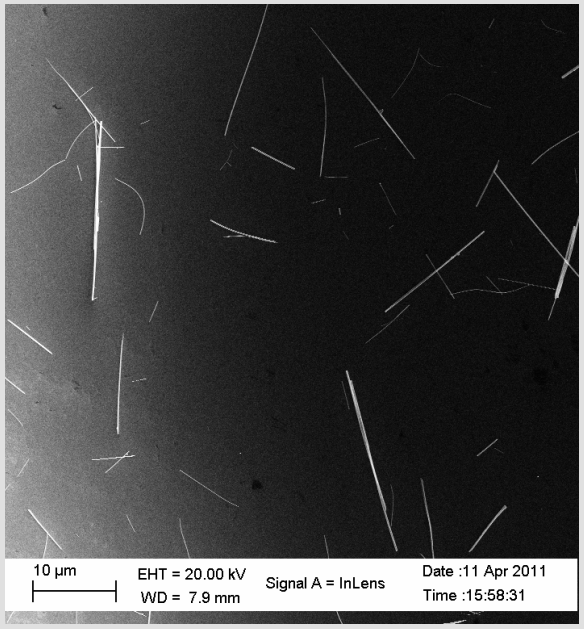


Collaboration Walter Schroeder(FZJ)

Science at the ESRF

XRF *nano*-imaging of Co-implanted ZnO *nano*-wires (NWs)





Small dimension structures:
- parallel optical guiding
- electrical contacting

Large surface to volume ratio:
- bio and chemical sensors

Anisotropic geometry:
- orientation dependent response

(spintronics, etc)

Open issues:

- **Trace elements in the *as-grown* NWs**
- **Co distribution in the Co-implanted ZnO NWs**
- **Co ions charge state (Co^{2+} state ?)**

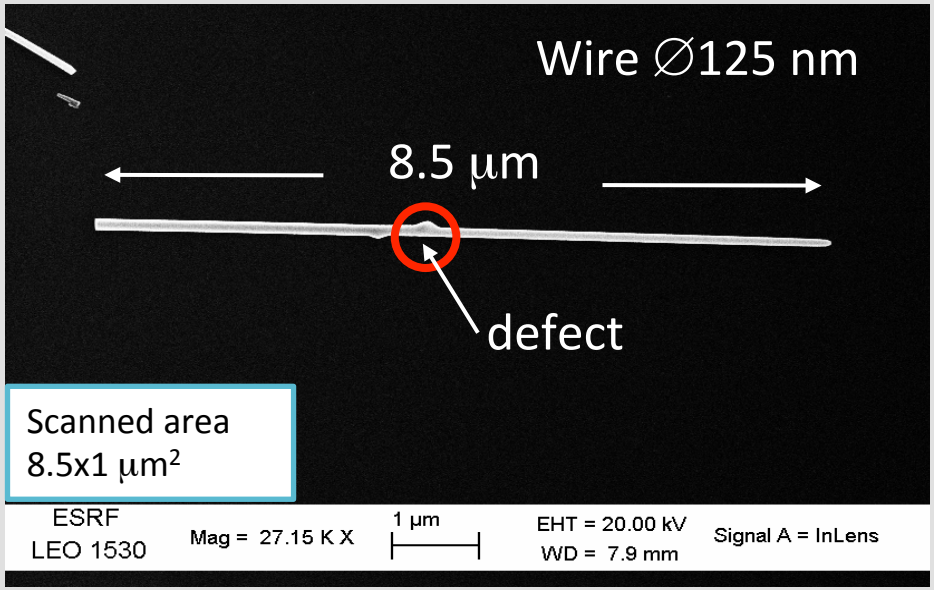
Samples: **CVD growth using VLS mechanism, dispersed on p-Si (100) substrates**

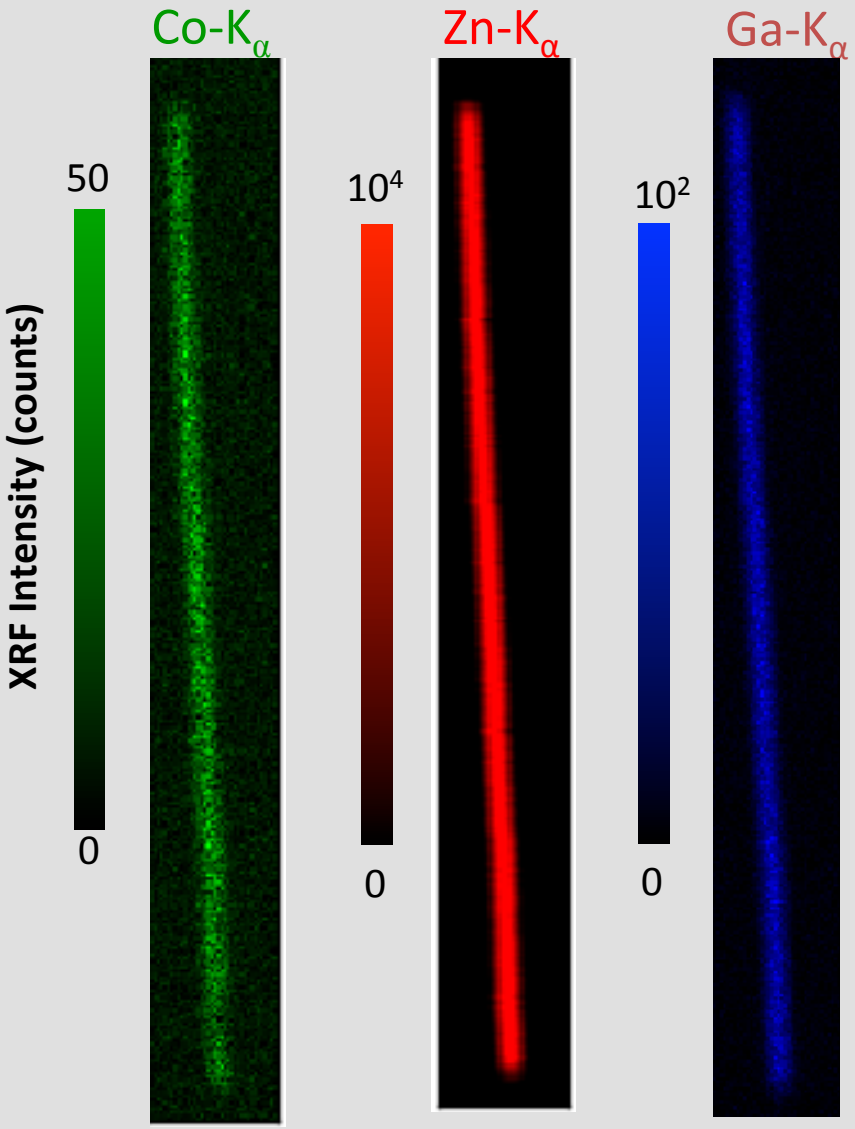
Science at the ESRF

XRF *nano*-imaging of Co-implanted ZnO *nano*-wires (NWs)

The *nano*-imaging station ID22NI: A flavor of NINA

- Beam energy: 17keV
- Beam size: 50 x 50 nm^2
- Acquisition time: 0.3 sec/point
- Step size: 25 nm

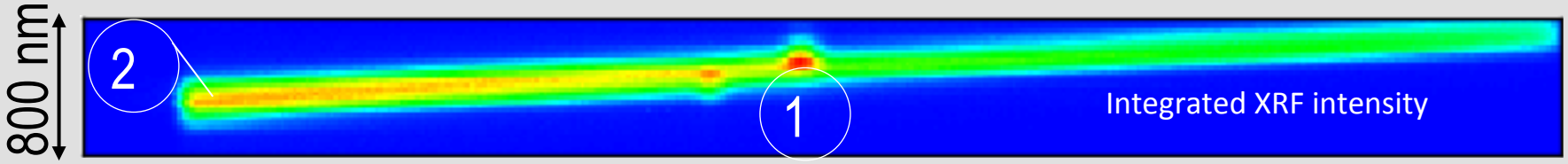


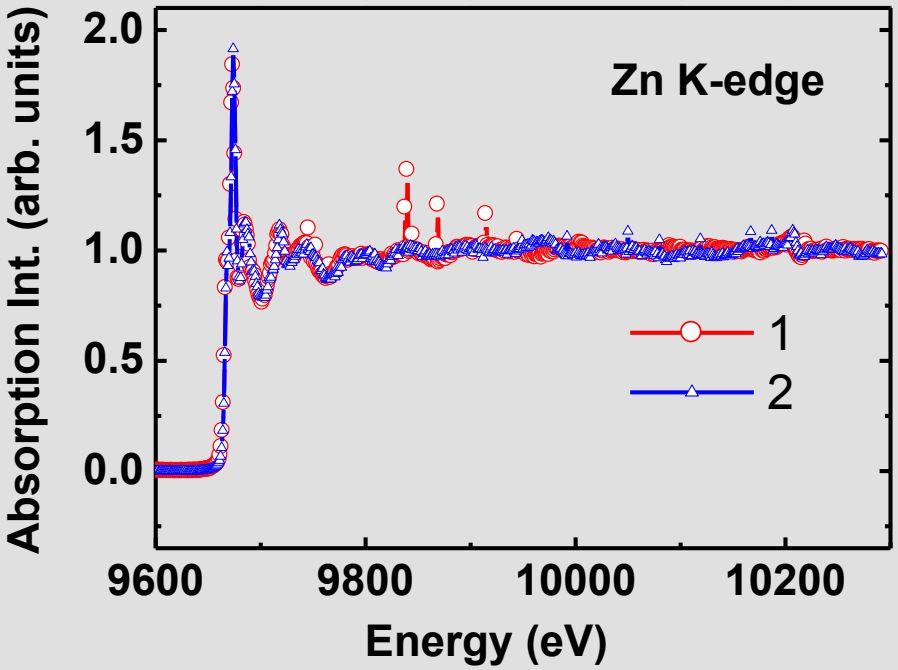


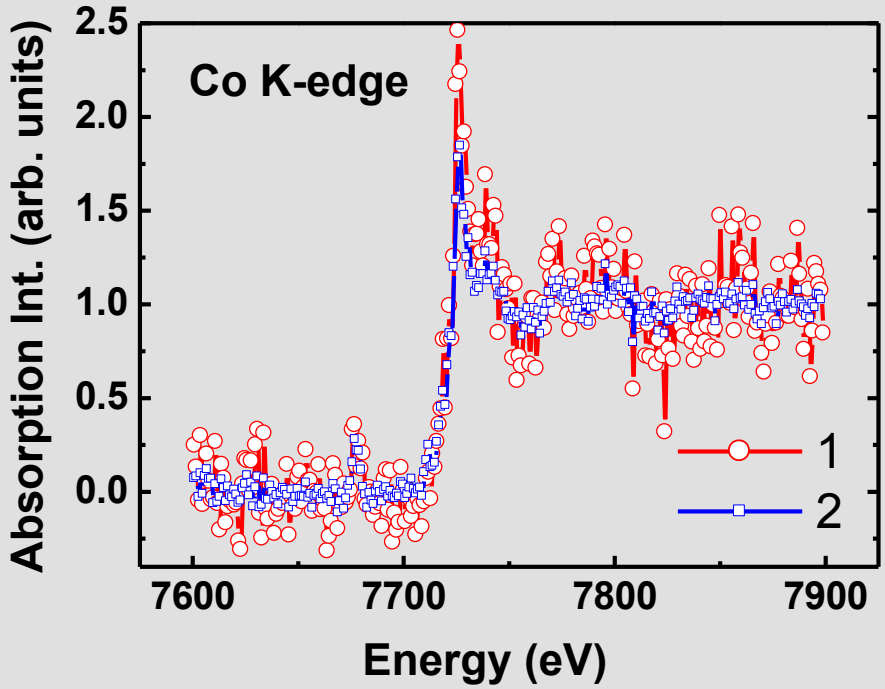
Science at the ESRF

XRF *nano*-imaging of Co-implanted ZnO nanowires (NWs)

nano-XAS on an individual NW







Beam size: 100 x 100 nm^2
Acquisition time: 0.5 sec/point
Step size: 1 eV

Co ions substitutional in Zn sites of wurtzite ZnO

Coherent Diffractive Imaging

coherent diffractive imaging in standard user operation

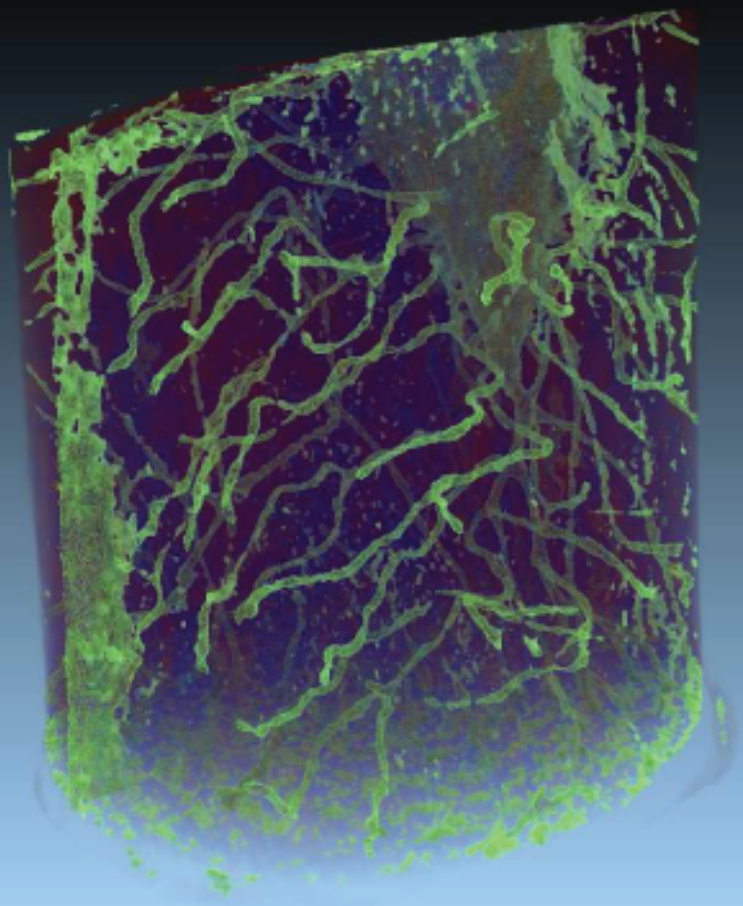
- Sample: bone,
  M. Dierolf *et al.*, *Nature* **467** (2010) 436

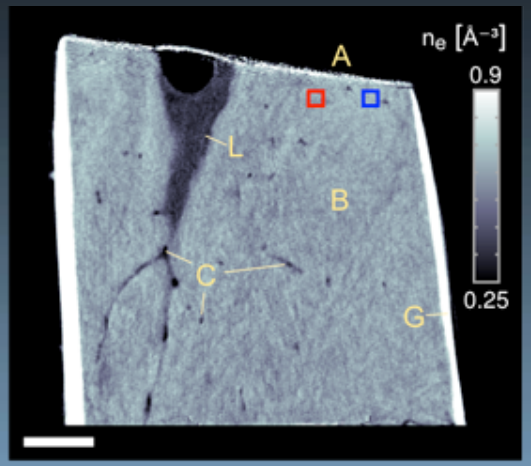
highly resolving

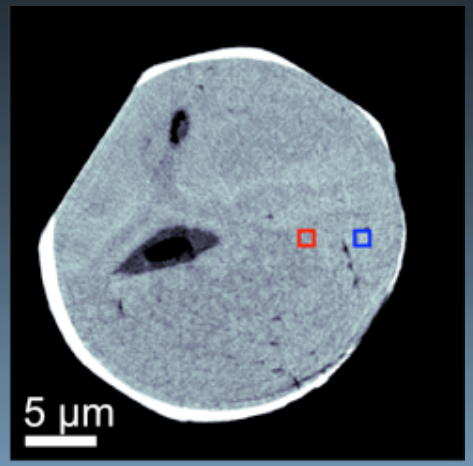
- voxel size $(65\text{nm})^3$
- resolution in 3D ~100nm
  in 2D ~ 20nm

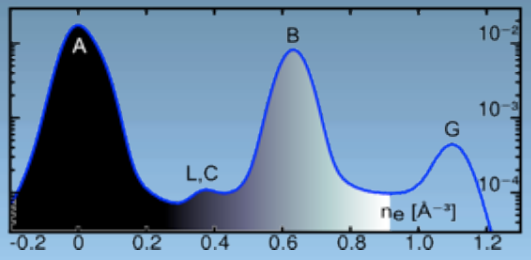
quantitative results

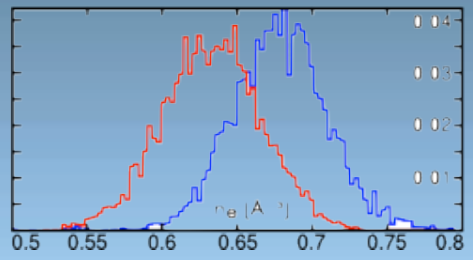
- uncertainty within voxel is 0.04 $e^-/Å^{-3}$
- significantly higher sensitivity for larger volumes, e.g., <0.002 $e^-/Å^{-3}$ for 1µm³











NSRRC is constructing the Taiwan Photon Source

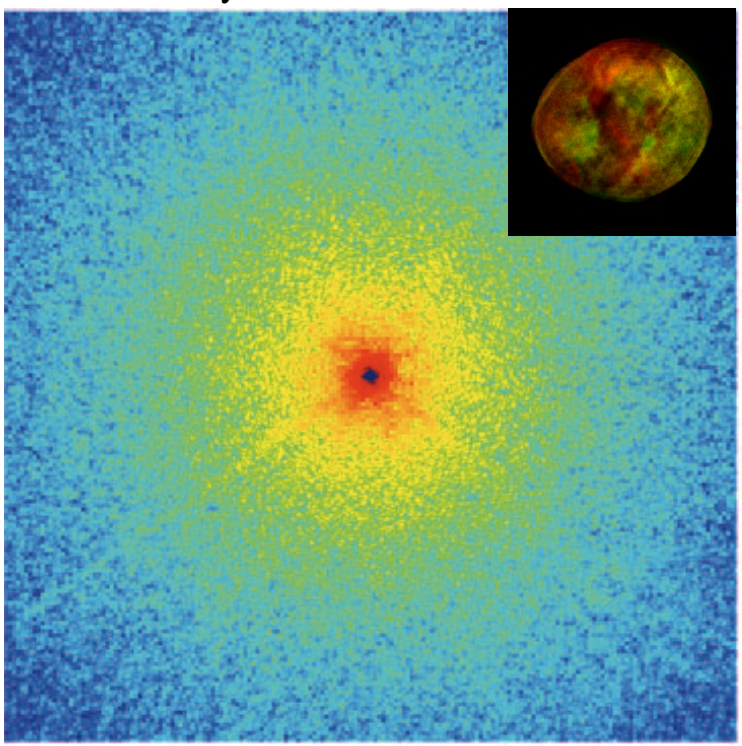
to be completed in 2013

ALBA a 3rd generation SL source with Storage Ring parameters:

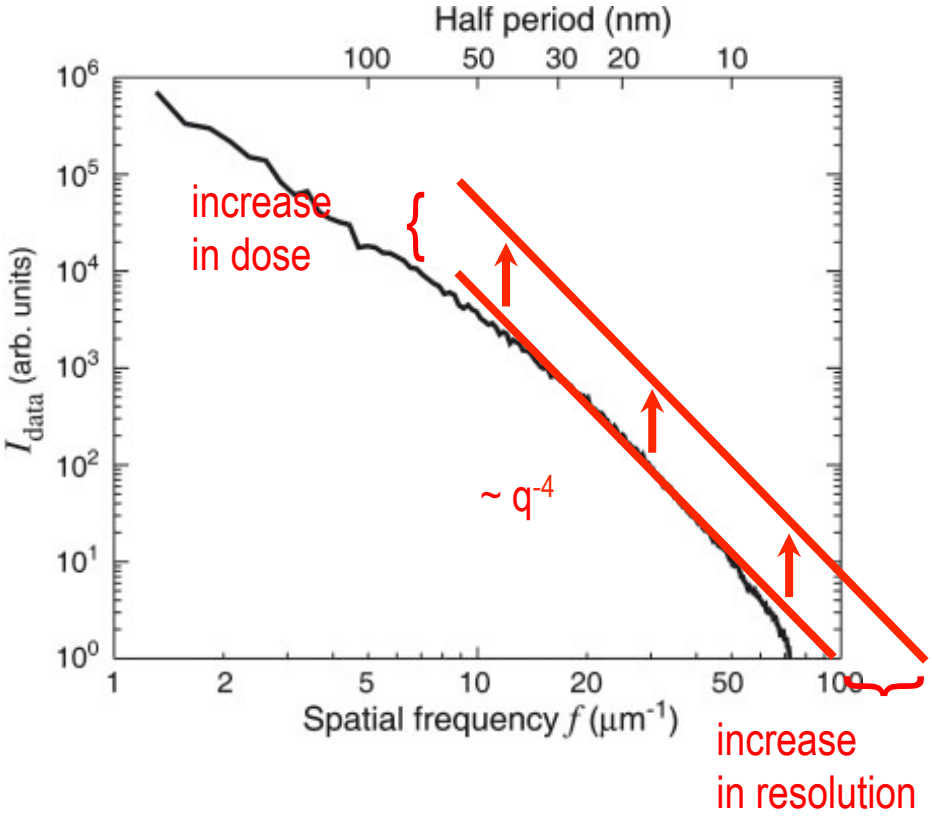
Emittance of 4.3 nm.rad; E= 3.0 GeV; I = 400 mA; C = 268.8 m; 4 straight sections (SS) of ca. 8 m; 12 SS of ca. 4.3 m; 8 SS of ca 2.58 m, and; 32 BM. Capacity for 33 beam-lines, currently 7 are funded (4 are X-ray beam-lines).

Resolution Limit of Coherent Imaging Techniques

Diffraction pattern of
freeze dried yeast cell:

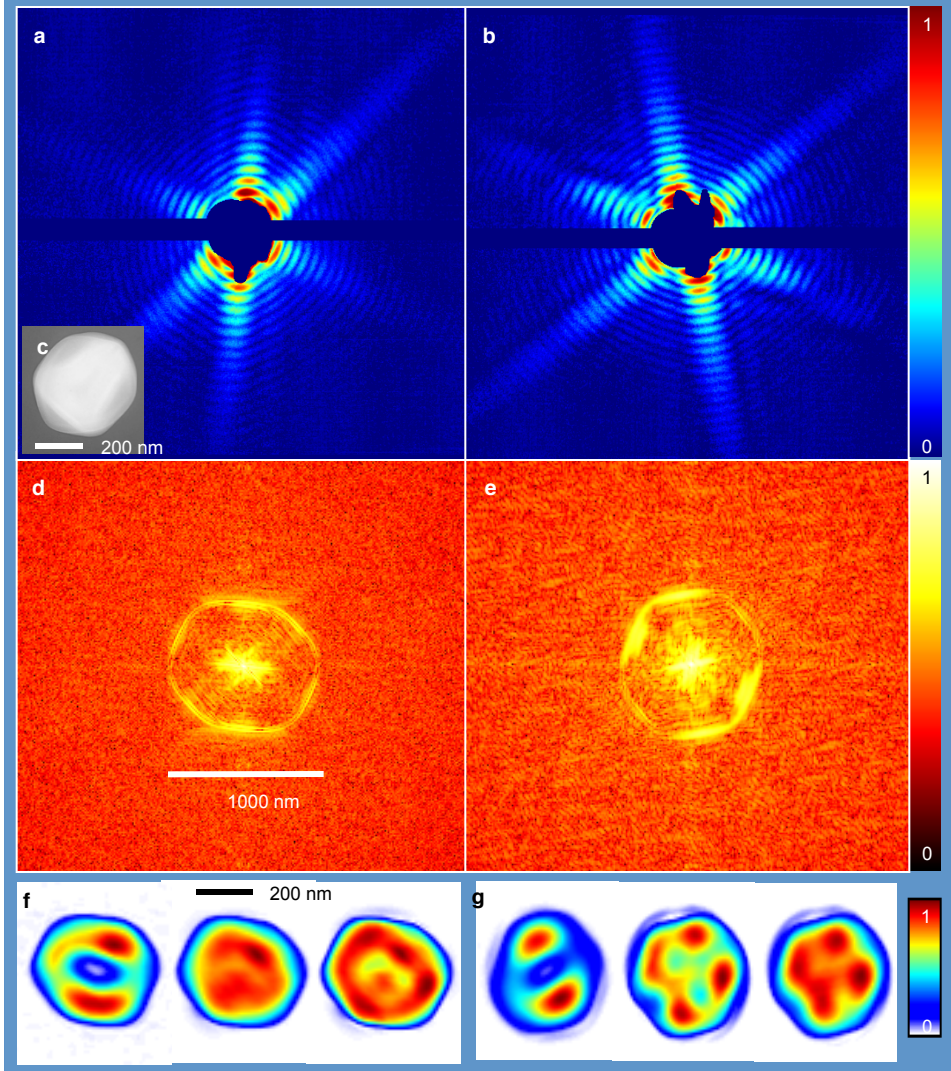


D. Shapiro, et al., PNAS 102, 15343 (2005)



Single mimivirus particles intercepted and imaged with an X-ray laser

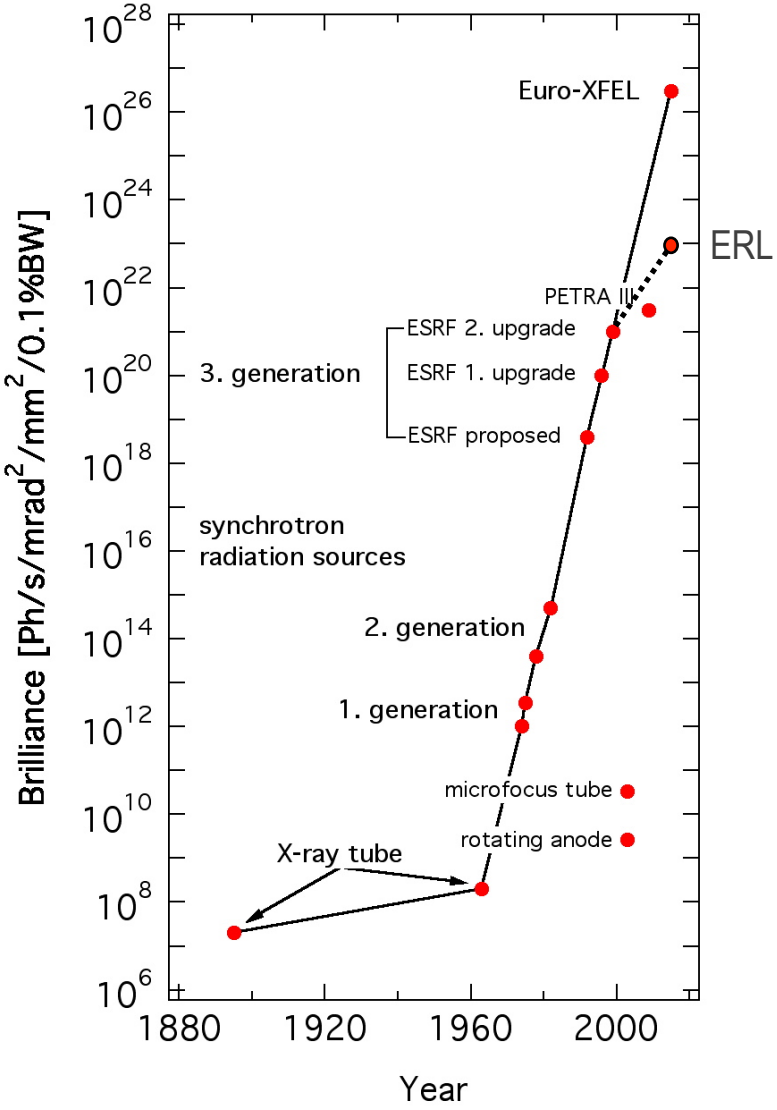
Beyond crystallography: A new world in structural sciences



- A very short and extremely bright coherent X-ray pulse can be used to outrun key damage processes and obtain a single diffraction pattern from a large macromolecule, a virus, or a cell *without the need for crystalline periodicity.*
- Mimivirus is the largest known virus, *comparable in size to a small living cell*. It is too big for structure determination by electron microscopy and it cannot be crystallised.
- The structure of the intact virus was recovered from the flash diffraction pattern alone.
- There was no measurable sample deterioration.
- Death-rays: We expect high-resolution structures in such experiments with shorter and brighter photon pulses focused to a smaller area.
- Resolution can be further extended by averaging for samples available in multiple identical copies.

M.M. Seibert, T. Ekeberg, F.R.N.C Maia et al., *Nature* **470**, 78–81 (2011)

High brilliance: High flux per phase space volume



Ideal for nanobeams

Small Source:
small geometric image
(diffraction limited focusing)

Small Divergence:
optic captures large fraction of
emitted radiation

X-ray FEL Parameters – Now and Future

(C. Pellegrini et al., summary of FEL workshops.)

Parameter	Now	Future
Photon energy, keV	Up to 10	**Up to 100**
Pulse repetition rate, Hz	≤ 120	10^2-**10^6**
Pulse duration, fs	~2-300	**<1-1000**
Coherence, transverse	diffraction limited	diffraction limited
Coherence, longitudinal	not transform limited	**transform limited**
Coherent photons/pulse	2×10^{12}-3×10^{13}	**10^9-10^{14}**
Peak brightness, ph/s mm^2 mrad^2 0.1% bandwidth	10^{33}	10^{30}-**10^{34}**
Average Brightness, ph/s mm^2 mrad^2 0.1% bandwidth	4×10^{22}	10^{18}-**10^{27}**
Polarization	linear	**variable, linear to circular**

red: parameter space to be developed

Summary

- Accelerator based x-ray sources have revolutionized protein crystallography: smaller and smaller crystals
- Real space 'imaging' limited by x-ray optics: approaching 10 nm
- Limits to diffractive imaging of single objects: dose x10 in resolution requires x10,000 in dose
- **New sources bring new science and they require new x-ray technologies: Optics, detectors, analysis…**

SLAC