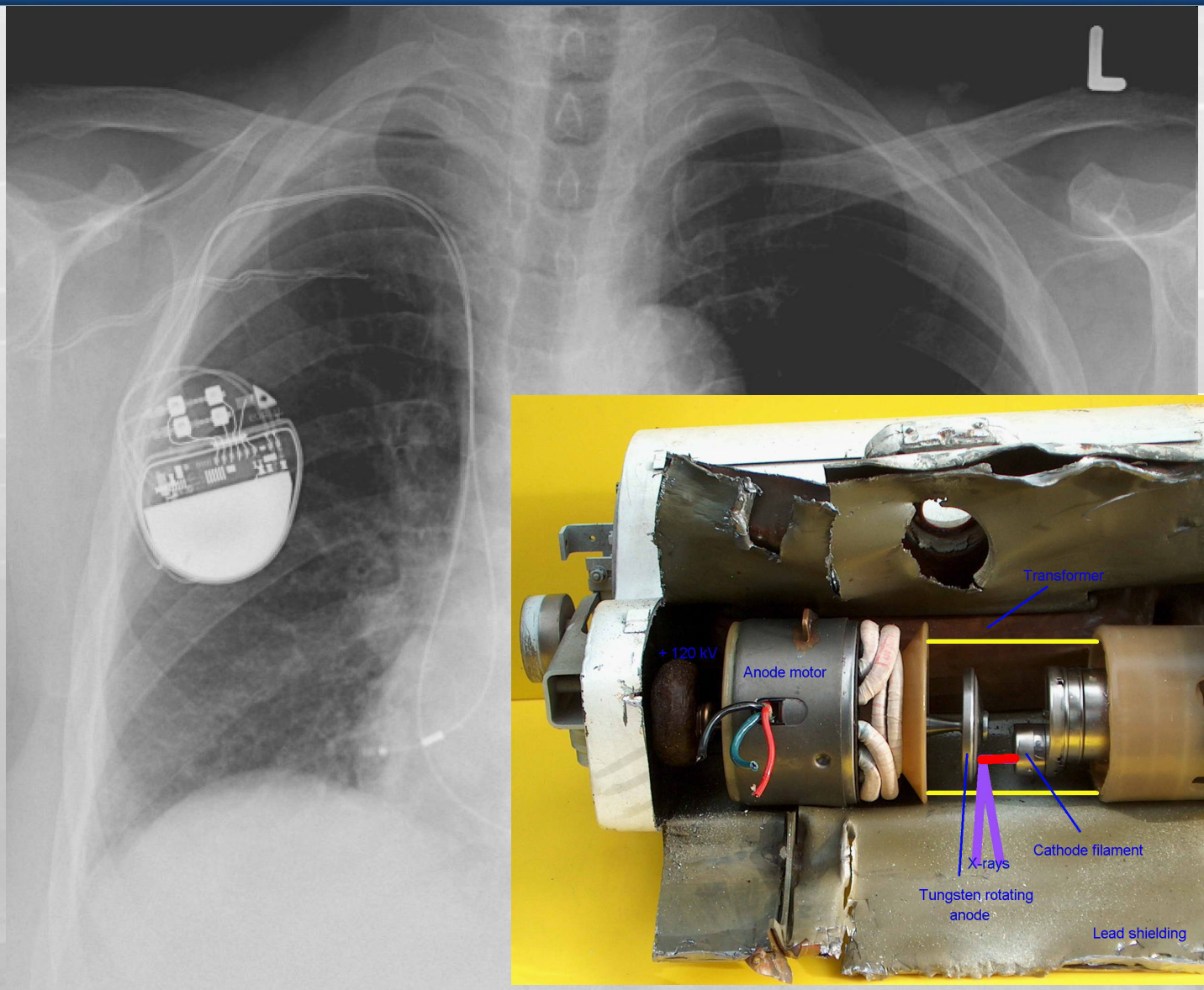
A Light for Science

ESRF

"X-ray science at synchrotron sources: new possibilities to understand complex materials"

Francesco Sette – ESRF BP 220 – 38043 Grenoble – France

- X-rays science and synchrotron radiation
- Science using synchrotron X-rays: materials for energy
- Upgrades of existing sources and future storage ring based synchrotron radiation sources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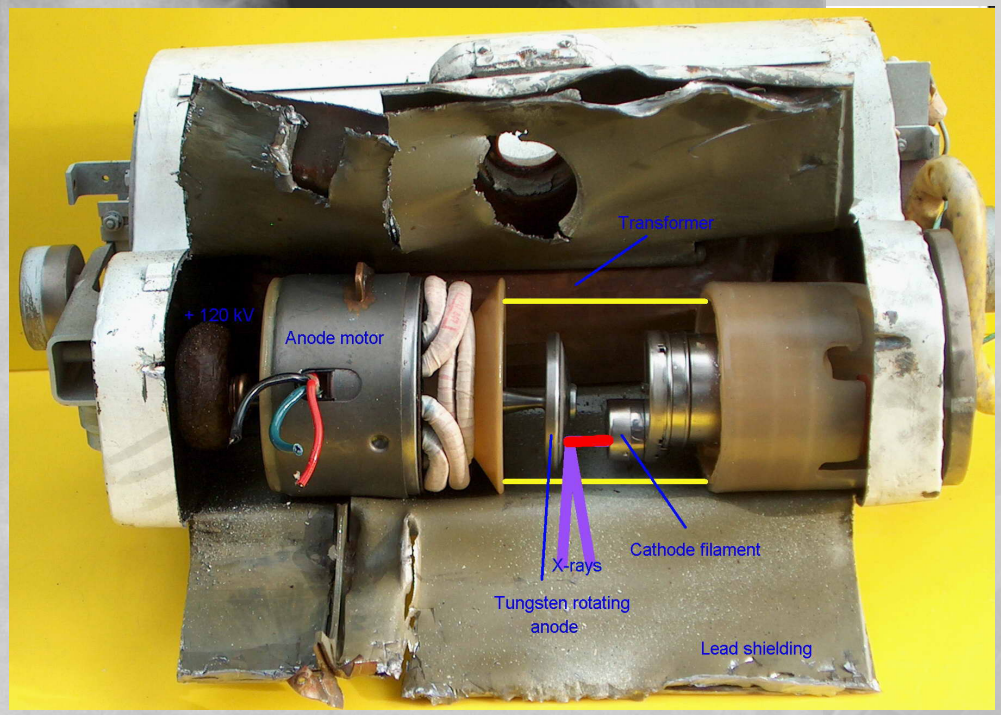
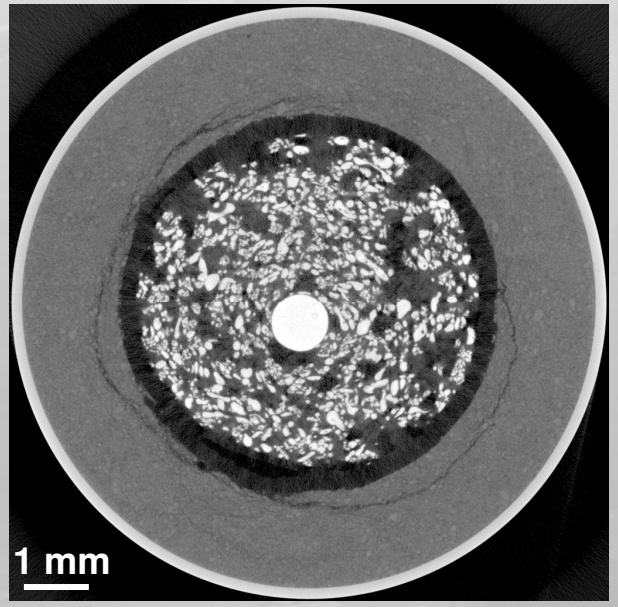
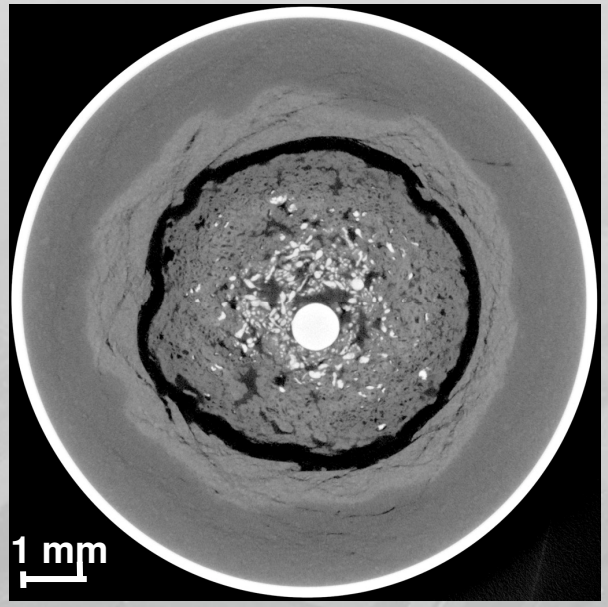

Transformer
+ 120 kV
Anode motor
X-rays
Cathode filament
Tungsten rotating anode
Lead shielding

A closer look at a battery with Synchrotron X-ray tomography

charged

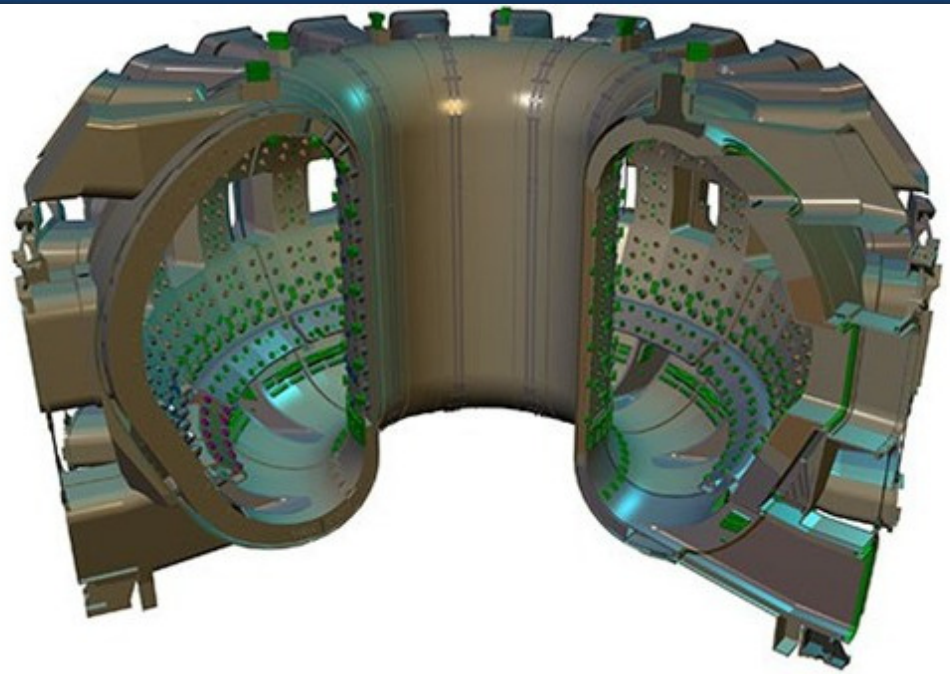


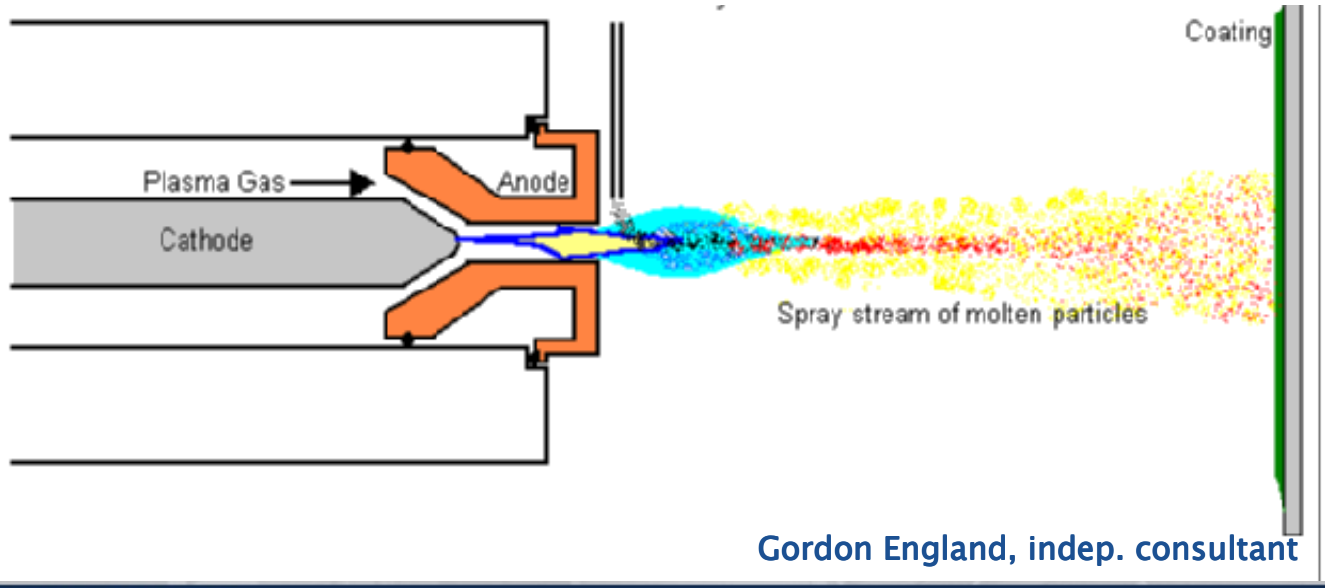
or empty?

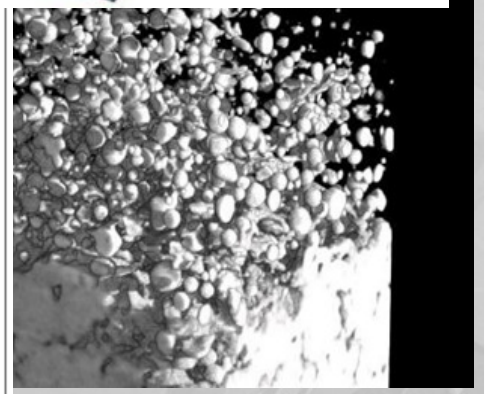


Tungsten coatings for the ITER vacuum vessel

Tu
ma
Photo







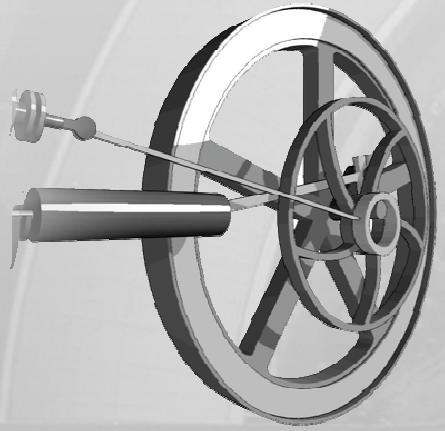
Gordon England, indep. consultant

SR is a unique tool to investigate materials

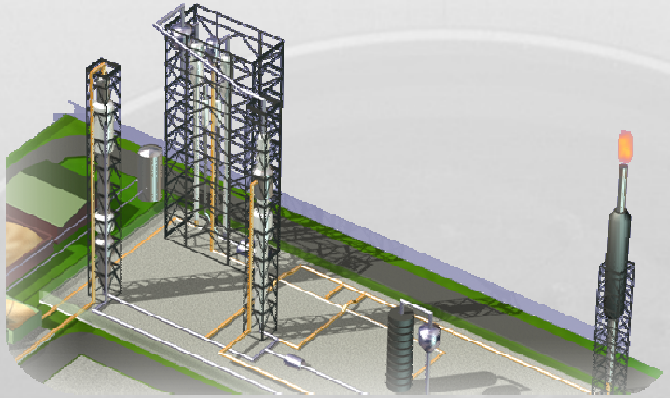
Technological progress is linked to understanding new materials

Stone-age

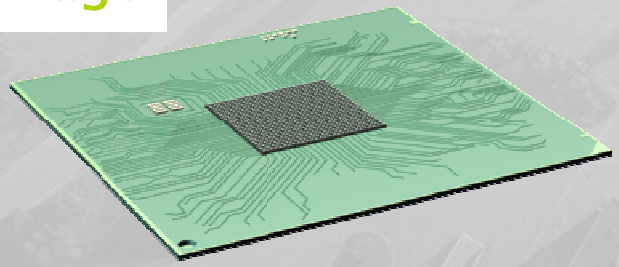
Bronze-age



Steel-age

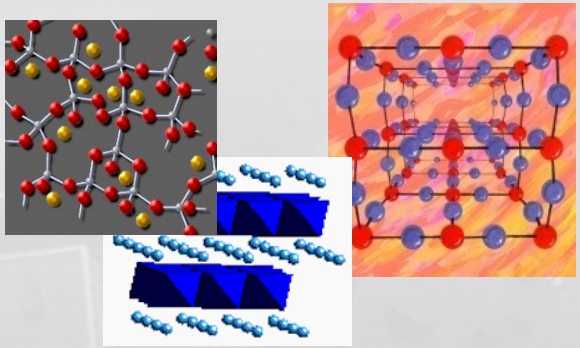


Oil-age



Silicon-age

The "new-age" of **Knowledge Based Materials**



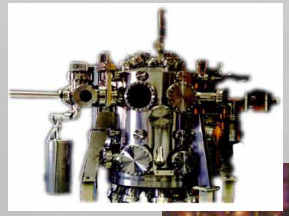
Structure

Characterization

Properties

Processing

Performance



Condensed matter, Materials and Living Matter: CHARACTERIZATION

- Many Laboratory Techniques: Electron Microscopies, Optical Methods, IR, NMR, Acoustic Methods, Tribology, X-rays ……
- Increasing need for techniques with *nano*-metre spatial resolution, and:

X-rays – as electrons – are ideally suited
to achieve *sub-nano*-metre spatial resolution:

- *No diffraction limit* (visible light)
- *X-ray Synchrotron nano-imaging*

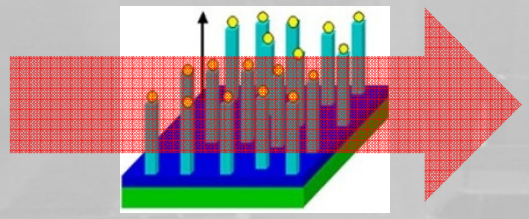
achieved already *resolution below 10x10 nm²*

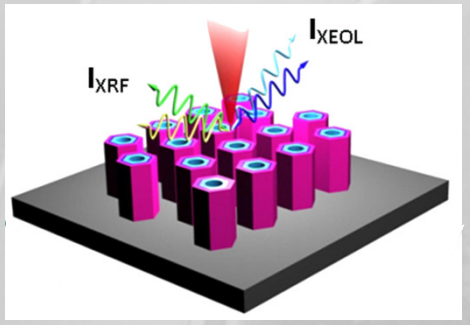
X-RAYS AT STORAGE RINGS and FREE ELECTRON LASER

Nanoscience / Nanotechnology with X-ray beams

Large beam on nanoscale objects

average properties



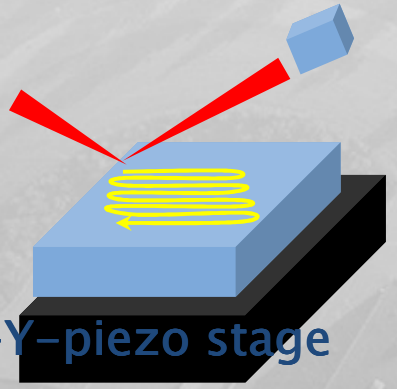


Small beam on nanoscale objects

individual properties

Small beam on large objects

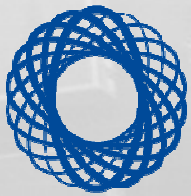
local properties scanning



Investigating matter, materials and living matter

Fields of application

Understanding matter down to the single atom links many scientific disciplines at Synchrotrons:

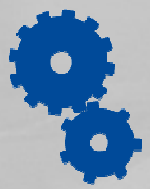


Solid-state physics

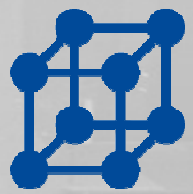
Chemistry

Medicine

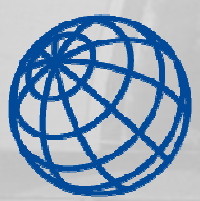
Life sciences



Engineering



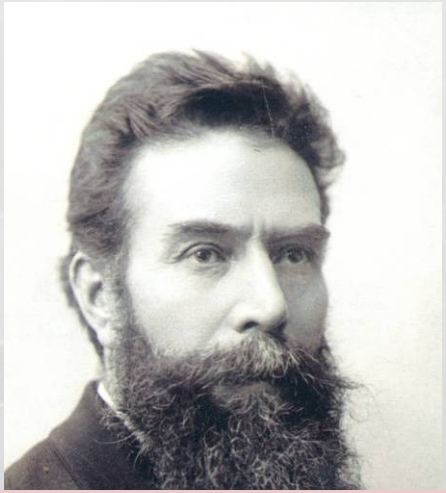
Material science



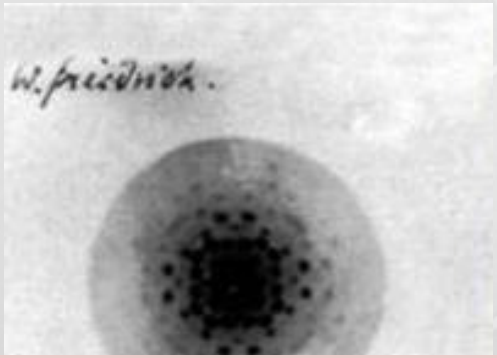
Earth sciences

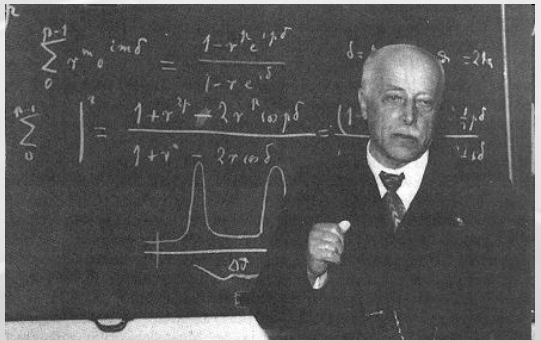
Environment

Cultural heritage

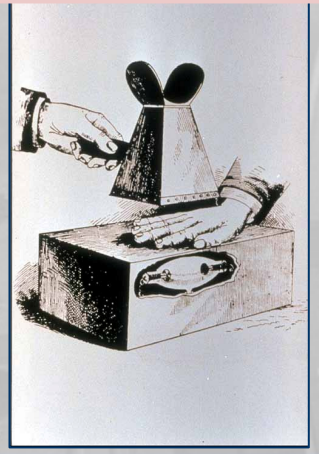


X-rays

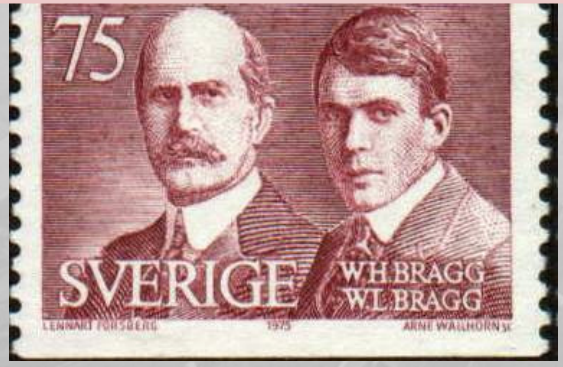




2012
X-ray Diffraction: 100 years

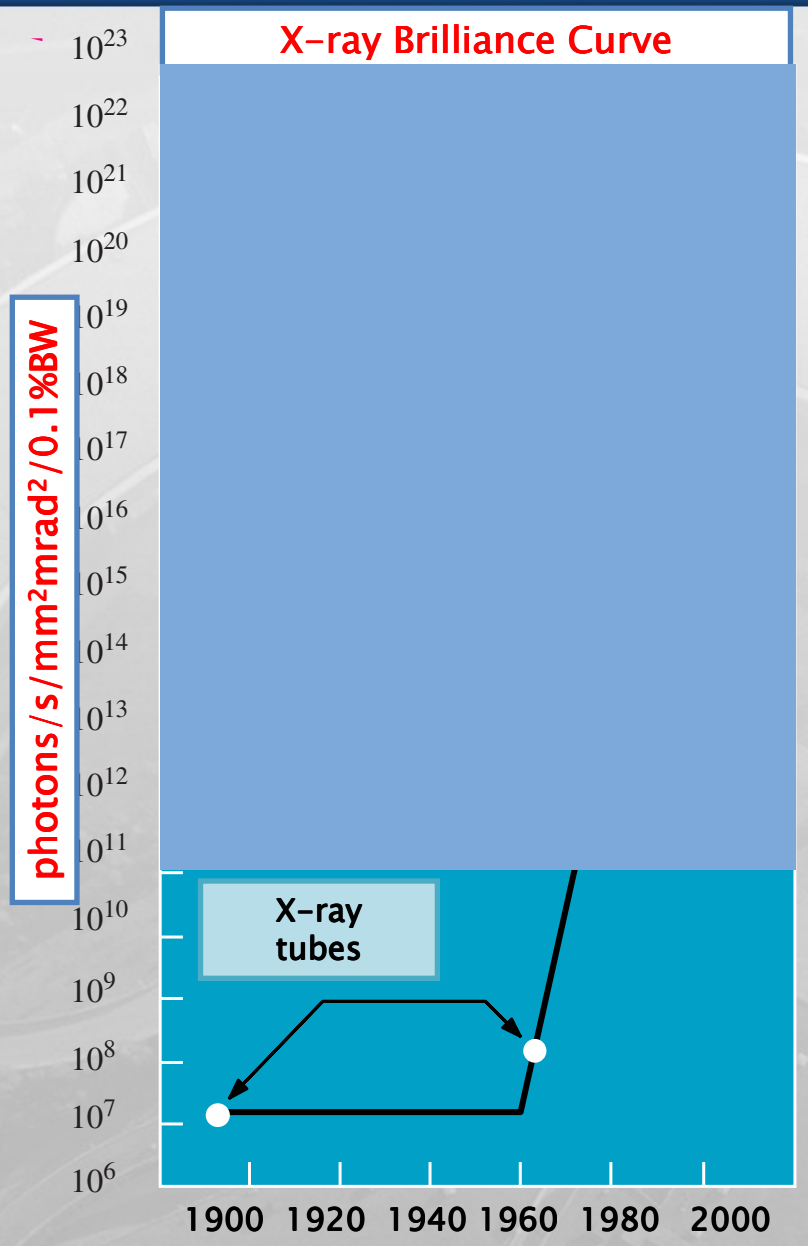


discovered in 1912-13 that X-rays can be used to study atomic structures and properties of materials. They received Nobel Prizes in physics in 1914 and 1915, respectively.



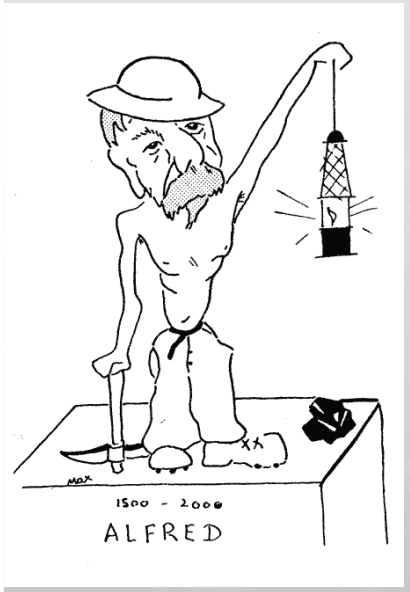
Conventional X-ray Sources

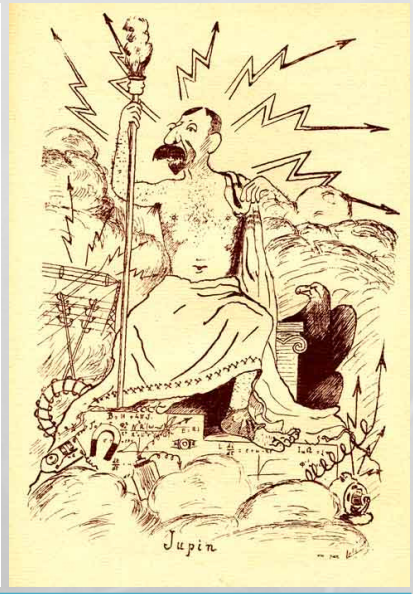
(before Synchrotron Radiation)



Relativistic electrons and light

Alfred-Marie LIENARD (1869-1958)



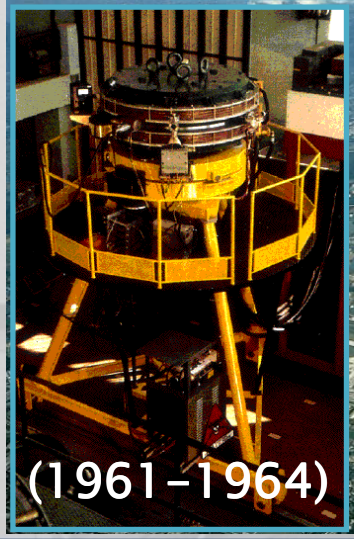


Bruno Touschek (1921-1978)

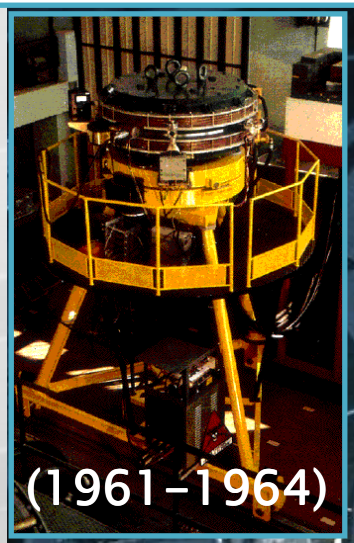
An electron submitted to a force can loose a considerable fraction of its kinetic energy by electromagnetic emission

"L'Éclairage Électrique, 16(27), pp. 5-14 (1898)

Storage Rings: from AdA to LHC



The European Synchrotron Radiation Facility: the 1st Third Generation Source

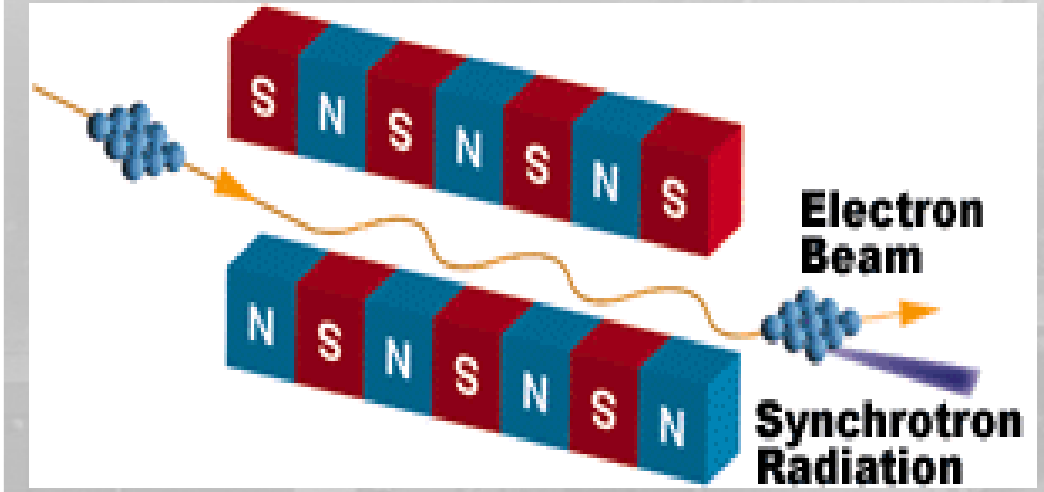


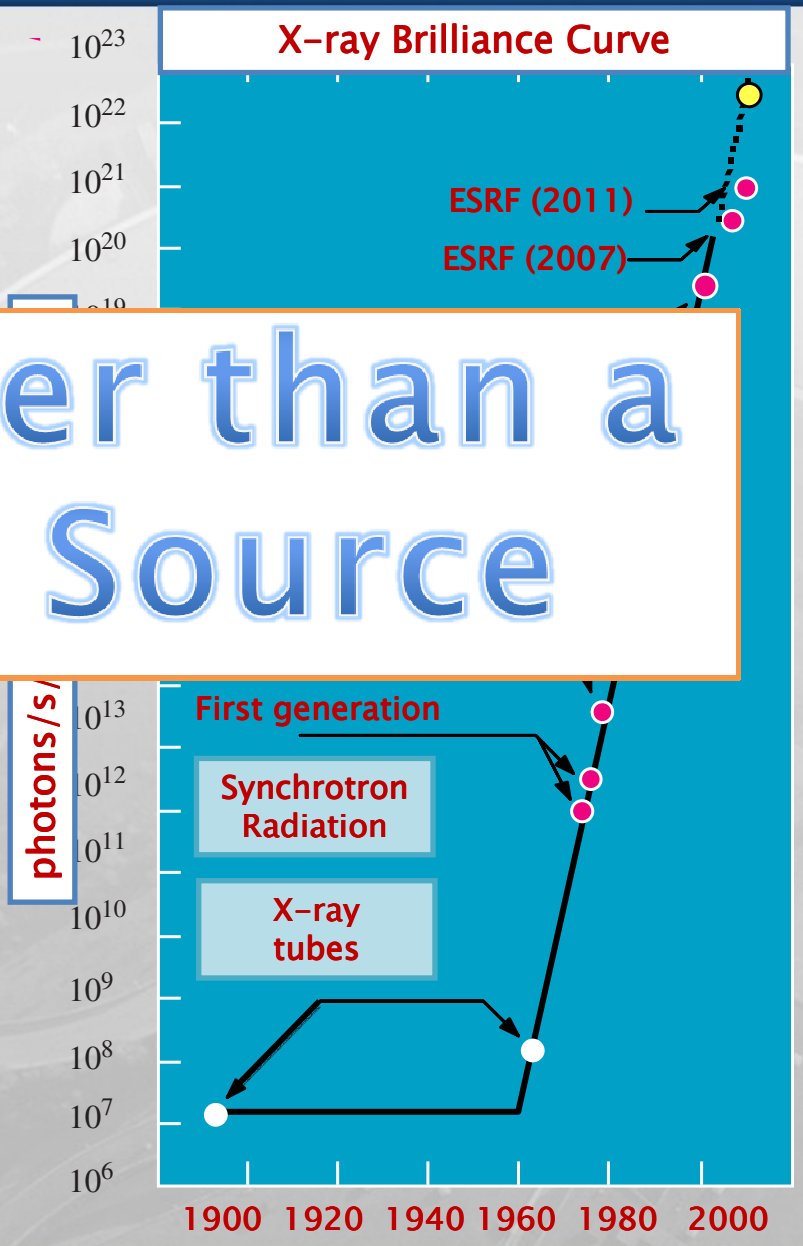
(1992-2XXX)

Synchrotron Radiation:

A Revolution in the use of X-rays

~10^{11} brighter than a Laboratory Source





Major X-Ray sources in the world

Hard X-ray sources: APS (USA) – ESRF (FR) – Spring 8 (JP) – Petra III (DE)

New SR sources: Soleil (FR) in 2006, Diamond (UK) in 2007, Petra-III (DE) and SSRF (CHN) in 2009, ALBA (ES) in 2010, Jordan, Poland, Russia, Sweden, Taiwan, USA.....

New plans also in Brasil, China, India, Japan, Korea .....

The ESRF today

ESRF: 19 Members and Associates Countries

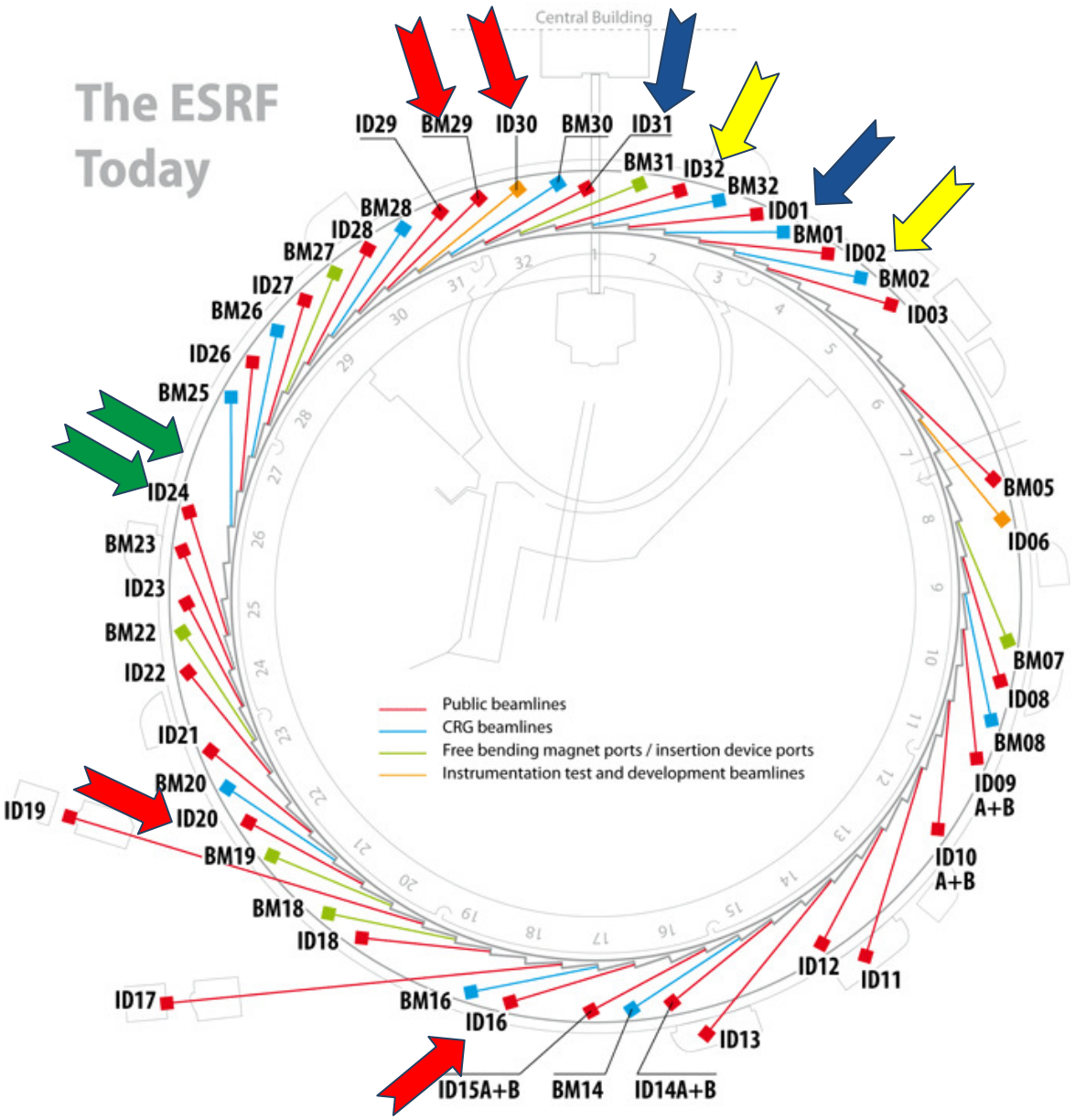
Contributions to ESRF Budget

Members

France	27.5%
Germany	25.5%
Italy	15%
UK	14%
BeNeSync (Belgium, The Netherlands)	6%
NordSync (Denmark, Finland Norway, Sweden)	4%
Spain	4%
Switzerland	4%
	100%

Scientific Associates

Portugal	1%
Israel	1%
Austria	1%
Poland	1%
Central-Sync (Czech Republic, Slovakia, Hungary)	1.05%



42 Beamlines portfolio

New Beamlines 2009 – 2015

Imaging
Diffraction
Spectroscopy

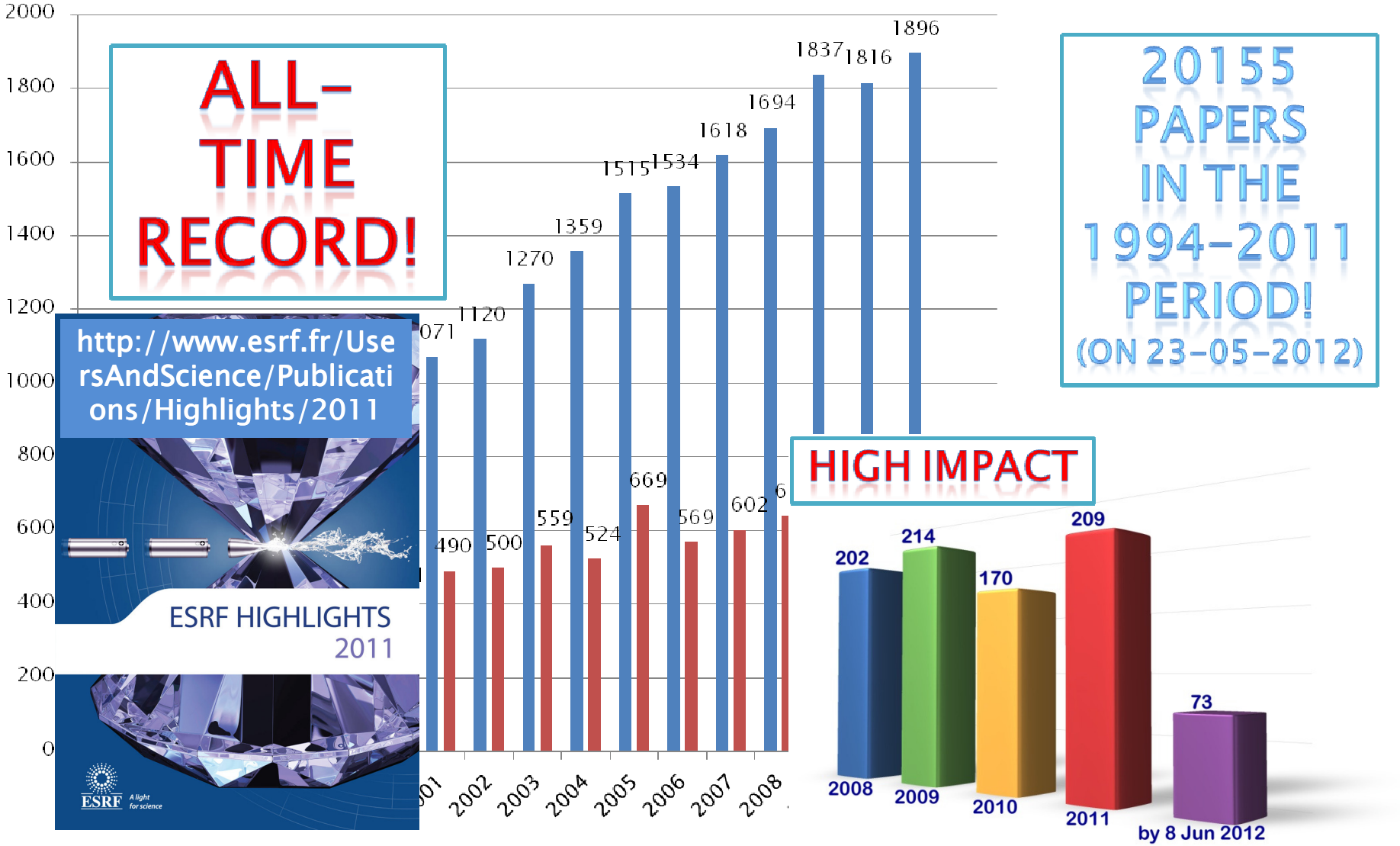
Fundamental Research

Energy
Life Science
nano–Materials

Refereed Publications from work at the ESRF

ALL-TIME RECORD!

20155 PAPERS IN THE 1994-2011 PERIOD! (ON 23-05-2012)



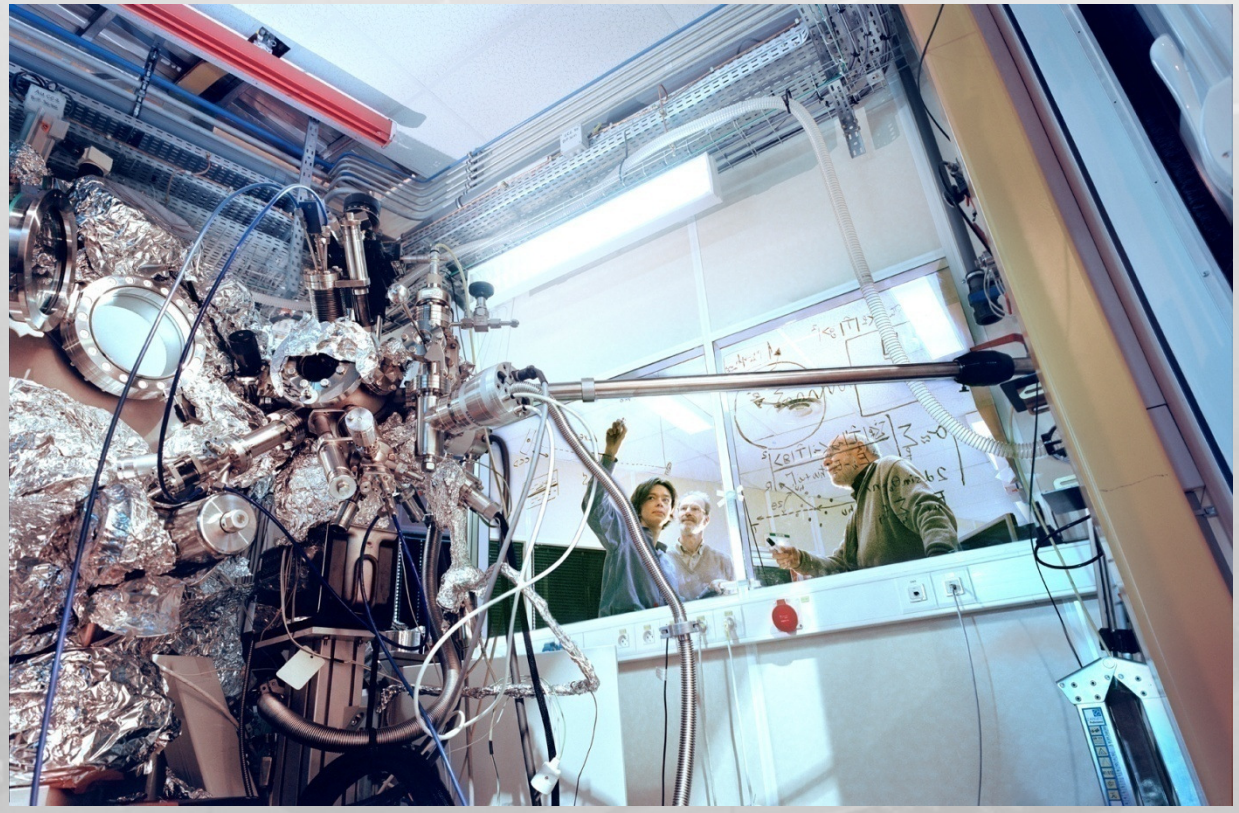
http://www.esrf.fr/UsersAndScience/Publications/Highlights/2011

HIGH IMPACT

Eighteen Years of Operation of the ESRF:
A bright and productive Synchrotron Radiation source

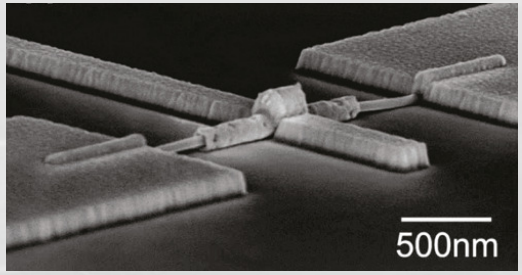
- ~ Serves 5000 Users/year (~7000 visits)
- ~ Receives 2000 Proposals/year
- ~ Provides 1500 Experimental Sessions/year
- ~ Provides 42 Synchrotron Radiation Beamlines
- ~ 1900 Refereed Scientific Publications/year from work at ESRF
- ~ Transfer of Advanced SR Technology and Training of Expert Staff

Nano-Science using synchrotron X-rays



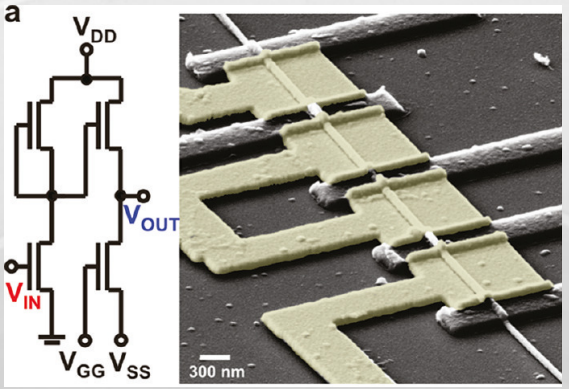
NANOWIRES (NWs):

Building blocks { Functional units
Wires for interconnection



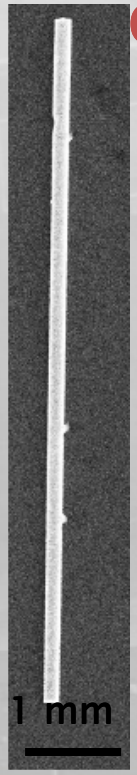
Storm *et. al.*, Nano Lett. 12, 1 (2012)

Single Transistors → Integrated Circuits



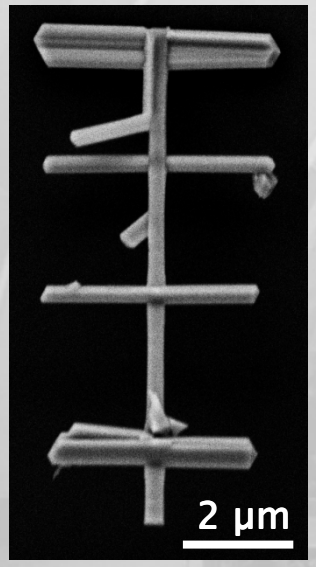
Kälblein, *et. al.*, Nano Lett. 11, 5309 (2011)

Co-doped ZnO NWs



InGaN NWs

Ga_2O_3/SnO_2 NWs

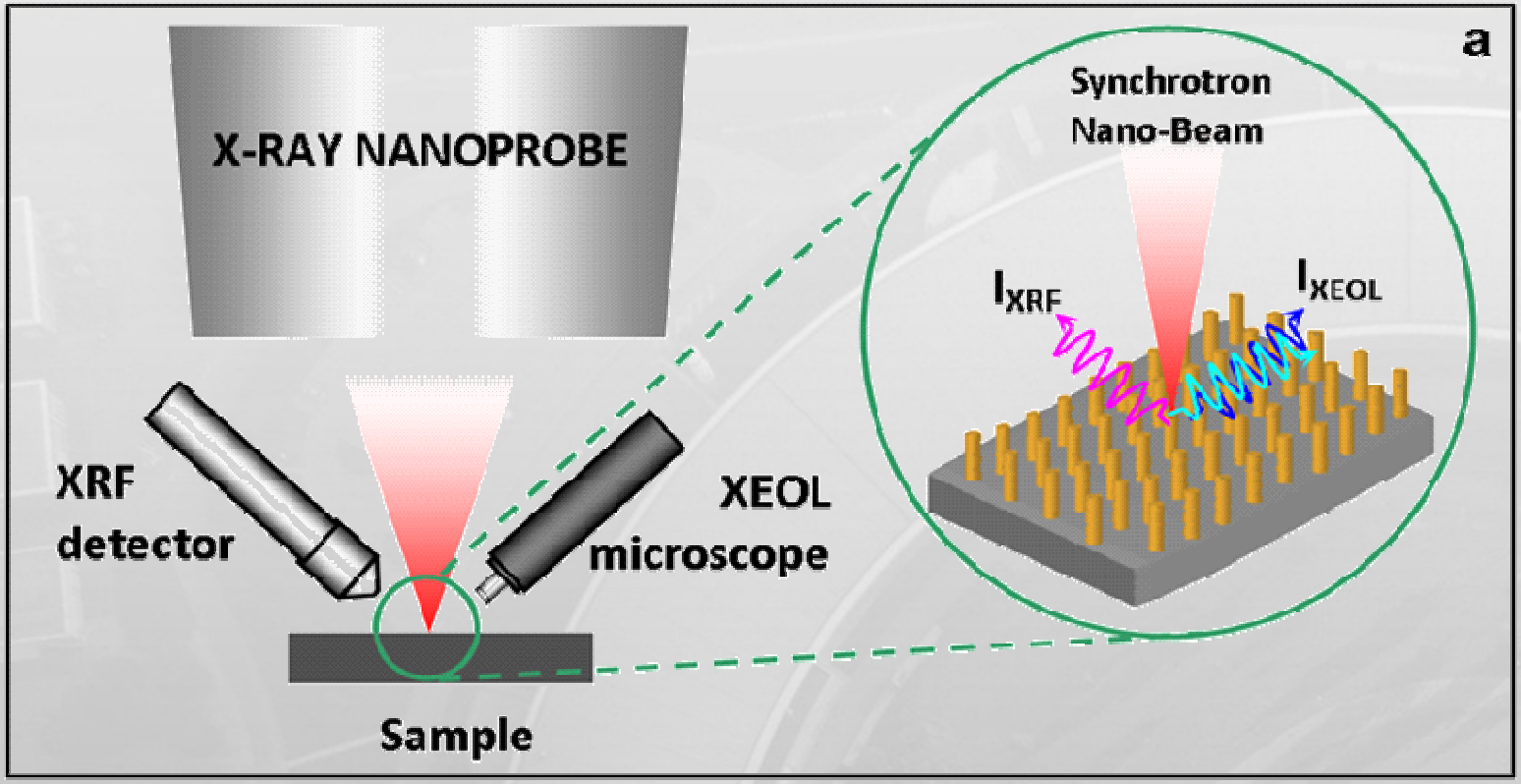


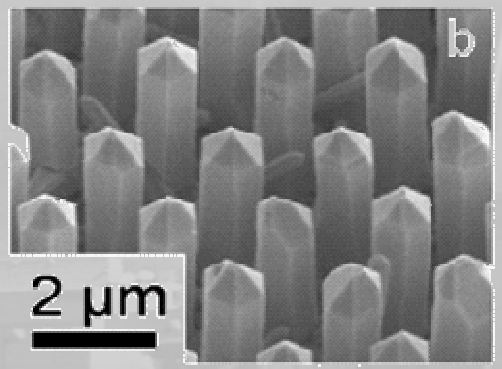
- Achromatic X-ray lens
- Nanometer X-ray beam
- Wide energy range:
  - In K-edge (27.98 keV)
  - Ga K-edge (10.38 keV)
  - Zn K-edge (9.67 keV)
  - Co K-edge (7.72 keV)

Challenge for local structural studies

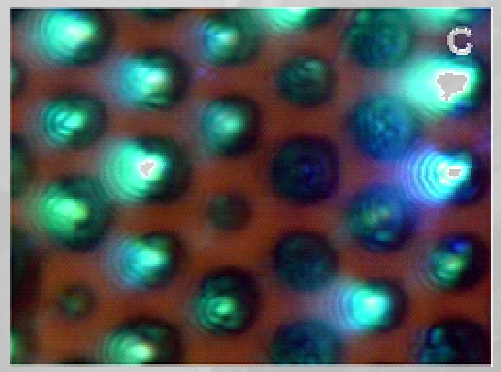
nano–LEDs --- G. Martinez-Criado et al.

Properties ↔ Device Function

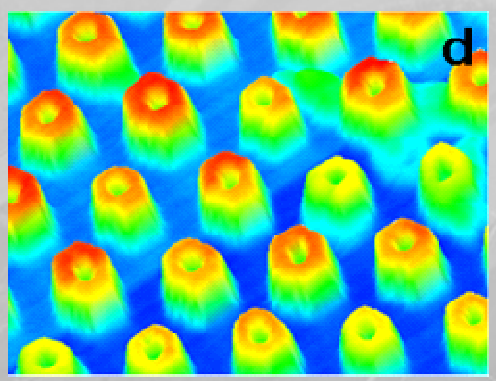




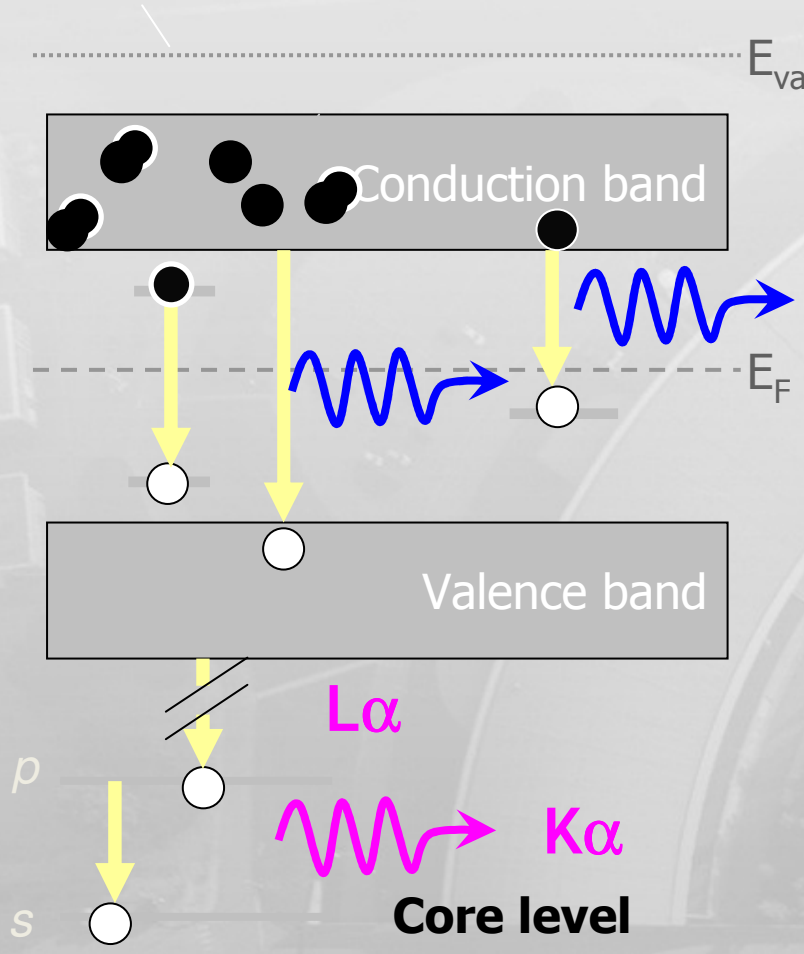
SEM



Electroluminescence

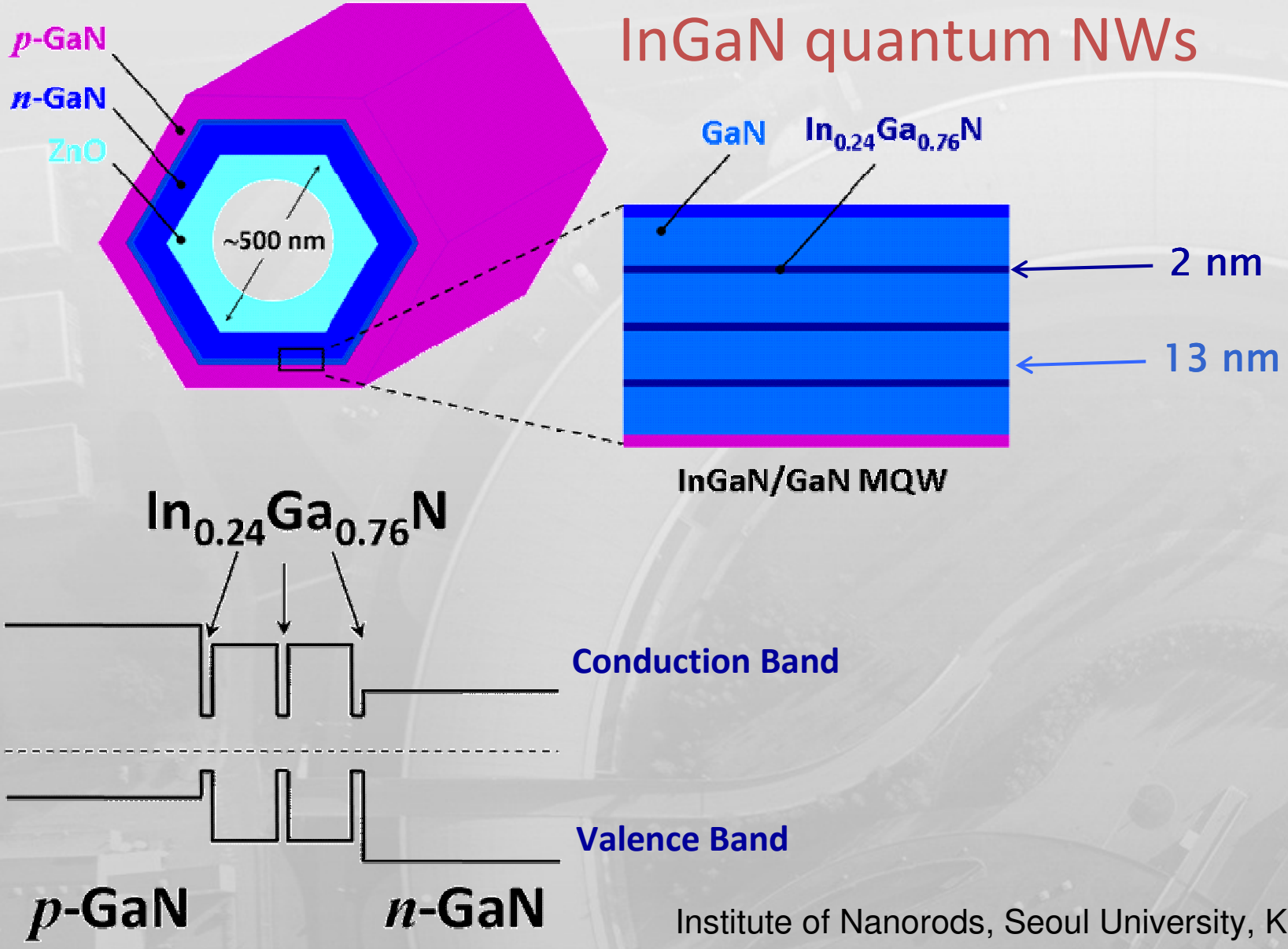


XRF Intensity - Ga K_α



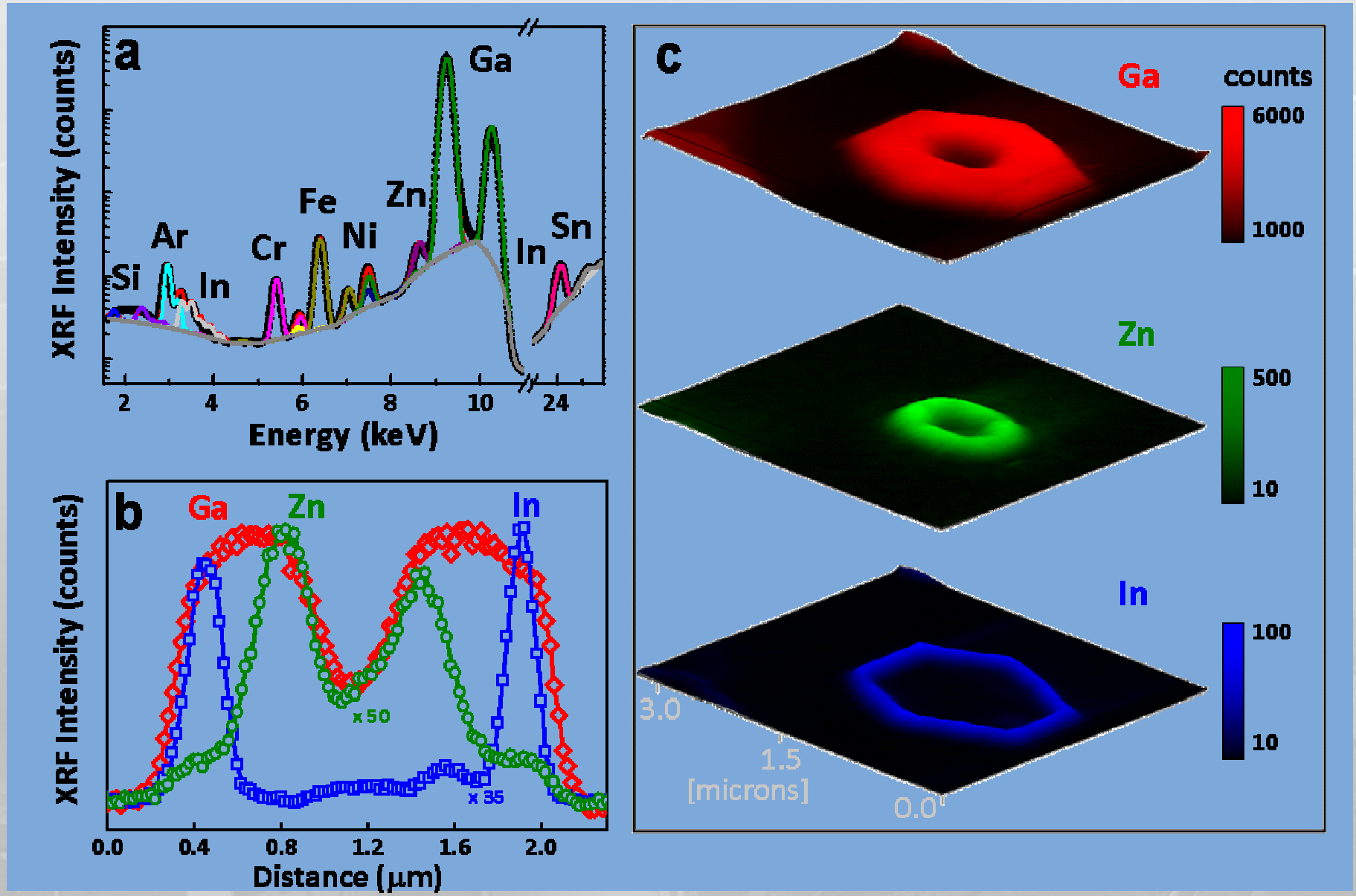
X-ray Excited Optical Luminescence

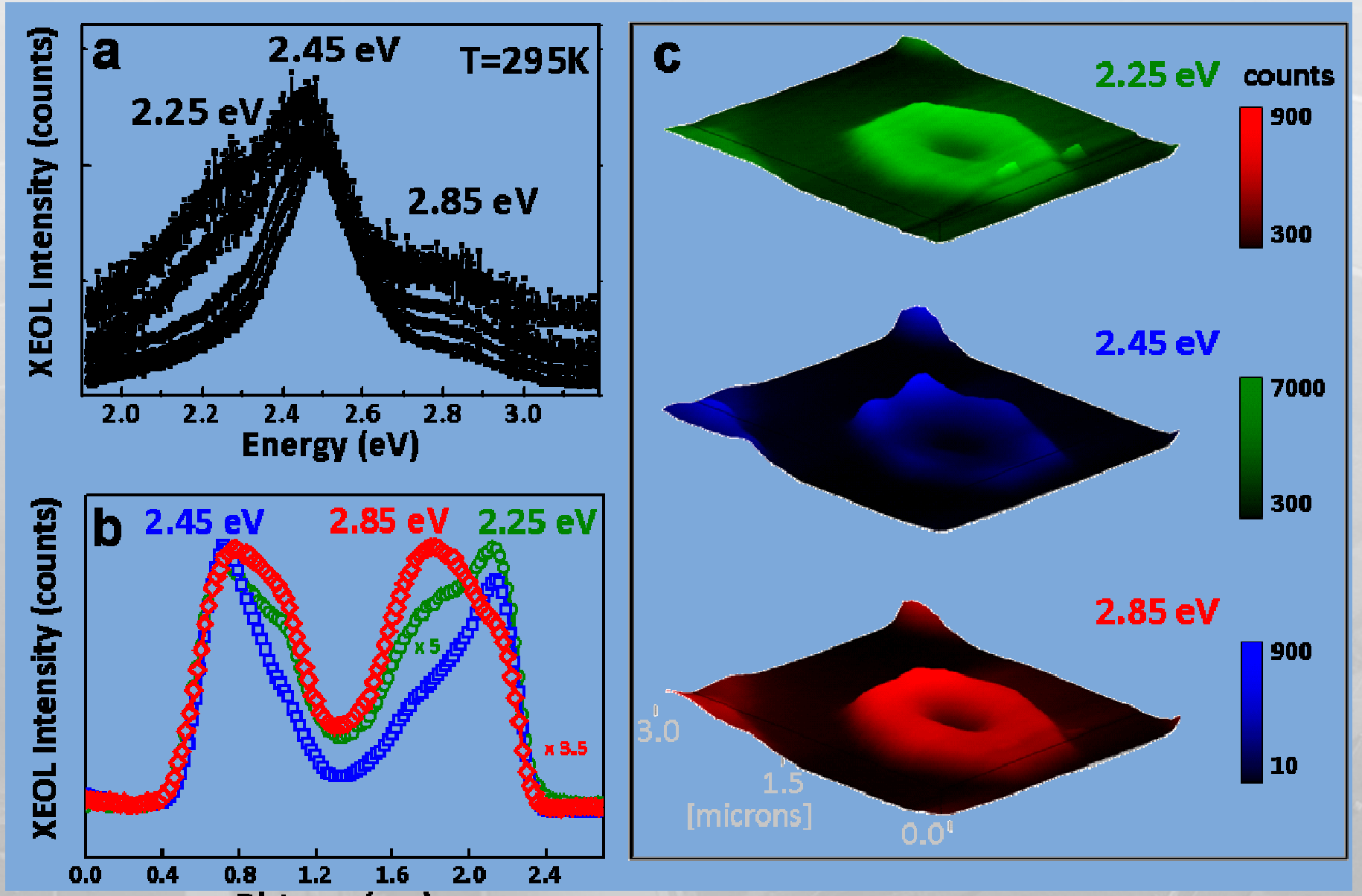
X-Ray Fluorescence



Institute of Nanorods, Seoul University, Korea

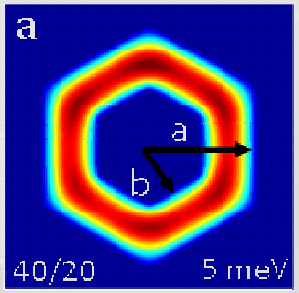
nano-LEDs: *nano*-XRF

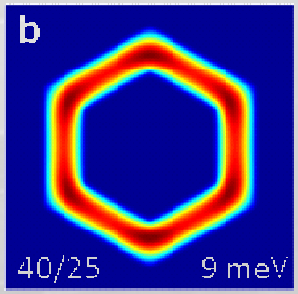


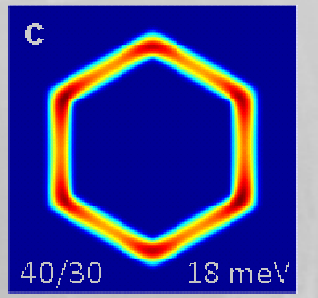

a
2.45 eV
T=295K
2.25 eV
2.85 eV
XEOL Intensity (counts)
2.0
2.2
2.4
2.6
2.8
3.0
Energy (eV)
b
2.45 eV
2.85 eV
2.25 eV
x 5
x 8.5
XEOL Intensity (counts)
0.0
0.4
0.8
1.2
1.6
2.0
2.4
Distance (μm)
c
2.25 eV
counts
900
300
2.45 eV
7000
300
2.85 eV
900
10
3.0
1.5
[microns]
0.0

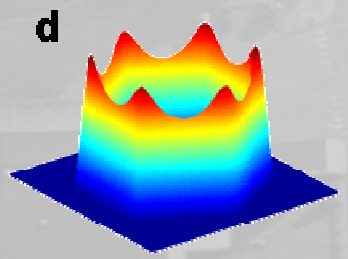
Confinement Effects

Theory

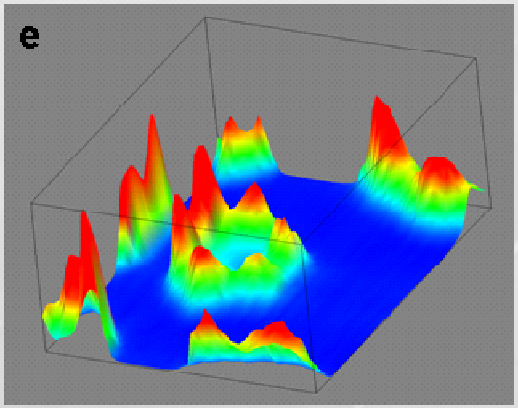


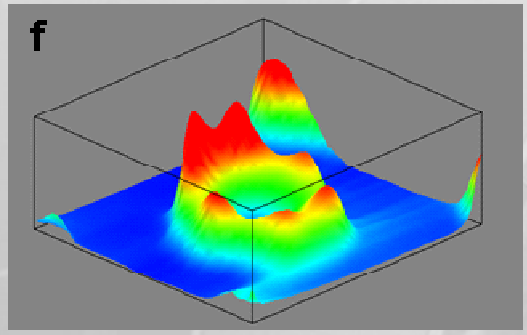


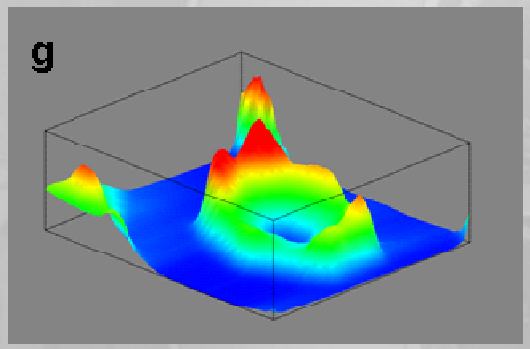




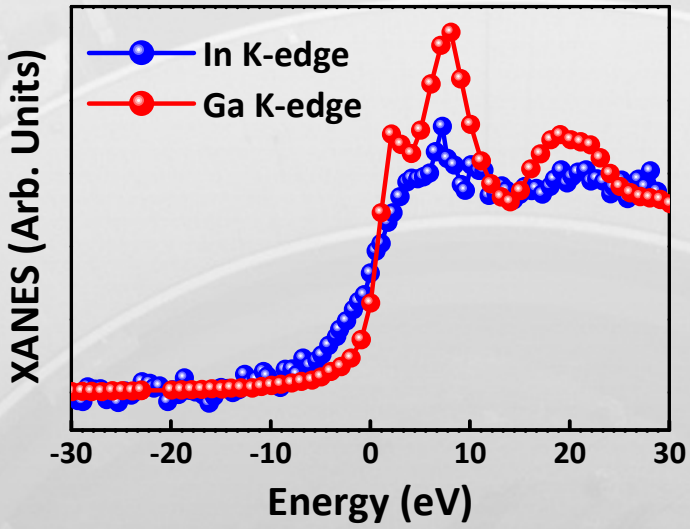
Experiment

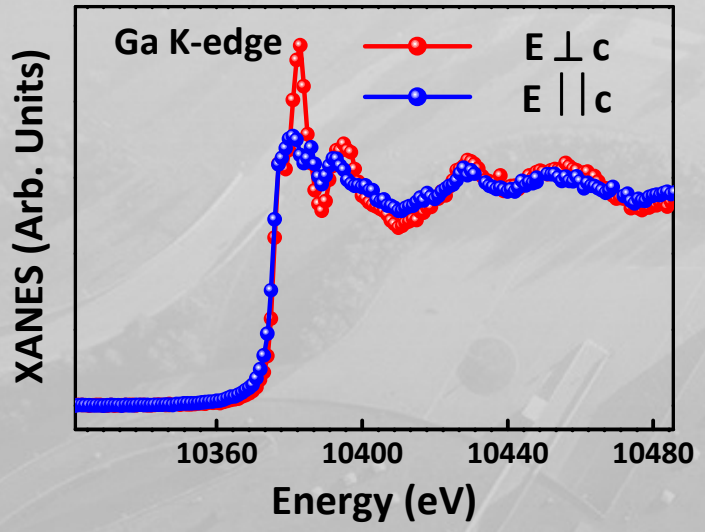




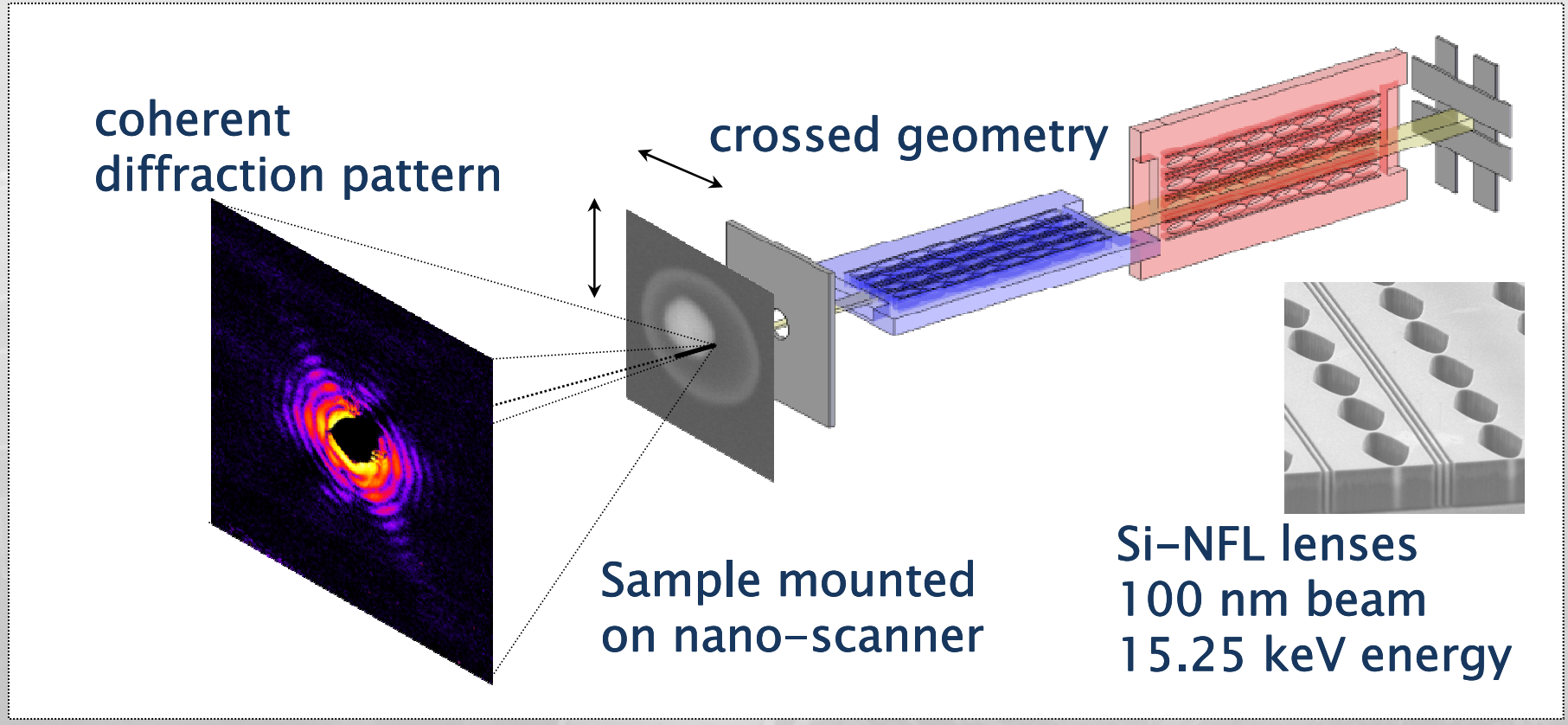


Nano-XAS

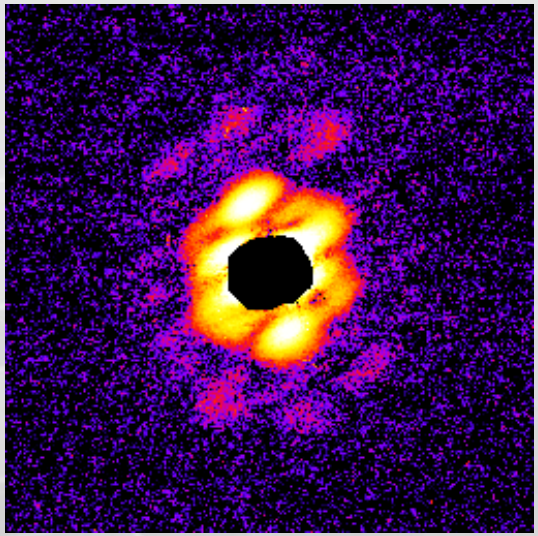




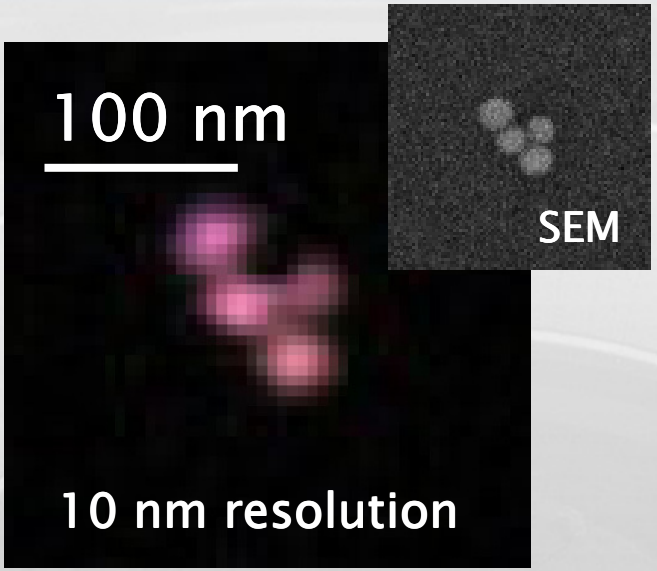
X-ray Imaging: *nano*-scale and coherent X-rays

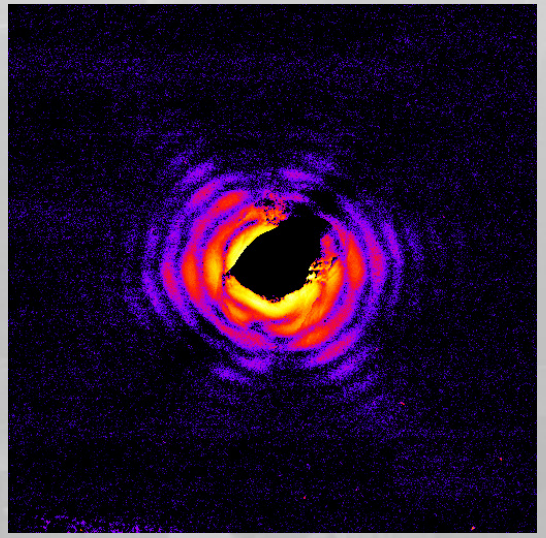


- *nano*-beam coherent X-ray diffraction imaging (CXDI)
- 100 nm beam generating gaussian illumination function
- *nano*-focusing silicon compound refractive lenses (NFL)

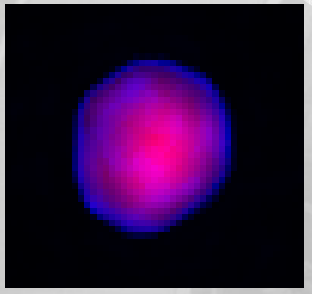


reconstruction
via
iterative
phase retrieval



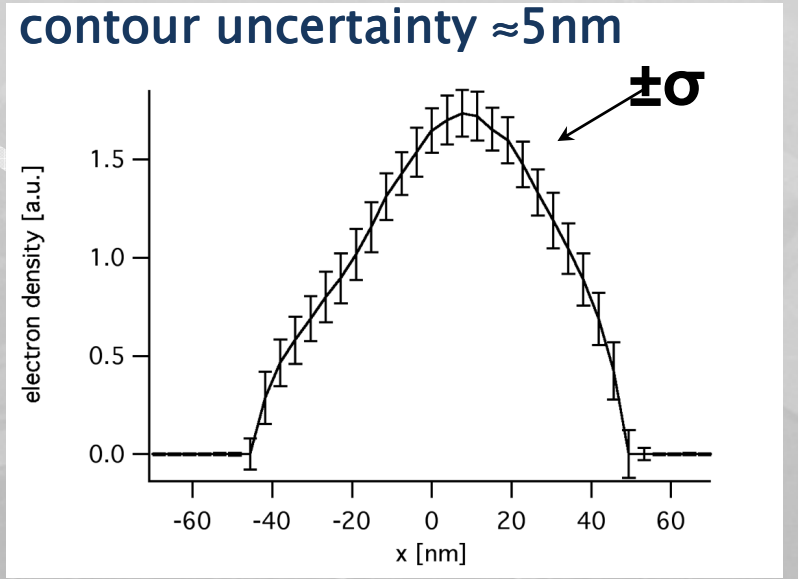


gold *nano*-particle



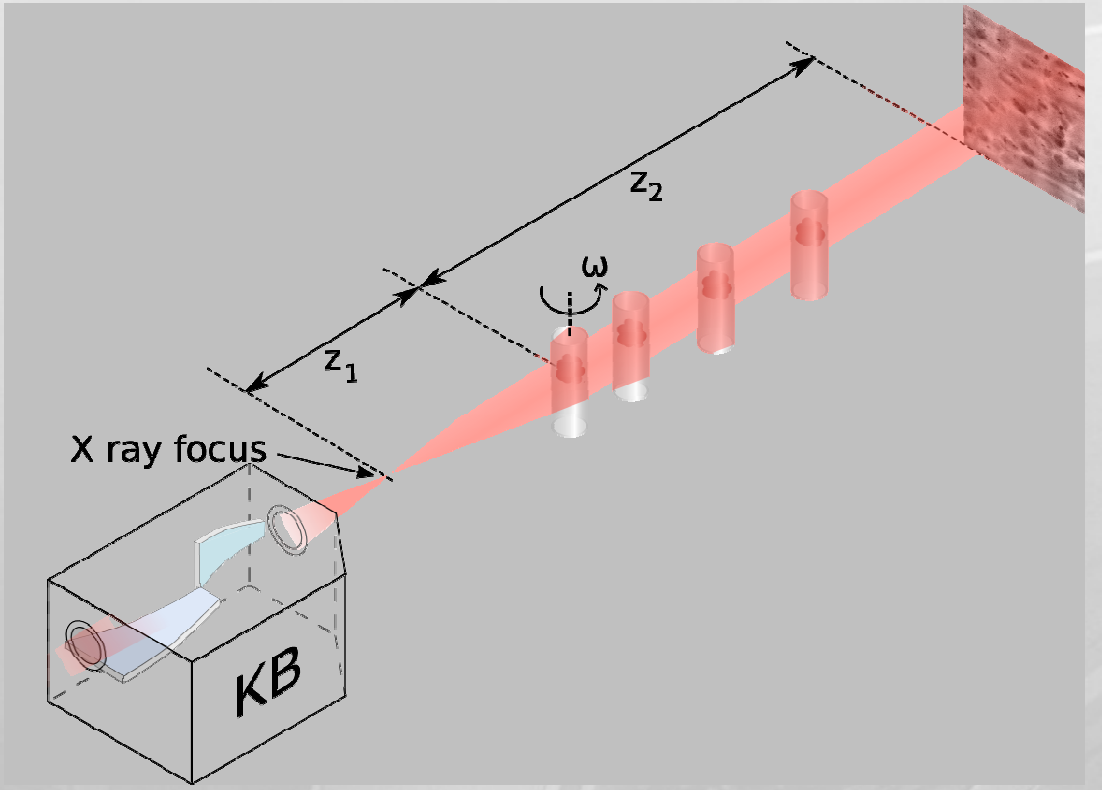
reconstructed
projection
5 nm resolution

contour uncertainty ≈5nm



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

Phase-contrast *nano*-tomography

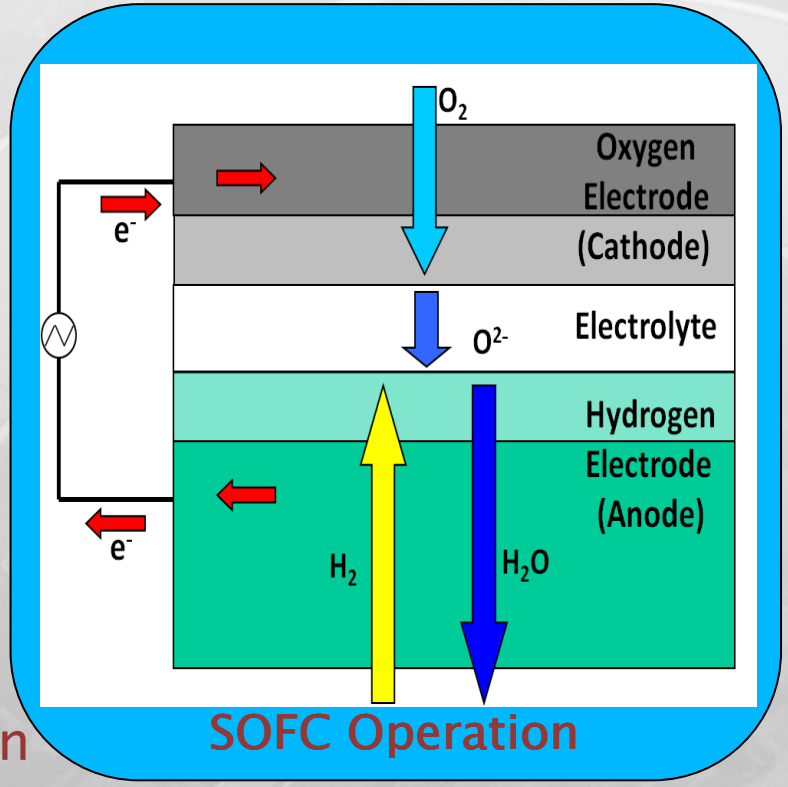


- Zoom *non*-destructively into a region of interest of a device, tissue,...
- Keep sample preparation and measurement straightforward
- Magnified phase contrast imaging
  - Quantitative reconstruction of the electron density
  - Very high sensitivity

X-ray *nano*-tomography of materials for energy production

Solid Oxide Fuel Cells (SOFCs):

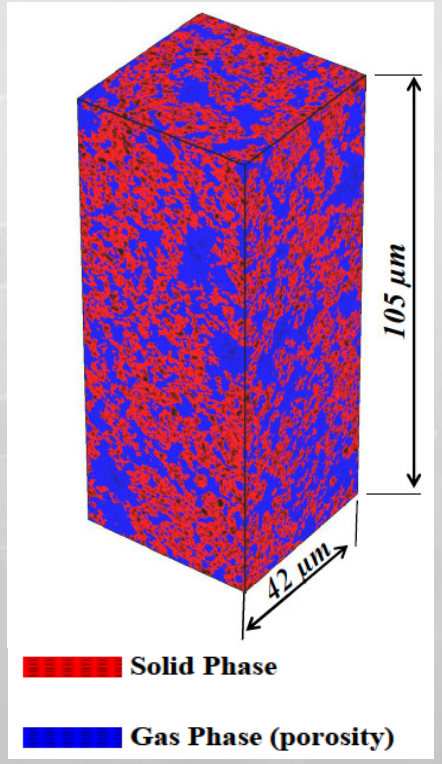
- High-performance electrochemical devices for energy conversion
- Reduced greenhouse gas
- Made of ceramic materials
- Operated at high T (~800°C)



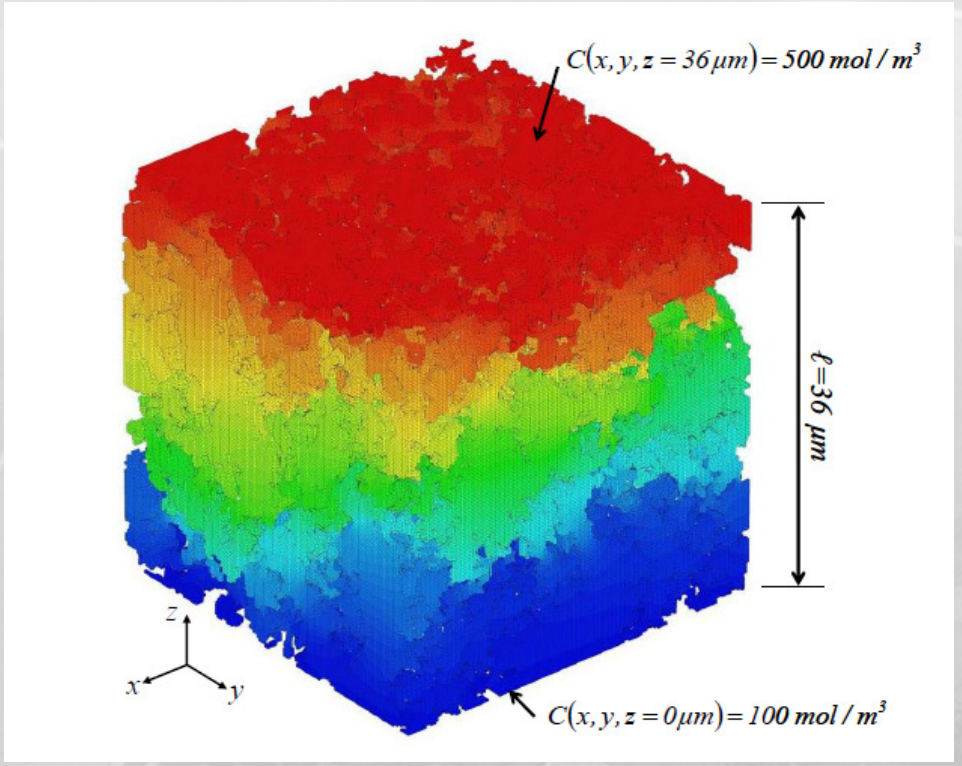
Very high efficiency but rapid degradation
Reasons related to high T operation:

- residual stresses
- elements diffusion
- *microstructure evolution*

X-ray Nano-Tomography of SOFCs



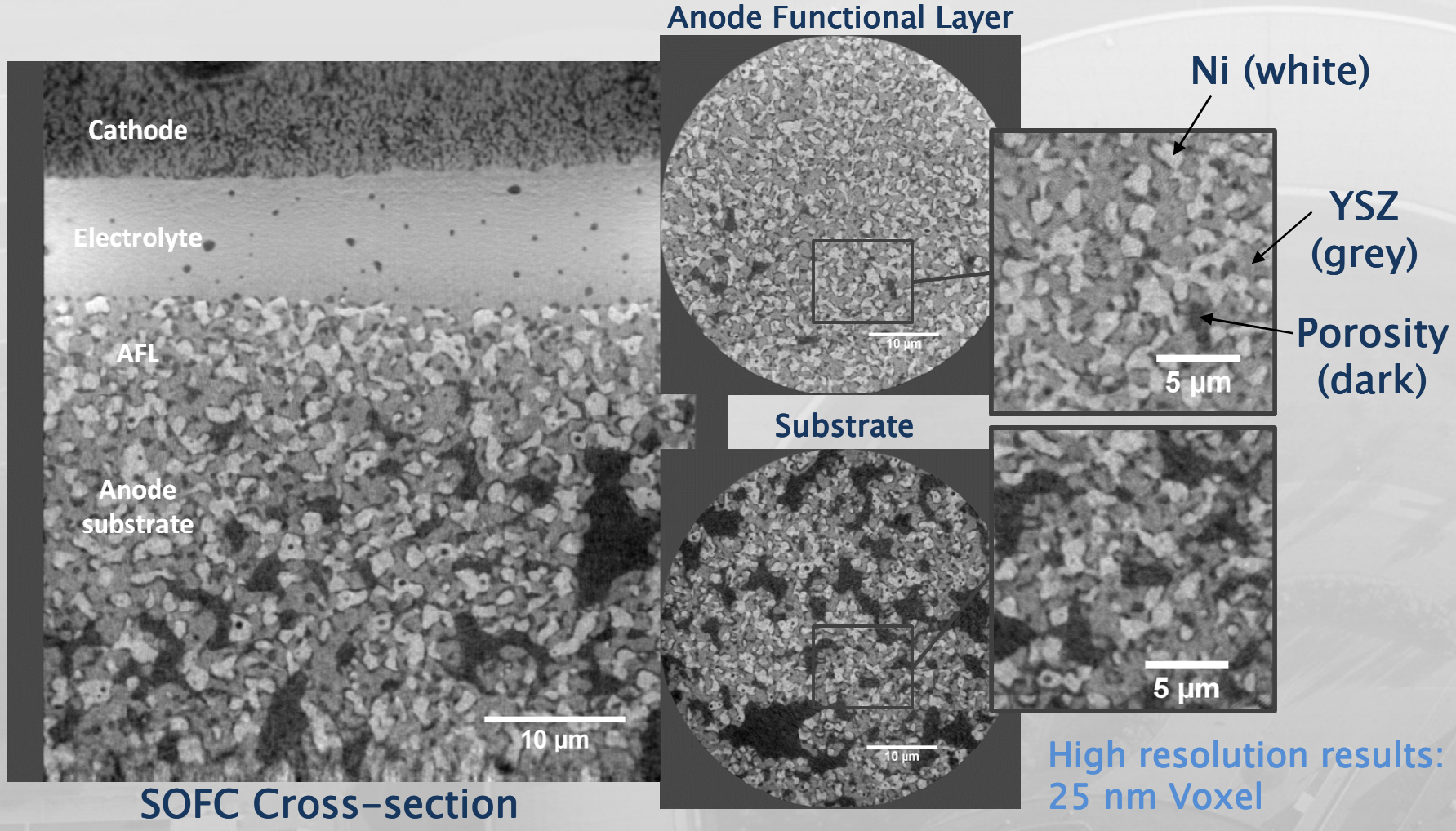
Reconstructed 3D microstructure



Concentration field in the percolated porosity

- Porous network in the hydrogen electrode
- Possibility to calculate the gas diffusion and transport in the anode

X-ray Nano-Tomography of SOFCs

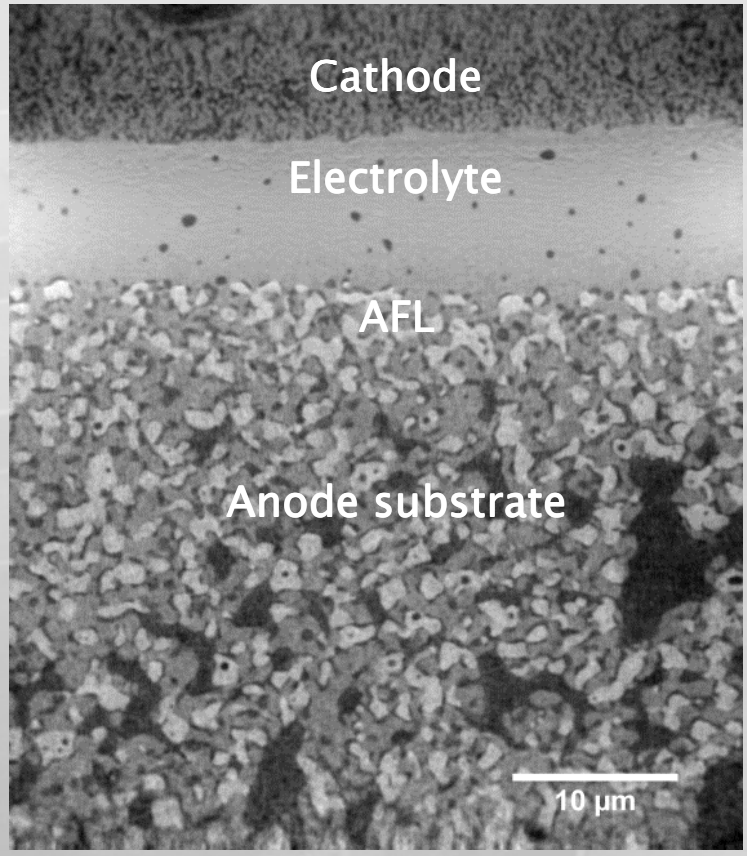


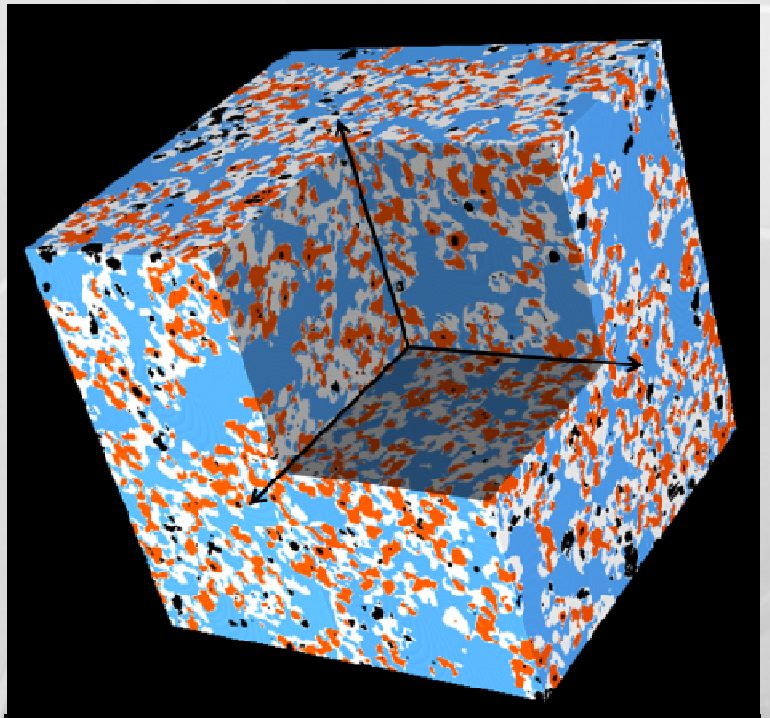
SOFC Cross-section

High resolution results:
25 nm Voxel

- Individual phases (Ni /YSZ) and micro and nano-porosity revealed

X-ray Nano-Tomography of SOFCs



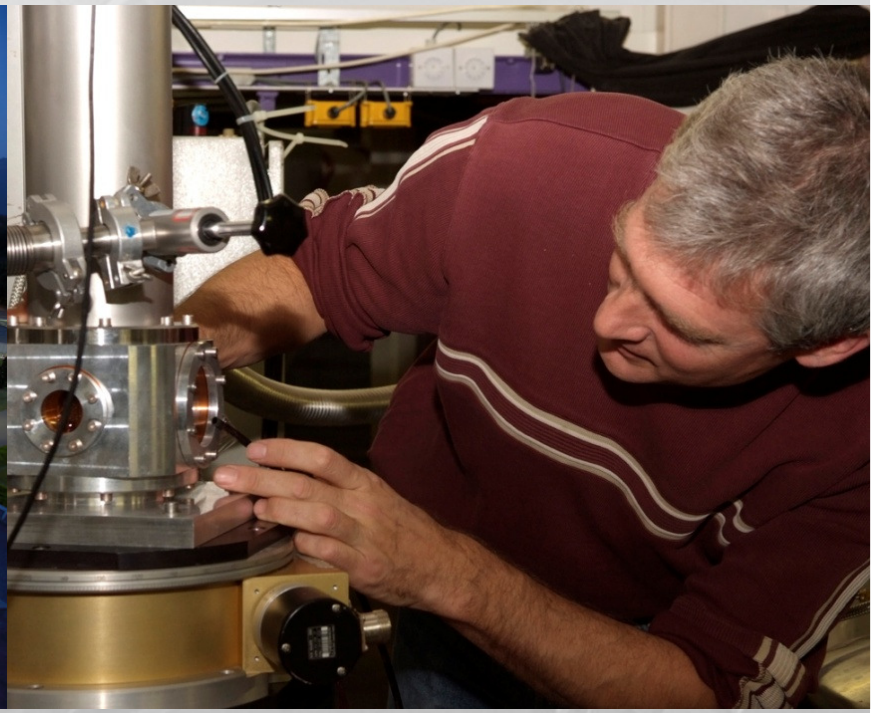


3D reconstruction after segmentation: 36x36x36 µm^3 (red: Ni, white: 8YSZ, blue: pores, black: non-percolating network)

- For the first time, the 3D reconstruction of the 3 phases (pores, Ni and YSZ) on a representative volume was obtained

Upgrades of existing sources and future storage rings for synchrotron radiation (ESRF, APS, Spring 8, ...)

New, better science



Upgrade Programmes: X-ray *nano*-beams for science

Objective: new, better science in areas linked to societal challenges and to the development of new key materials.

New instruments and a brighter and more stable source for users

	Advanced materials	Health & life sciences	Energy research
Nano-science and *nano*-technologies	●		●
Pump-probe experiments & time-resolved science	●	●	●
X-ray imaging for the *nano*-world	●	●	
Structural biology and soft matter	●	●	
Science at extreme conditions	●		●

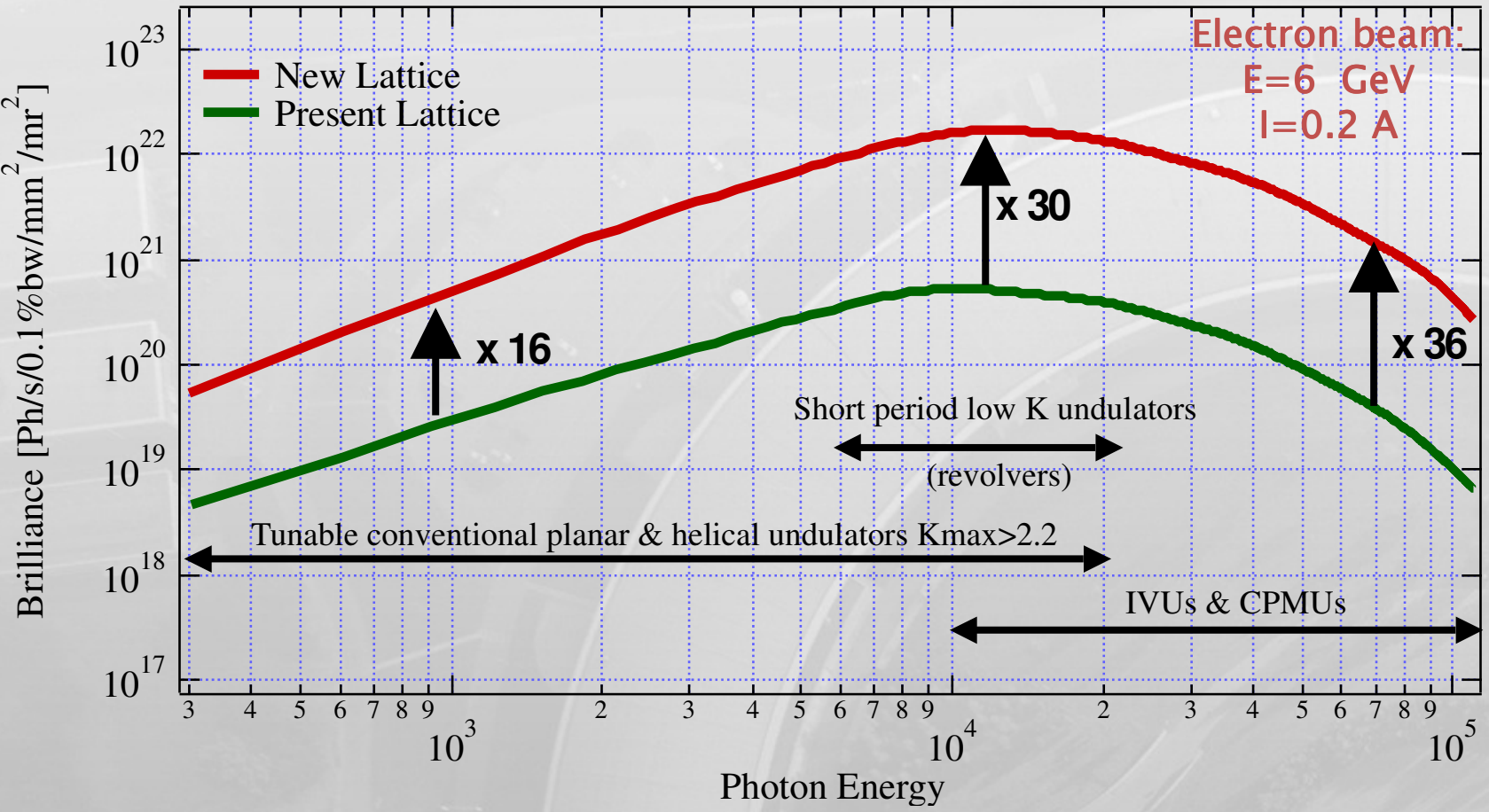
Upgrade Programmes

Four Pillars:

- Accelerator and source upgrades
  - Stability and reliability (orbit feedbacks, RF, ...)
  - Increased Brightness (Ids, current, ε_x, ...)
  - Timing and time resolution to the *p*s timescale
- New state-of-the-art beamlines
- Enabling technologies
  - Optics, detectors, precision *micro*- and nano- mechanics
  - Data handling, real-time analysis and storage
  - Automation, remote control, ...
- Experimental Hall extension for long beamlines

ESRF Upgrade Programme
Phase I (2009–2015): well advanced
Phase II (2015–2019): proposal in preparation

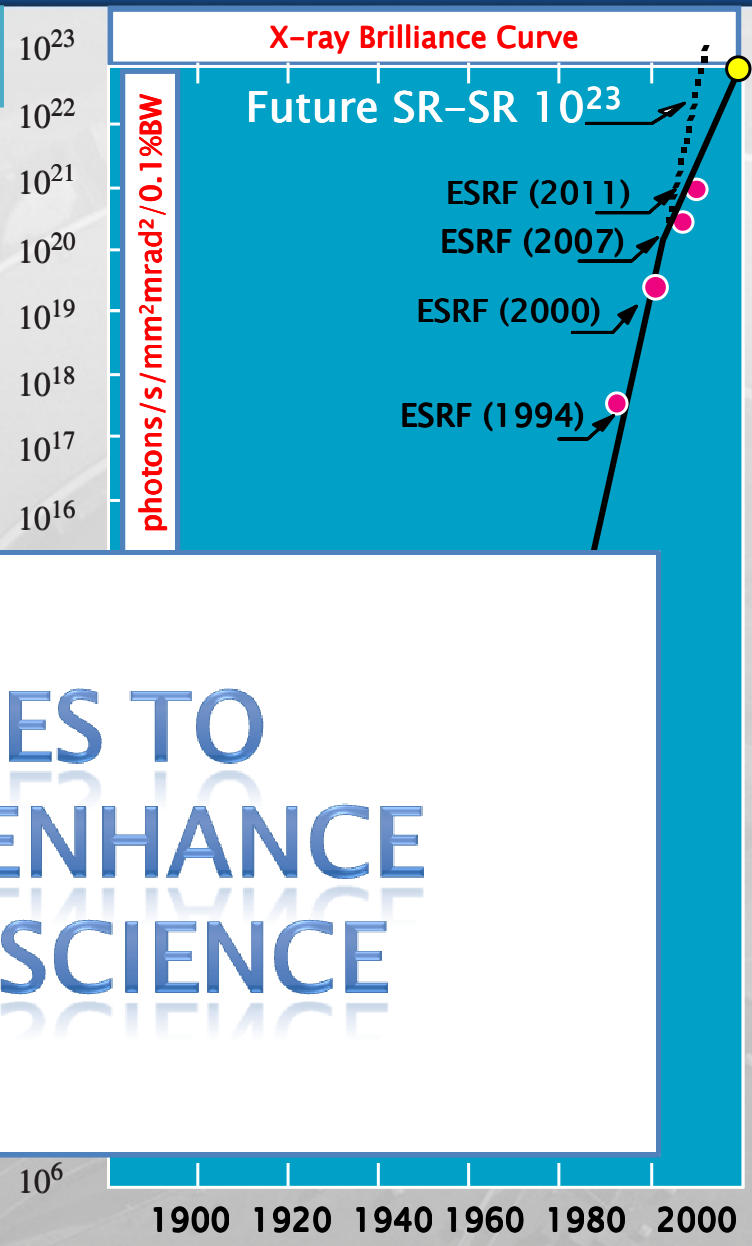
- Accelerator and source
- Beamlines
- Enabling technologies

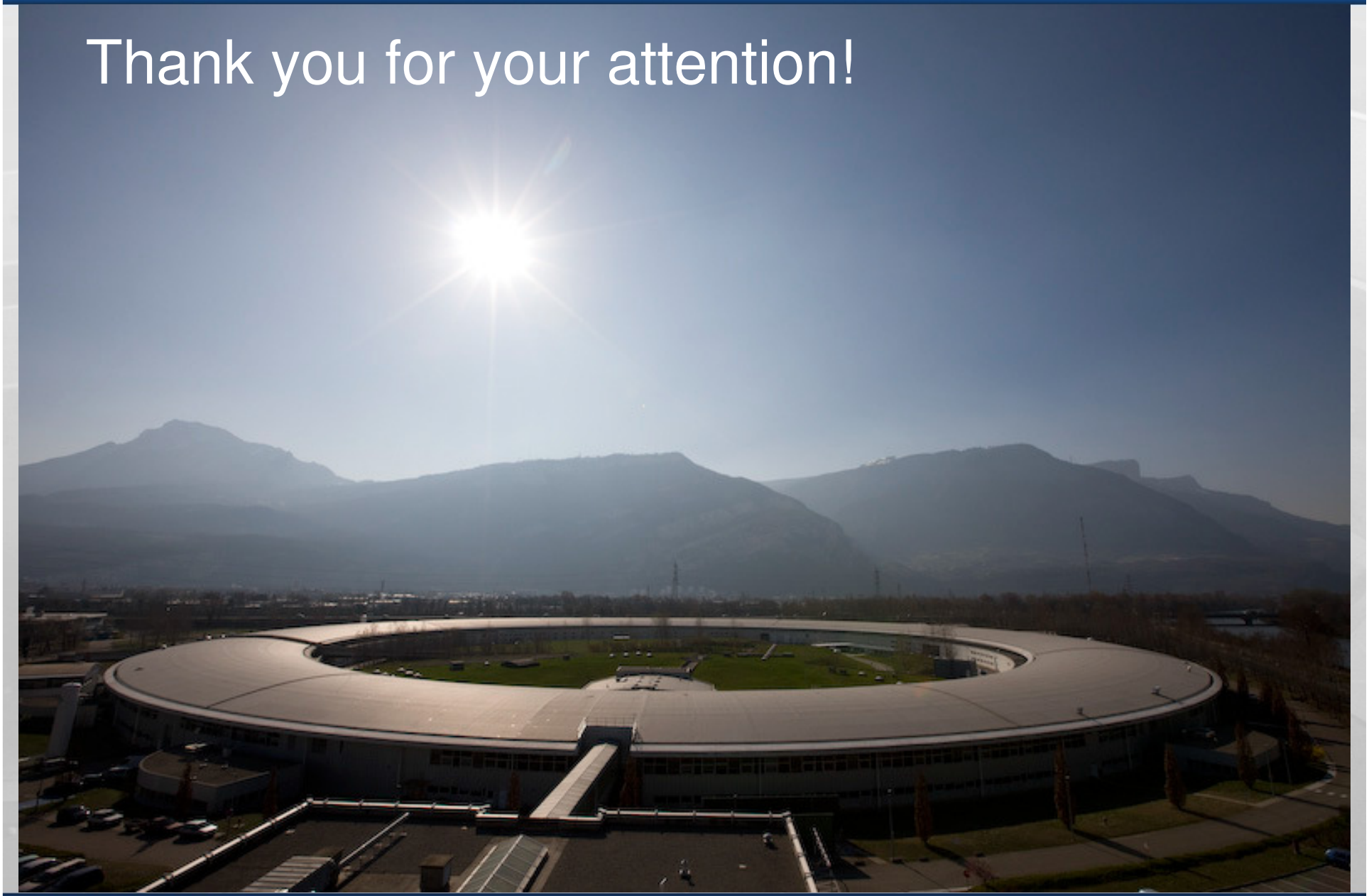


	Emittance	Coulping [%]	Energy spread [%]
Present	4 nm	0.12	0.1
New lattice	0.13	1	0.09

The SR Grand Challenges

- To be increasingly useful:
  - Powered by scientific excellence
  - Faithful and committed Users' service
- To enlarge the Users' Community:
  - More then PHOTONS!
- C
  -
  -



OPPORTUNITIES TO QUALITATIVELY ENHANCE SYNCHROTRON SCIENCE

Thank you for your attention!