ESRF

Next Generation X-ray Analyses

and the ESRF Upgrade Programme

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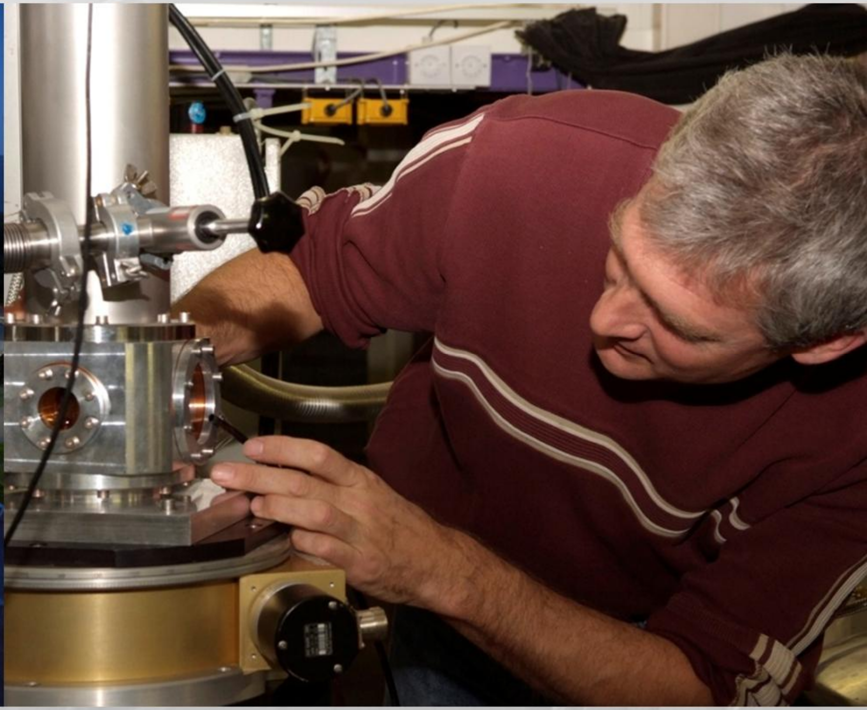
- ✓ **Science at brighter X-ray sources**
- ✓ **Concluding remarks**

Major synchrotrons in the world

ESRF is the world's first 3rd generation hard X-ray source

Upgrades of existing sources and future storage rings for new synchrotron science (ESRF, PETRA III, APS, Spring 8, …)

New, better science

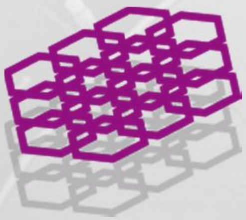


ESRF Upgrade Programme: X-ray *nano*-beams for science

Objective: a new generation of instruments for *frontier and applied science* in condensed matter, materials, and living matter.

New beam lines and a brighter, and more coherent and stable source

Advanced materials



Health & life sciences

Energy research

- Science and technology at the atomic scale
- Biology and time-resolved science
- Soft matter and imaging of biological samples
- Materials and chemistry
- Earth environment and extreme condition science

ESRF UP Phase II

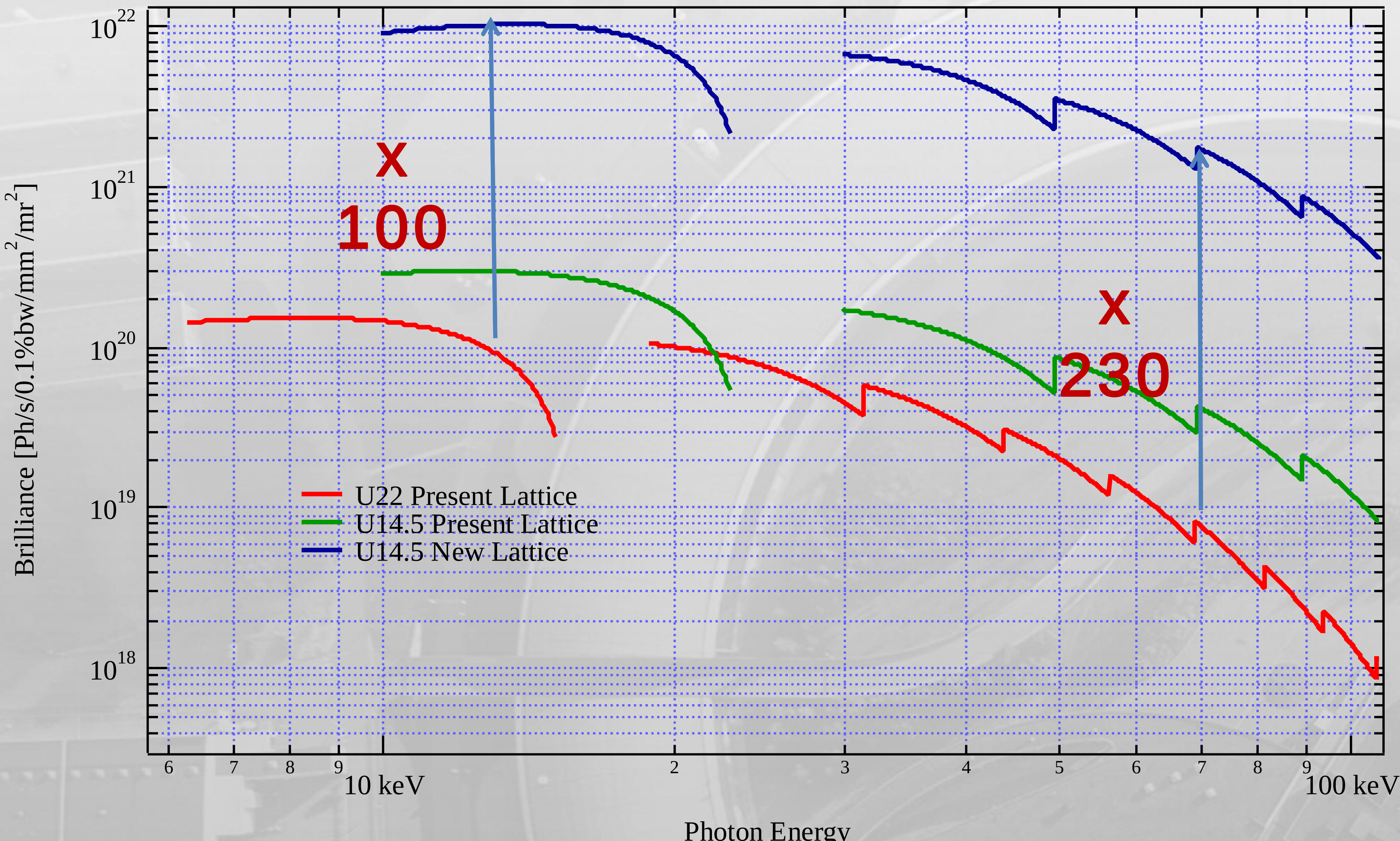
A NEW LOW-HORIZONTAL-EMITTANCE LATTICE
from 4 *nm* to ~0.1 *nm*

- Increased brightness and coherent fraction *(x30 ++ on IDs and x60 on BMs)*
- Substantial reduction of the total power on beam line optics
- Power density increase by not more than a factor of ~2 (IDs) and ~6 (BMs)

2 m IVUs & CPMUs:

U22 Min. Gap 6 mm, K_{max}=1.7

U14.5 Min. Gap 4 mm, K_{max}=1.7 (CPMU)



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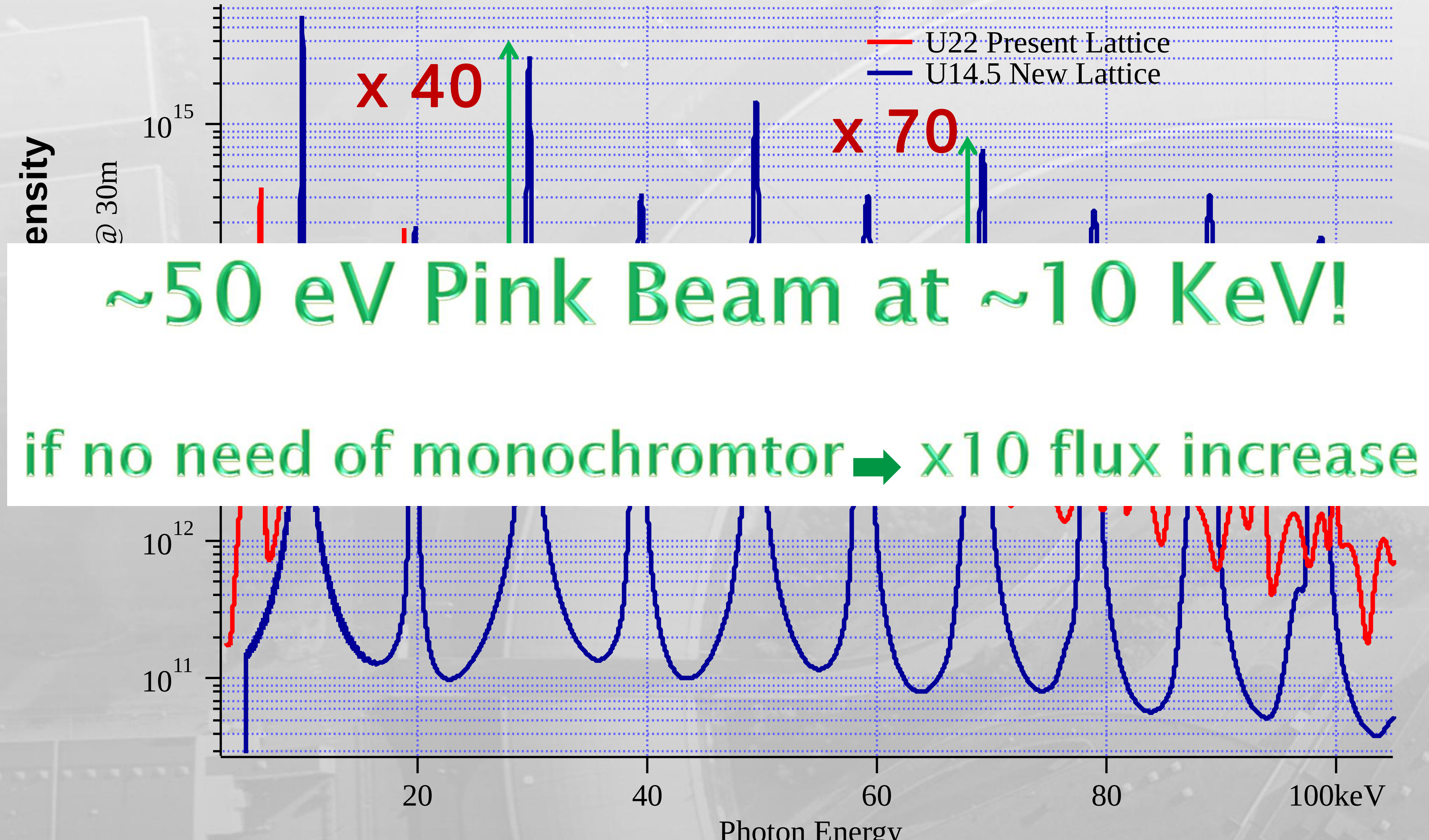


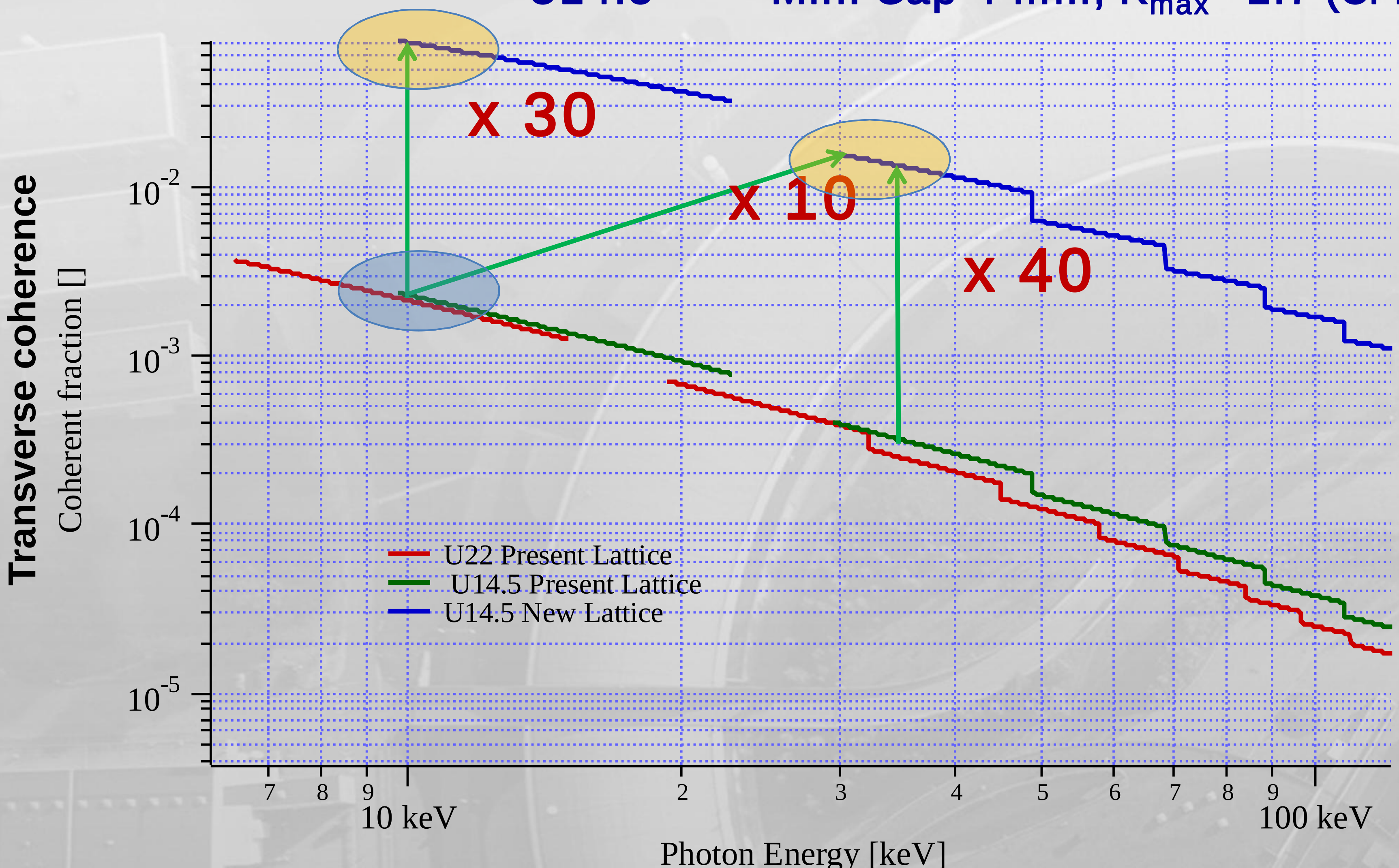
~50 eV Pink Beam at ~10 KeV!

if no need of monochromtor ➡ x10 flux increase

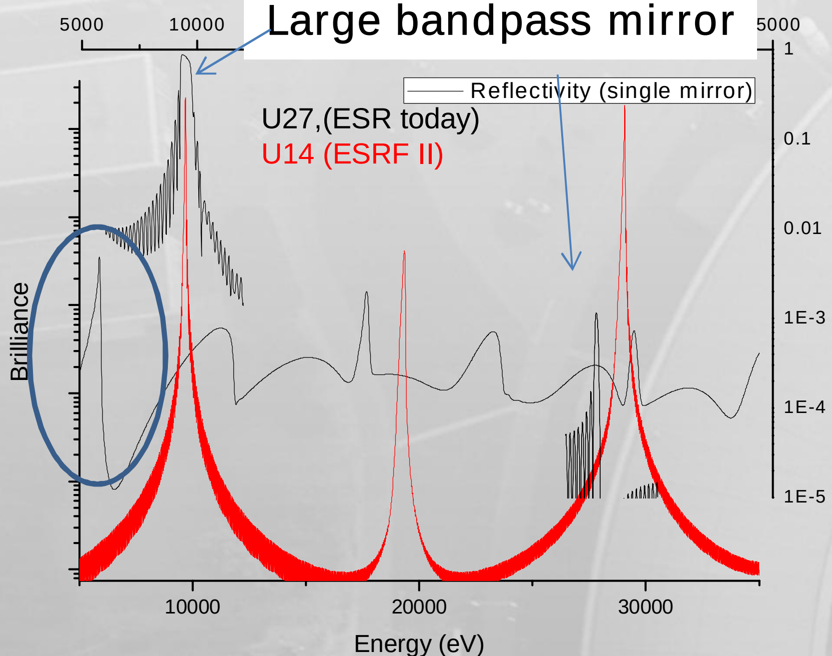
2 m IVUs & CPMUs:

U22 Min. Gap 6 mm, K_{max}=1.7

U14.5 Min. Gap 4 mm, K_{max}=1.7 (CPMU)

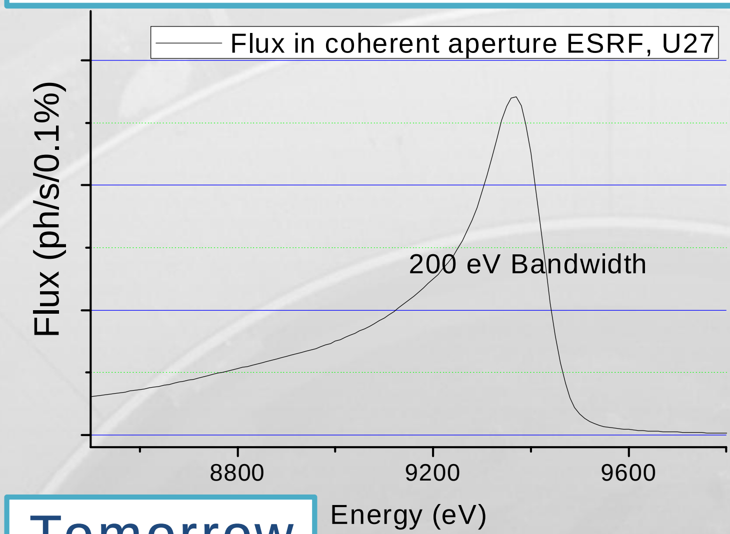


Coherent flux for short coherence lengths (nano particles!):

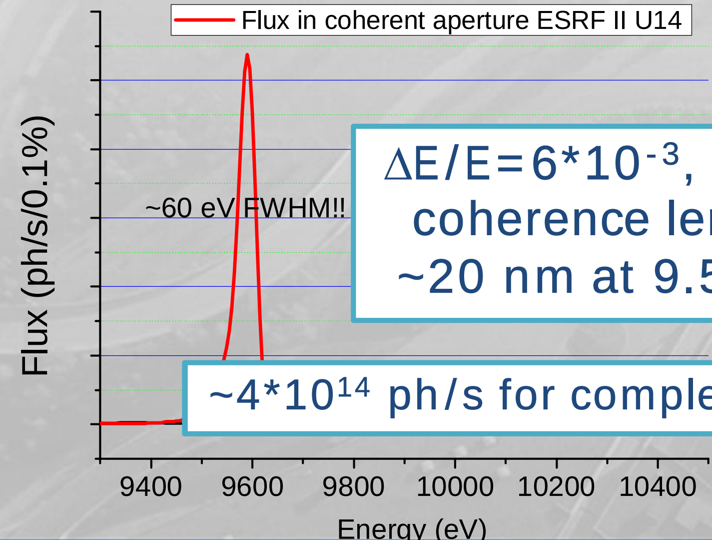


Pink beam (20 nm long. coh. length) coherent flux x2000 !!

Today's flux inside the coherent aperture at 9.5 keV:



Tomorrow



$\Delta E/E = 6*10^{-3}$, Long. coherence length ~20 nm at 9.5 keV

~4*10^{14} ph/s for complete line

ESRF Upgrade Programme

X-rays for science in the mesoscopic regime

beyond optical imaging – approaching e-microscopy

Advanced materials | Health & life sciences | Energy research

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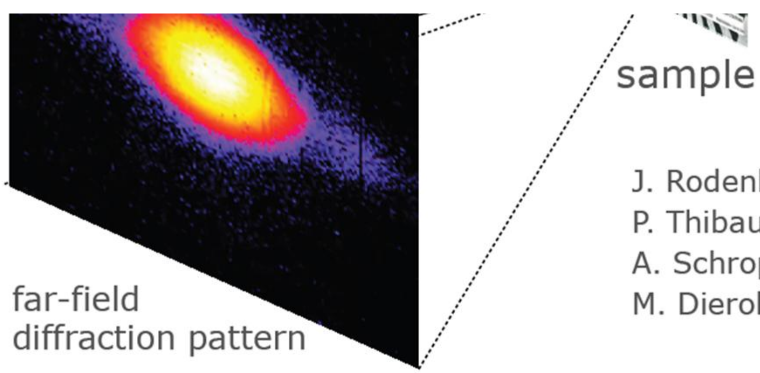
Scanning Coherent X-Ray Diffraction Imaging: Ptychography

- Sample is raster scanned through confined beam
- At each position of scan: diffraction pattern is recorded

This works also in full field!
Strongly reduced dose!!



limited by scattering power and fluence on sample.

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),
M. Dierolf, et al., *Nature* **467**, 436 (2010).

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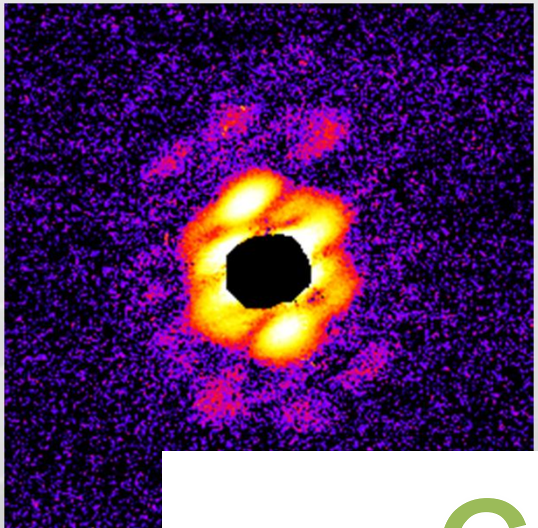
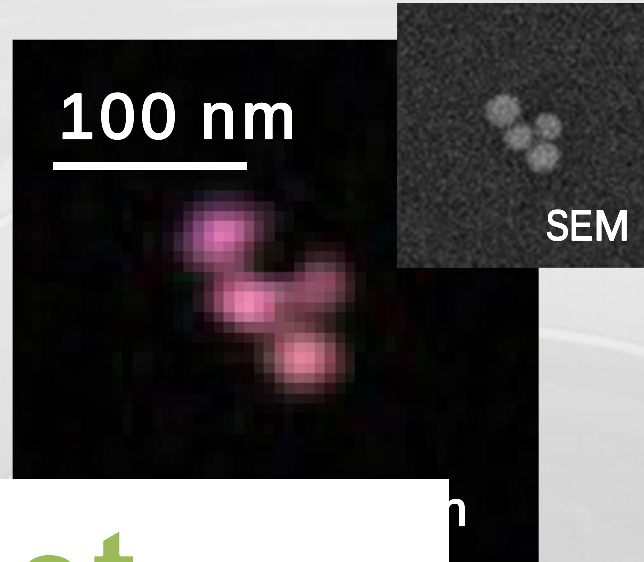
Science at brighter X-ray sources:

courtesy Christian Schroer

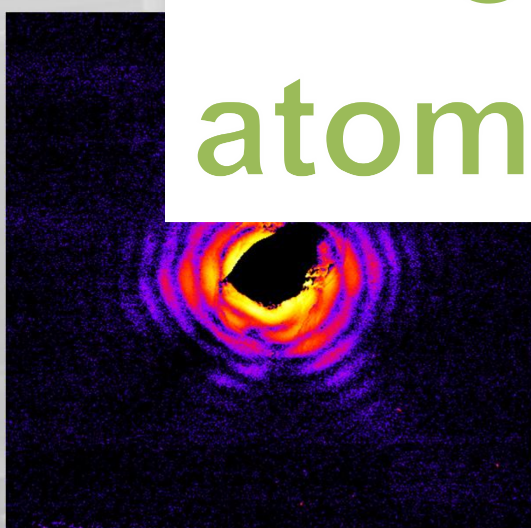


reconstruction
via

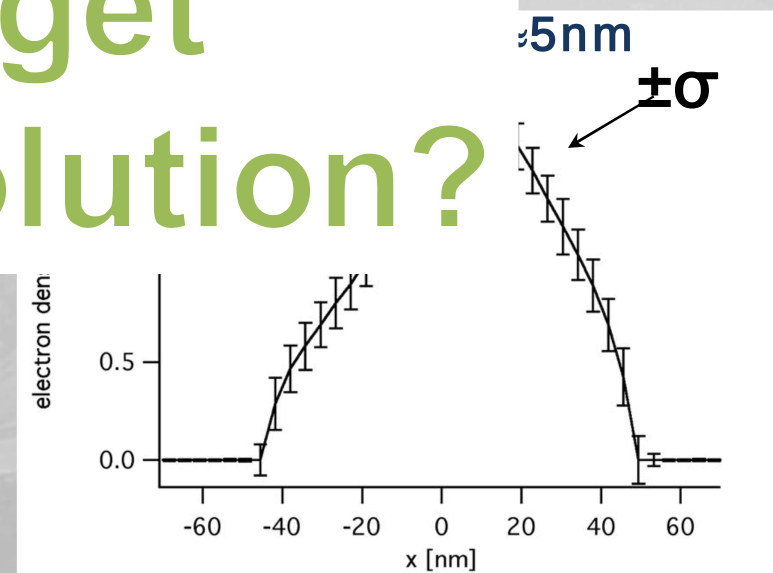
iterative
phase retrieval



Can we get atomic resolution?



reconstructed
projection
5 nm resolution



C. G. Schroer et al. Phys. Rev. Lett. 101, 090801 (2008)

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Route to Atomic Resolution...

Signal in diffraction pattern:

$$I_c \cdot t \cdot \frac{d\sigma}{d\Omega} \cdot \Delta\Omega_{\text{focus}} \propto \underbrace{Br}_{\text{source}} \cdot \frac{\Delta E}{E} \cdot \underbrace{NA^4 \cdot T}_{\text{optic}} \cdot t$$

speckle size:

$$\Delta\Omega_{\text{focus}} = \pi \frac{\lambda^2}{4d_t^2} = \pi NA^2$$

fluence:

$$I_c \cdot t \propto Br \cdot \frac{\Delta E}{E} \cdot NA^2 \cdot T \cdot t$$

Improvements (compared to current experiment):

- upgraded sources (DLSR):

  $Br \rightarrow \times 40 - \times 1000$ — $10 - 10^3$

- improve optic:

  $T \rightarrow \times(7 \cdot 100)$ — $10 - 10^3$

  efficiency (7), relaxing spatial filtering of source (100)

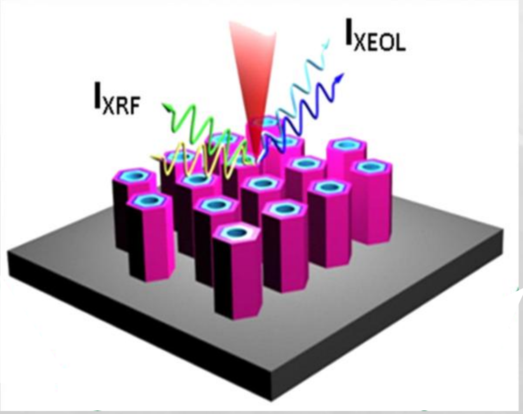
  $NA \rightarrow \times 10$ (10 nm focus) — 10^4

overall improvement: 10^{10}

Gordon X-Ray Conference 33

Isolated nano-scale objects are starting to be well characterized thanks to recent developments in *nano*-beams and coherent scattering techniques (*Ptychography*, *Coherent X-ray Diffraction Imaging (CXDI), etc.).*

The next big challenge is the development of new methodologies for characterizing *embedded nano*-scale objects within *multi*-scale structural hierarchies.

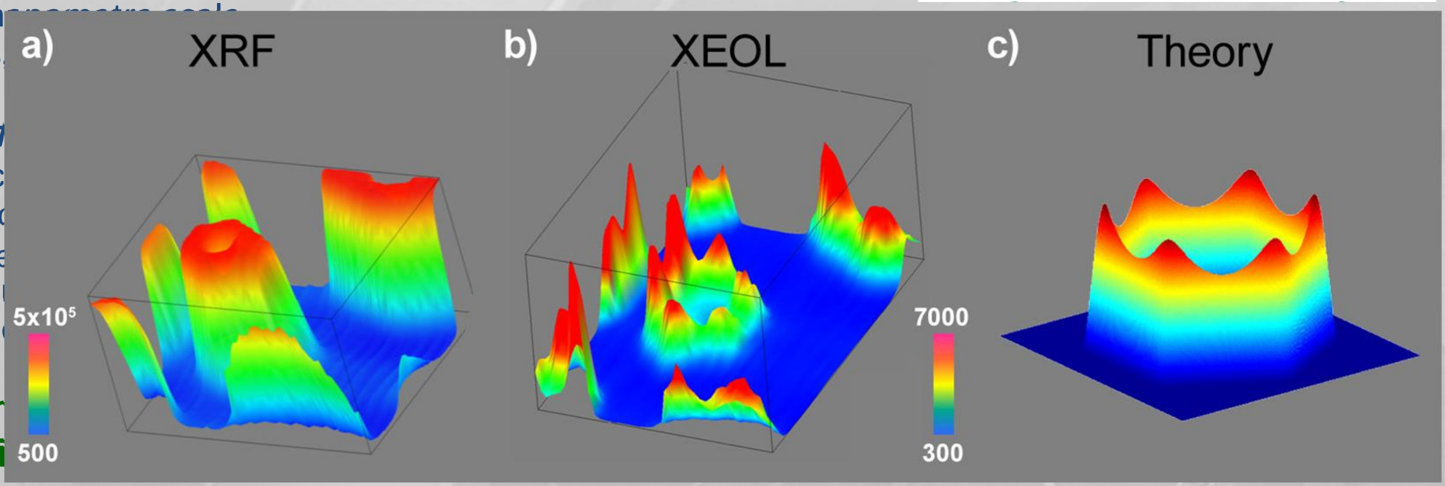


- ***Multi-scale materials modelling and nano-engineering in bulk materials:***
  Structured materials (metals, ceramics, building materials, rocks, ice, bones, ...) are hierarchically organized on several length scales, from mm to nm.
  Modelling, control and assembly representative at all scales simultaneously to understand and predict material properties, microstructure, processing ......

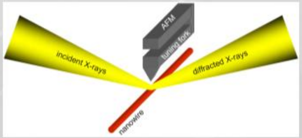
- ***Self-assembly of 3D systems:***
  Self-organization in bio-minerals, photonic crystals and colloidal crystals (to name a few), gives rise to 3D mesoscopic structures with hierarchical organization down to the n
  In situ conditions (solvents

- ***In-operando studies o***
  Localized phenomena (crac
  electrochemical cells, ...) g
  in a wide range of industrial se
  The ability to directly *see* s
  conditions could guide pro

**New tools such as the h
new exciting opportuni**

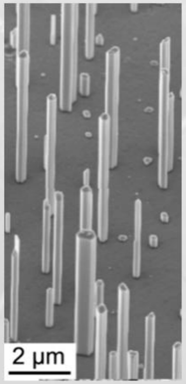


CXS from crystal defects



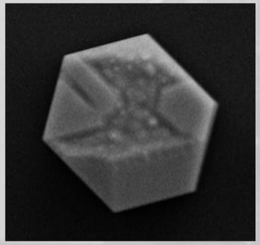
ANR
MECANIX

MECANIX ANR-11-BS10-01401

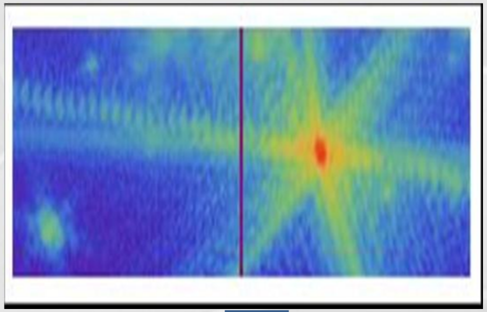


(001)

Al_2O_3 Substrate

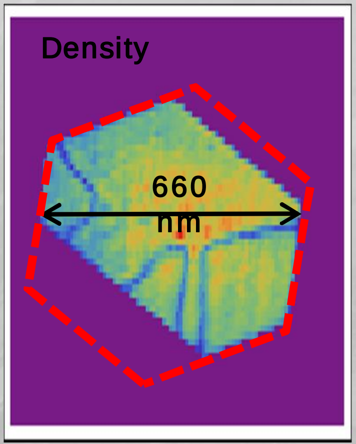


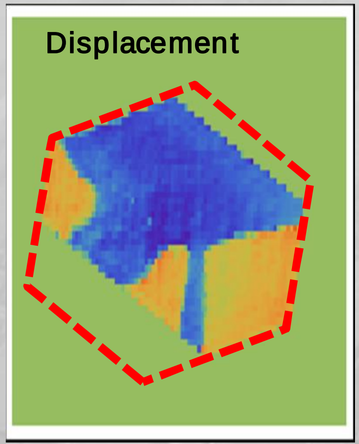
2D Diffraction Pattern



Phase retrieval algorithm

Pixel size : 16 nm X 8 nm





2 domains with a displacement shift of 0.56 Å

Structural Biology provides tremendous impact on Life Sciences, giving fundamental insights into how viruses work, cells synthesize proteins, and proteins generate energy.
This allows breakthrough applications in biotechnology and pharmaceutics.

Modern Biology aims to understand in an integrated way how cells and organisms live, interact, and react on each other and on a changing environment. This requires improved methodologies to obtain 3D structures of membrane proteins and their complexes with other proteins.

• ***The smaller the better***:
Small crystals are more perfect, and give better diffraction data sets. Data sets from sub-micron size crystals (if sufficient X-rays are available!).

• ***The more the better***:
Crystallography yield time- and space-averaged structures, and so conformational differences in proteins are often lost in crystals. “Perfect” tiny crystals help. An attractive strategy is to to study many micron-size crystals with only one mosaic block, and couple to many other “imaging” techniques (e-m, SAXS, MD, ... and Coherent imaging).

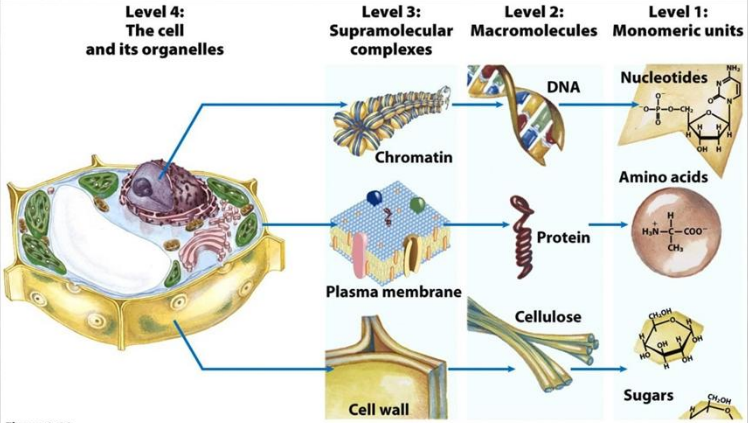
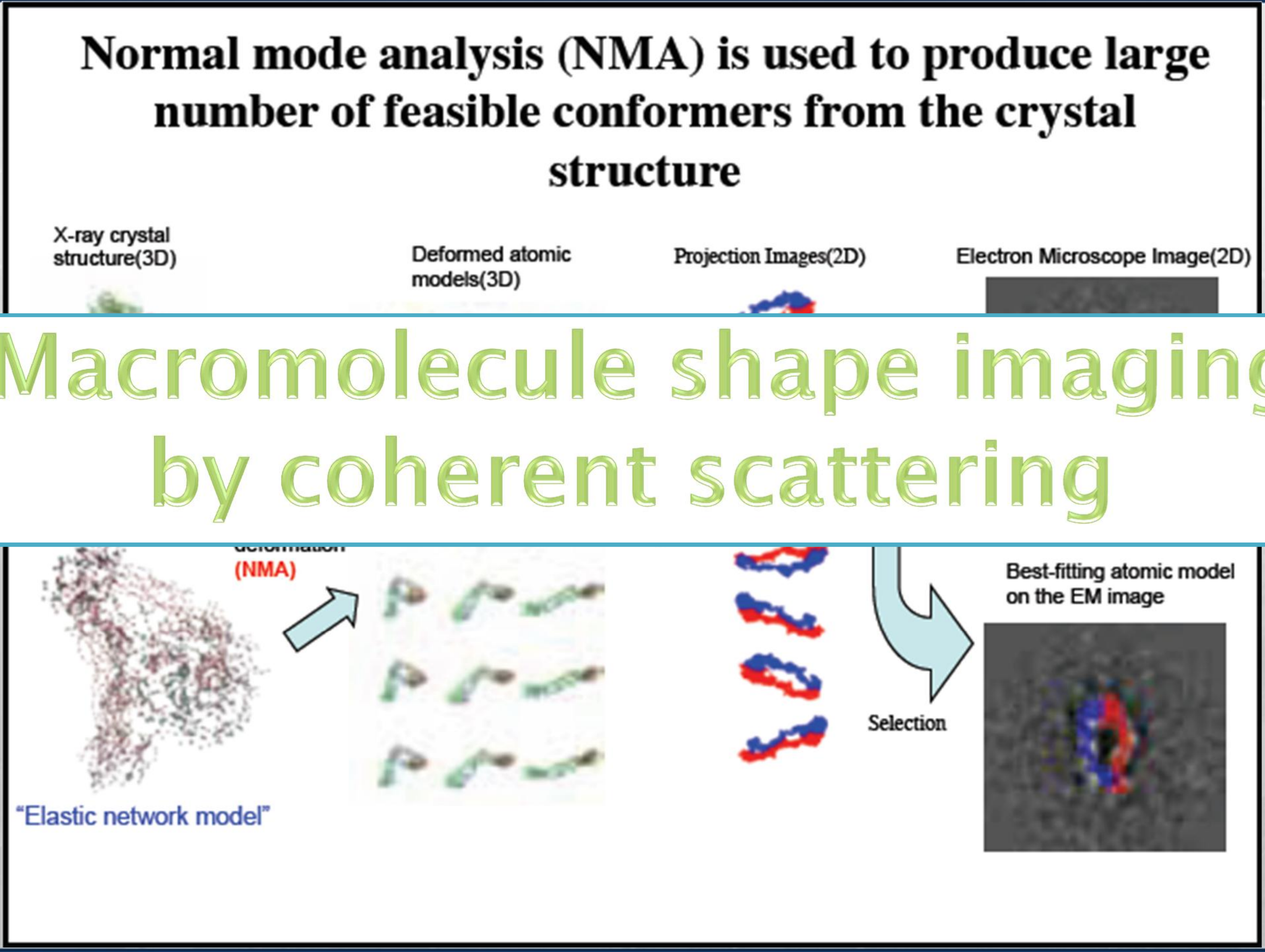


Figure 1-11
Lehninger Principles of Biochemistry, Fifth Edition
© 2008 W. H. Freeman and Company

• ***Time-resolved studies***:
Rapid protein dynamics can be studied by time resolved diffraction studies on fully reversible photo-inducible reactions in protein crystals. To study crystals not returning in their resting/initial configuration - in micron-sized protein crystals at a time-scale of micro-seconds – a flux density increase by 5 orders of magnitude (new source and tighter focus) is required.

All of the above-mentioned challenges can be drastically improved and often met with a new brighter X-ray source, new instrumentation and its automation, and new strategies for data collection, analysis and storage.



Macromolecule shape imaging by coherent scattering

Fast measurements, improved penetration, higher coherent flux

- ***From functional Soft Matter to Biology*:**
Soft matter systems exhibit many interacting degrees of freedom leading to structural organization and dynamics over extremely broad spatial and temporal scales. To tackle biologically relevant problems (biological recognition, *nano*-diagnostics, *nano*-medicine) X-ray diffraction is very well adapted if one can manage radiation damage. *nano*-beam serial scanning diffraction, *nano*-scale manipulation and *nano*-fluidic devices will enable new powerful strategies to elucidate processes occurring in hitherto unchartered spatial and temporal regimes.

- ***Evolutionary Developmental Biology***
3D visualization of the formation of organs in individual has involved so far *post-mortem* imaging. Developmental studies done on the same specimen at different growth stages will be by far more interesting if it would be possible keeping the animals alive, and developing low-dose approaches. A higher coherent photon flux density at high X-ray energy would allow to substantially reduce the dose.

- ***Biomedical Imaging*:**
X-ray imaging is often the only way to study inner organ functions, as hearing and feeding behaviour. Real-time X-ray cinematography of organ function, including gas distribution in lungs, and perfusion and micro-vascular permeability in brain, lung and other organs and tissues, is highly challenging at small length scales. There is currently a trade-off between spatial and temporal resolutions, and both are difficult to achieve simultaneously.

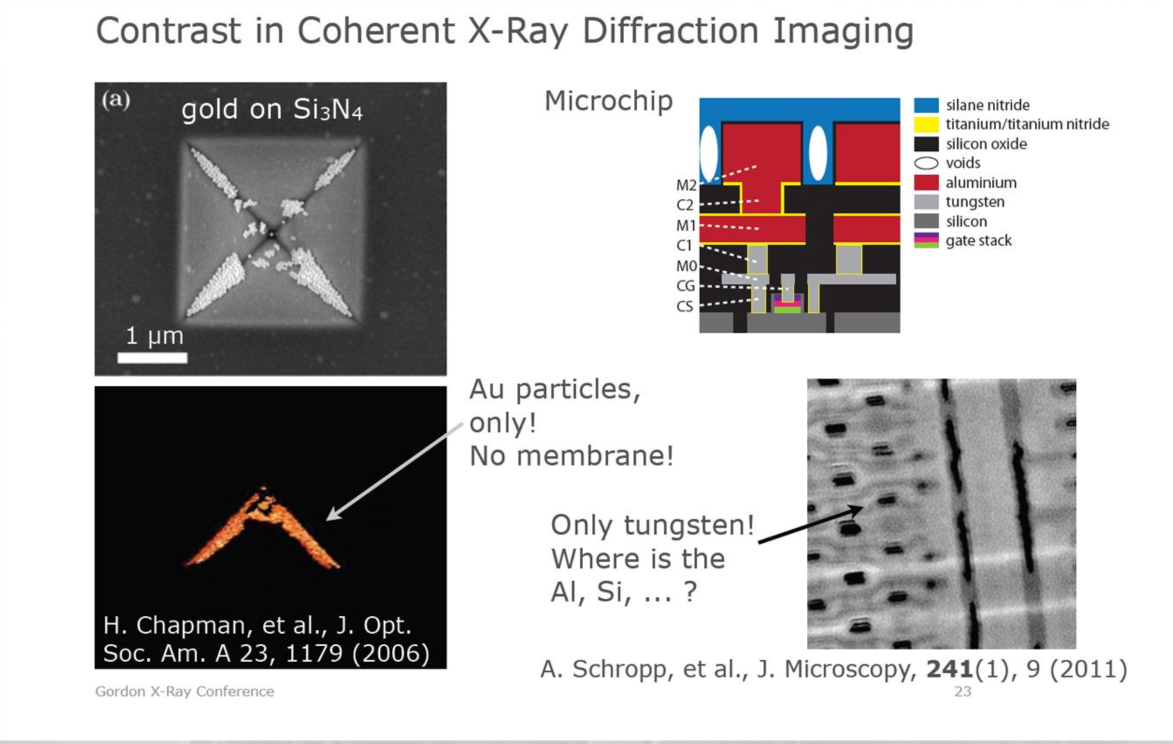
Clear need for a substantially brighter and more coherent source and a new generation of beamlines

***Low dose in-vivo* μCT: crocodile embryo**

Materials and chemistry

Key societal challenges such as the need for sustainability drive the never ending push to produce new functionalities with less input of energy and raw materials resources.

- ***Catalysis***:
  The potential for reduction of the energy footprint and environmental impact of current production methods is intimately related to the performance of existing and new catalysts. Understanding catalysts in action as close as possible to industrial conditions (temperature, pressure, chemical environment, relevant timescales, etc.) is therefore very important and challenging. Today we have access to the extreme ends, with a big gap in the nano-second to milli-second regime.



- ***Functional Materials***:
  Electronic devices with nanometre structures require analytical tools with very high space and time resolution.
  Today the in *operando* understanding of the electro-mechanics in electronics systems as MEMS and NEMS, the presence of defects, the transfer of matter in contacts during ultrafast motions are mostly deduced by electrical measurements studies without local information and direct visualisation. The need for tools to monitor a system in package is urgent, and a key point for the development of next generation devices.

The two areas indicated above will qualitatively grow with respect to present capabilities when the unprecedentedly bright *nano*-beams will become available with the new storage ring.

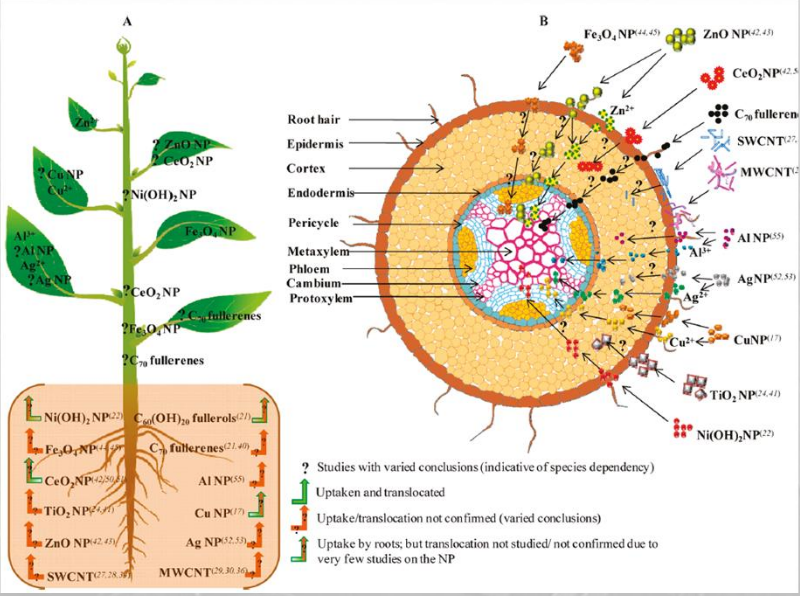
The study of natural processes (climate changes, evolution of the Earth's crust, living organisms), human controlled processes (natural resource management, pollution and remediation), extreme conditions (in planets, stars, earth interior) are increasingly studied using synchrotron X-rays.

Understanding natural processes is key to prudent management of natural resources. Synchrotron studies have contributed tremendously in clarifying many such processes, often reproducing exotic and extreme natural conditions. Many challenges still lay ahead.

- ***Structural and chemical complexity in natural processes:***

Bright *nano*-beams are successfully used in the study of complex interactions in hierarchical and heterogeneous samples relevant to environmental science (natural interfaces, microbial bio-films, plants and soils, etc). The combination of spectroscopy and imaging provide unique possibilities in terms of spatial and temporal resolution with atomic sensitivity.

Nano-particles, increasingly used in applications, raise concerns about potentially negative environmental and health effects.

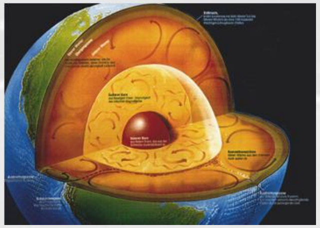


- ***Structure of materials at extreme conditions:***

The behaviour of elements at high pressures provides fundamental insight into the behaviour of high-density matter. A new generation of intense *nano*-beams will enable the efficient use of pressure cells operating in the multi-Mbar regime, providing opportunities: 1) to extend phase diagrams of materials; 2) to explore warm dense matter states; 3) to study the phase diagrams of highly correlated systems, frustrated magnetic systems, and 3D domain arrangement in ferroelectrics, magneto-strictors and multi-ferroics; 5) to determine the properties of minerals and their melts in planetary mantles and cores such as simple silicates and oxides (SiO_2, $MgSiO_3$, MgO) and iron alloys (Fe-S, Fe-Si, Fe-O, Fe-C).

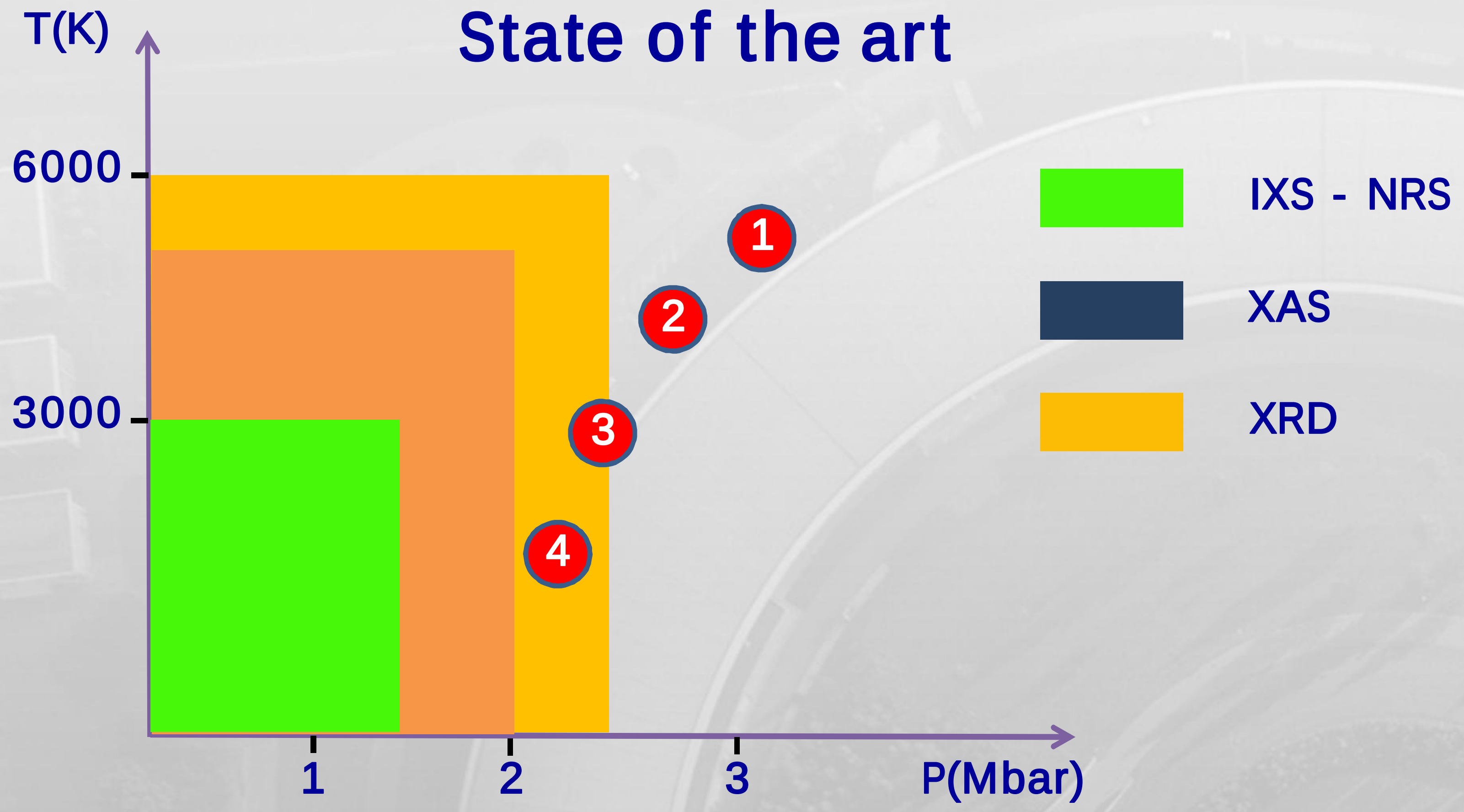
These areas will qualitatively grow with the much brighter *nano*-beams at the new X-ray source.

Earth interior; important unanswered questions:



1. What is the composition, structure, and chemistry of the inner core of the Earth?
2. What is the electronic and local structure in its liquid outer core?
3. What is the chemistry of light elements in the lower mantle and in the inner core of the Earth?
4. What is the rheology in the deep interior of the Earth and how does it correlate to the observed seismic discontinuities?

State of the art



Beyond current possibilities of present SR sources and high-pressure technology

Exoplanets

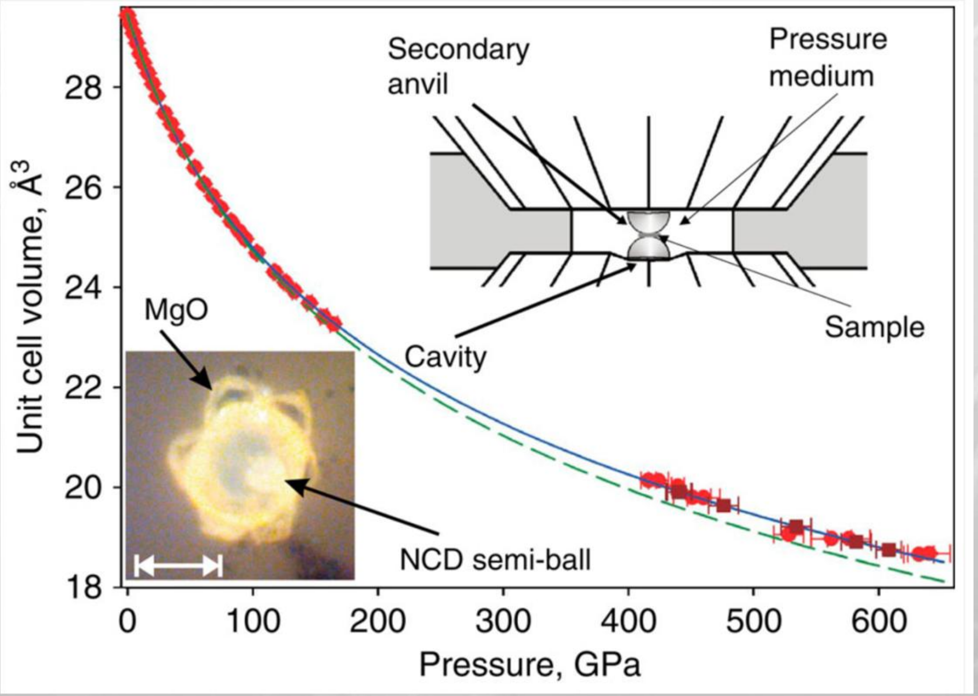
One of the most important discovery of the last 10 years in astronomy:

Existence of more than 1000 Exoplanets with a diversity of size, mass, composition, some of them potentially habitable.

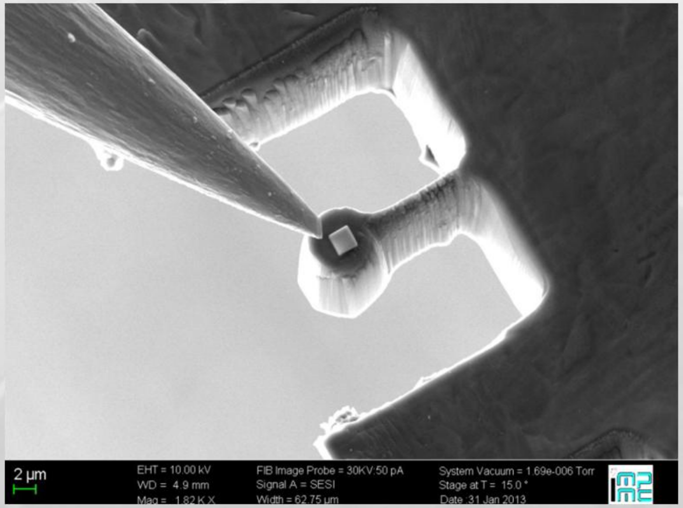
Progress in HP technology

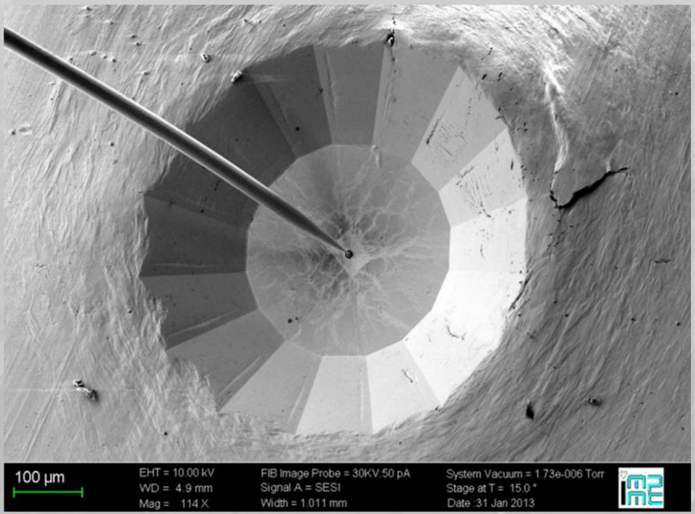
Advanced sample loadings

Double-stage diamond anvil cell: Maximum pressure beyond 6 Mbar!



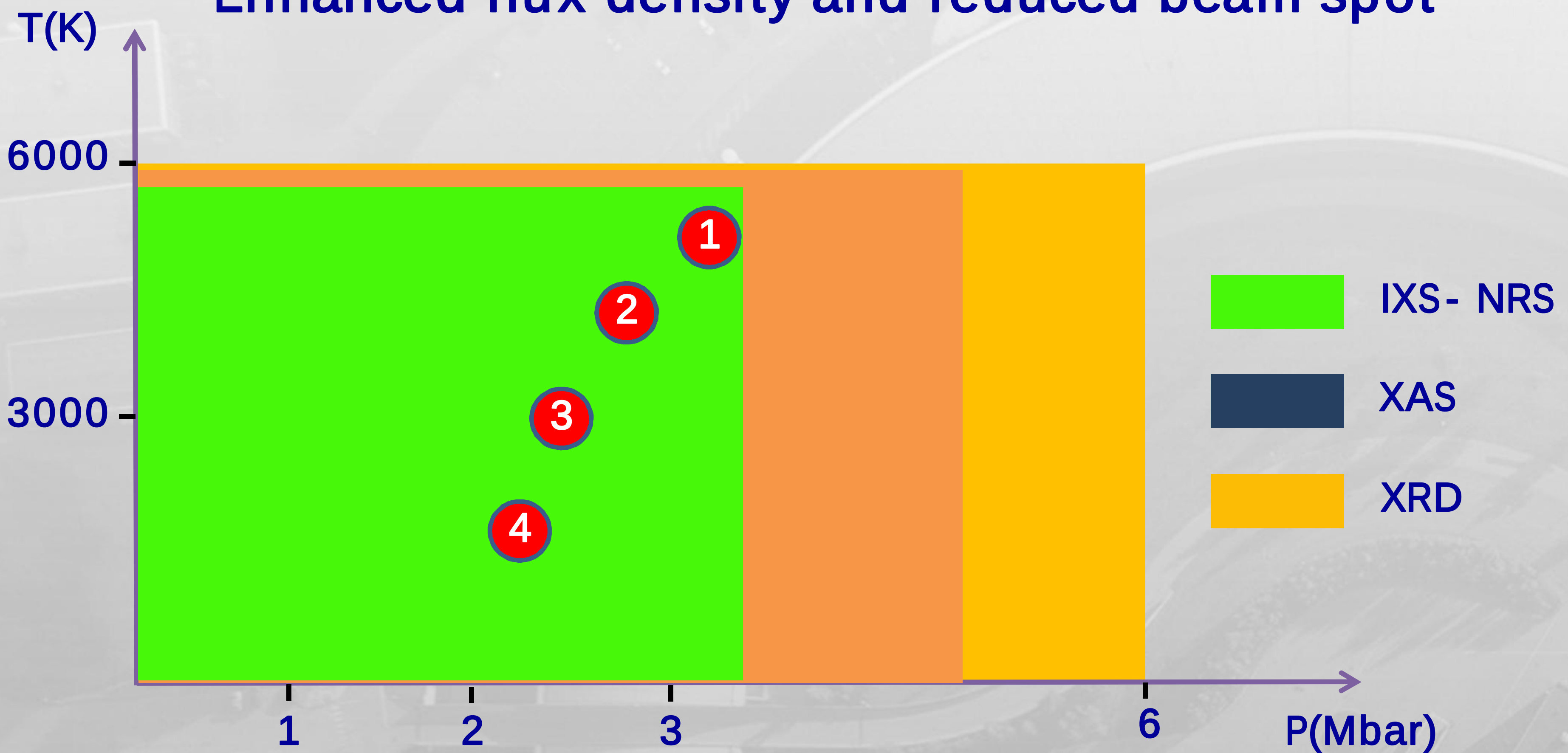
Dubrovinsky et al, Nat. Com. (2013)





Fiquet et al., Nat. Mat. (submitted)

State of the Art at the new ESRF X-ray source: Enhanced flux density and reduced beam spot



DSLR: Experimental Challenges

Managing Radiation Damage:

- New Detector Technologies
- Smart automation and data collection strategies
- Data management: flow control, collection, storage, analyses

Enabling Technologies:

- *nano*-mechanics, -positioning, –optics, etc.
- Software, software and software

DSLR: a new dimension for X-ray science

Opportunity to enter into a new era of X-ray Science to the benefit of investigations in condensed matter, materials and life sciences

Novel availability of brighter and highly coherent X-ray *nano*-beams

Couple diffraction, scattering and spectroscopy methods to microscopy and *3D* imaging with *nano*-meter spatial resolution

Thank you for your attention!

A Light for Science

ESRF