ESRF

The European Synchrotron

Introduction to Synchrotron Radiation

Ray Barrett

X-ray Optics Group

Instrumentation Services and Development Division

European Synchrotron Radiation Facility

OUTLINE OF THE PRESENTATION

Introduction

- The electromagnetic spectrum
- A brief history of X-ray science and synchrotron radiation
- Synchrotron radiation (SR) facilities world-wide

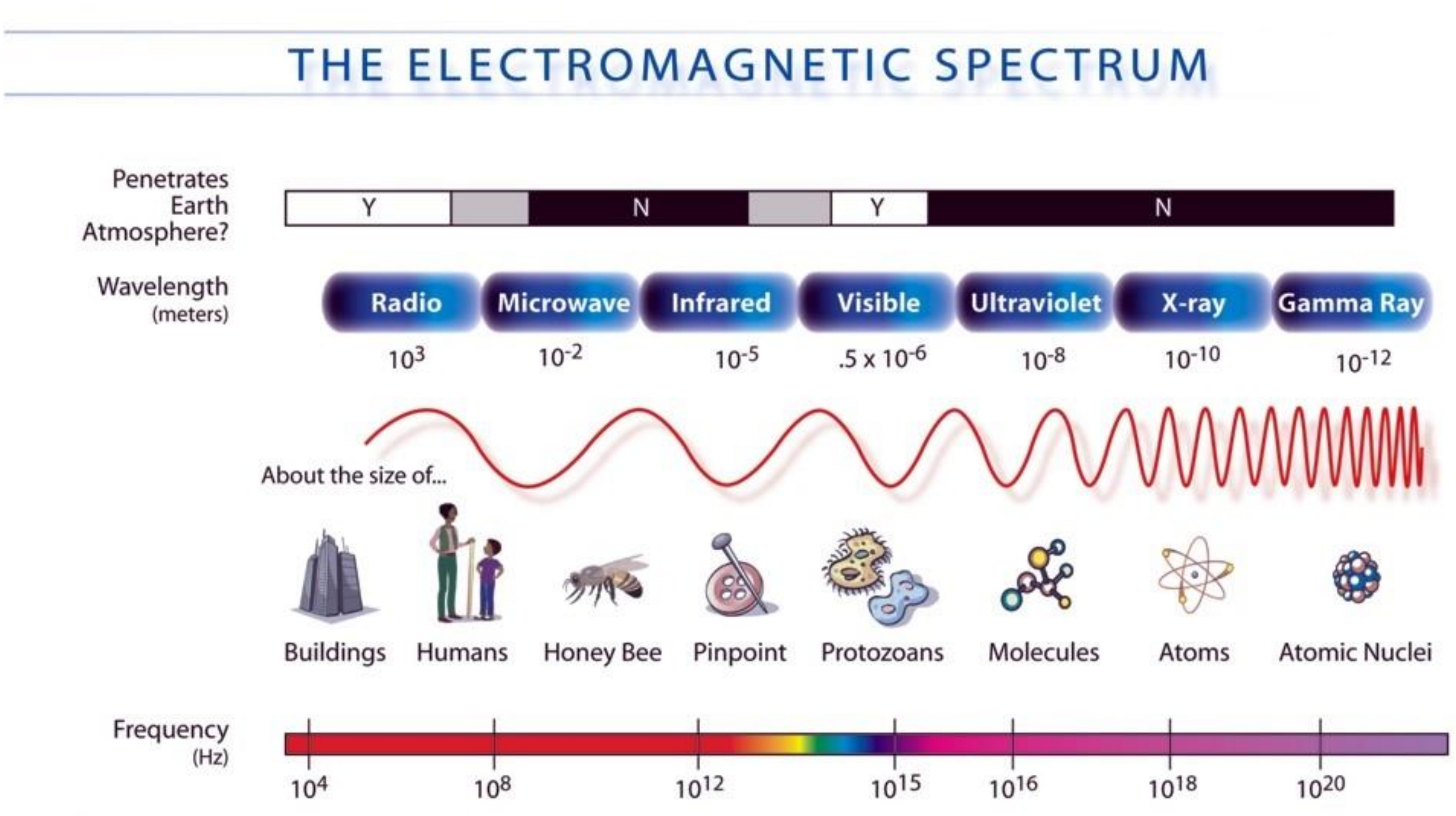
Production of synchrotron X-rays

X-ray beam transport, sample, and x-ray detection

X-ray techniques and applications

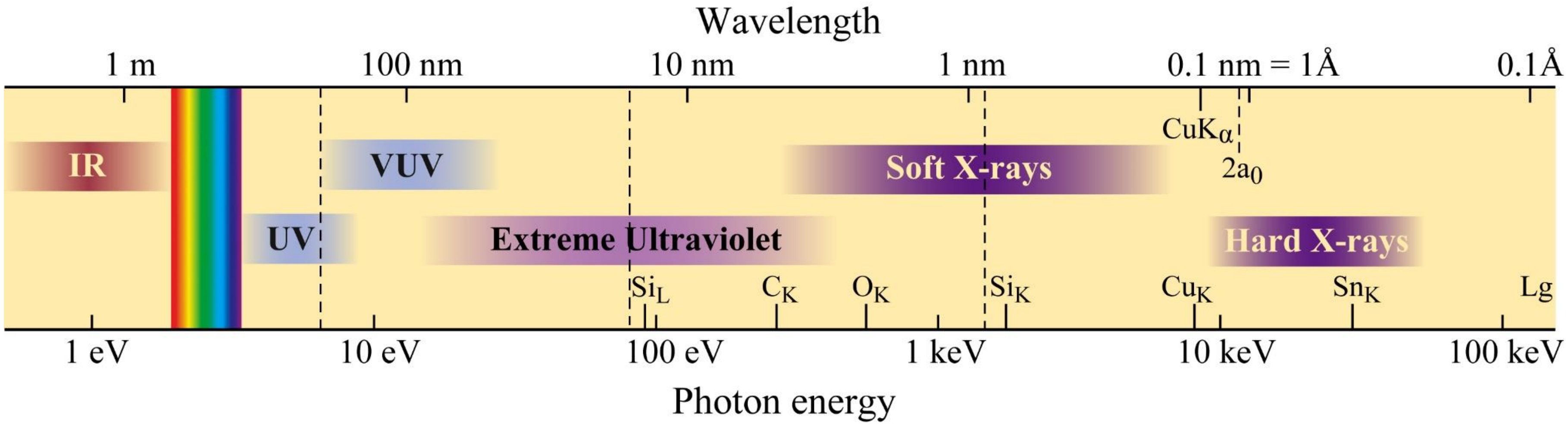
- Diffraction and scattering
- Spectroscopy
- Imaging

LIGHT



The European Synchrotron | ESRF

X-RAYS: THE SHORT WAVELENGTH REGION OF THE ELECTROMAGNETIC SPECTRUM



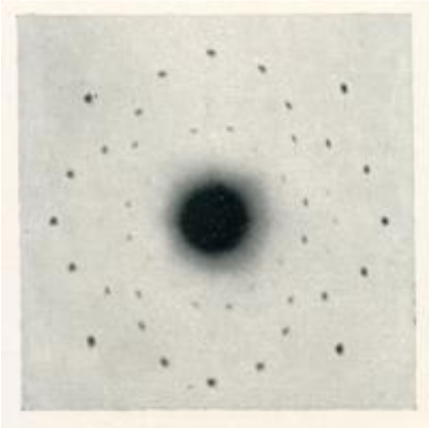
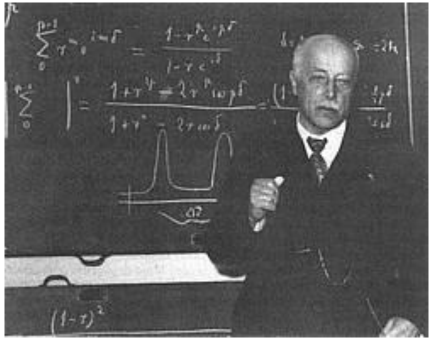
X-rays …..penetrate visible opaque objects
probe matter down to the atomic scale
allow for elemental, chemical, magnetic, site selectivity
can be focused down to 10 nm

$$\hbar\omega \cdot \lambda = hc = 1239.842 \text{ eV nm}$$

$$n = 1 - \delta + i\beta \qquad \delta, \beta << 1$$

X-RAY SCIENCE: A LONG SUCCESS STORY WHICH STARTED IN 1895

Wilhelm Conrad Röntgen (1845-1923)

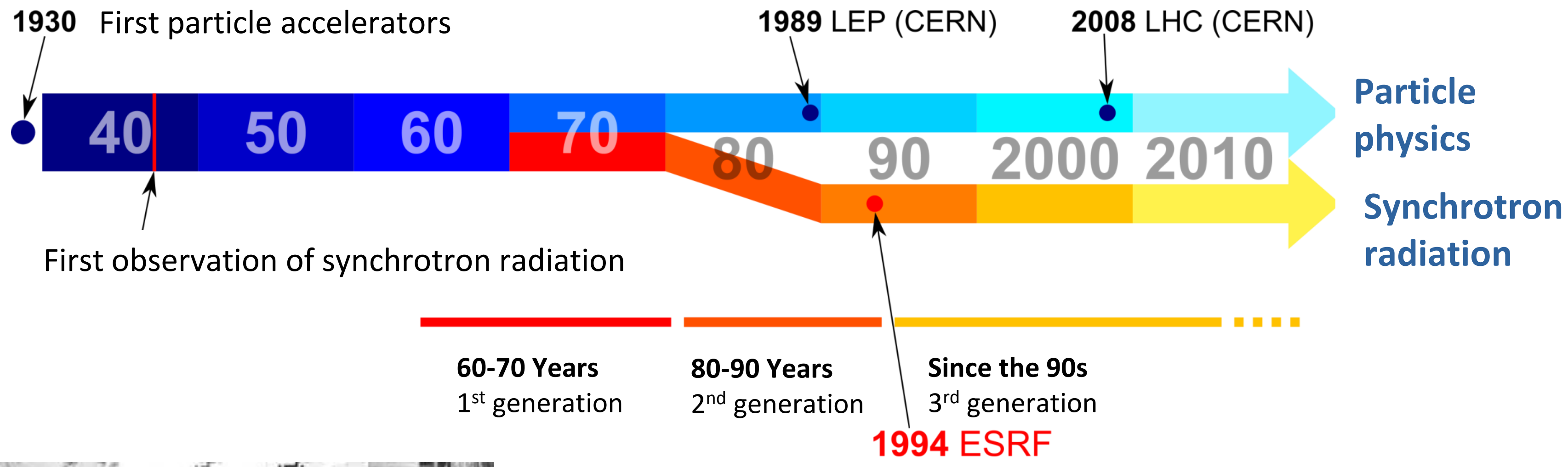



Max von Laue (1879-1960)

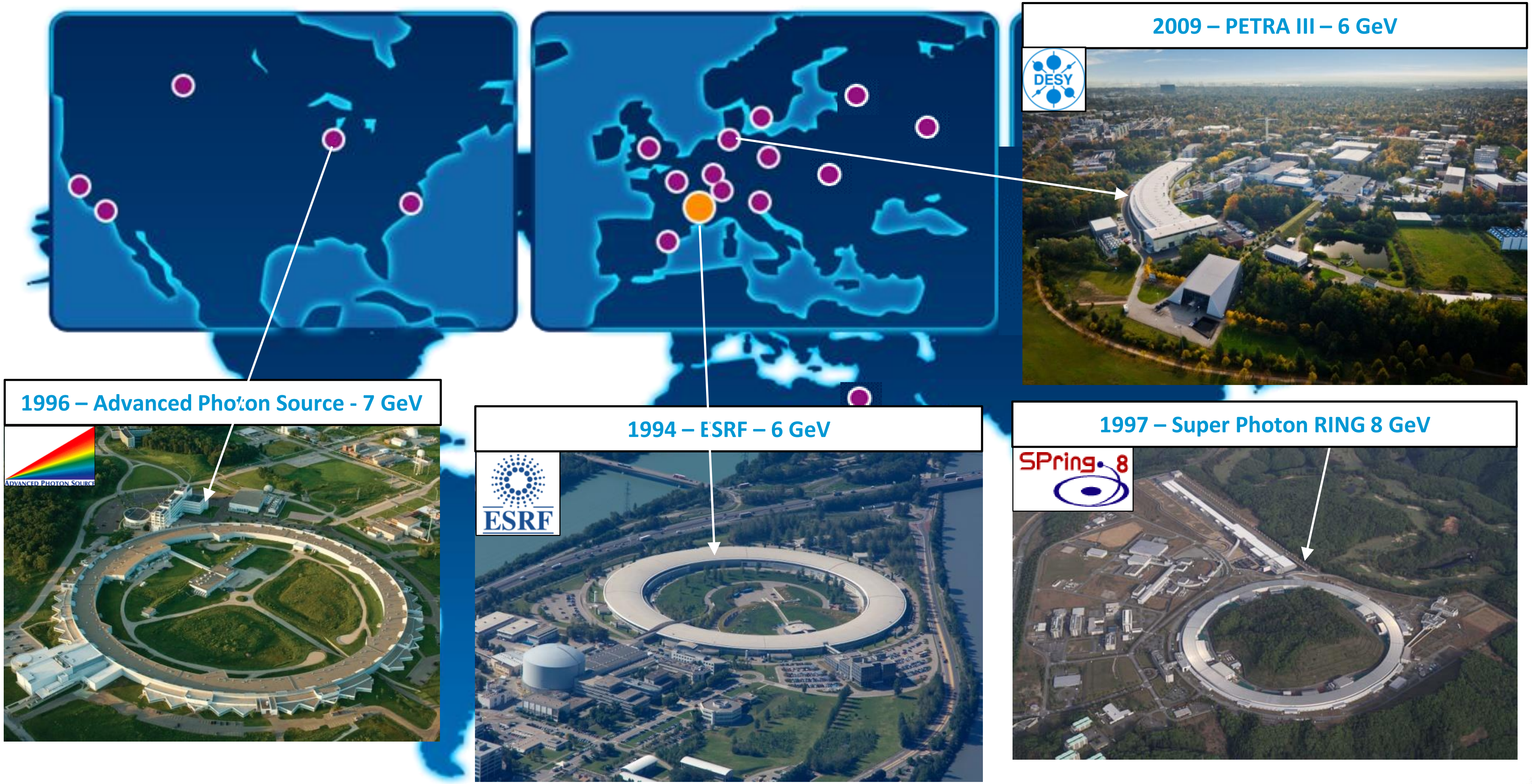
1947: First observation of synchrotron radiation at General Electric (USA).

..followed by decades of parasitic use of Synchrotron radiation on high-energy machines

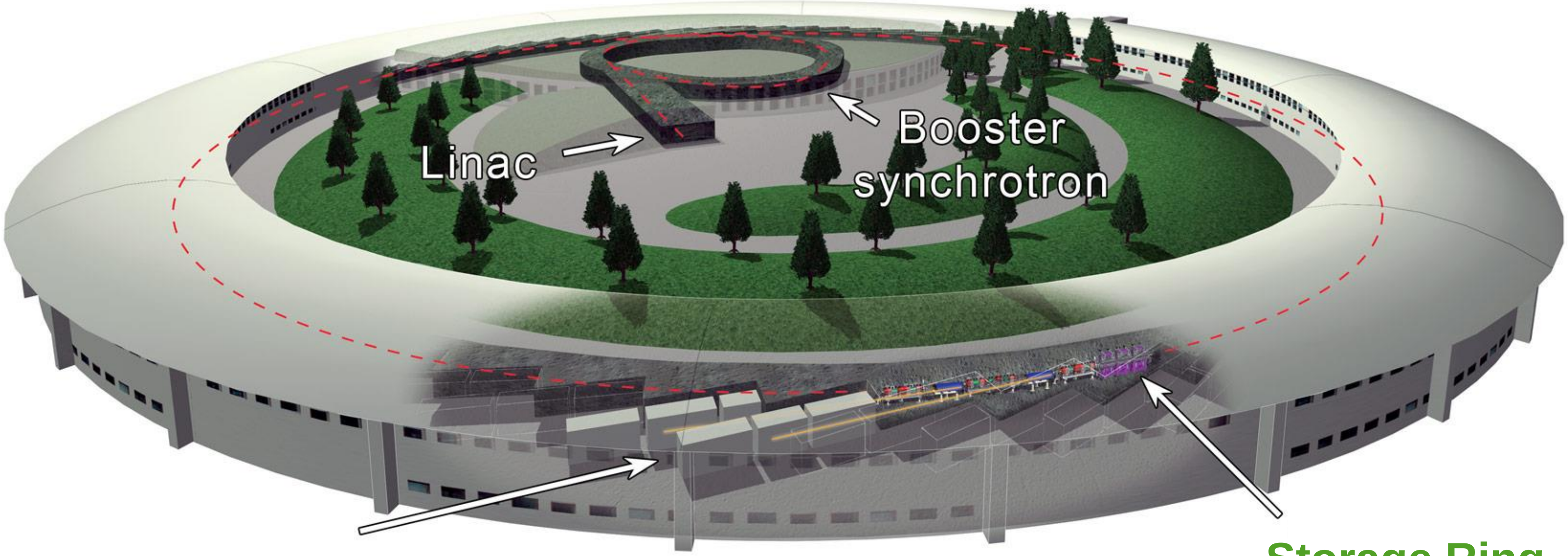
HISTORY OF SYNCHROTRON RADIATION SOURCES



MAJOR THIRD GENERATION SYNCHROTRON FACILITIES WORLDWIDE



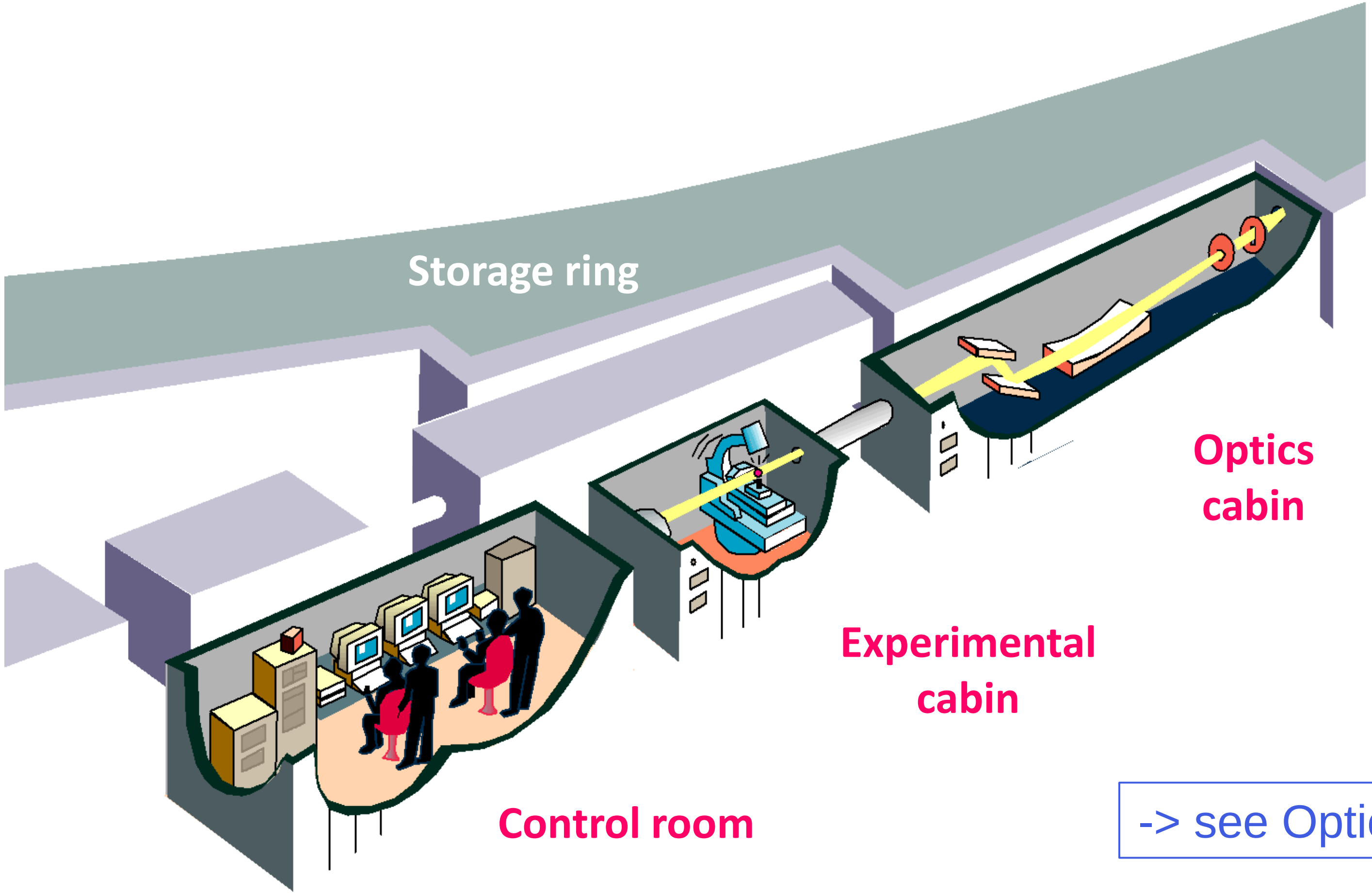
OPERATION OF A SYNCHROTRON SOURCE



Lines of light = beamlines

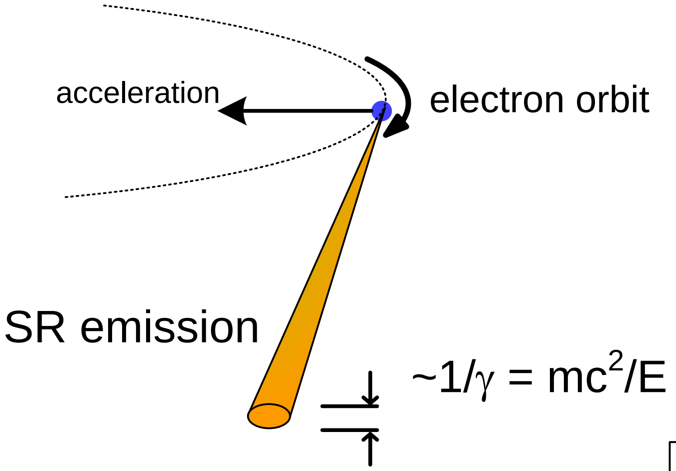
Storage Ring

A TYPICAL SYNCHROTRON BEAMLINE



-> see Optics lecture

Synchrotron radiation is produced by relativistic charged particles accelerated by magnetic fields. It is observed at particle accelerators.



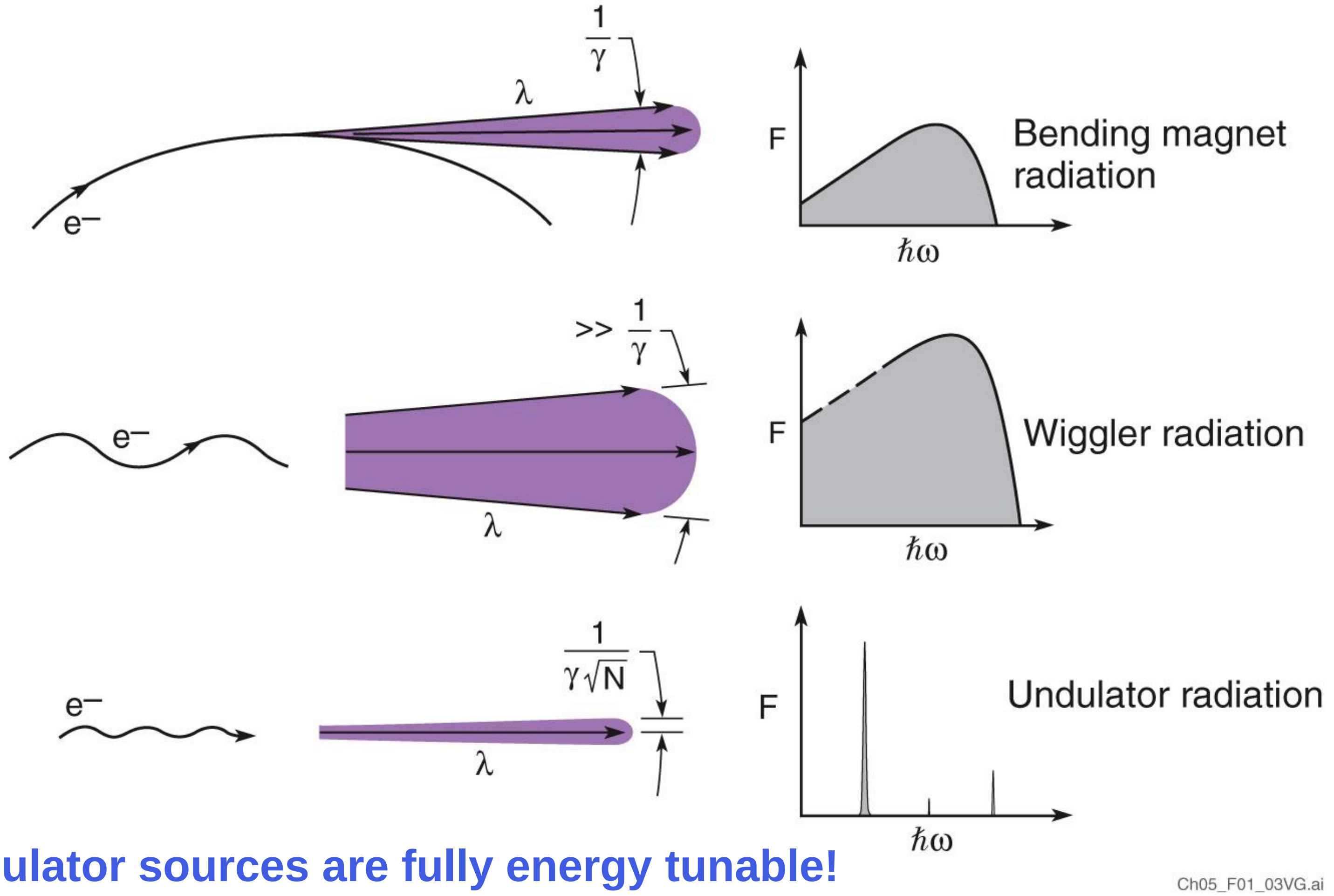
$$E_e = \gamma m c^2$$

$$\gamma = \frac{1}{\sqrt{1-\left(\frac{v}{c}\right)^2}}$$

BESSY II:	E_e = 1.7 GeV,	γ = 3 300,	s' = 330 μrad
ESRF:	E_e = 6 GeV,	γ = 11 800,	s' = 85 μrad

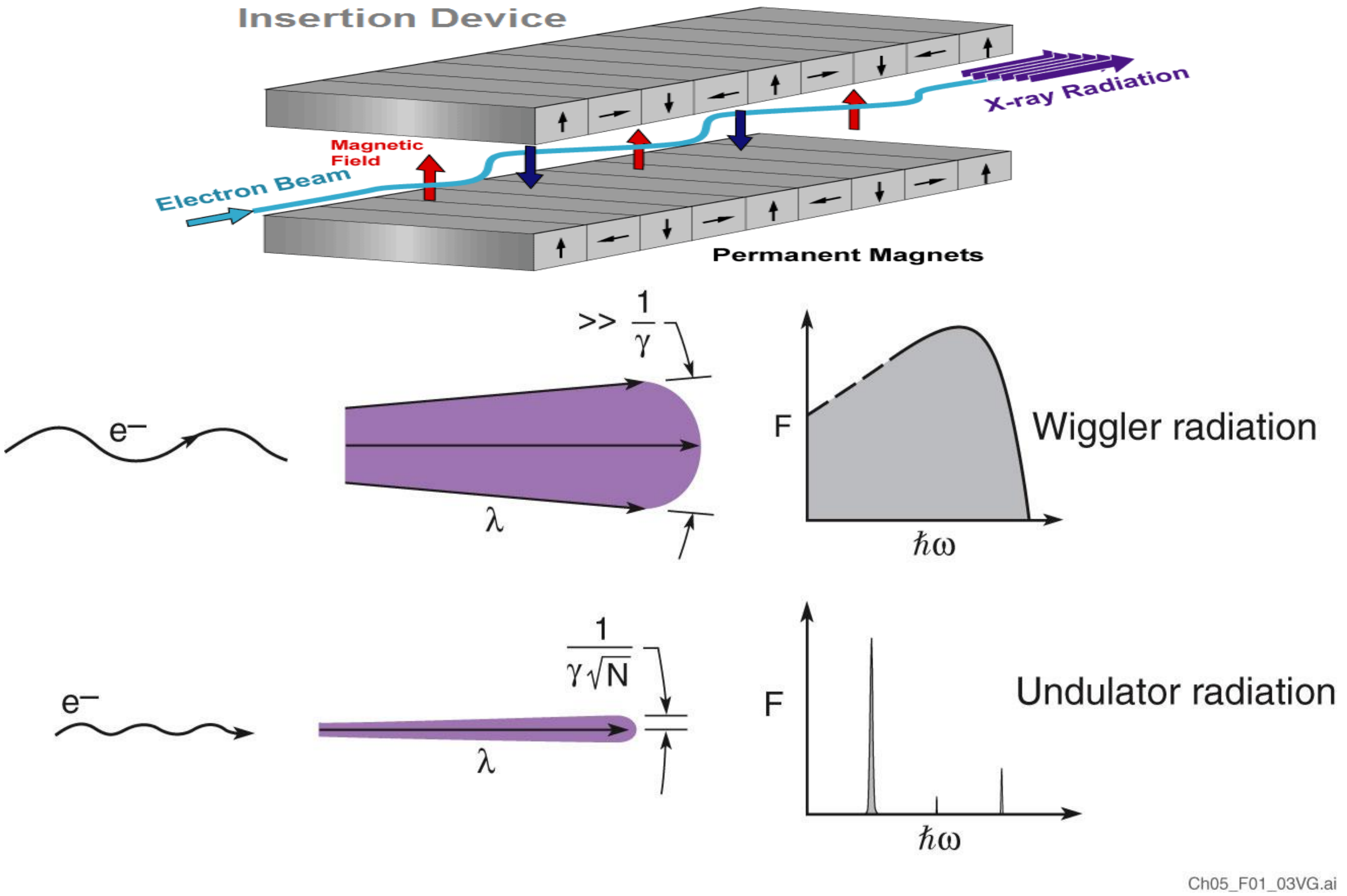
The emission is concentrated in the forward direction

3 DIFFERENT SOURCES OF SYNCHROTRON RADIATION



Undulator sources are fully energy tunable!

3 DIFFERENT SOURCES OF SYNCHROTRON RADIATION



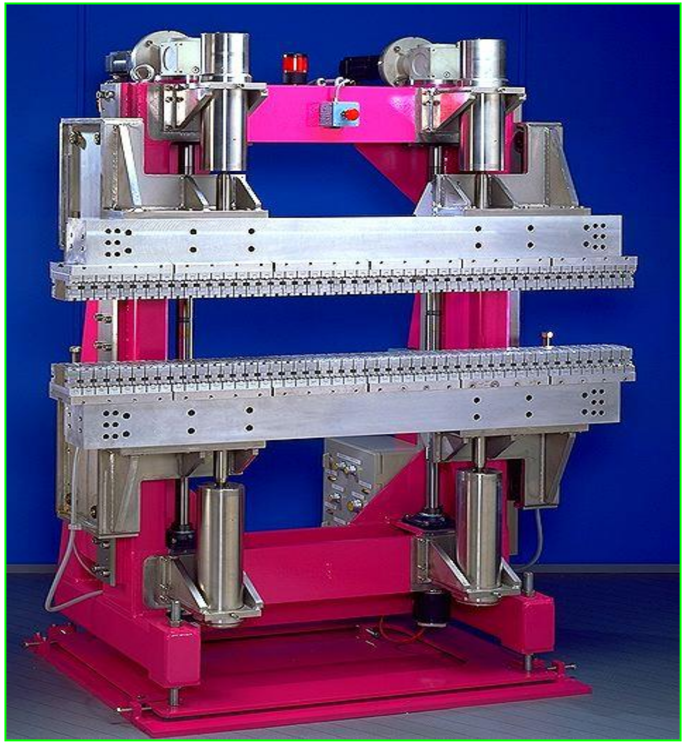
PERMANENT MAGNET UNDULATORS

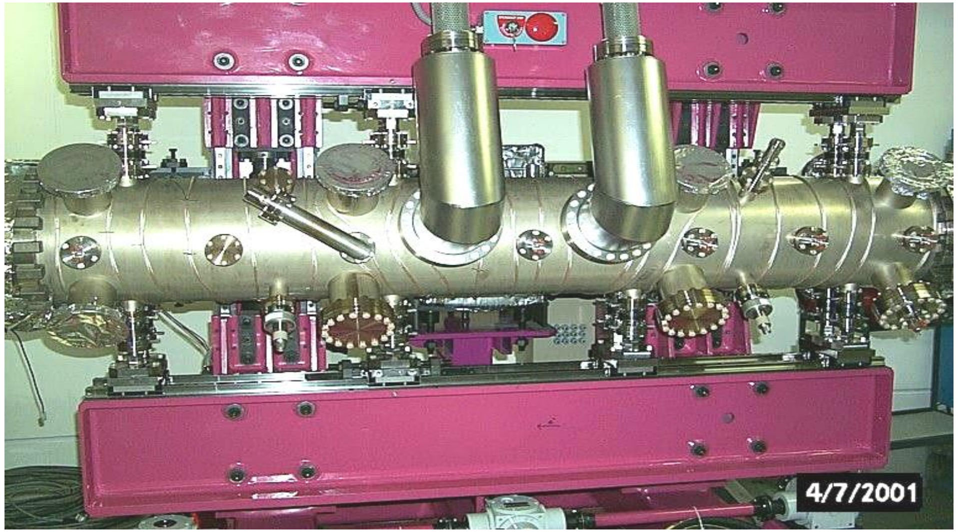
Arrays of rare earth magnets (NdFeB, SmCo)

Standard undulators

In-vacuum

Cryogenic





SYNCHROTRON RADIATION – THE FIGURES OF MERIT

SR Emittance = $\Delta A \cdot \Delta\Omega$

ΔA: source area
$\Delta\Omega$: solid angle

units: [nm·rad] -> [pm·rad]

$$\text{SR Brilliance} = \frac{\text{photon flux}}{\Delta A \cdot \Delta\Omega \cdot \Delta\lambda/\lambda}$$

$\Delta\lambda/\lambda$: spectral bandwidth

units: $\frac{\text{number of photons}}{\text{s}\cdot\text{mrad}^2\cdot\text{mm}^2\cdot 0.1\%\text{bandwidth}}$

Both emittance and brilliance are **invariant quantities** in phase space → optical techniques cannot improve them!

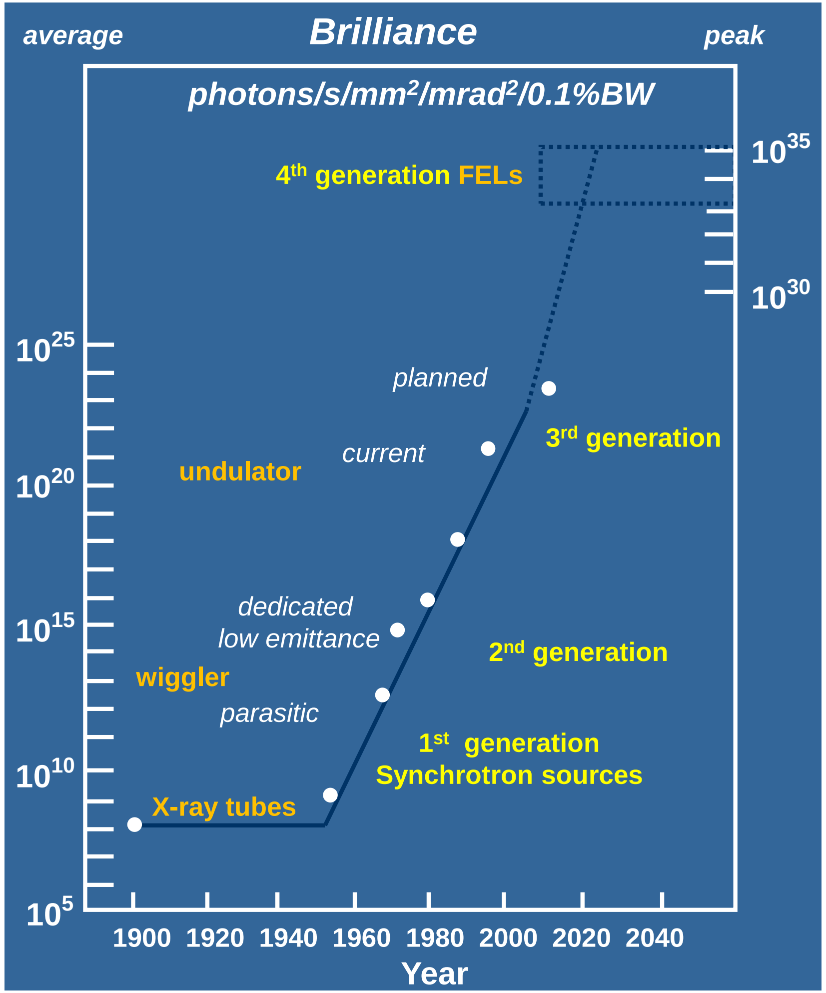
SR X-rays:

Large energy tunability (infrared -> γ-rays)

Polarisation tunability

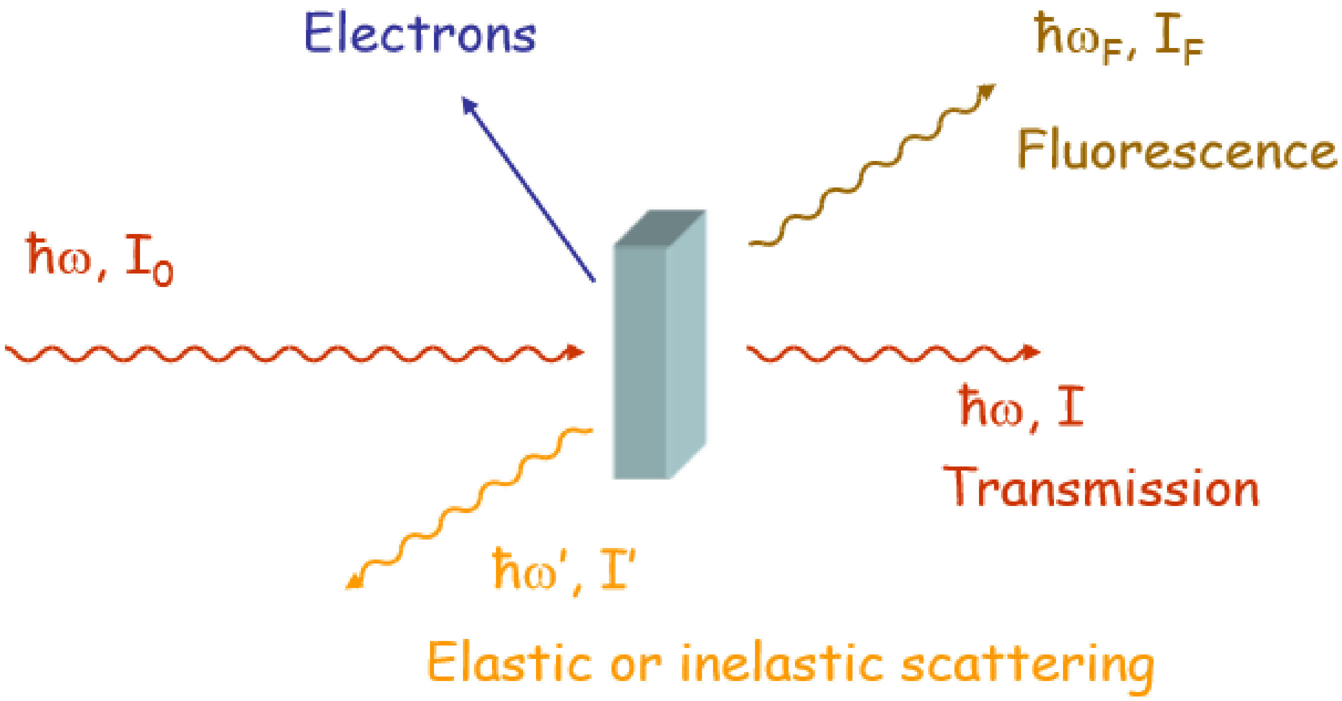
High spatial coherence

Pulsed emission (e^- bunches)



Fundamental and applied studies on materials and living matter

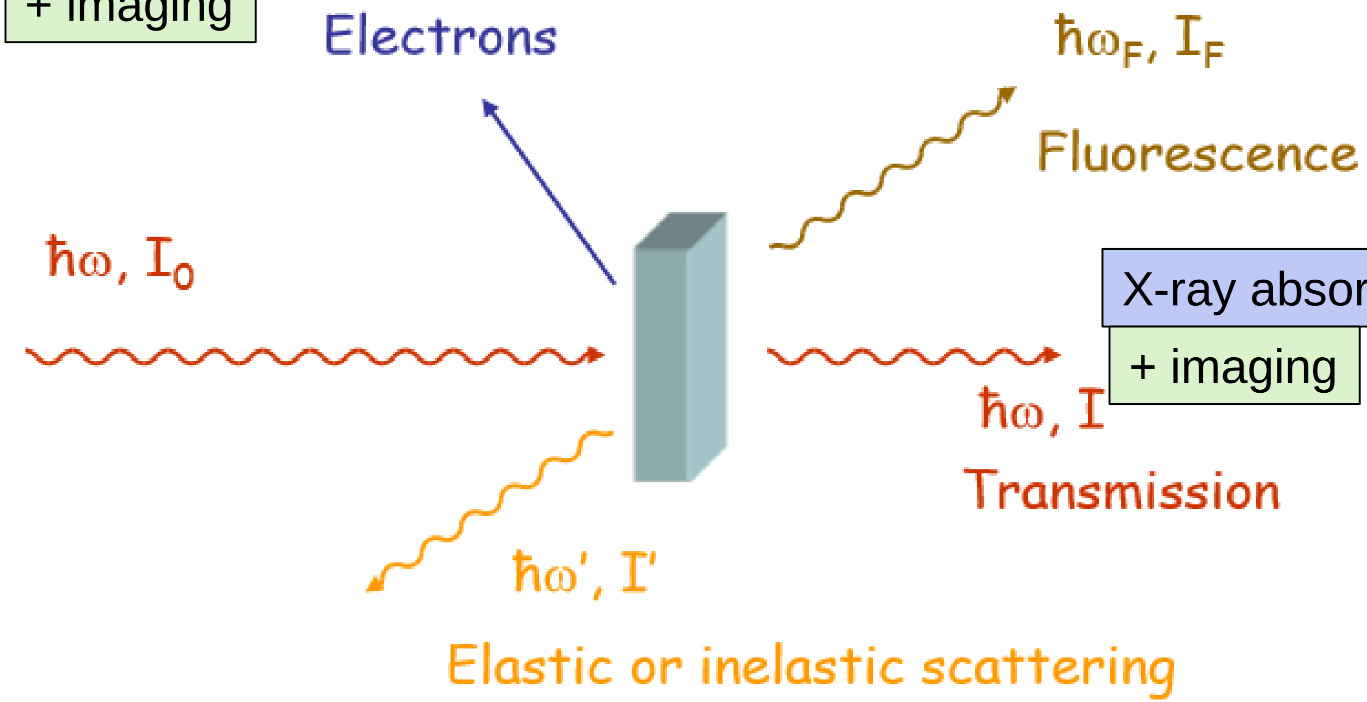
X-RAY – MATTER INTERACTION



X-RAY – MATTER INTERACTION: THE CLASSICAL TECHNIQUES

Photoemission spectroscopy

+ imaging



X-ray absorption spectroscopy

+ imaging

X-ray diffraction and elastic scattering

+ imaging

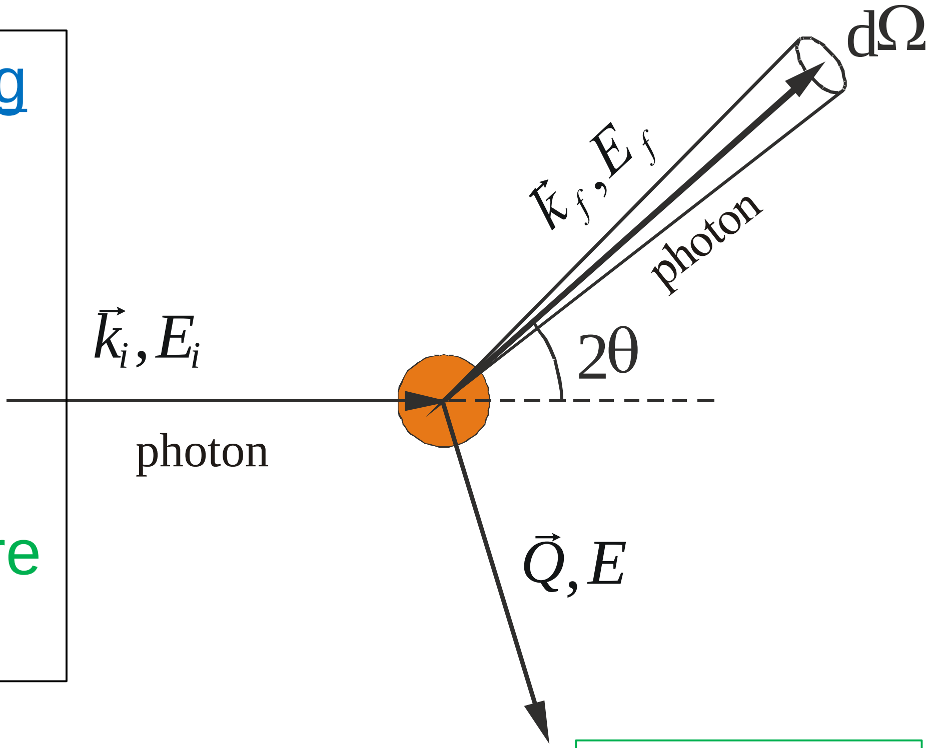
Elastic scattering

$E_f = E_i$

$|\vec{k}_f| = |\vec{k}_i|$

Q = 4π/λ·sin(θ)

"determine where the atoms are"



$|\vec{k}_i| = 2\pi/\lambda$

$\vec{Q} = \vec{k}_f - \vec{k}_i$

$E = E_f - E_i$

Inelastic scattering

$E_f < E_i$

$|\vec{k}_f| < |\vec{k}_i|$

Q = 4π/λ·sin(θ)

E = E_f - E_i

"determine where the atoms are and how they move"

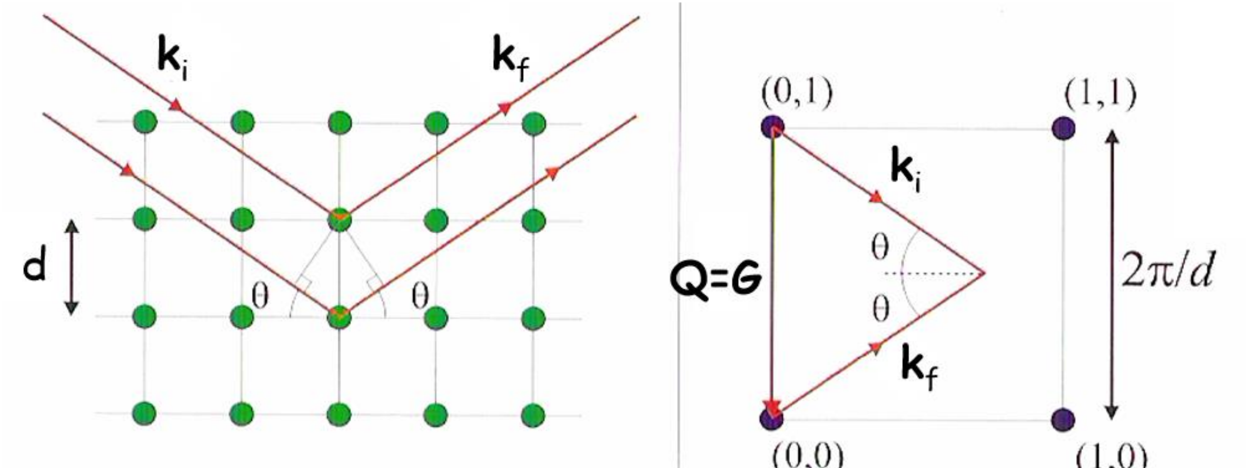
CRYSTAL DIFFRACTION: BRAGG'S LAW AND LAUE CONDITION

A **single crystal** is a **periodic array of atoms in 3D**.

Description as the Fourier transform of a 3D array of **δ-functions** in **reciprocal space**: $G_{hkl}(\vec{Q})$ with integers h,k,l (Miller indices).

Diffraction only occurs when $\vec{\boldsymbol{Q}} = \vec{\boldsymbol{G}}$: **Laue condition**

This is geometrically equivalent to $\mathbf{n \cdot \lambda = 2d_{hkl} \cdot sin\theta}$: **Bragg law**



d is the lattice spacing of planes hkl

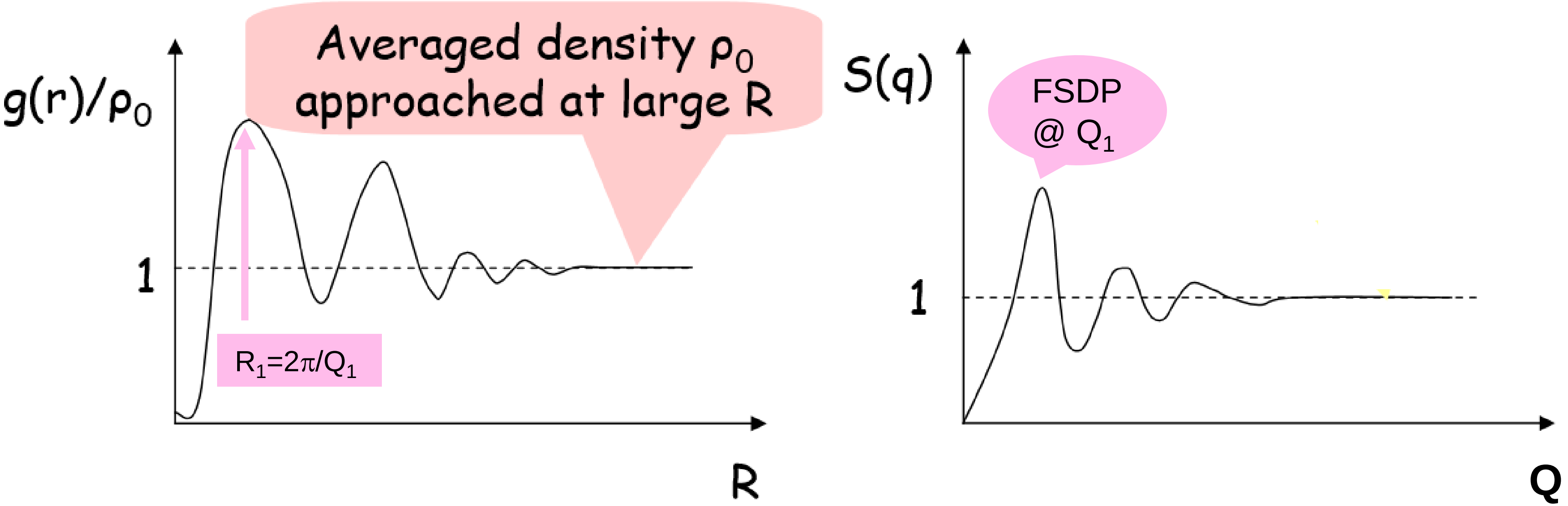
$|\vec{Q}| = 4\pi \cdot \sin(\theta)/\lambda$

or

$|\vec{Q}| = 2\pi/d_{hkl}$

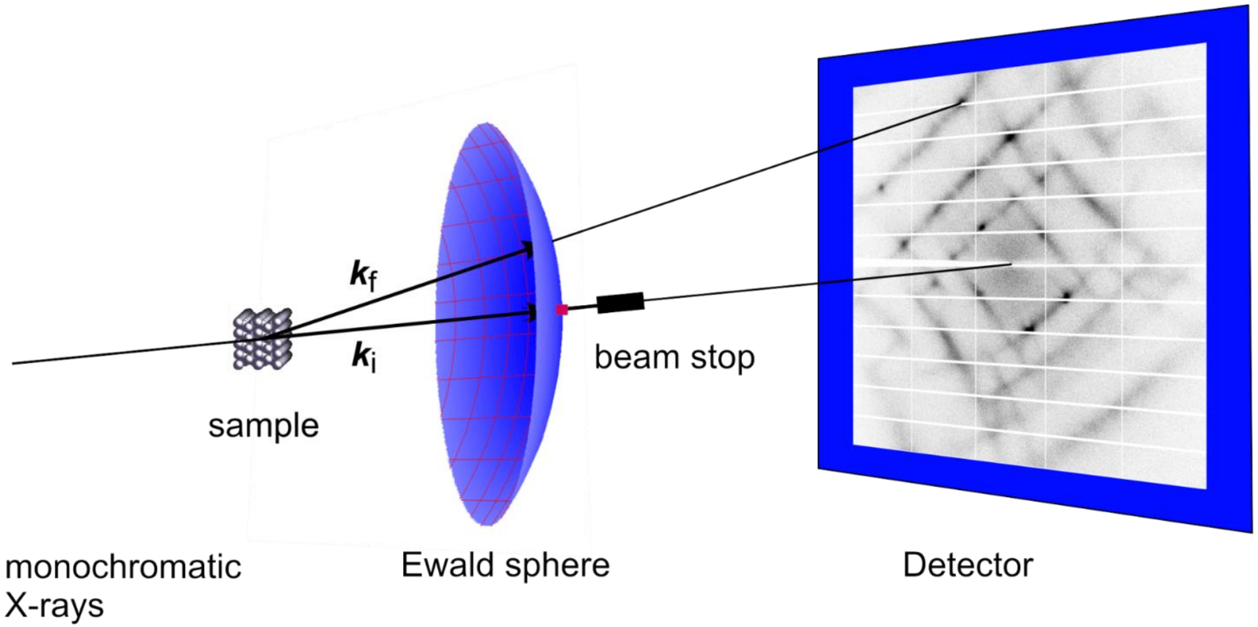
The **disordered structure** is described by the **pair correlation function g(r)** given by the probability of finding a particle at a distance r from another particle.

We determine the **static structure factor S(Q)**, which is the Fourier transform of g(r).



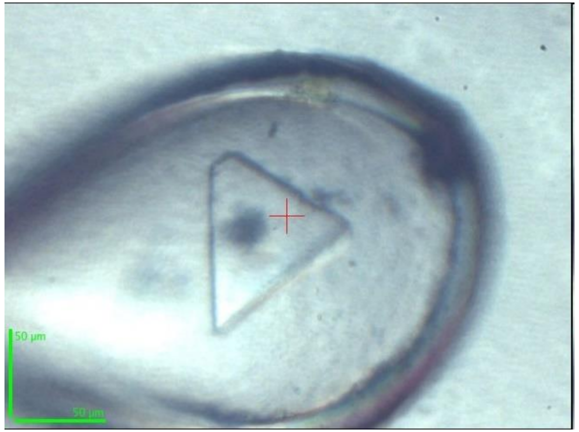
The first "sharp diffraction peak" (FSDP) at Q_1 corresponds to the average distance R_1 to neighboring atoms

X-RAY DIFFRACTION AND ELASTIC SCATTERING



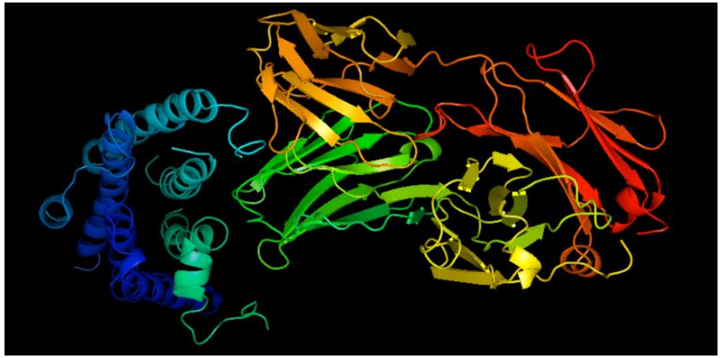
Large area, low noise, high dynamic range detectors are essential!

-> see lecture by Pablo Fajardo



G-Protein Coupled Receptors

800 different proteins controlling body functions and drug transit across membrane

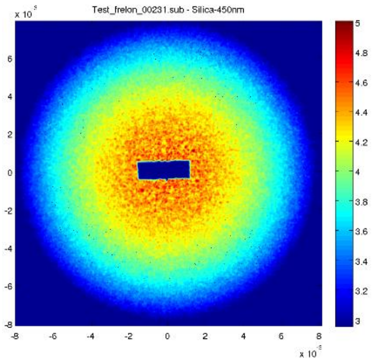


Crystals thought impossible to be made

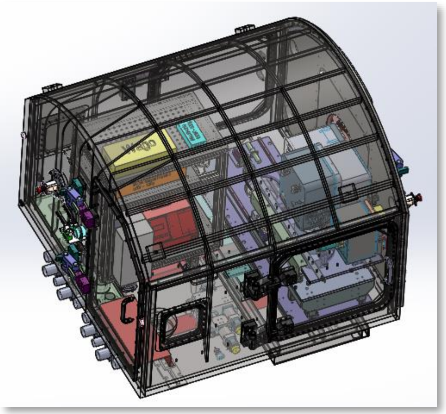
Rasmussen et al, Stanford & Cambridge

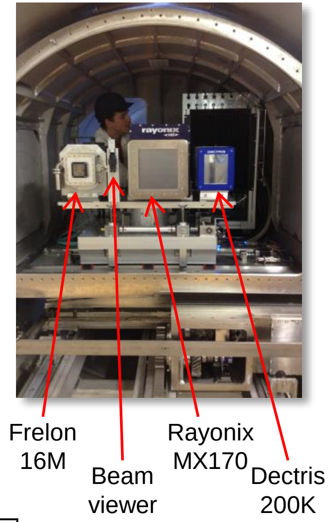
-> see EMBL lectures

ID02 – THE ULTIMATE SMALL ANGLE X-RAY SCATTERING STATION



Dilute silica colloids of 450 nm in size





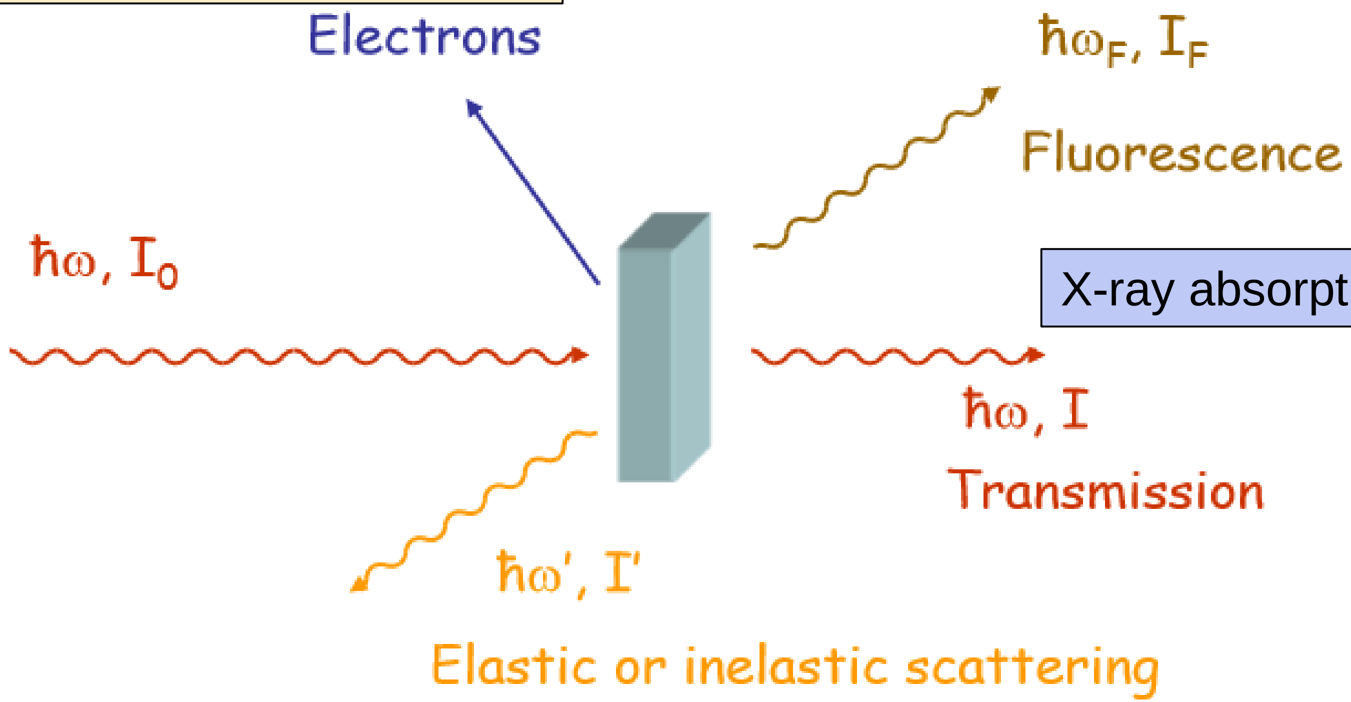
Large Q – range: 10^{-3} – 50 nm^{-1}

Typical length scales: 3 μm – 0.6 Å

X-RAY SPECTROSCOPY

Photoemission spectroscopy

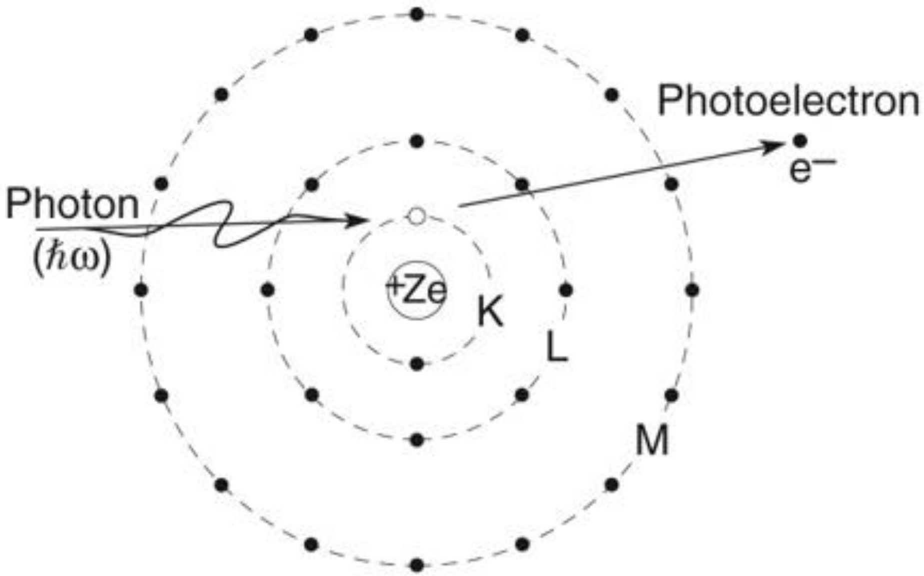
X-ray emission spectroscopy

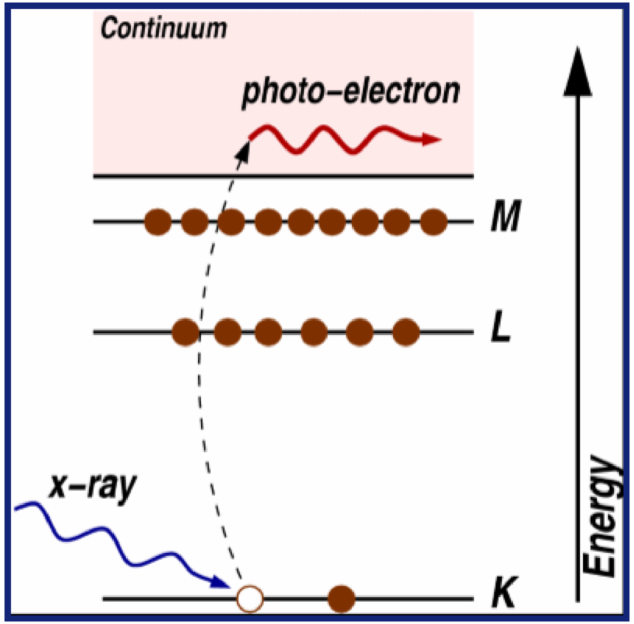


X-ray absorption spectroscopy

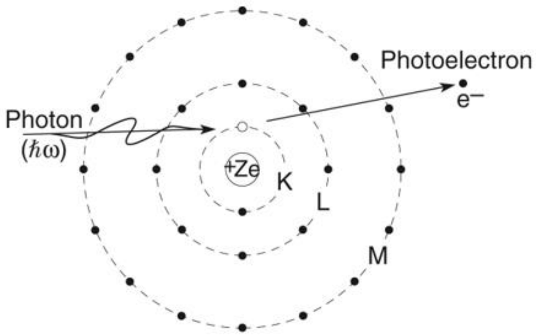
Resonant (in)elastic x-ray spectroscopy

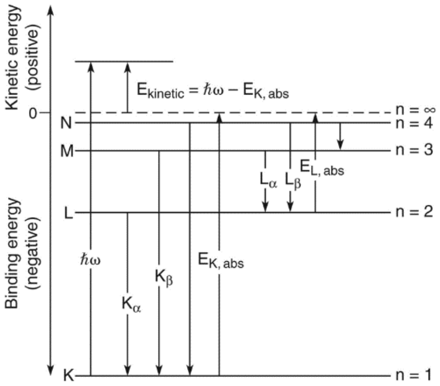
An X-ray is absorbed by an atom when the energy of the X-ray is transferred to a core-level electron (K, L, M, ….shell) which is ejected from the atom = photo-electric absorption





Coverage of many characteristic absorption edges of almost all elements





element selectivity: the characteristic binding energies are different for different elements

chemical sensitivity: for the same element the binding energy depends on the valence state

spin selectivity: the absorption coefficient is different for for circularily polarized X-rays in magnetic materials

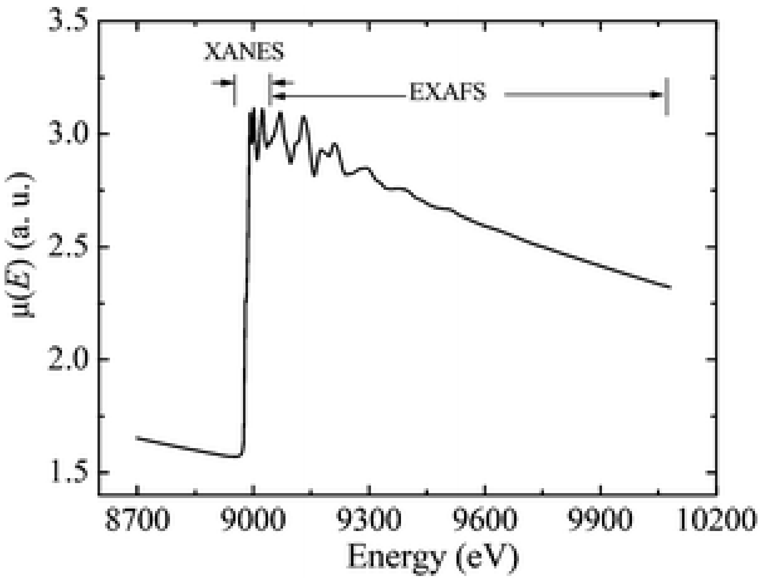
bulk sensitivity: photons, especially at high energies

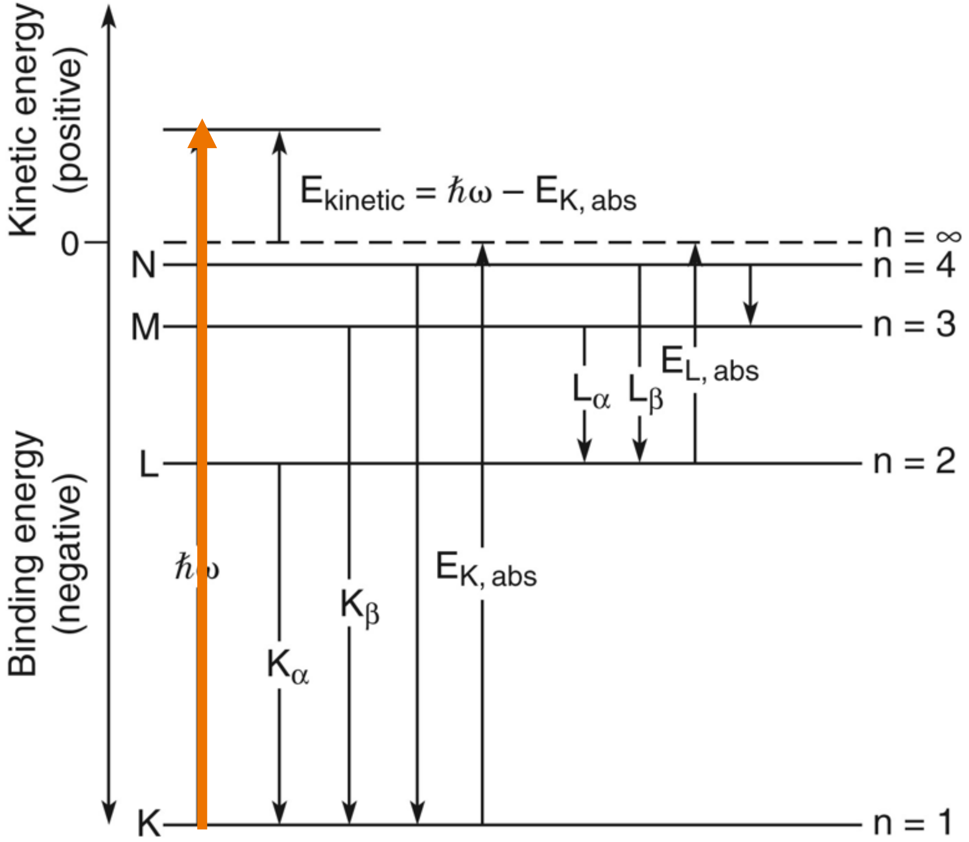
surface sensitivity: electrons

+ spatial selectivity via imaging techniques

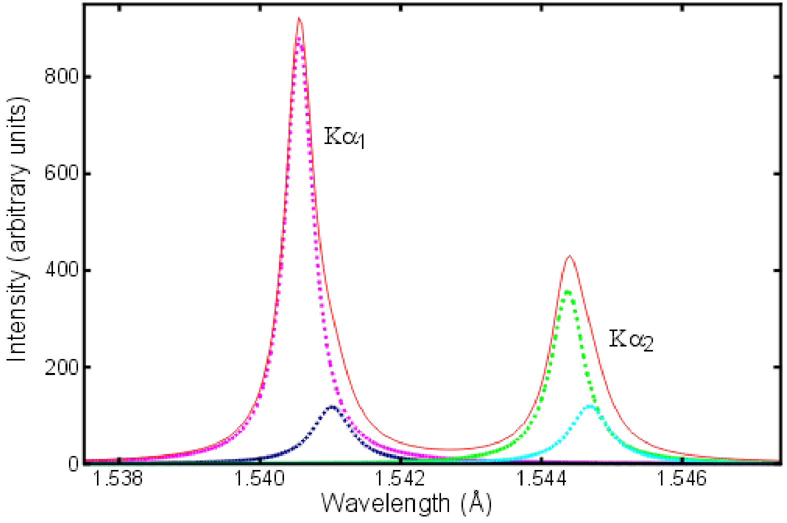
X-RAY ABSORPTION

Cu K-edge absorption



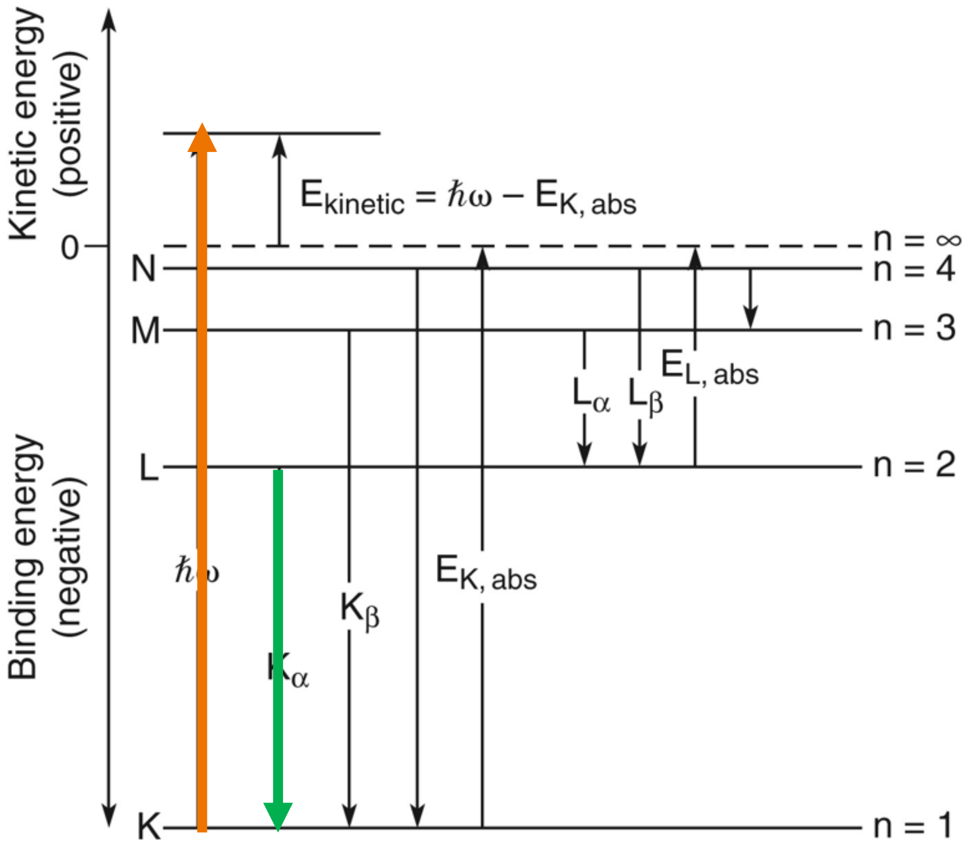


Cu K-$\alpha_{1,2}$ emission (fluorescence)

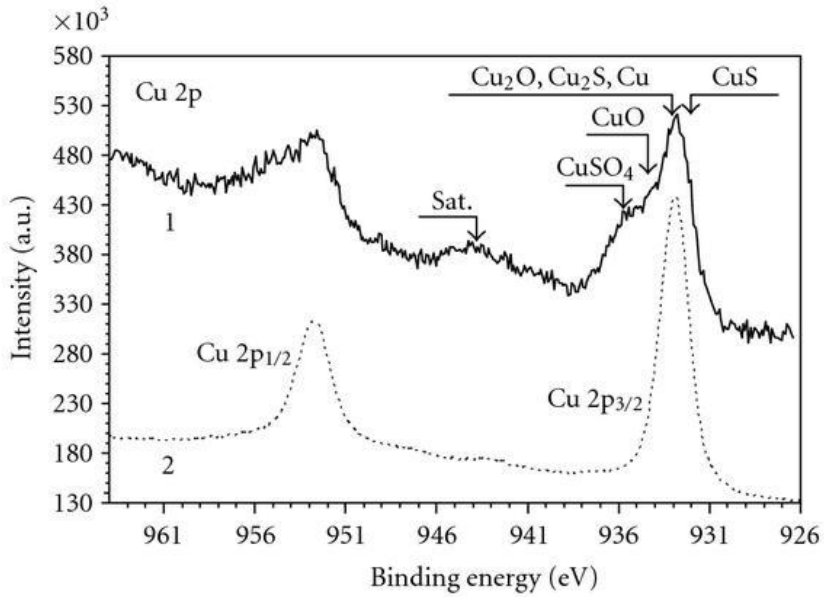


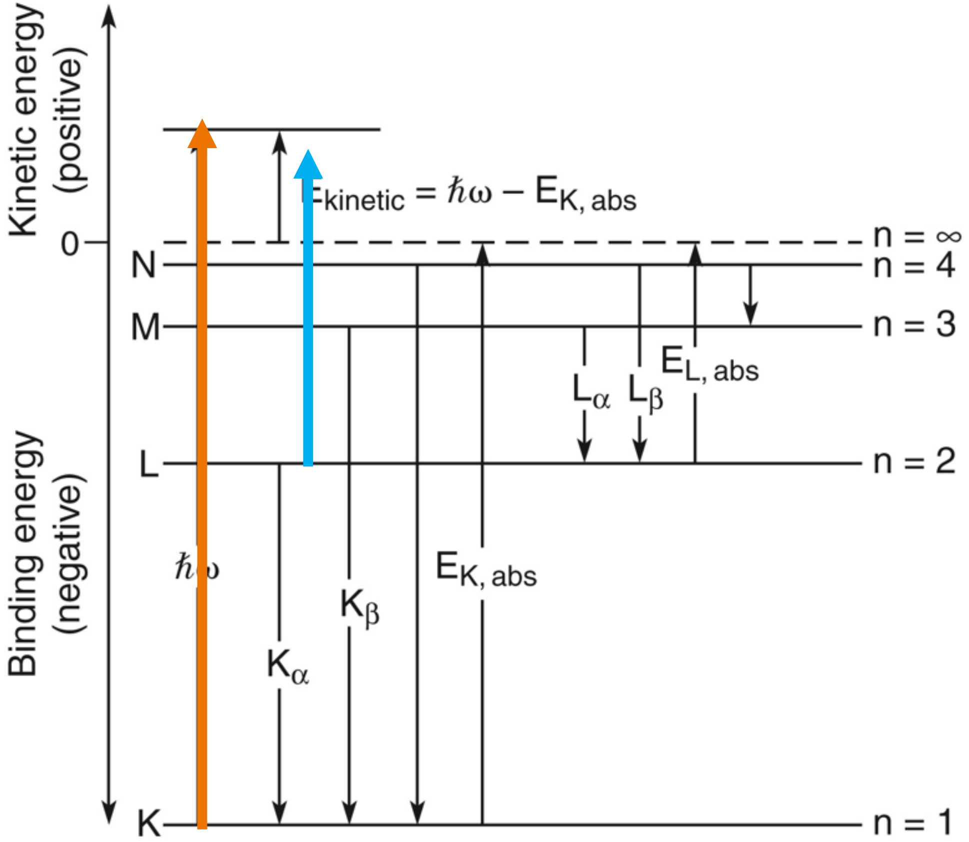
$2p_{3/2}$ -> 1s: Cu $K\alpha_1$

$2p_{1/2}$ -> 1s: Cu $K\alpha_2$

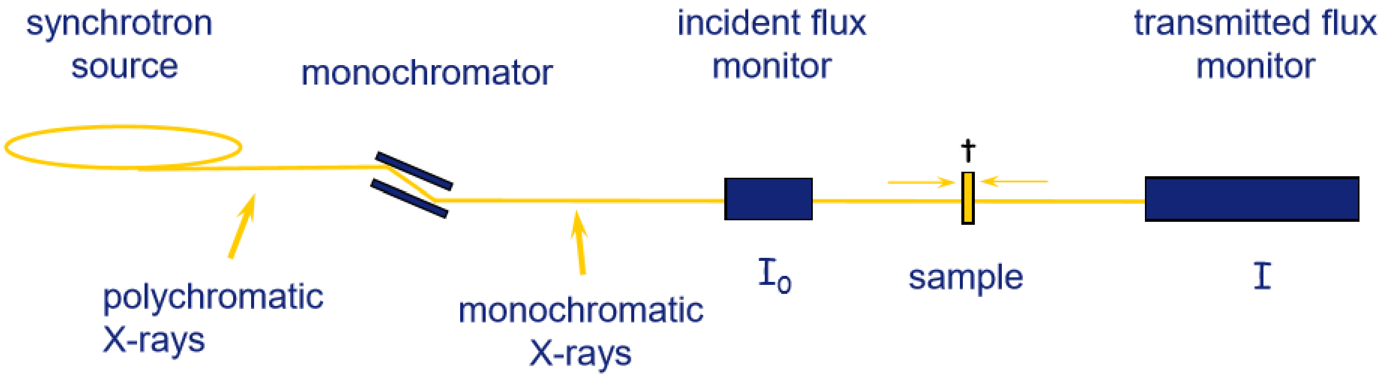


Cu L_2 ($2p_{1/2}$) and L_3 ($2p_{3/2}$) photoemission

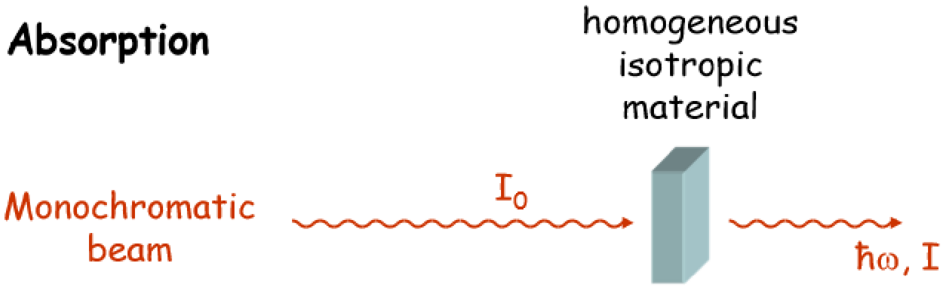




X-RAY ABSORPTION – AS SIMPLE AS REASONABLY POSSIBLE



Absorption



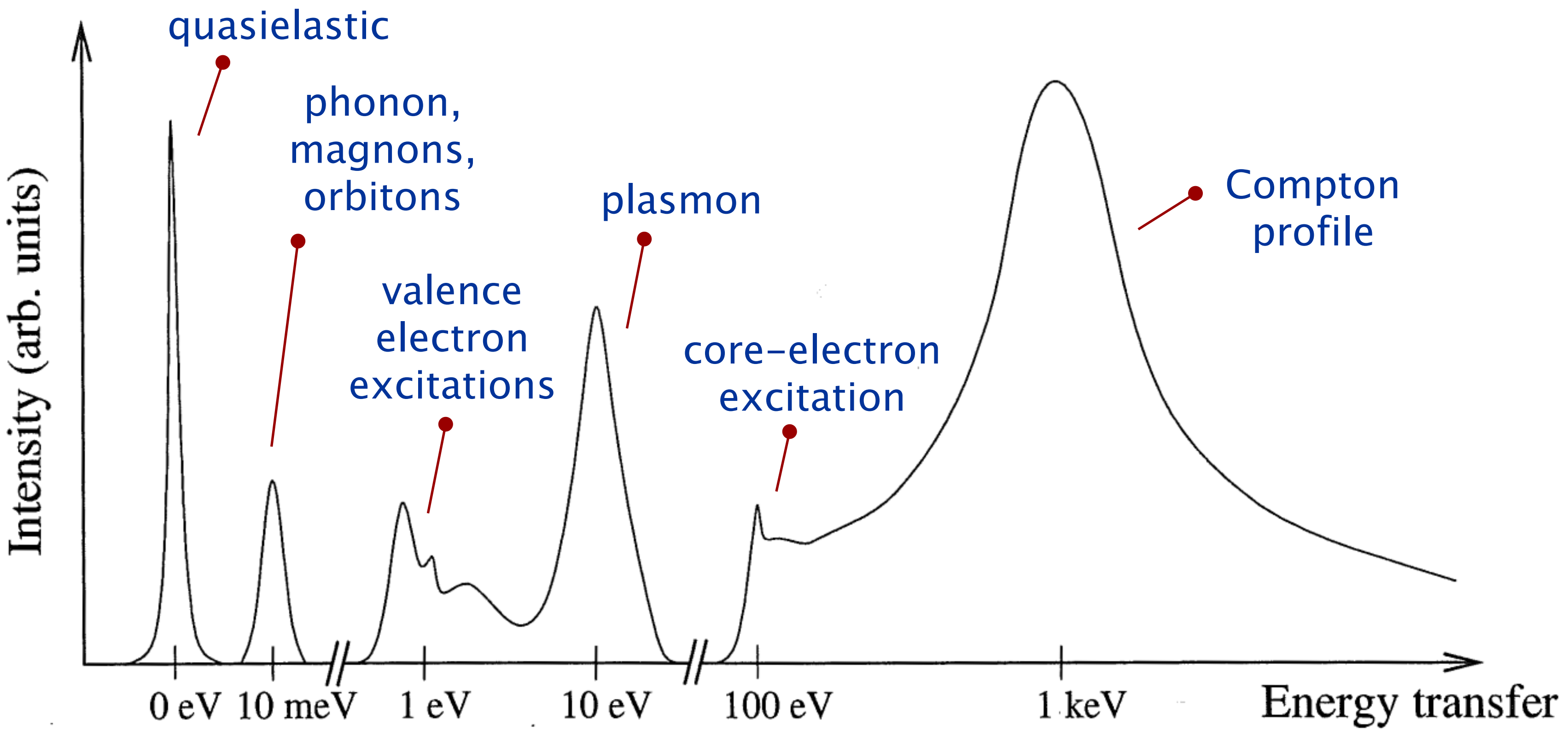
$I = I_0 \exp(-\mu D)$

μ: total linear absorption coefficient
D: sample thickness

Beer-Lambert law :

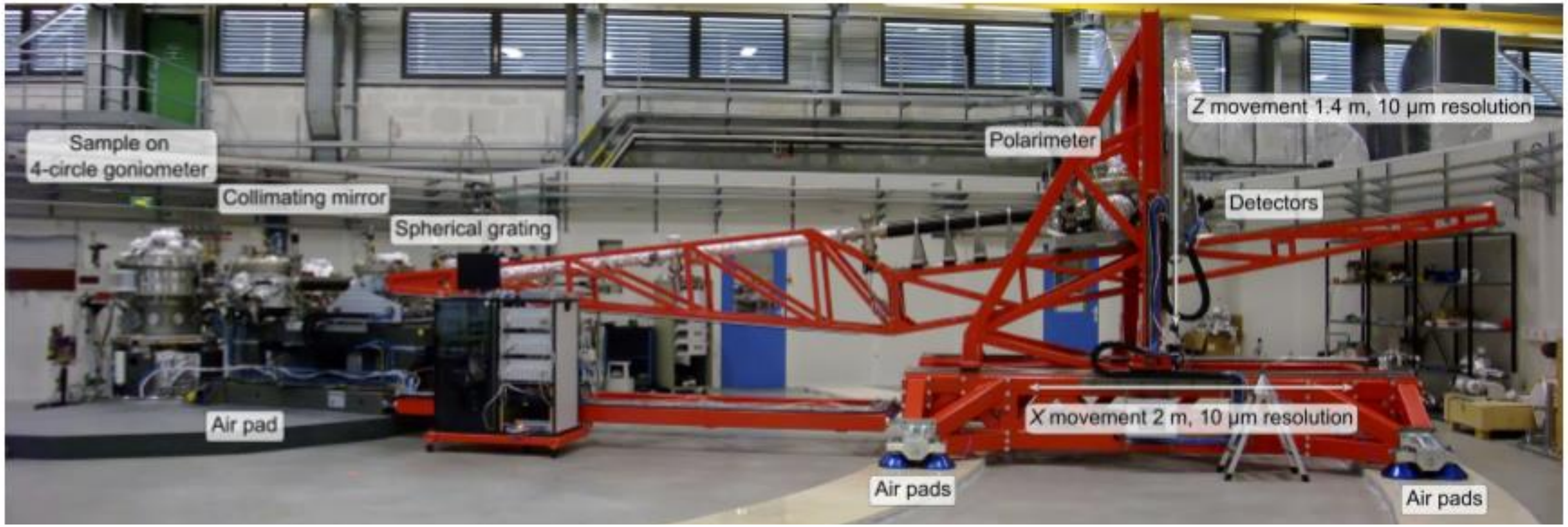
$\ln(I_0/I) = \mu D$

X-RAY SPECTROSCOPY USING INELASTIC X-RAY SCATTERING (IXS) TECHNIQUES



S. Galombosi, PhD thesis, Helsinki 2007

RIXS SPECTROMETER ON ID32 (AND ID20) AT THE ESRF



Soft X-ray RIXS

Resolving power: 50 000; 32 meV at the Cu L_3-edge

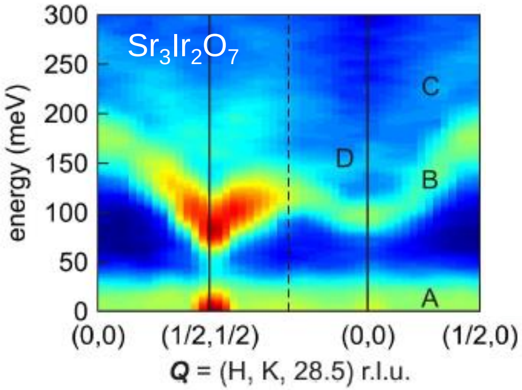
Beam size on sample: 30 x 4.5 μm^2 (hor. x vert.)

Hard X-ray RIXS

Resolving power: $4.5 \cdot 10^5$; 25 meV at Ir L_3-edge

Beam size on sample: 30 x 4.5 μm^2 (hor. x vert.)

Magnon dispersion in correlated electron systems

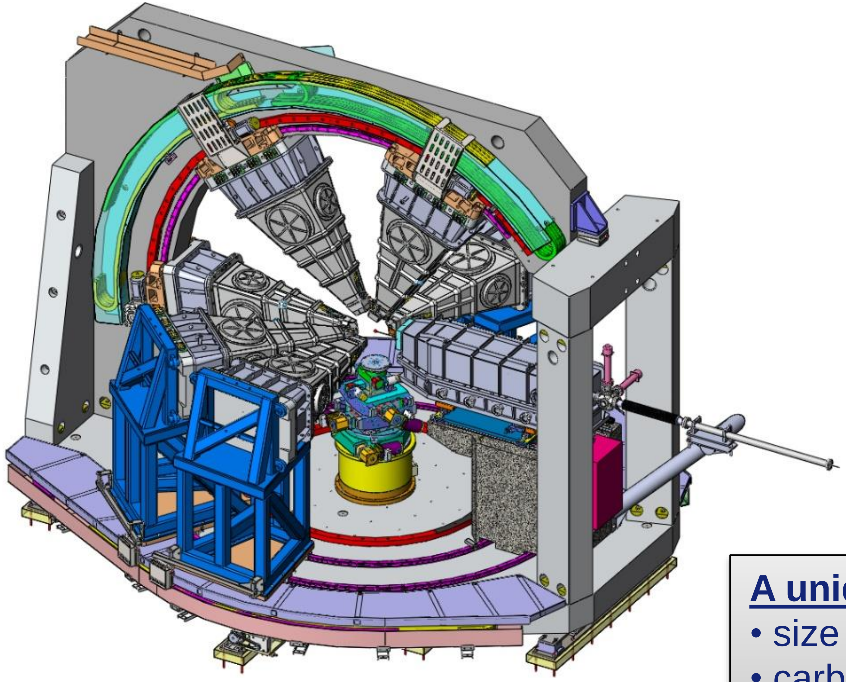


L. Braicovich et al., Phys. Rev. Lett (2010)

M. Moretti et al., Phys. Rev. B (2015)

THE X-RAY RAMAN SPECTROMETER ON ID20 AT THE ESRF

X-ray Raman spectroscopy: Soft x-ray absorption spectroscopy in the hard x-ray range



<u>A unique challenging instrument</u>
- size + precision + stability
- carbon fibre composite technology
- 72 analyser crystals = 216 motors
- Integration of 6 MAXIPIX detectors

APPLICATIONS OF X-RAY SPECTROSCOPY

Chemistry and Catalysis

Magnetism

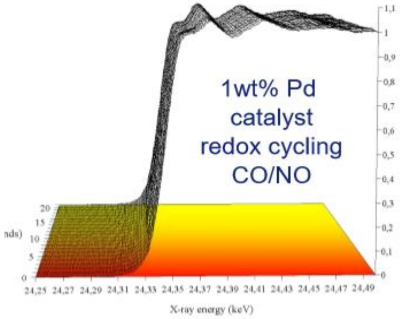
Superconductivity

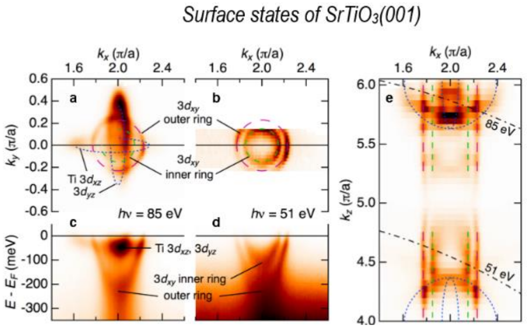
Earth & Planetary science

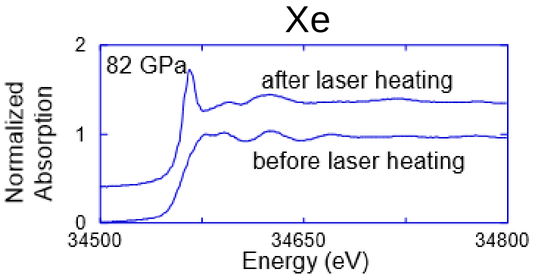
Environment

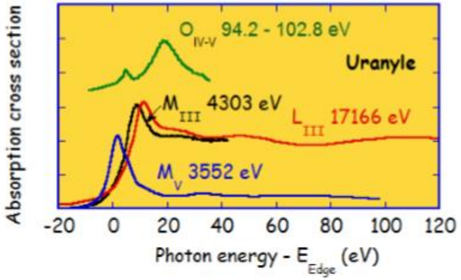
Cultural heritage

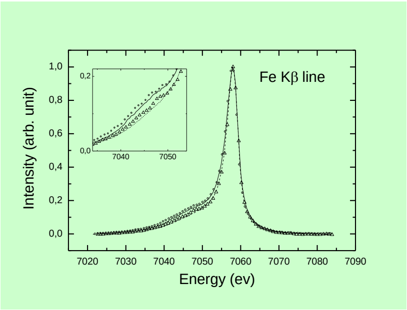
…….

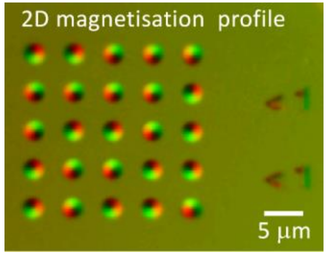




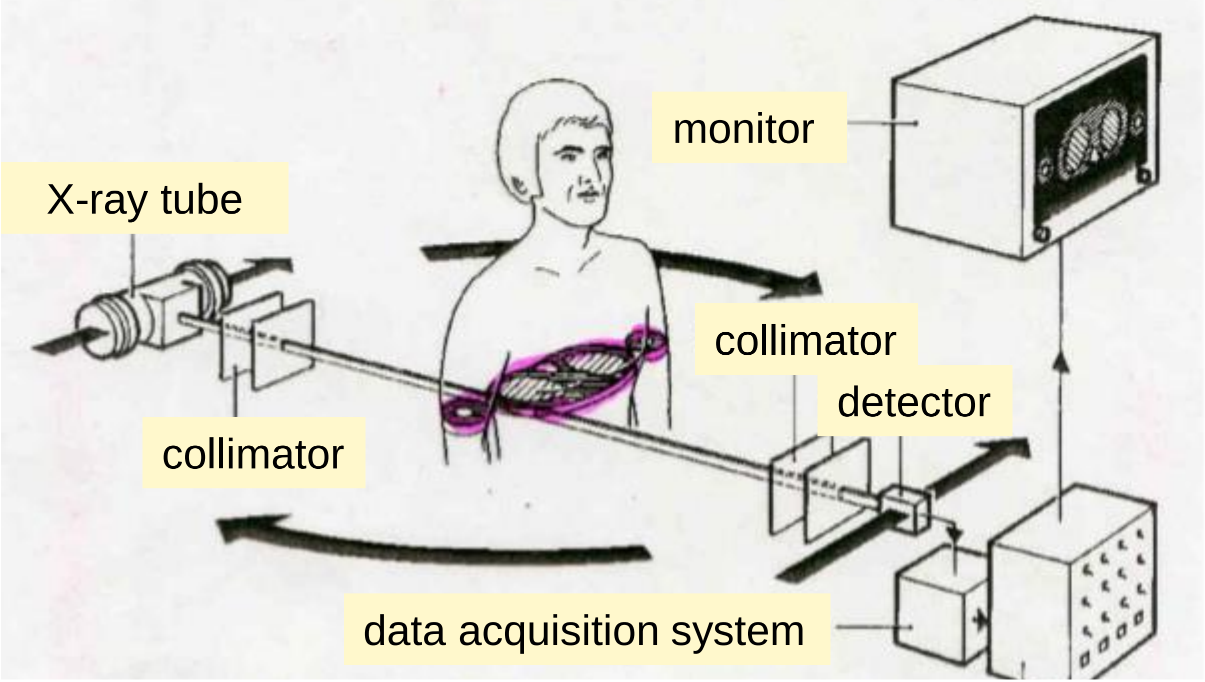








X-RAY IMAGING – X-RAY RADIOGRAPHY



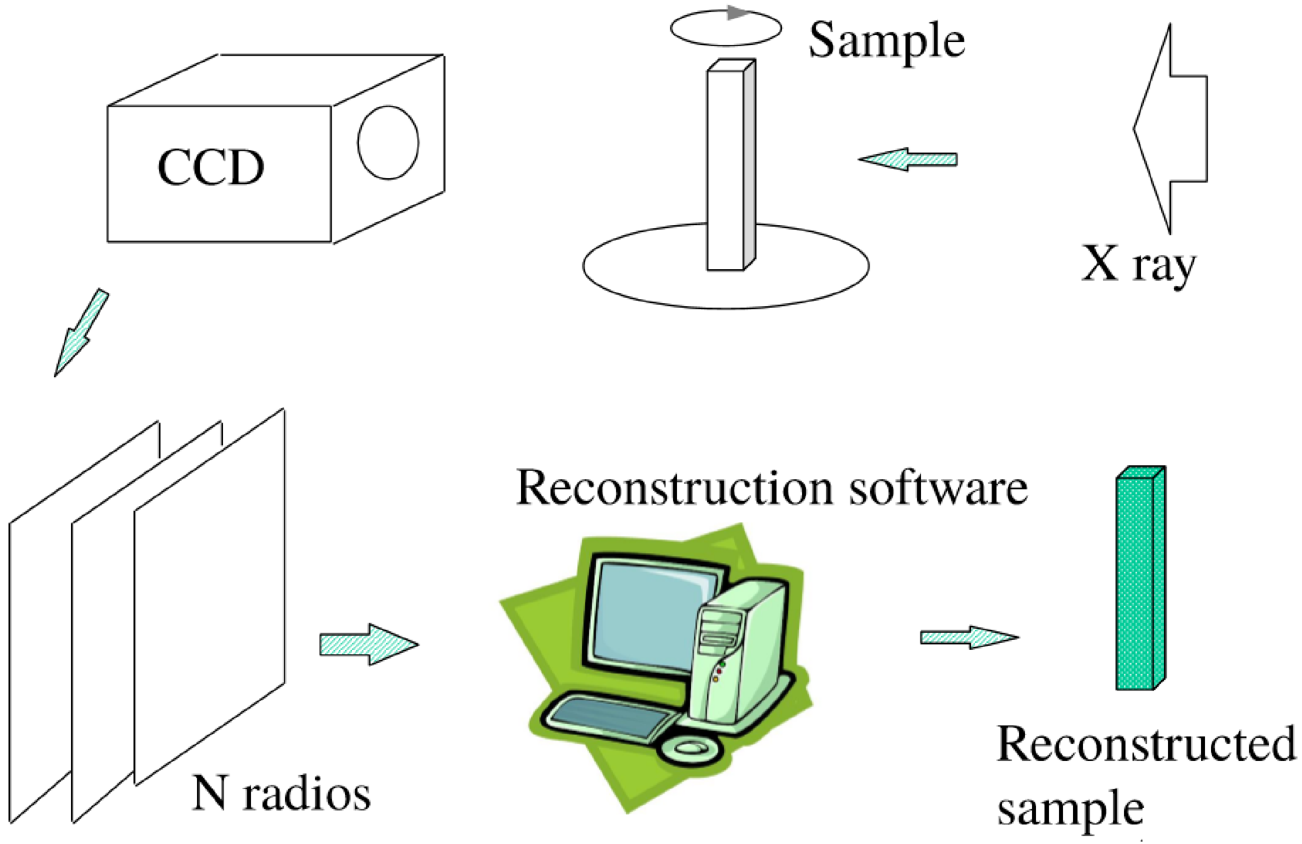
X-ray radiography: the oldest X-ray imaging technique (see hand of Röntgen's wife)

good lateral resolution

easily available in any lab or hospital

but no depth resolution = no three dimensional information

360º rotation of the sample; at each Δθ take an image; 3D reconstruction from N radios



$N \cdot \Delta\theta = 360º$

compute intensive

A TYPICAL MICROTOMOGRAPHY ENDSTATION (ID19 @ ESRF)

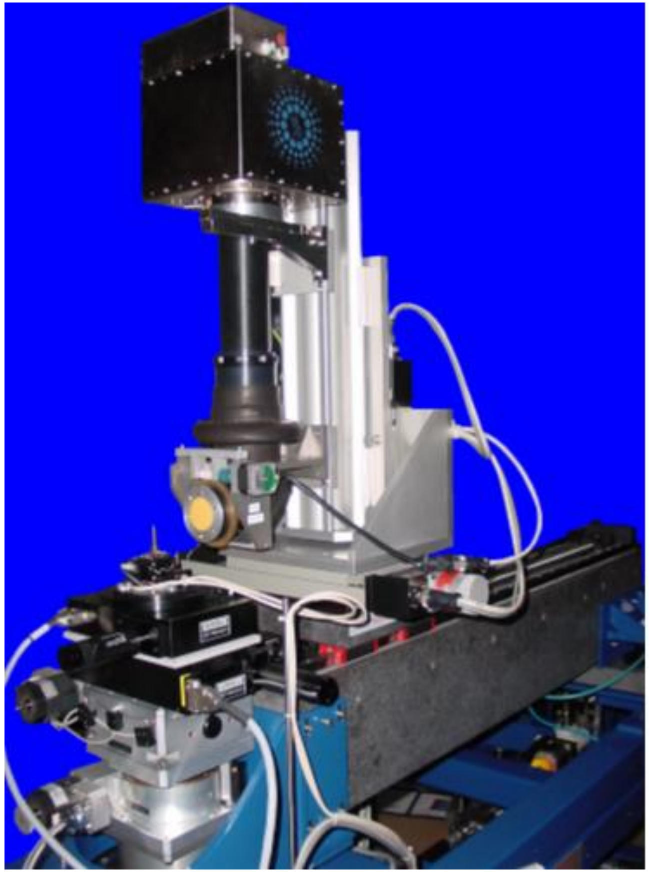
- Long distance (145 m)

  → coherence (phase contrast)
- Multilayer monochromator:

  $\Delta\lambda/\lambda \sim 10^{-2}$
- High resolution detector system

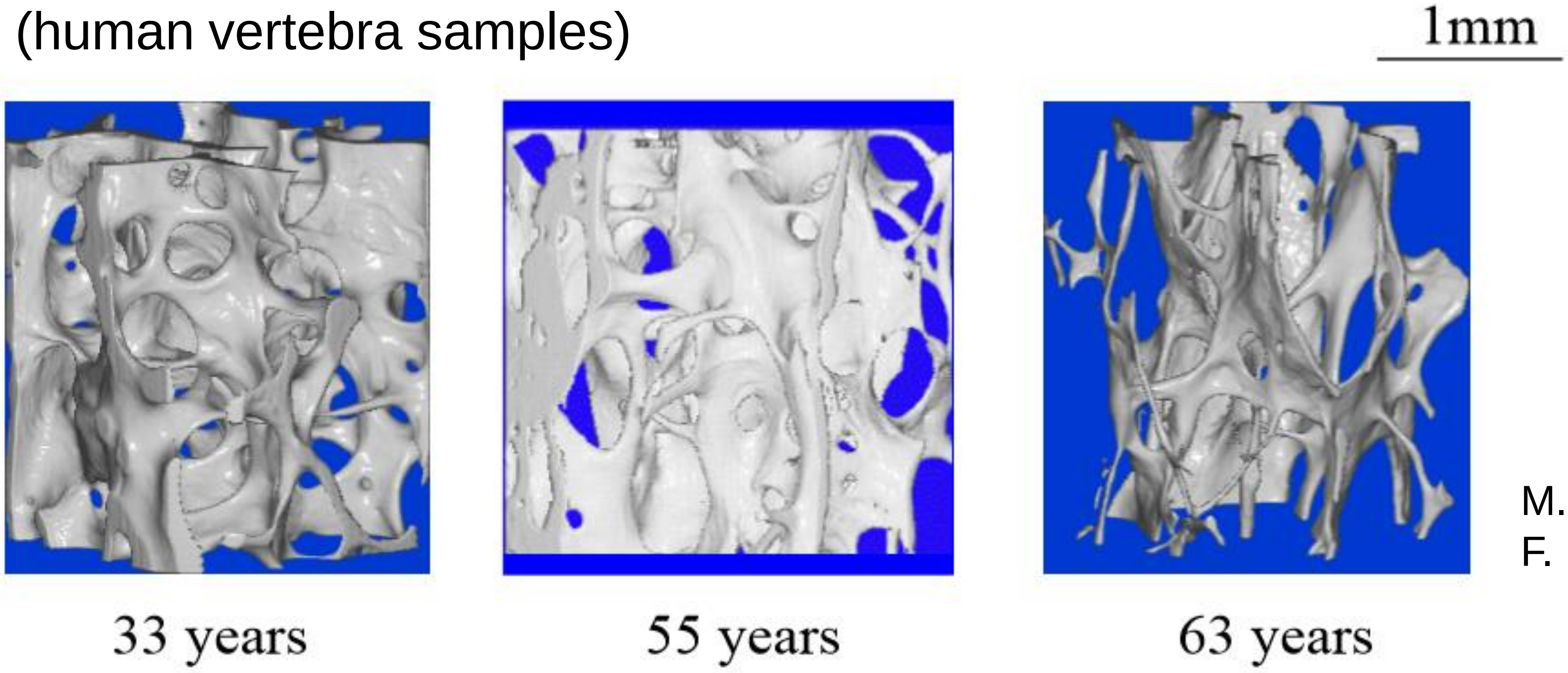
  14 bit, 1024^2 and 2048^2

  CCD, 60 ms readout, 1 µm.
- Dedicated µ-tomography set-up
- Sample environment: fatigue machine, cold cell, furnace, …



X-RAY IMAGING – BONE STUDIES

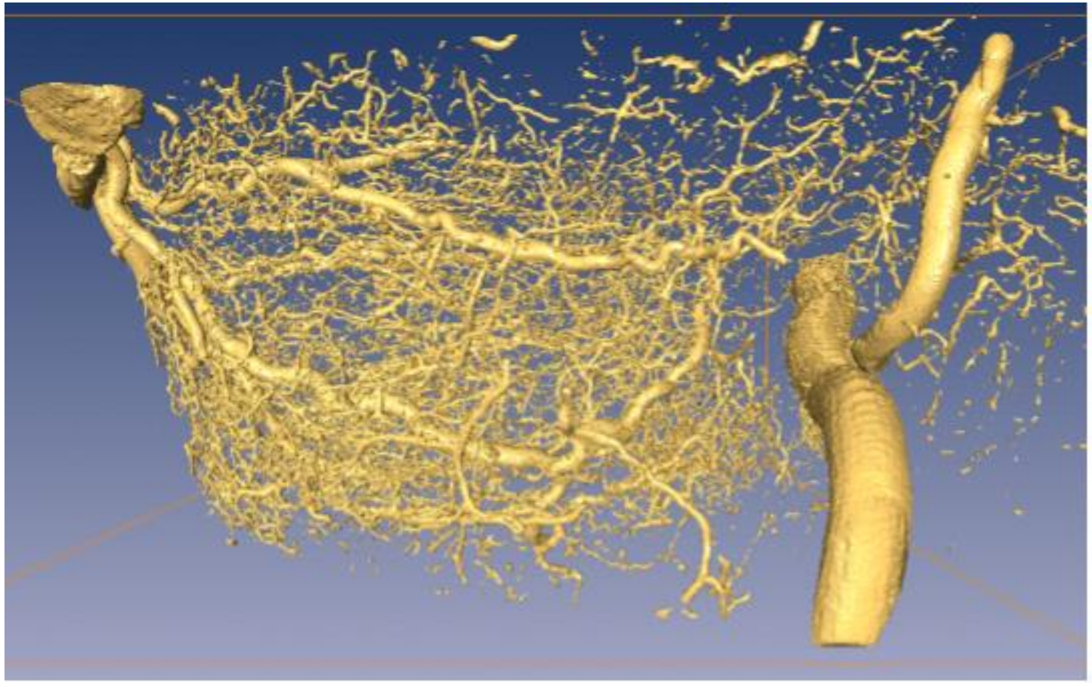
3D-visualization of bones attained by Osteoporosis
(human vertebra samples)



M. Salomé,
F. Peyrin 1998

Images $(512)^3$, voxel size=6.7 µm

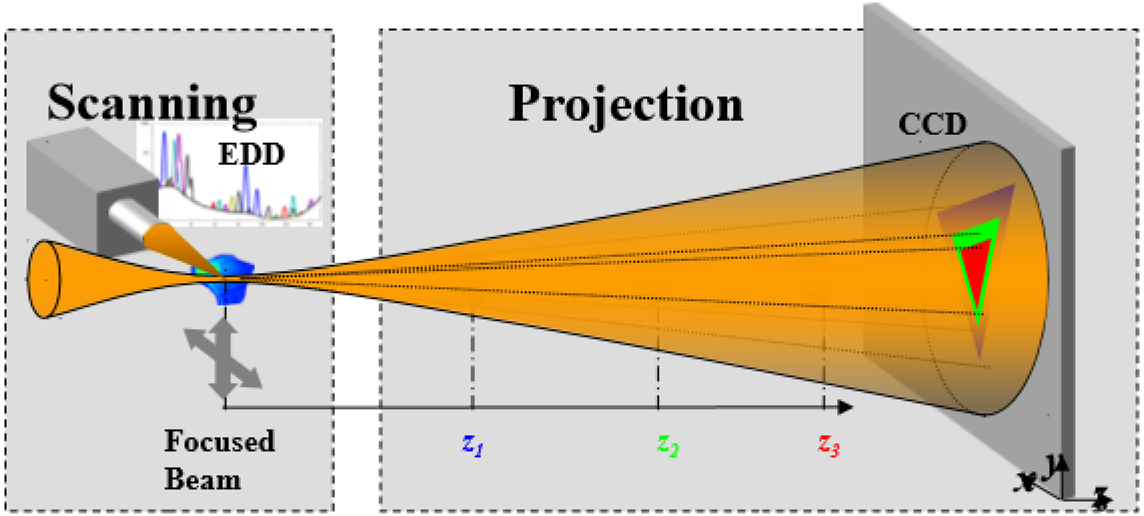
Characterization of brain vascular network and transport after injection of barium



F. Plouraboué et al.

Sample: rat cortical brain	Barium injection: 600 mg/mL
Sample size: 1.5x1.5x1mm^3	Take two data sets:
Voxel size: 1.4 μm^3	above and below of the Ba K-edge (20 keV)

NANO-PROBE IMAGING (EXAMPLE ID16A @ ESRF)

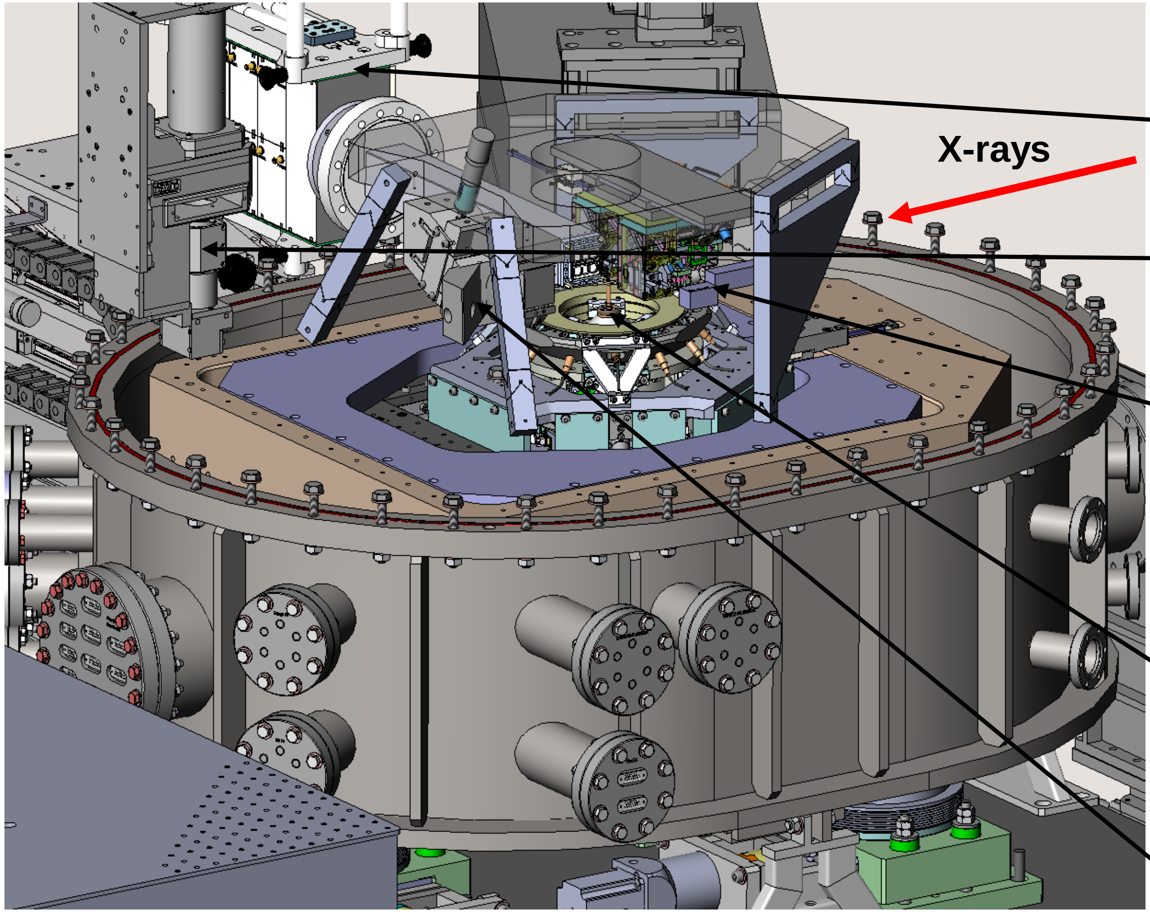


Focus ~ 30 nm
Flux > 10^{11} ph/s
E = 17 keV or 33.6 keV
ΔE/E 1%

Height error over 360°:
< 10 nm
Focus stability:
Δy (Δz) over 8 h: 6 (30) nm

Nano-endstation must be compatible with vacuum operation at cryogenic temperatures

END-STATION FOR NANOPROBE IMAGING (ID16A @ ESRF)



Large solid angle, high through-put fluorescence detectors

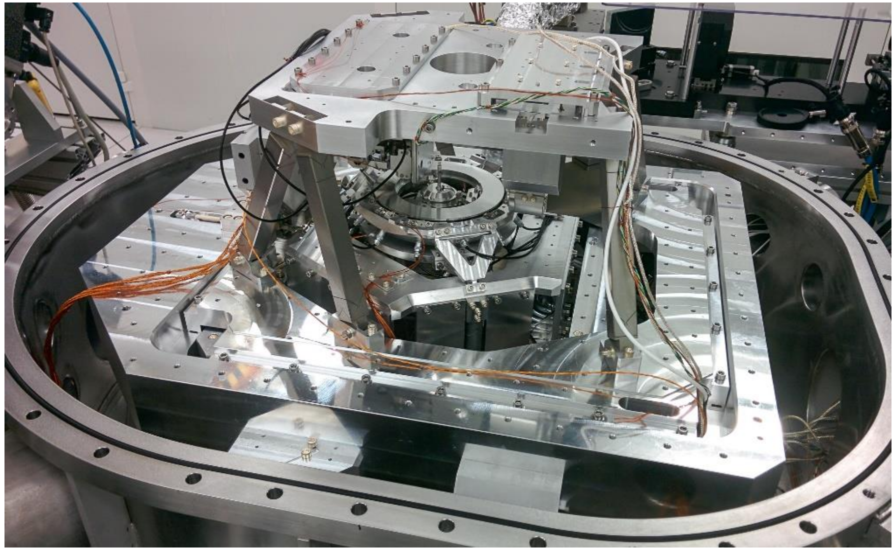
High-resolution imaging detector

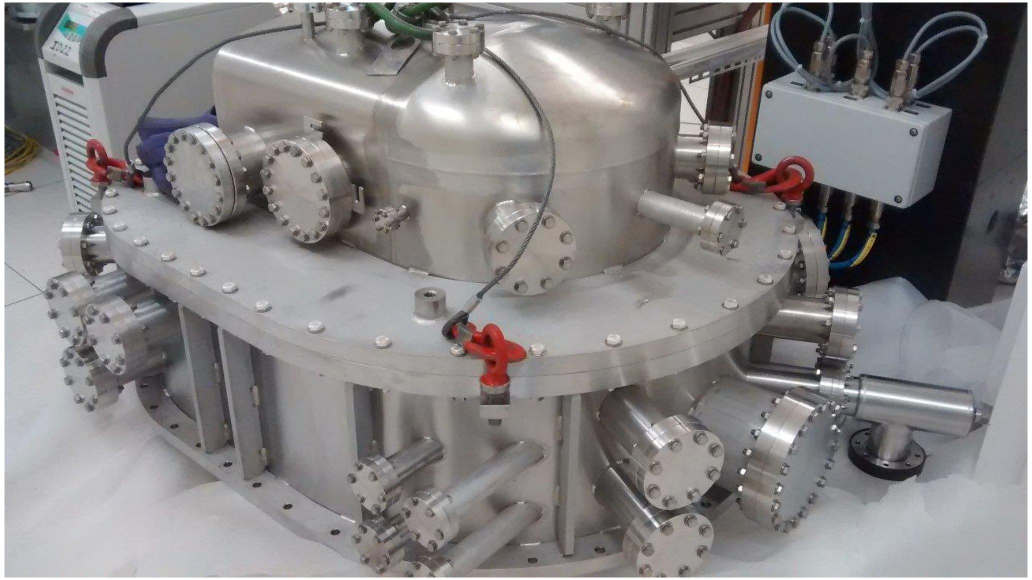
Nano-focusing optics: Fixed curvature, multilayer coated KB optics

3D nano-positioning of Metrologic reference under control of capacitive sensors

On-line microscope optics

END-STATION FOR NANOPROBE IMAGING (ID16A @ ESRF)

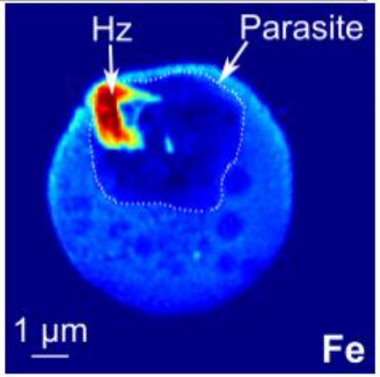




NANO-IMAGING – SCIENTIFIC TOPICS

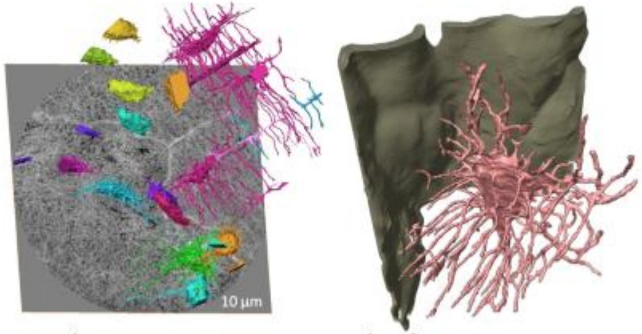
Nano Imaging

Biomedical Research:
Sub-cellular processes



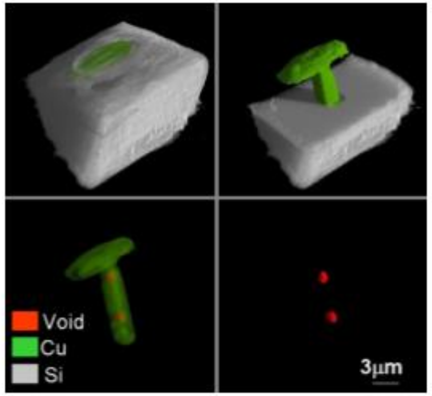
Anti-malarian drugs
Dubar et al, *Chem. Commun.*

Biomedical Engineering:
Tissue-level



Bone ultrastructure; Langer et al, *PloS One*

Nano/Micro-Technology:
3D Integration

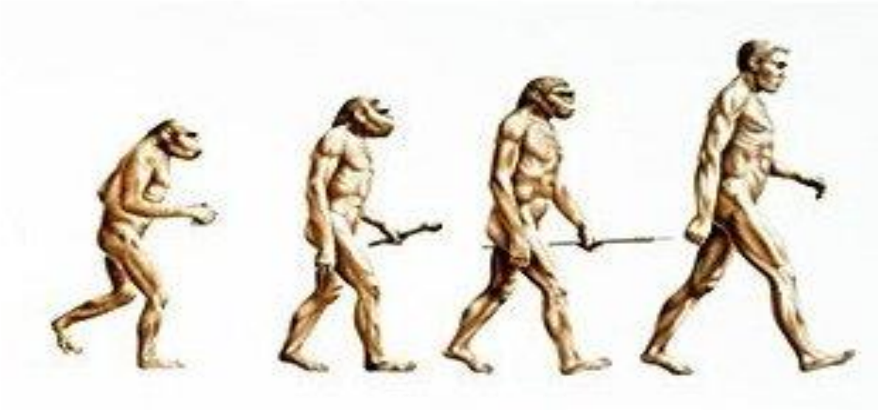


Voids in Through-Si-via
Bleuet et al (CEA-Leti)

The Malapa Synchrotron Project and Sediba

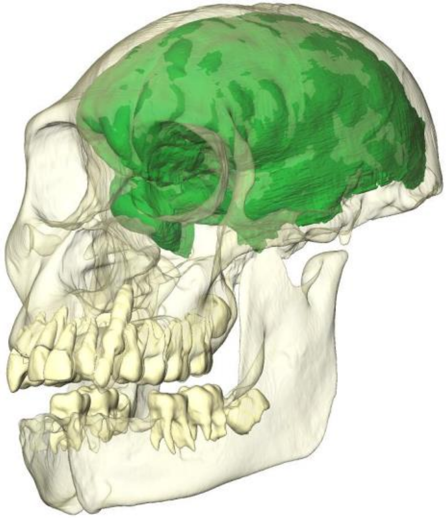
New hominid species (age 1.9 million years) have been discovered in August 2008 in South Africa by Pr. Lee Berger and colleagues.

Species showing intermediate character between *Australopithecus* and *Homo-Genus*.



9 April 2010 : Four papers describe the discovery of a new hominid species

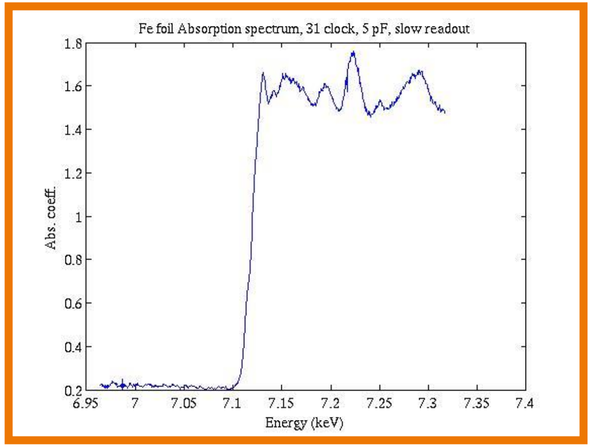
IMAGING APPLICATIONS IN PALAEONTOLOGY



"The many very advanced features found in the brain and body ...make it possibly the best candidate ancestor for our genus, the genus Homo."
Lee Berger, Wits U, Johannesburg

K.J. Carlson et al.; Science (2011)

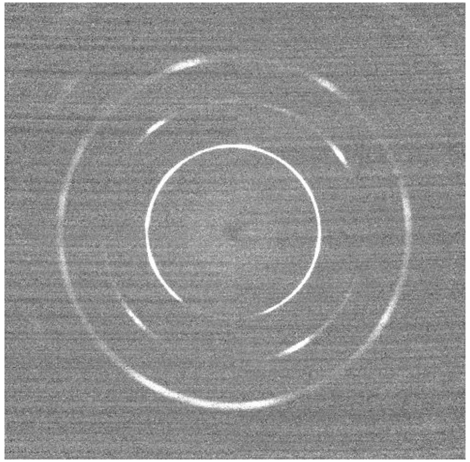
XAS at ID24

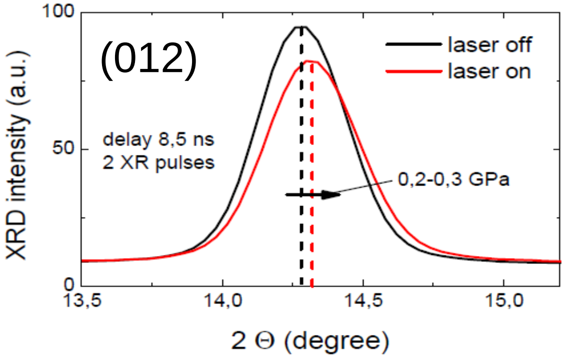


1 bunch:
100 picoseconds

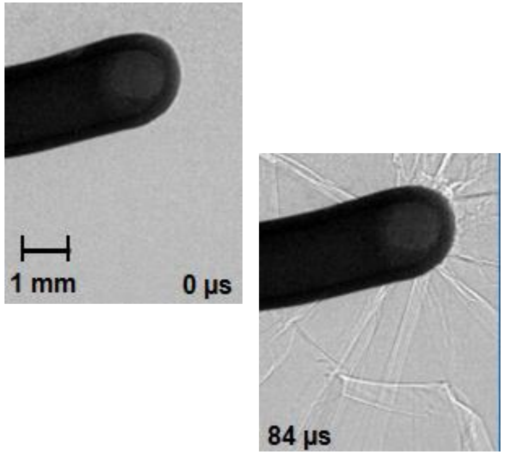
XRD at ID09A

Bismuth





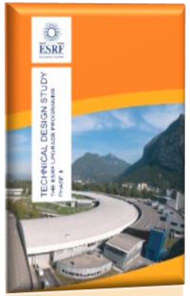
XRI at ID15A/ID19



breaking glass with single bunch resolution

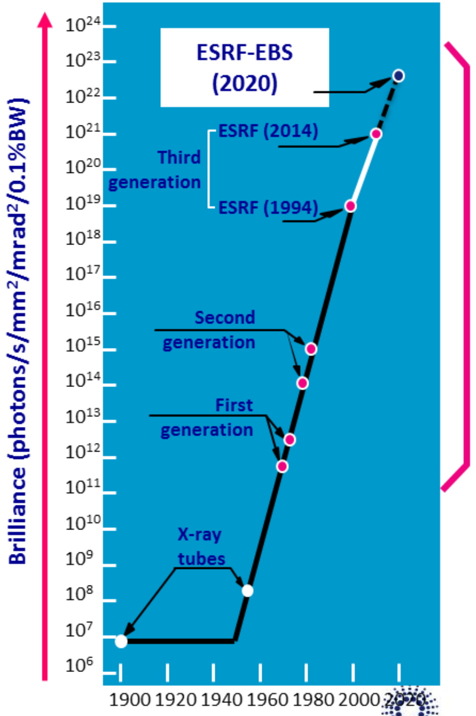
ESRF UPGRADE PROGRAMME: AN AMBITIOUS PROGRAMME TO PREPARE THE FUTURE

Purple
Book
January
2008



Orange
Book
January
2015

e-beam emittance values:
(smaller is better!)
ESRF: 4 nm.rad
EBS: 0.13 nm.rad



ESRF UPGRADE PHASE I
180 M€ (2009-2015):
ESFRI ROADMAP 2006-2016
IN TIME – WITHIN BUDGET

- 19 new beamlines, many specialised on *nano*-beam science
- Upgrade and renewal of facilities and support laboratories

ESRF-EBS
EXTREMELY BRILLIANT SOURCE

ESFRI

European Commission

ESRF-EBS
Extremely Brilliant Source
150 M€ (2015-2022):
ESFRI LANDMARK (2016)

revolutionary design
for a new generation of
synchrotron source storage rings

Smaller, more intense X-ray beams

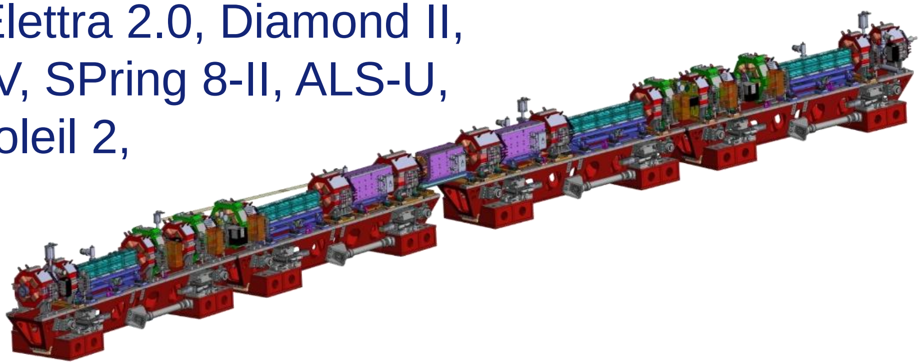
Replacement of the 32 cells in the ring with new arcs. The more complex sequencing will make it possible to create an X-ray source with a very low emittance (less divergence and smaller in size)

- 14 Vacuum chambers (7 today)
- 31 magnets (19 today)
- 10 correction magnets and 10 beam position monitors

x32

ESRF is the pioneer in performing such an upgrade – other sources also have upgrade plans :

APS-U, Elettra 2.0, Diamond II, PETRA-IV, SPring 8-II, ALS-U, SLS-2, Soleil 2,



Schedule EBS Machine

Jan 2015 - Dec 2019	Execution phase
Jan 2015 - Dec 2015	Design phase ✓
Jan 2016 - Dec 2018	Procurement phase ✓
Oct 2017 - Dec 2018	Assembly phase ✓
Jan 2019 - May 2019	Dismantling phase (...)
Apr 2019 - Dec 2019	Installation phase
Dec 2019 - Feb 2020	Beam commissioning
Feb 2020 - Aug 2020	Beamline commissioning
Sept 2020	Restart operation with users

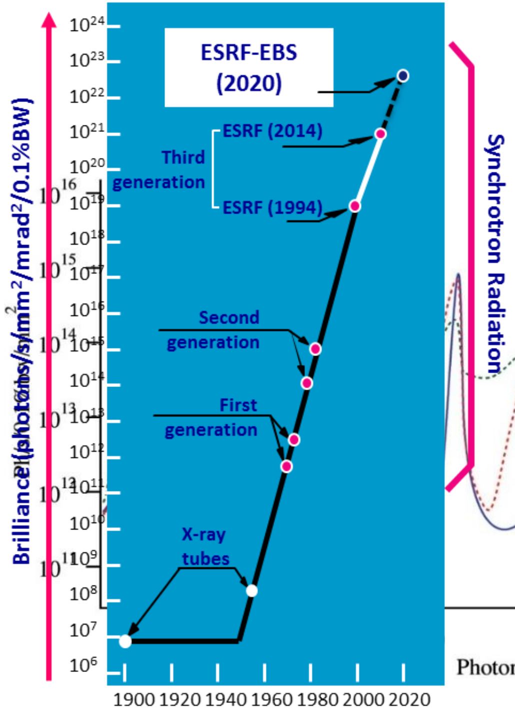
ESRF X-RAY SOURCE PARAMETERS: CURRENT AND ESRF-EBS LATTICES

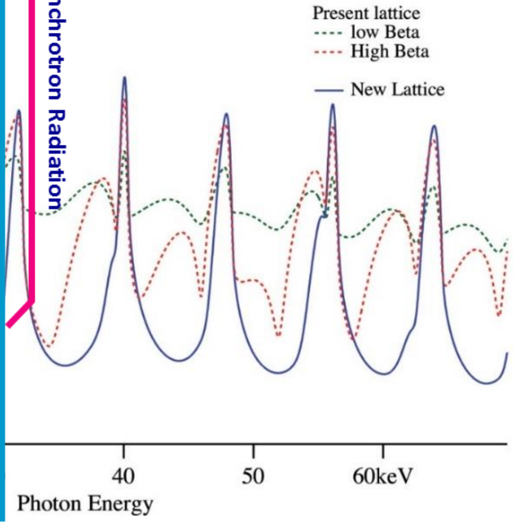
e-beam emittance values:
(smaller is better!)
ESRF: 4 nm.rad
EBS: 0.13 nm.rad

X-ray beam characteristics: 2m U18 undulator at 10 keV

Lattice	RMS size (µm)		RMS divergence (µrad)	
	H	V	H	V
Current low β section	49	5.0	107	6.1
Current high β section	410	4.9	11.5	6.1
EBS lattice	30	5.1	7.4	6.1

relatively little change





Improved spectral purity
~1.5x reduction in undulator peak width

ESRF → ESRF-EBS!
Top up operation
⇒ 'constant' power
✓

Susini et al., J. *Synch. Rad.* **21**, (2014) 986–95

SUMMARY

Experiments at synchrotrons become faster, more complex, and more diversified

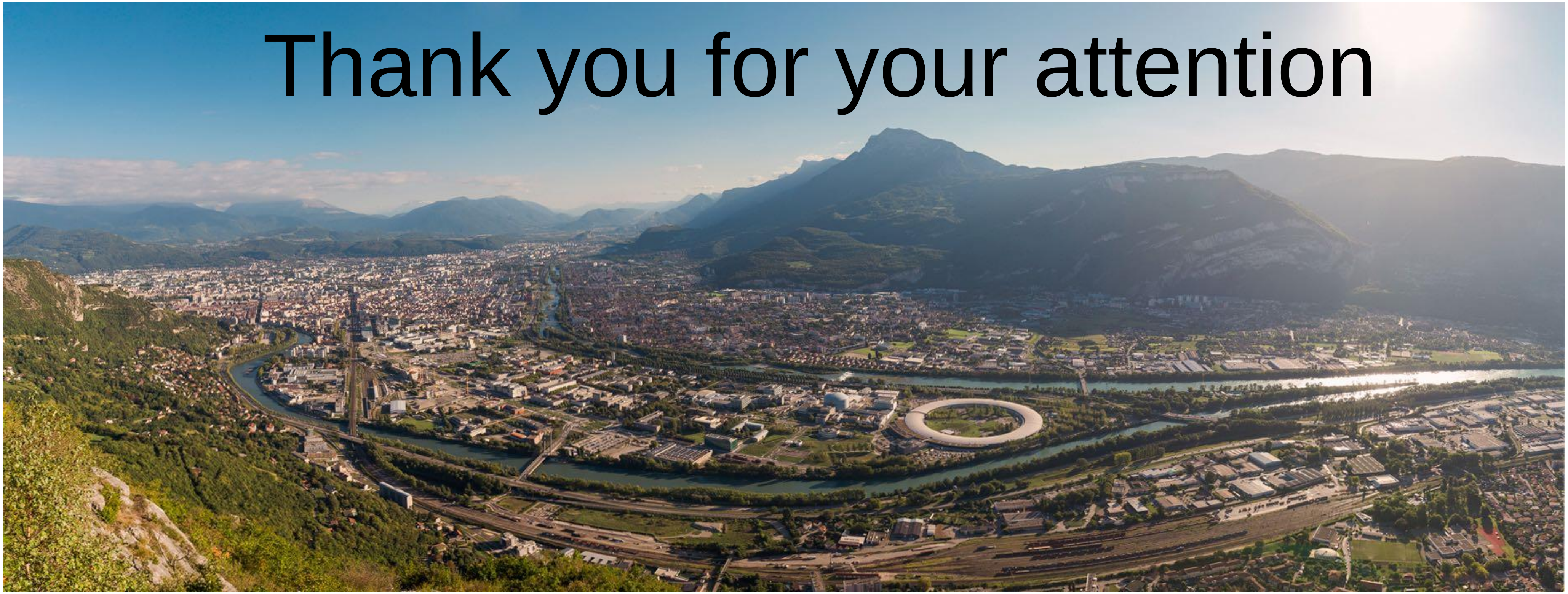
Major challenges for:

- stability of the storage ring
- x-ray optics
- experimental endstations
- detectors
- data acquisition systems, data analysis, data management

Instrumentation developments have to keep pace with this!

FURTHER READING

- D. Attwood and A. Sakdinwat, *X-Rays and Extreme Ultraviolet Radiation* (Cambridge, UK 2016); available at Amazon.com.
- J. Als-Nielsen and D. McMorrow, *Elements of Modern X-ray Physics* (Wiley, New York, 2009), Second edition.
- A. Hofmann, *Synchrotron Radiation* (Cambridge, UK, 2004).
- P. Duke, *Synchrotron Radiation* (Oxford, UK, 2000).
- J. Samson and D. Ederer, *Vacuum Ultraviolet Spectroscopy I and II* (Academic Press, San Diego, 1998). Paperback available.


Thank you for your attention