Synchrotron radiation-based imaging

An introduction

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Outline

- 1 – Introduction: synchrotron radiation and imaging
- 2 – Basic Interactions
- 3 – Radiography: absorption, phase
- 4 – Microtomography
- 5 – Microbeam-based techniques
- 6 – Bragg diffraction based techniques

  (DEI, “topography”, diffraction tomography)

- 1 – **Introduction: synchrotron radiation and imaging**
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  (DEI, “topography”, diffraction tomography)

Imaging

- **reveals inhomogeneities and singularities in the sample**
- **through variations in reflection, transmission, …behaviour for the probe used**

Images can be made with

light, electrons, ultrasound, nuclear magnetic resonance, neutrons, …

and X-rays !

Making images with different probes is valuable because it can yield different information

X-ray Imaging

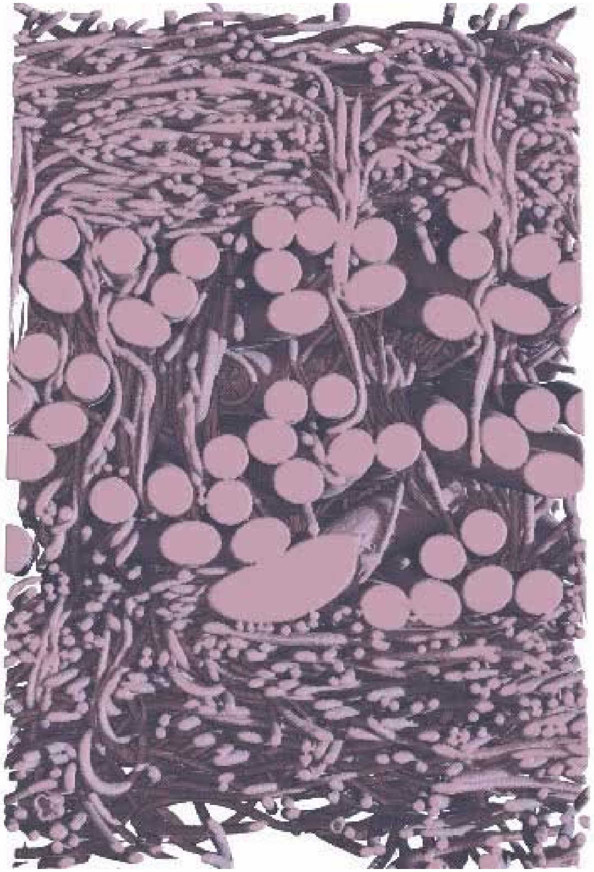
X-ray imaging is not actually a new technique....

Variation of absorption

First X ray made in public. Hand of the famed anatomist, Albert von Kölliker, made during Roentgen's initial lecture before the Würzburg Physical Medical Society on January 23, 1896.

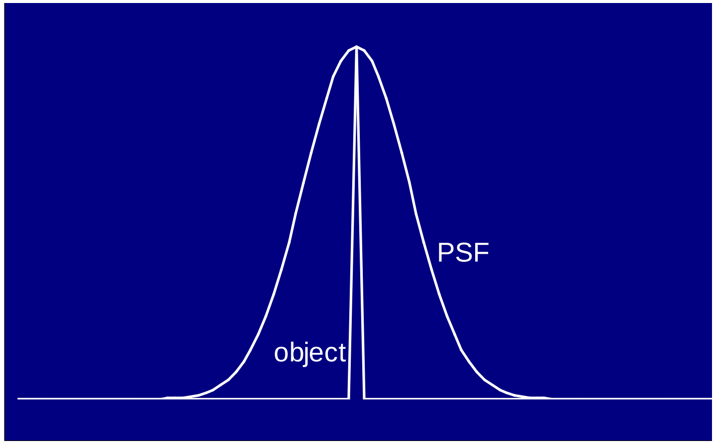
X-RAY IMAGING

Substantially renewed by the use of Synchrotron Radiation



Microtomographic image of an industrial felt

The observed image integrates the detection process



The observed size feature is a function of its physical size within the x-ray beam exiting the sample and the

Point Spread Function

of the used detector

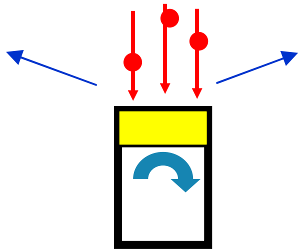
Also depends on the statistical or instrumental noise, geometry, …

⇨ **importance of devoting time to detectors**

⇨ John Morse's talk

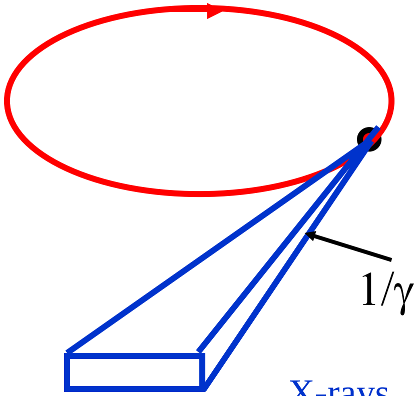
Synchrotron radiation

⇨ The amount of X-rays produced by X-ray generators is limited by the heat load on the anode



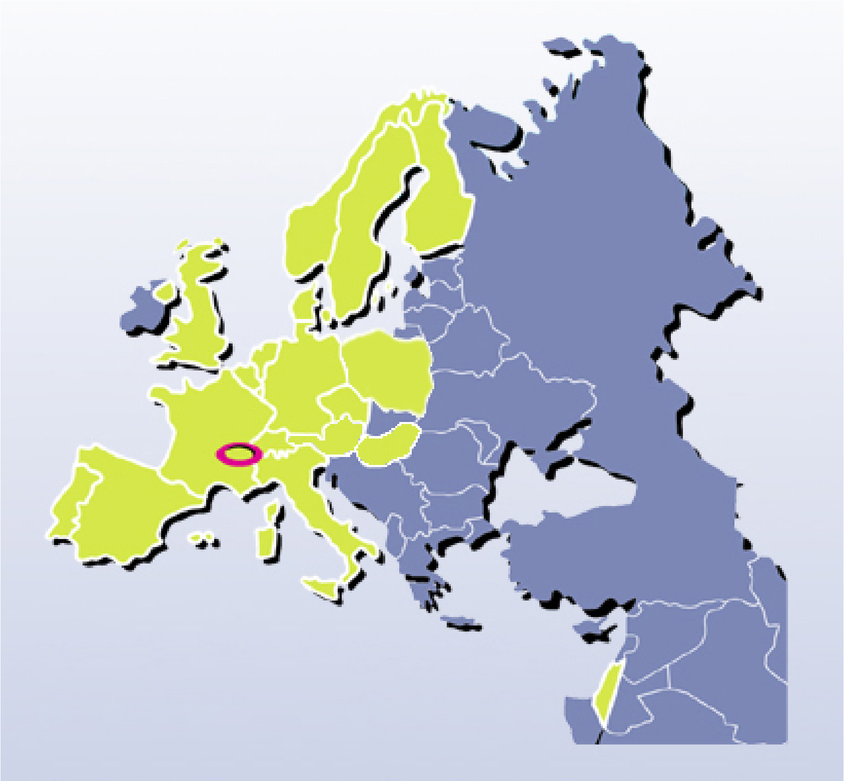
⇨ Another way of obtaining X-rays is by using the radiation emitted by accelerated **relativistic** ($E_e = \gamma\, E_{rest}$, with γ #10^3–10^4) electrons

⇨All the emission is concentrated in a narrow cone of aperture $1/\gamma$



A modern synchrotron radiation X-ray source

Cooperation between 18 countries

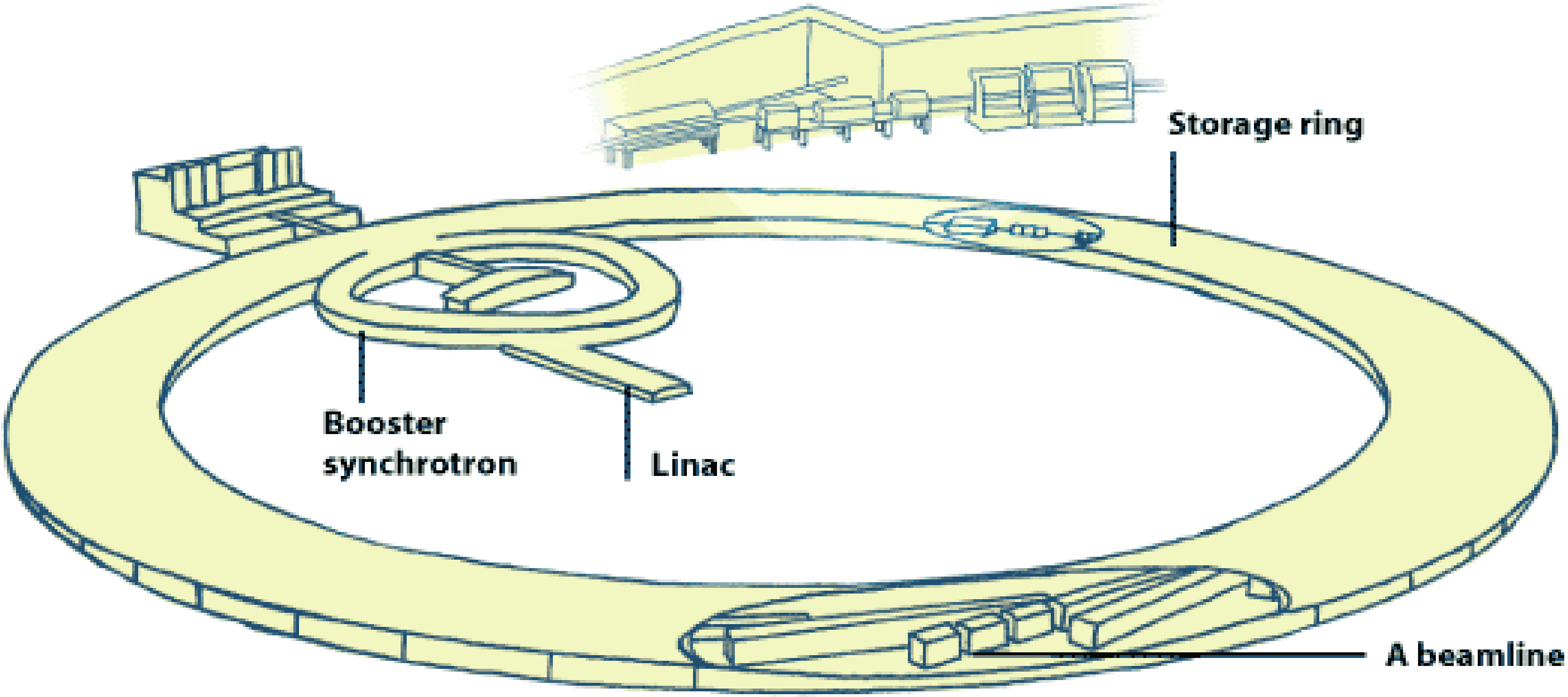


The ESRF is a French company financed by 18 countries.

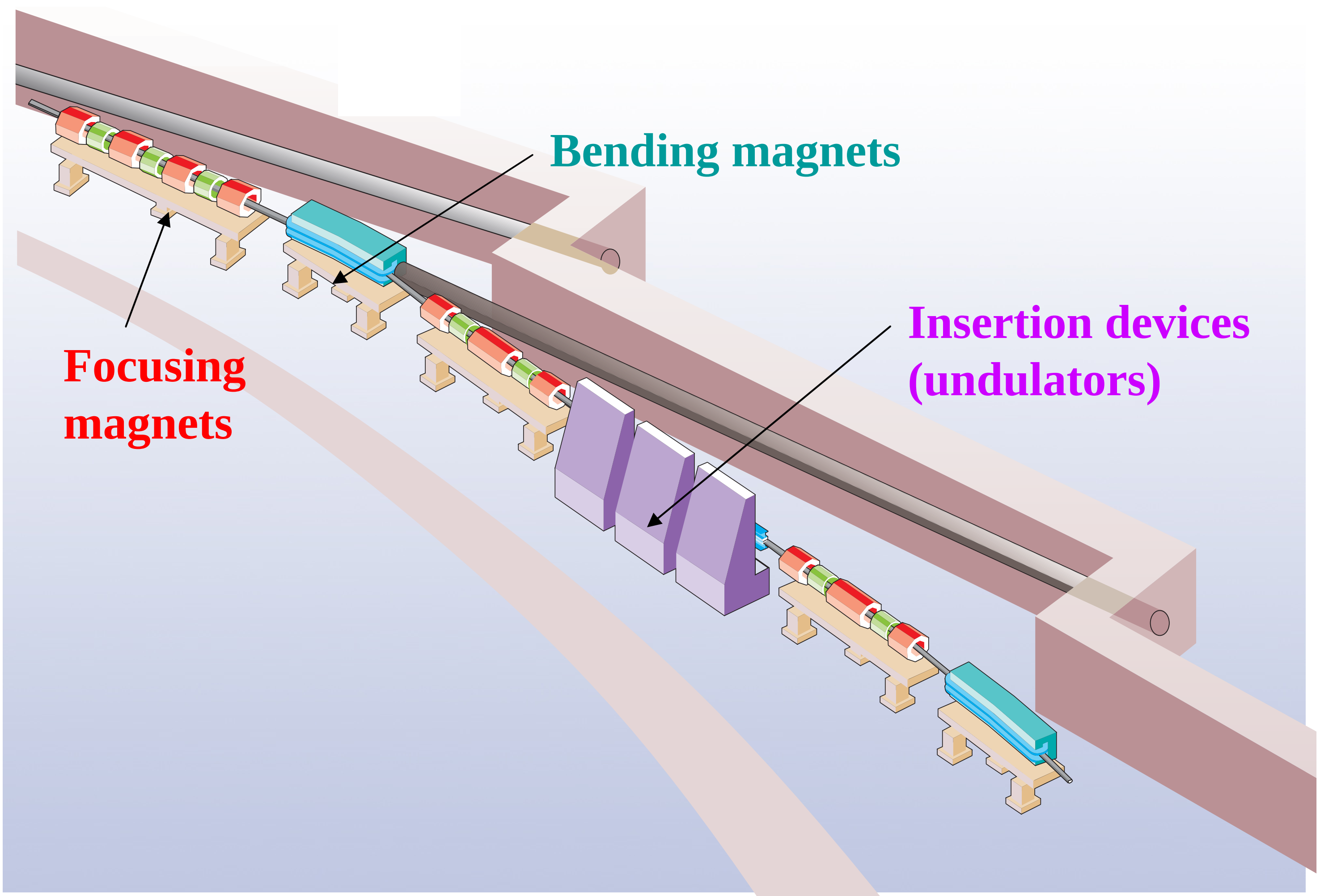
Annual budget : 75 MEuro.

Budget breakdown	%
France	27,5
Germany	25,5
Italy	15
United Kingdom	14
Spain	4
Switzerland	4
Benesync (Belgium, Pays-Bas)	6
Nordsync (Denmark, Finland, Norway, Sweden)	4
Scientific partners :	
Portugal	1
Israel	1
Austria	1
Czech Republic	0,44
Hungary	0,20
Poland	0,60

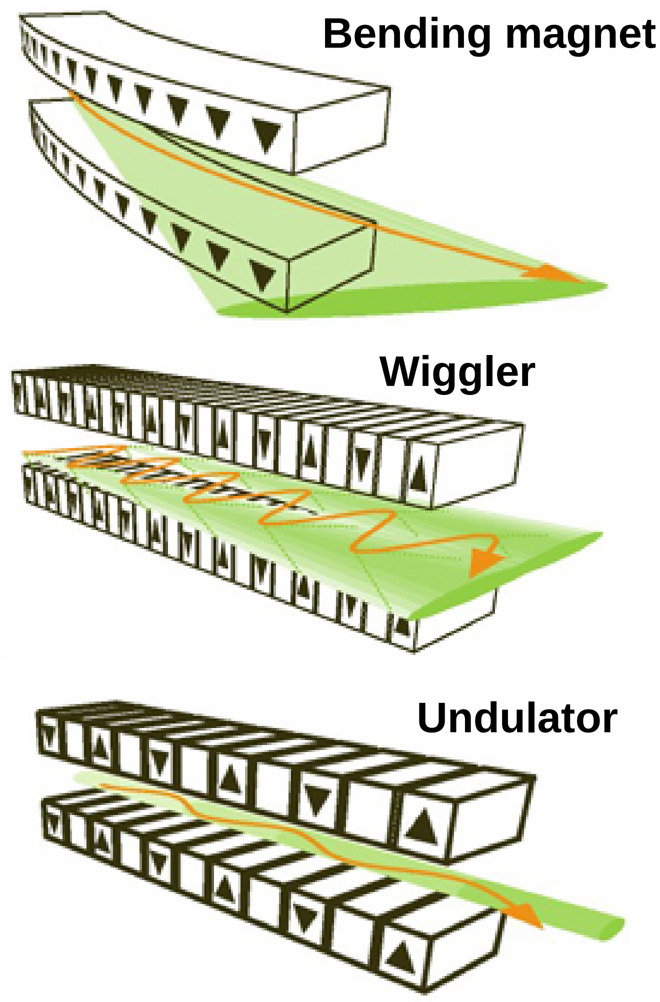
Accelerators and Experimental Hall



Inside the Storage Ring



SR sources and Spectra



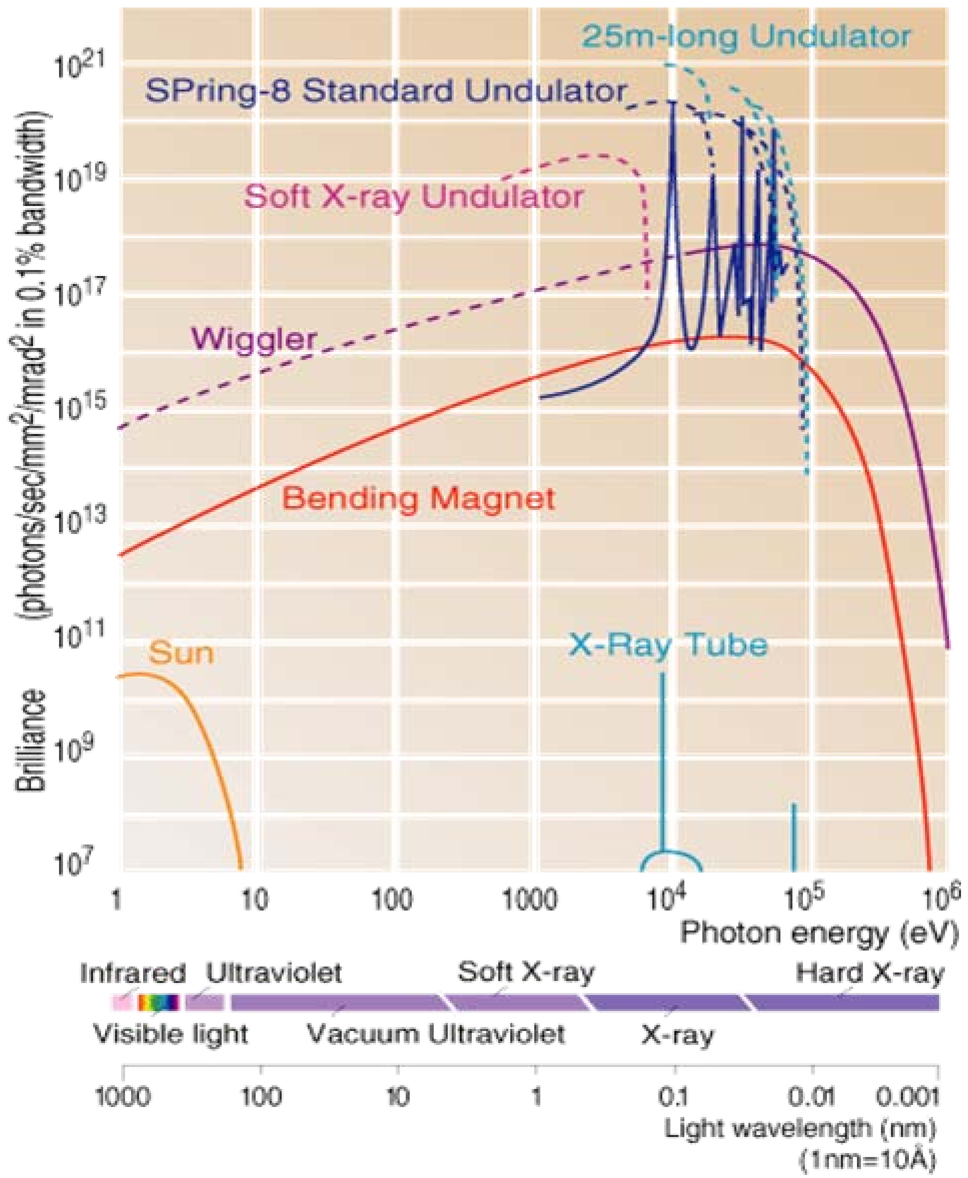


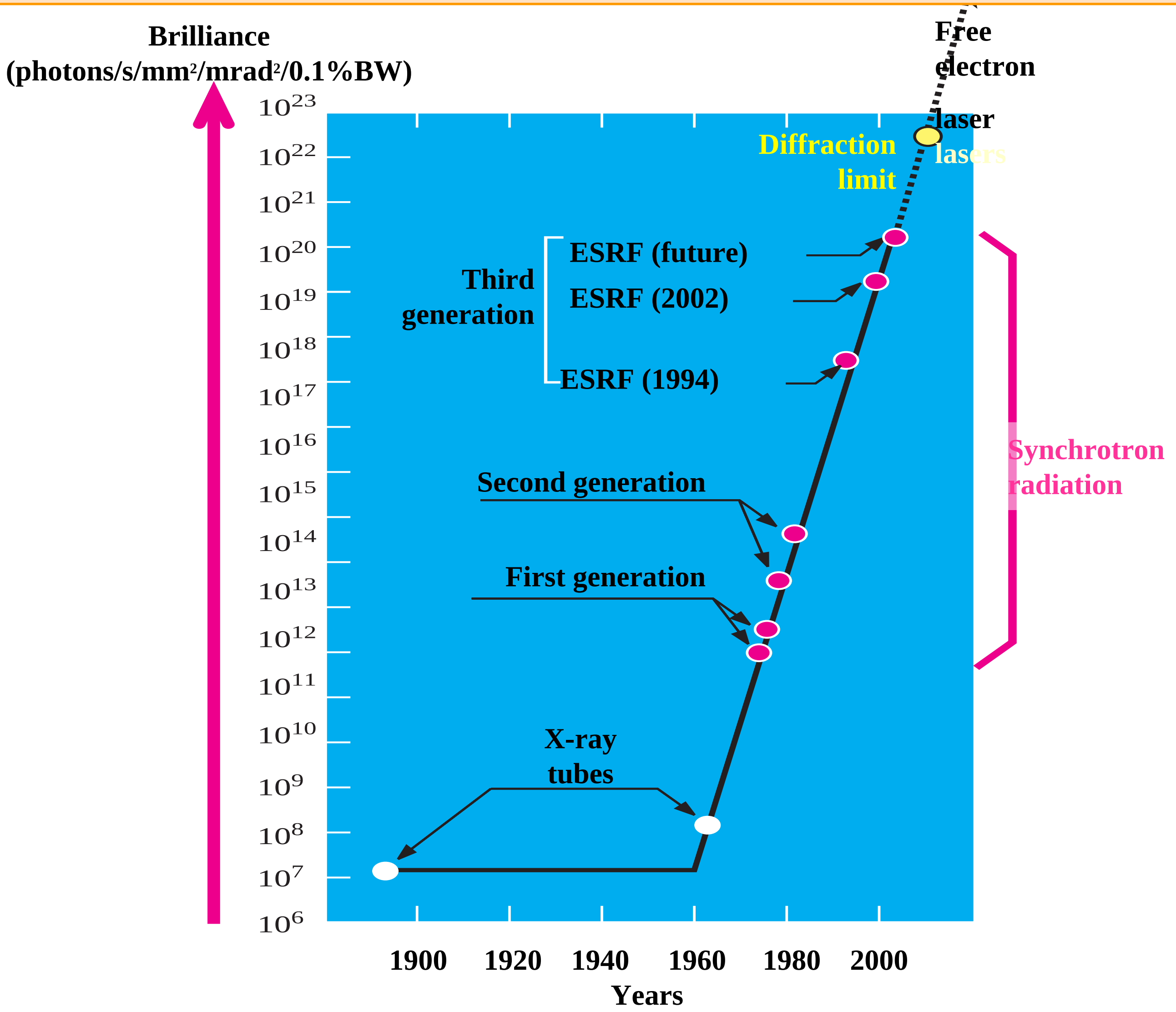
Figure of merit of the source

$$\textbf{Brilliance} = \frac{(\text{Photons/sec})}{(\text{mrad})^2\,(\text{mm}^2\ \text{source area})\,(0.1\%\ \text{bandwidth})}$$

Present SR sources

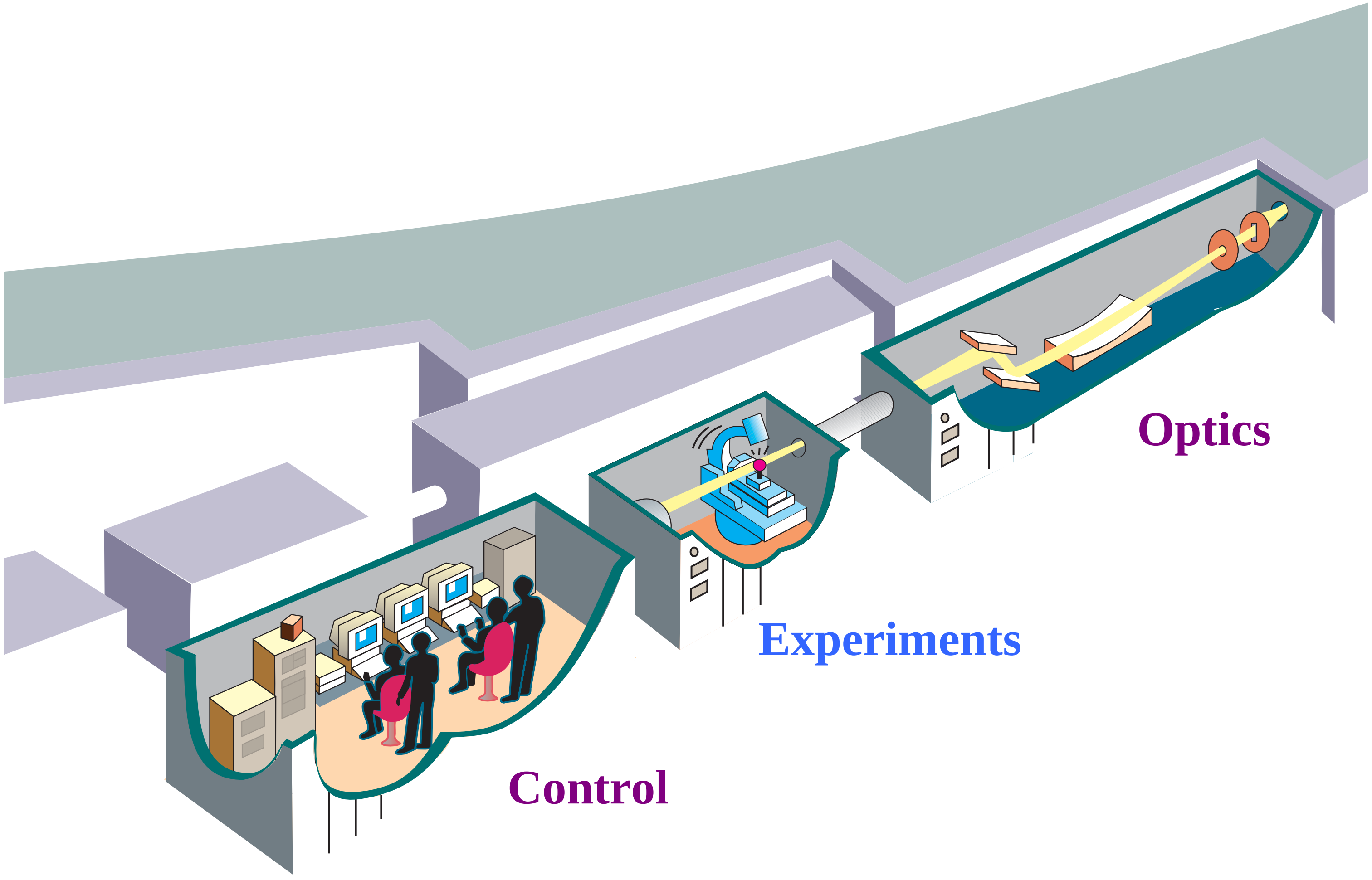
~10^{10} times more brilliant than X-ray generator

History of Brilliance

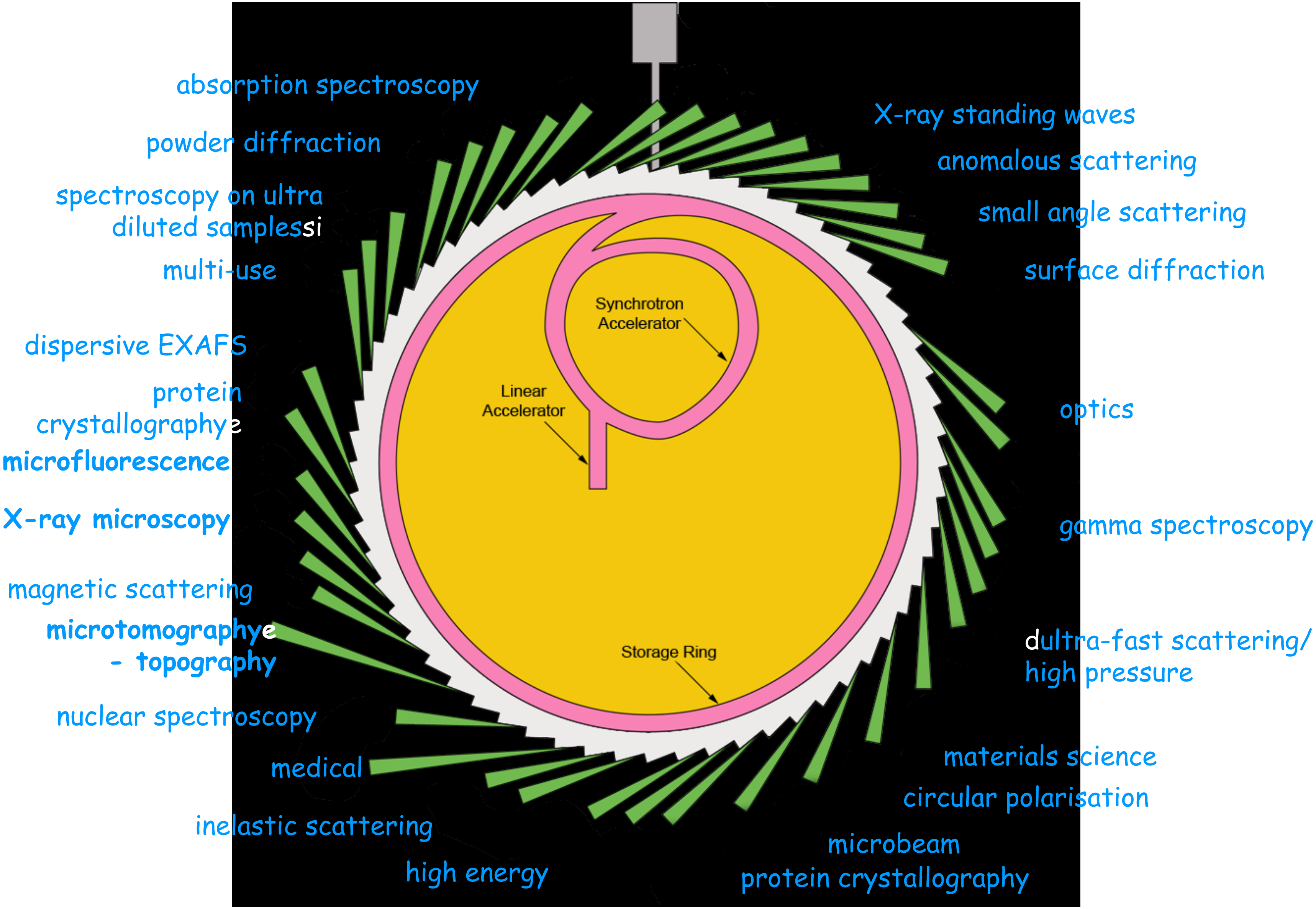


A Typical Beamline

A Typical Beamline



Beamlines



absorption spectroscopy
powder diffraction
spectroscopy on ultra diluted samples
multi-use
dispersive EXAFS
protein crystallography
microfluorescence
X-ray microscopy
magnetic scattering
microtomography - topography
nuclear spectroscopy
medical
inelastic scattering
high energy
X-ray standing waves
anomalous scattering
small angle scattering
surface diffraction
optics
gamma spectroscopy
ultra-fast scattering/ high pressure
materials science
circular polarisation
microbeam
protein crystallography
Synchrotron Accelerator
Linear Accelerator
Storage Ring

SR features important for X-ray imaging

Brilliance

- **High Flux**
  - monochromatic beam
  - Real time experiments
  - weak signals
- **Coherence**
  - Phase contrast images
  - Coherent Diffraction Imaging

Tunability

- Use of absorption edges
- Use of the best energy for a given experiment

The « divergence » of the beam to be considered is the **angular size of the source α = s/L,** typically 10^{-4} m/ 10^{2} m ⇨ ~ 1 µrad

COHERENCE

When is a beam « coherent »?

⇨ the SR source is not coherent

⇨ the SR beam is partially coherent

- **Temporal coherence** ⇨ finite bandwidth of the beam

  Longitudinal coherence length $L_L = \frac{1}{2} (\lambda^2/\Delta\lambda)$

- **Spatial coherence** ⇨ finite extent in space

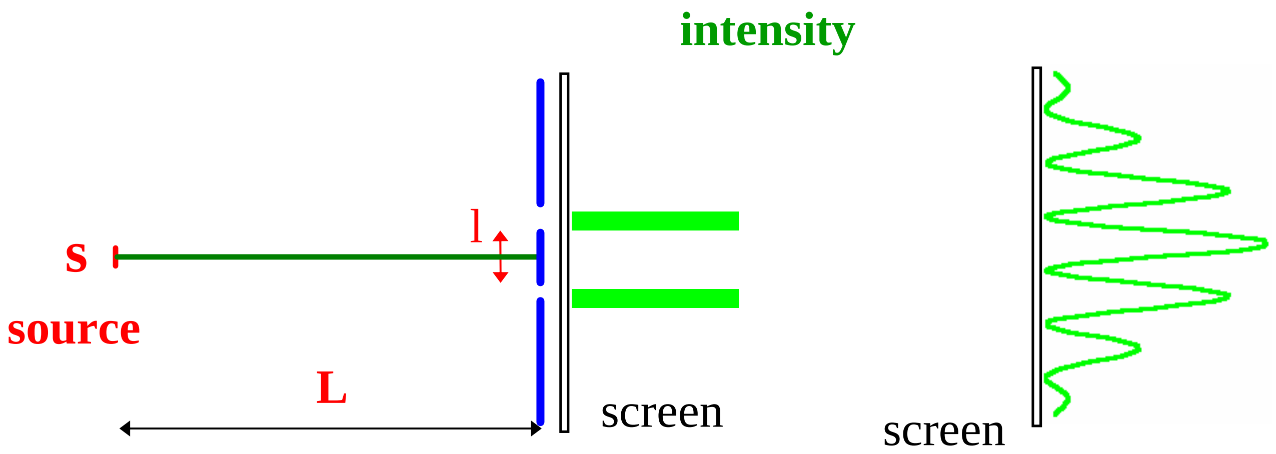
  Transversal coherence length $L_T = \frac{1}{2} (L\lambda/s)$

L : source-to-sample distance **s** : source size

ESRF $\Rightarrow$ partially coherent beam

Young's double slit experiment: the source is not a coherent one

transversal coherence length L_T : $L_T = \lambda L / 2s$



If screen is very close $\Rightarrow$ absorption image

s size of the source
L distance source-sample
l slit separation
λ wavelength of the radiation

For visible light $L_T \sim$ 1mm
For X-rays usually $L_T <$ 1 µm
For ID19, at the ESRF:
L = 150 m, s ≈ 50 µm $\Rightarrow$ $L_T \approx$100 µm

X-ray transmission imaging

Interaction of X-rays with matter: complex refractive index

$$n = 1 - \delta + i\beta$$

Absorption part expressed by β $\leftrightarrow$ $\mu = 4\pi\,\beta/\lambda$

λ wavelength, μ linear absorption coefficient

Phase variation $\Delta\phi$ related to δ

$$\Delta\phi = 2\pi\,\delta\,t/\lambda$$

3rd generation SR beams are coherent because the angular size of the source is very small

Index of refraction

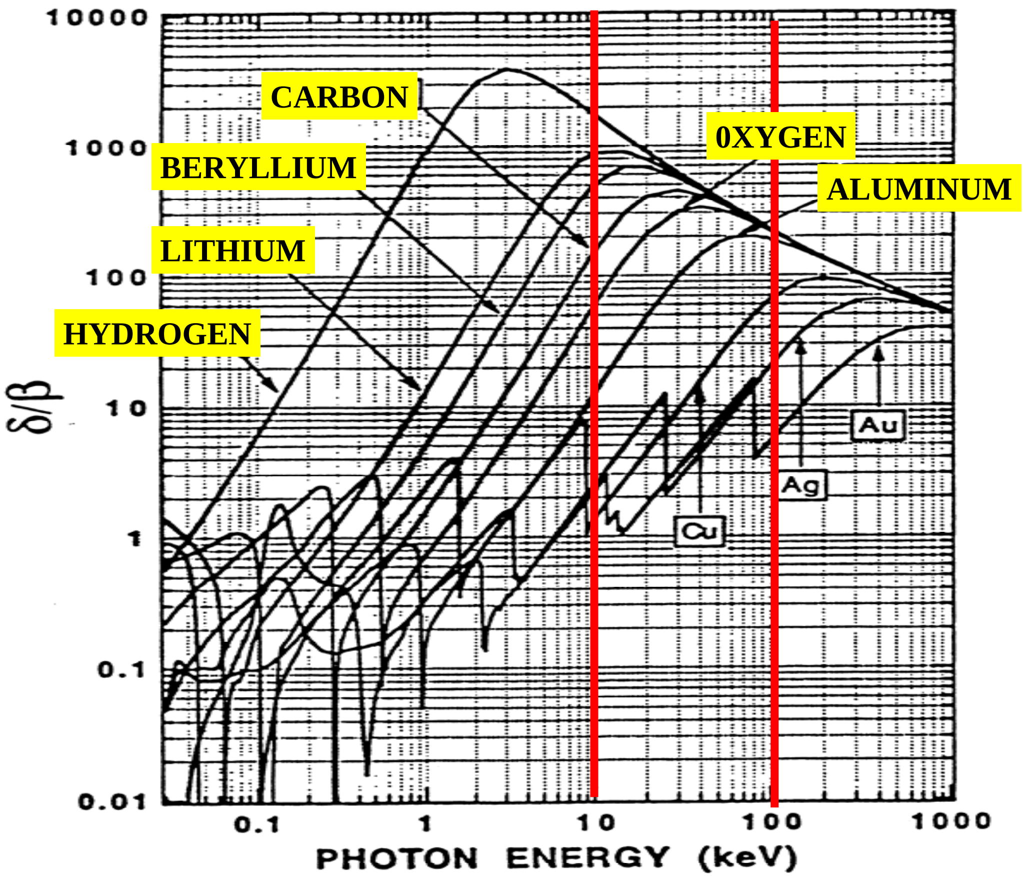
$n = 1- \delta + i\beta$

Ratio phase and amplitude (δ/β) as a function of the X-ray energy

10→1000 in the

hard X-ray range

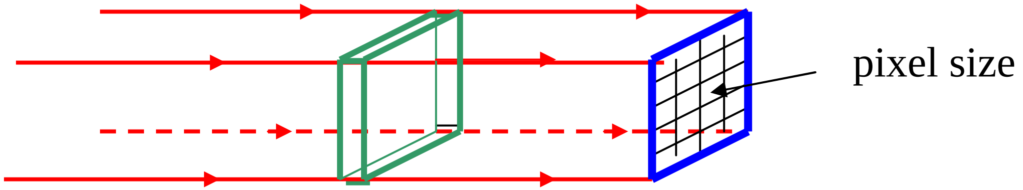
enhanced sensitivity



What type of imaging do we perform at SR sources?

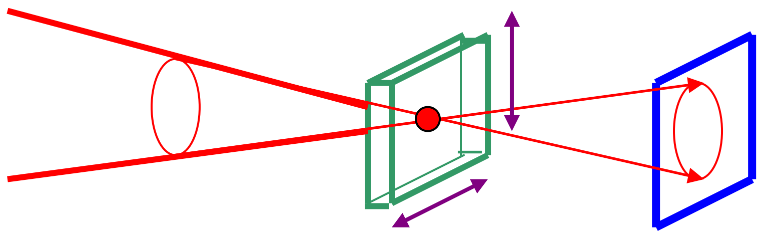
Imaging with a parallel and extended beam

the spatial resolution is mainly a function of the detector

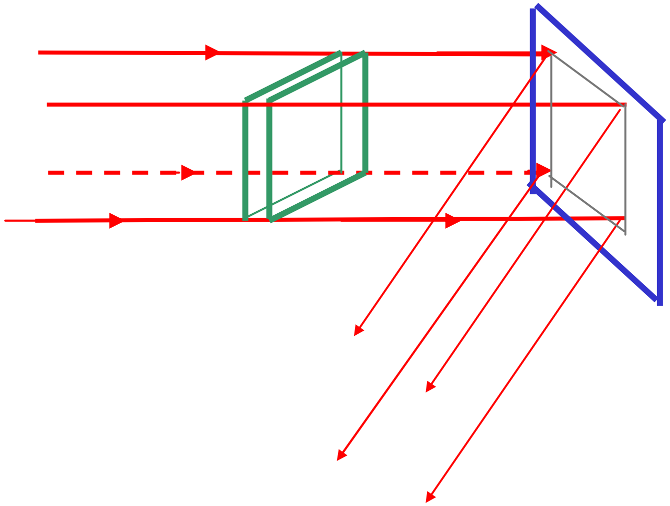


Imaging with a microbeam (« scanning »)

the spatial resolution is mainly a function of the spot size



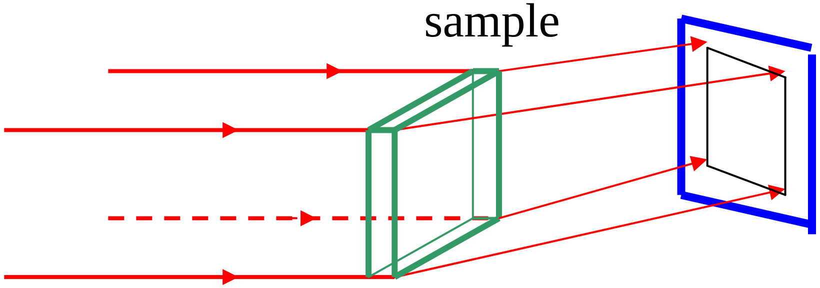
Analyzer based imaging (DEI, …)



Shows variations of density (index of refraction) through the refraction they entail

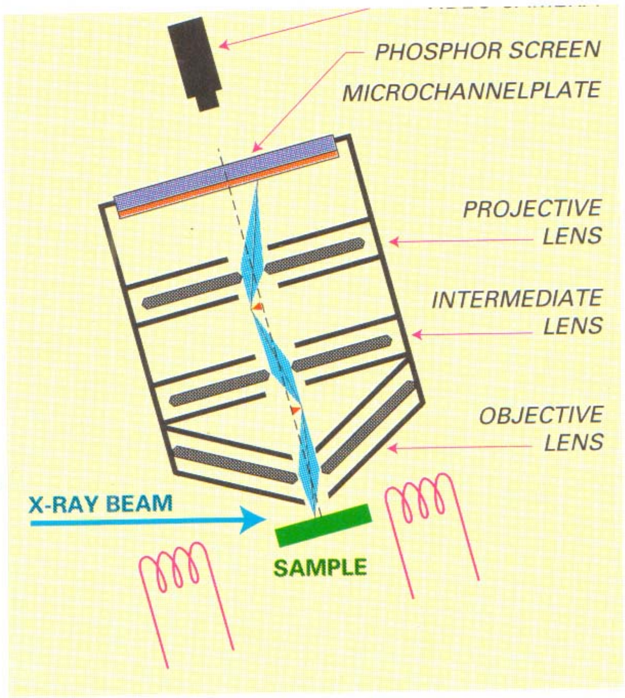
Important in medical imaging (cartilages, …)

Bragg diffraction imaging (topography, Rocking Curve Imaging)



Shows defects, domains, phases, … in the crystal through their associated variation of distortion

PEEM imaging



and, in addition,

Coherent diffraction imaging

…and all combinations of imaging techniques,

which are not covered in this introduction, but that you'll see during this course

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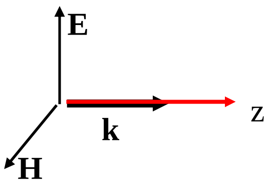
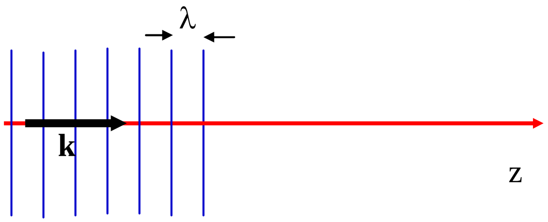
  (DEI, “topography”, diffraction tomography)

Monochromatic beam, basic processes
X-rays interactions with matter

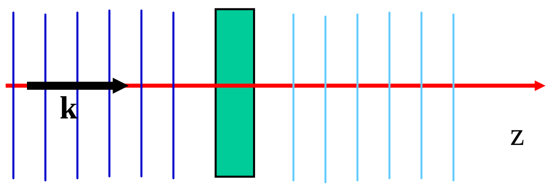
Monochromatic beam: electromagnetic transverse waves, $k = 2\pi/\lambda$, $\lambda \sim 10^{-10}$ m



E (keV) = 12.4 / λ (Å)



Absorption

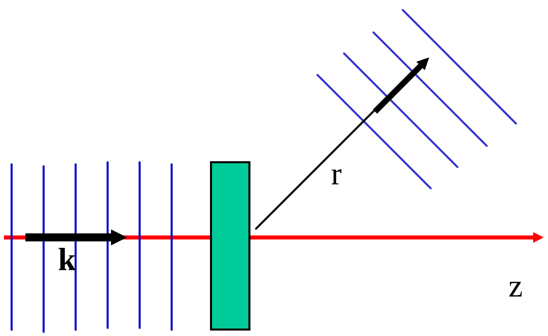


Below E ~ 100keV, mainly photoelectric effect

Scattering

Elastic (Bragg diffraction, for instance)

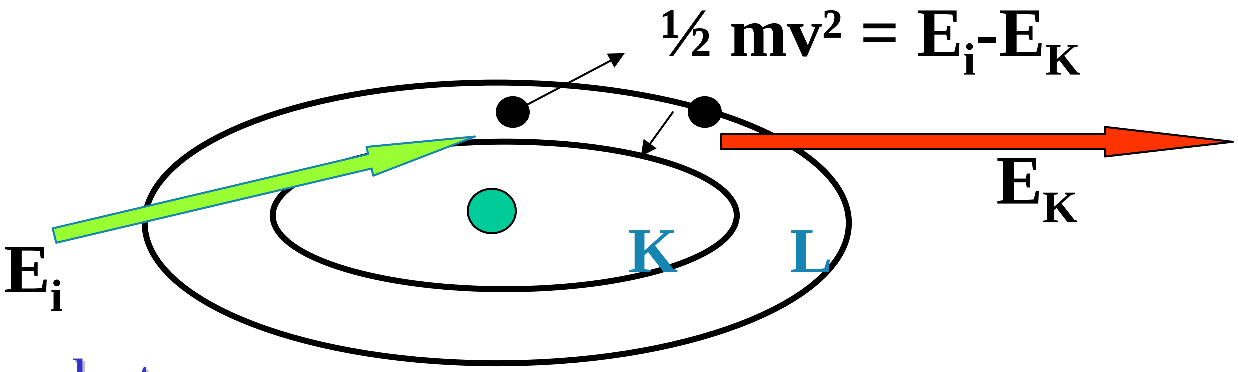
Inelastic (Fluorescence, for instance)



Refraction (actually related to scattering)

X-ray imaging utilizes several of the basic interactions of X-rays with matter

Absorption (mainly through **photoelectric effect** on deep shell electrons (K, L, M...)**)**



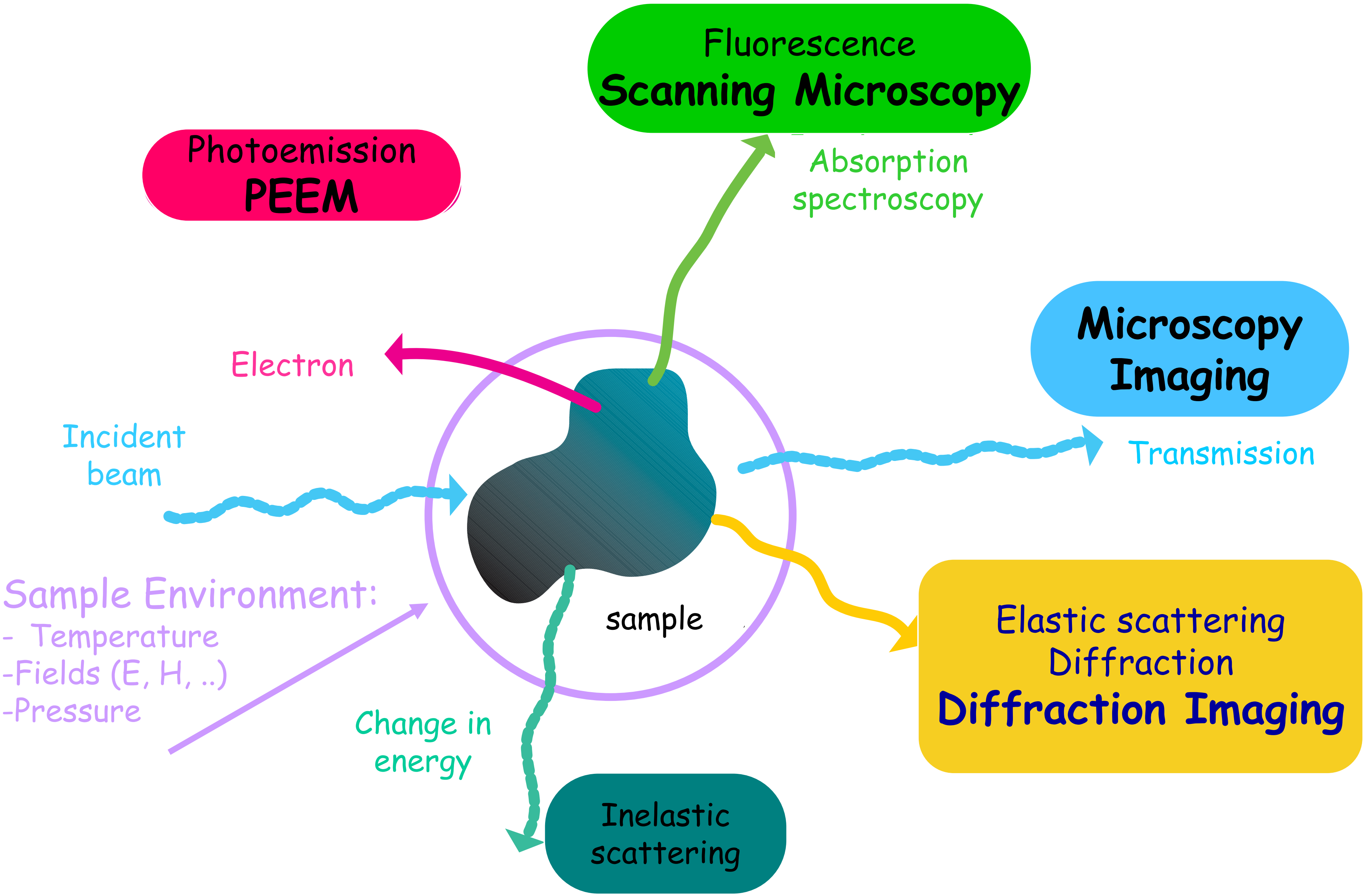
Fluorescence photons

Refraction (or, more generally, modification of wave front)

Bragg diffraction

Photoemission…..

The techniques : light-matter interaction



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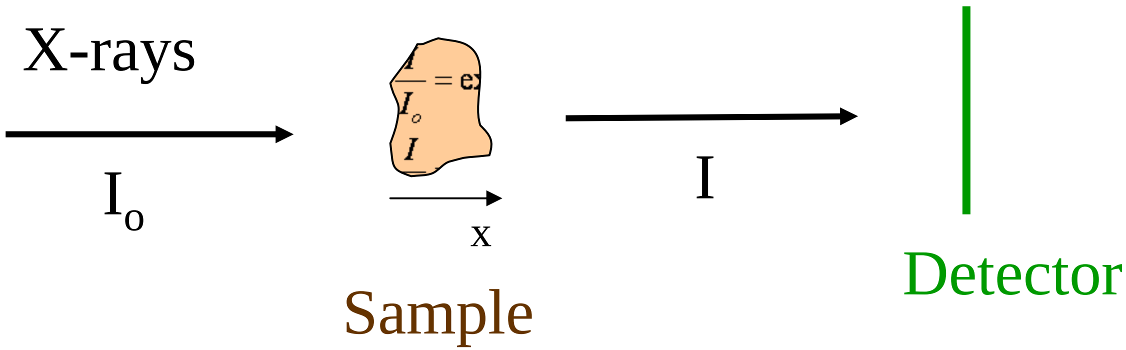
  (DEI, “topography”, diffraction tomography)

Radiography

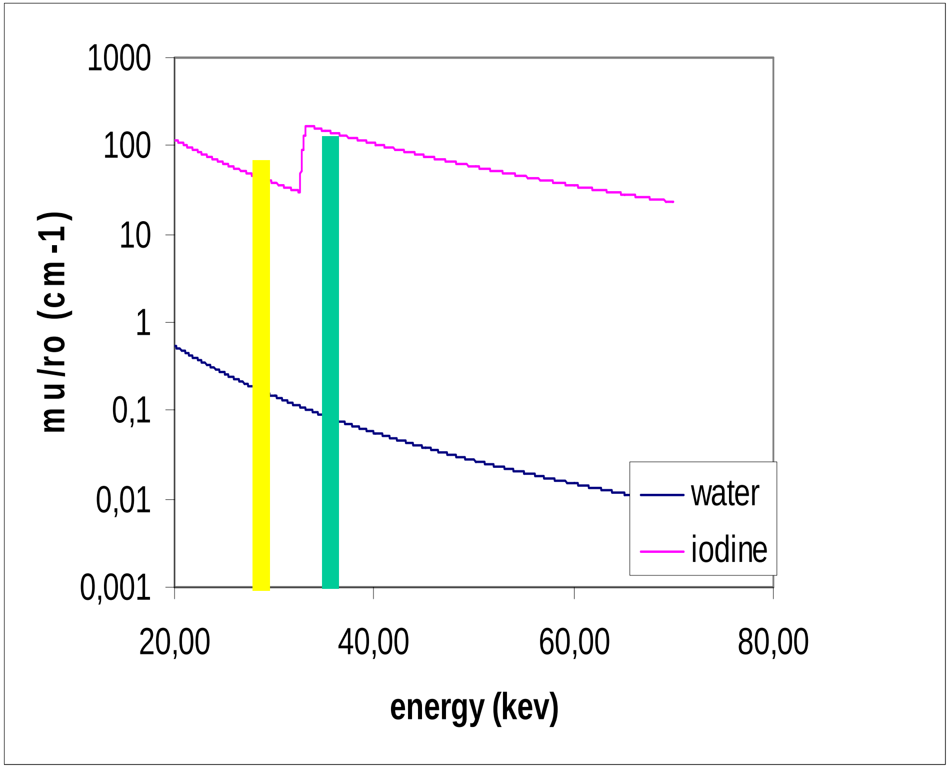


Lambert-Beer Law:

$$\frac{I}{I_o} = \exp \int -\mu dx$$

μ, absorption coefficient

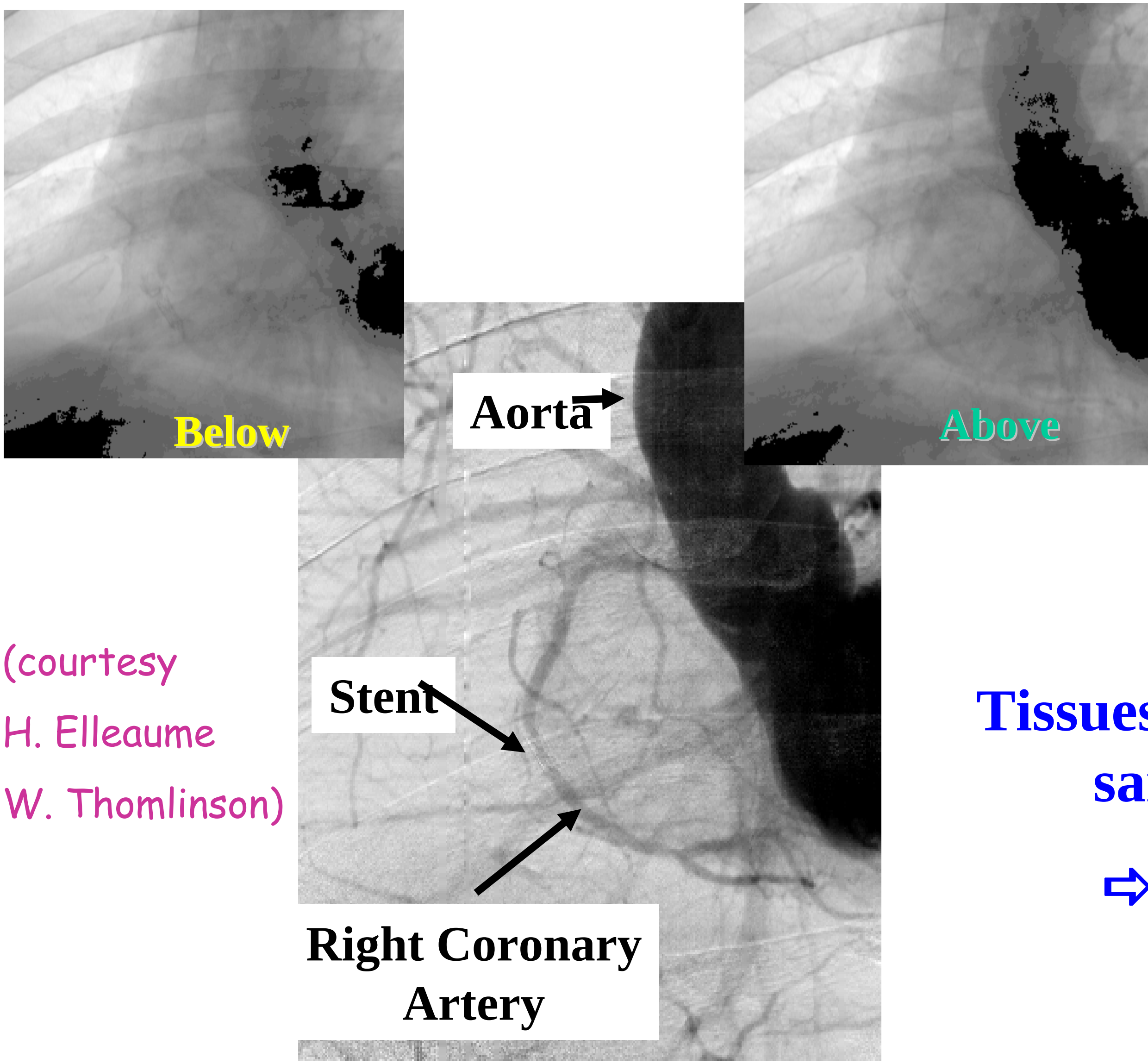
Using the absorption edges
(« chemically selective radiography »)



A way to visualize a given element within a sample is to record 2 images, above and below the element absorption edge, and to substract the images.

The other elements display, for these two very close energies, nearly the same absorption

Medical imaging: angiography, first human patient at the ESRF (January 2000)



Dual Energy Imaging

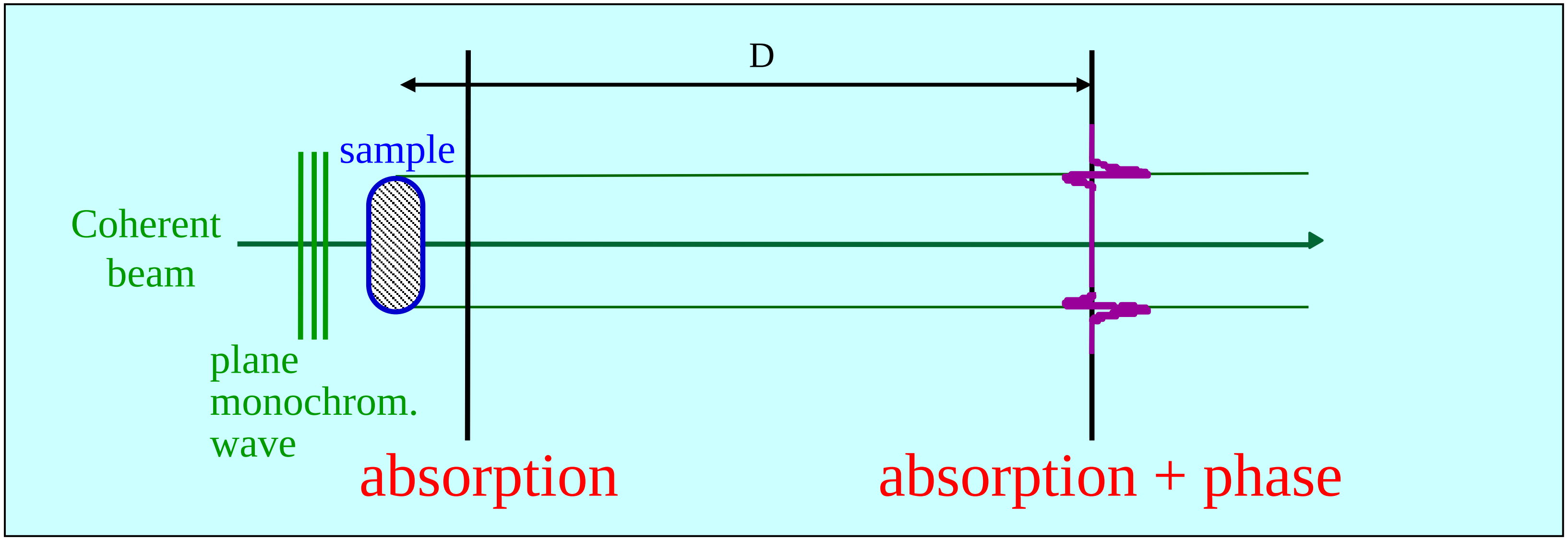
subtraction of images recorded above and below the “absorption edge” of iodine

(courtesy
H. Elleaume
W. Thomlinson)

Tissues display nearly the same absorption

⇨ iodine is seen

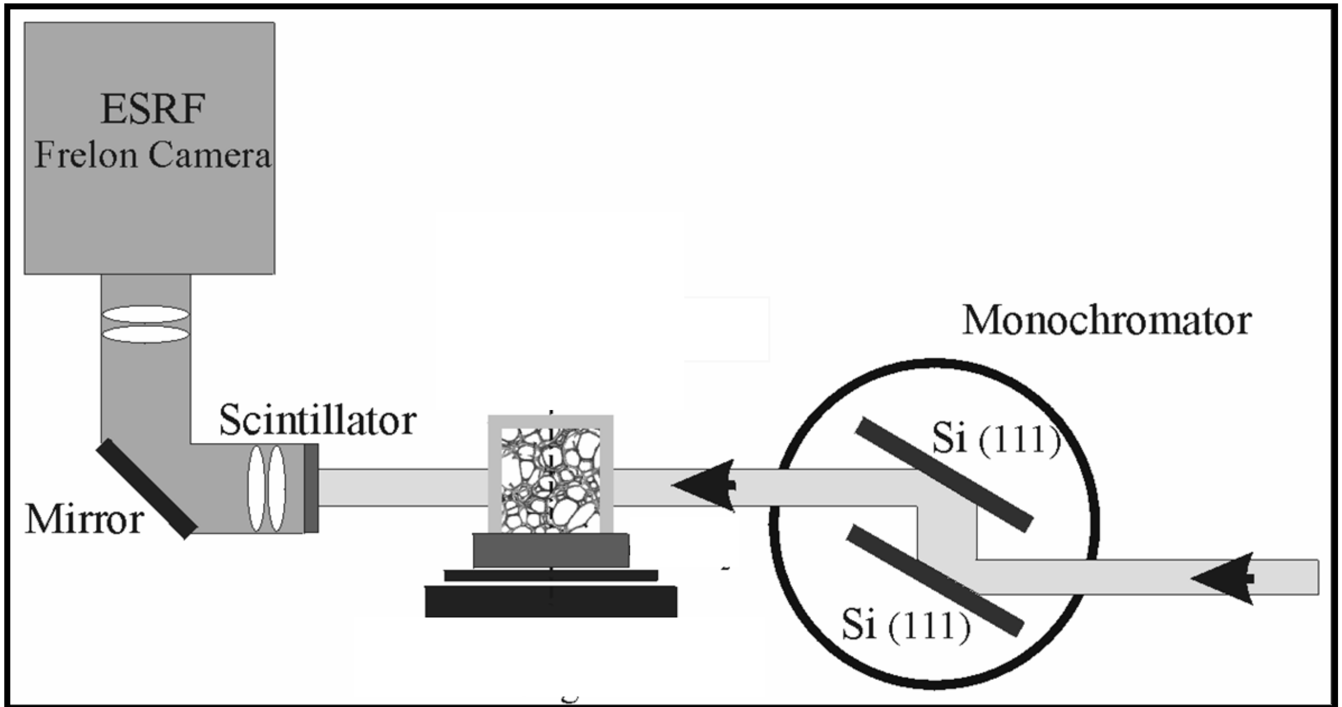
Using the coherence of the beam

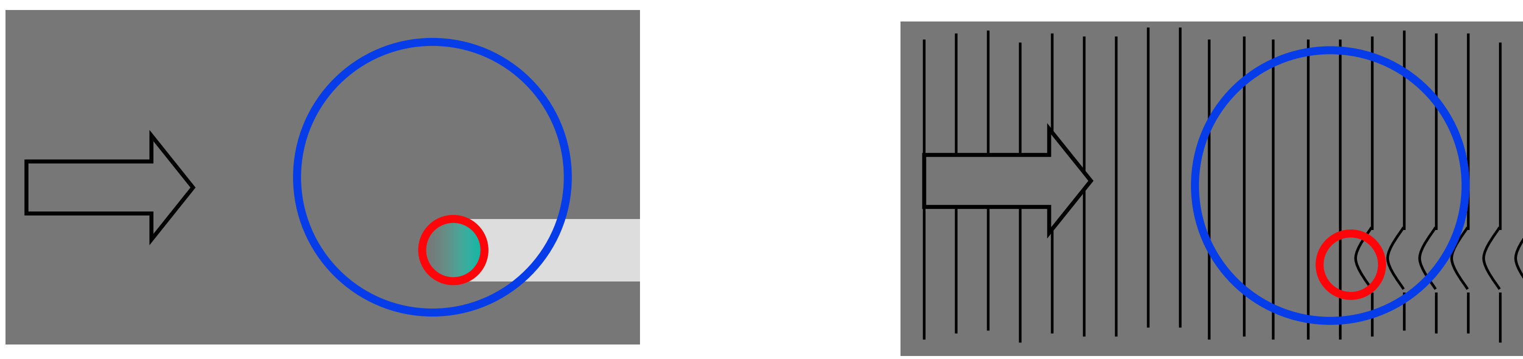


Phase Sensitive Radiography: experimental layout

Depending on the distance D

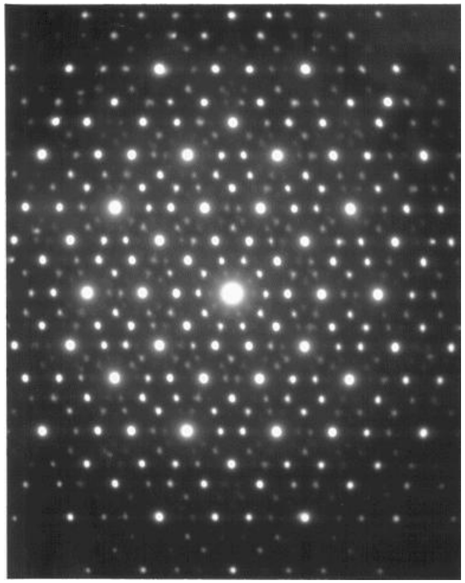
- ⇨ Absorption only
- ⇨ "edge detection"
- ⇨ "hologram"





Coherent beam ⇨ Phase Contrast Imaging: enhanced sensitivity

SR–based radiography as to investigate the porosity of **quasicrystals**

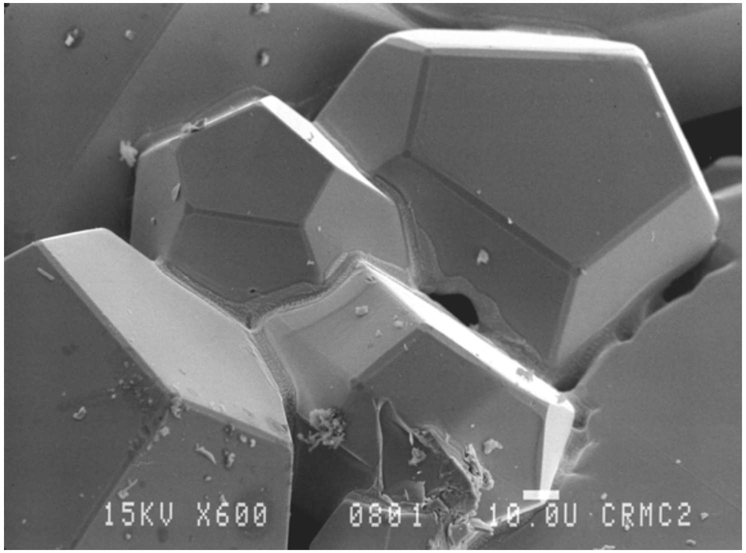


Shechtman, Blech and Gratias, 1984

→*quasiperiodic* translational order

→ orientational symmetries forbidden for crystals (fivefold for instance)

→ sharp diffraction patterns

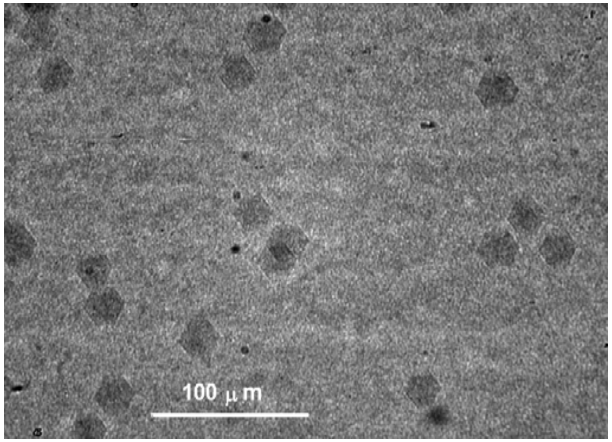


Al Pd Mn displays unusually big pores (~ 10µm)

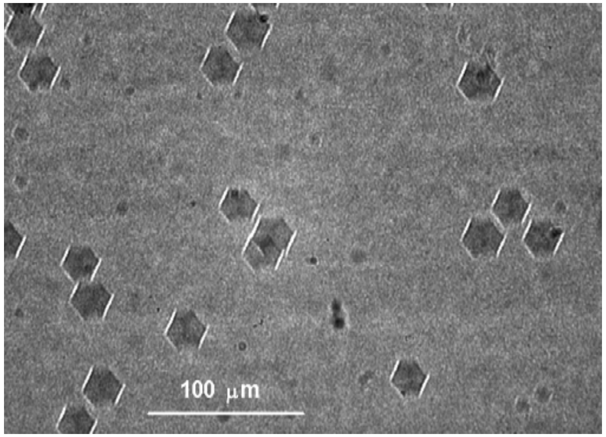
Phase Sensitive Radiography on a AlPdMn quasicrystal grain

- Resolution = 1µm

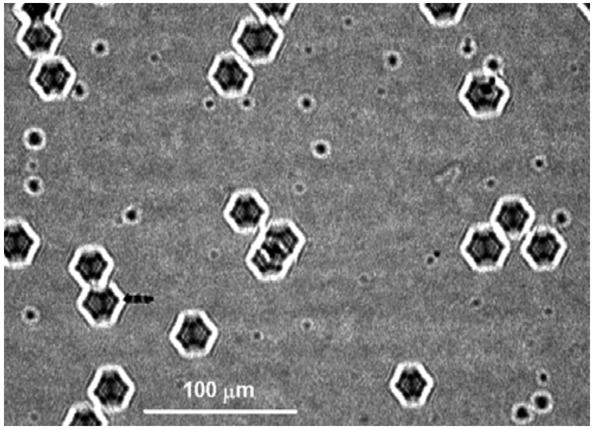
λ = 0.35Å



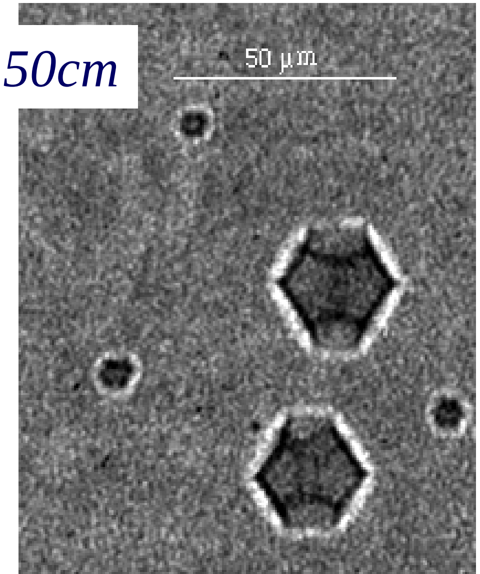
D = 1.3cm



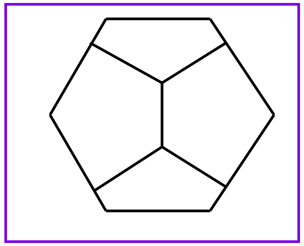
D = 10cm



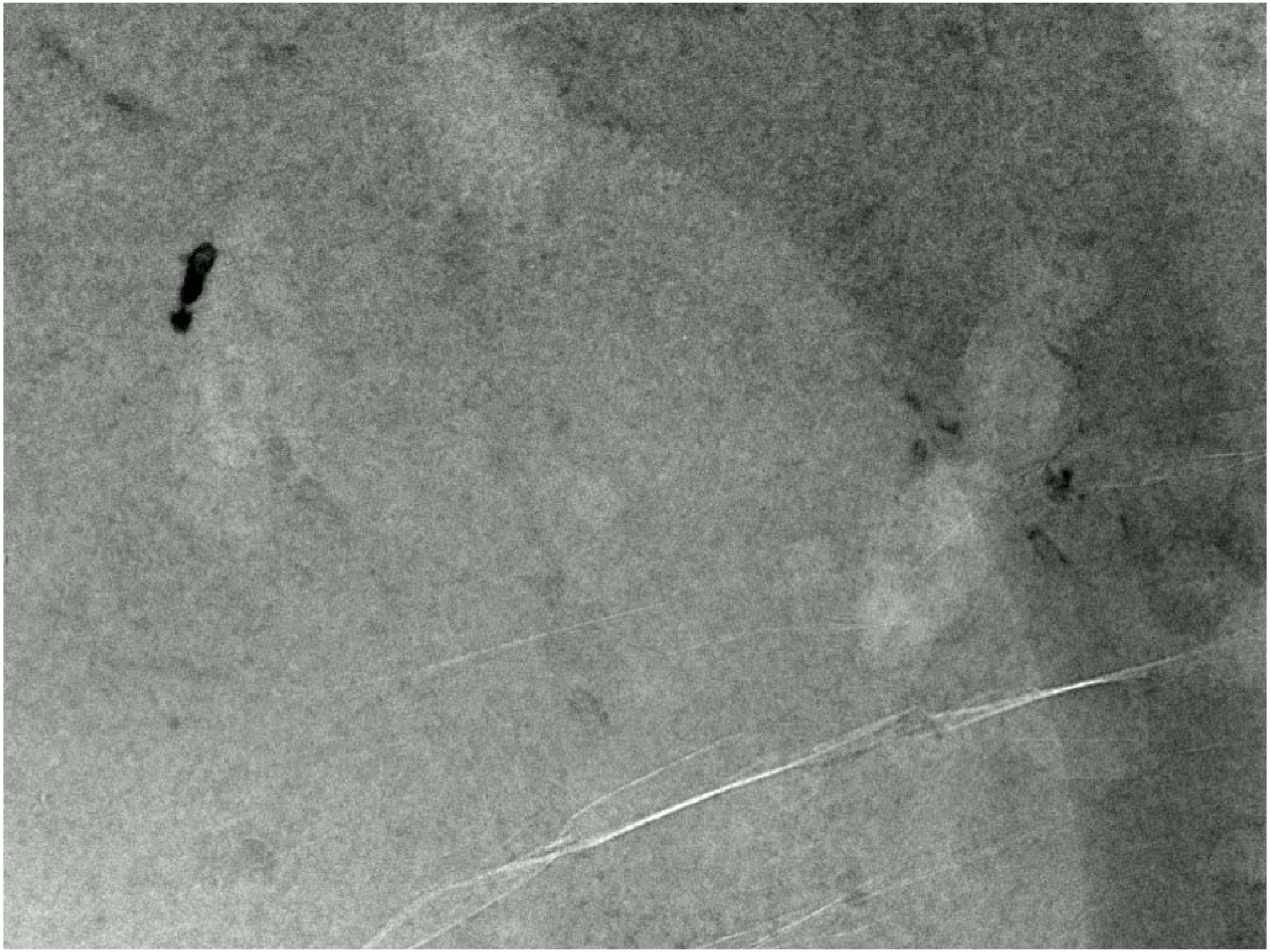
D = 50cm



dodecahedron seen along the 2-fold axis



Phase contrast imaging on fossil insects in 10^8 years old opaque ambers



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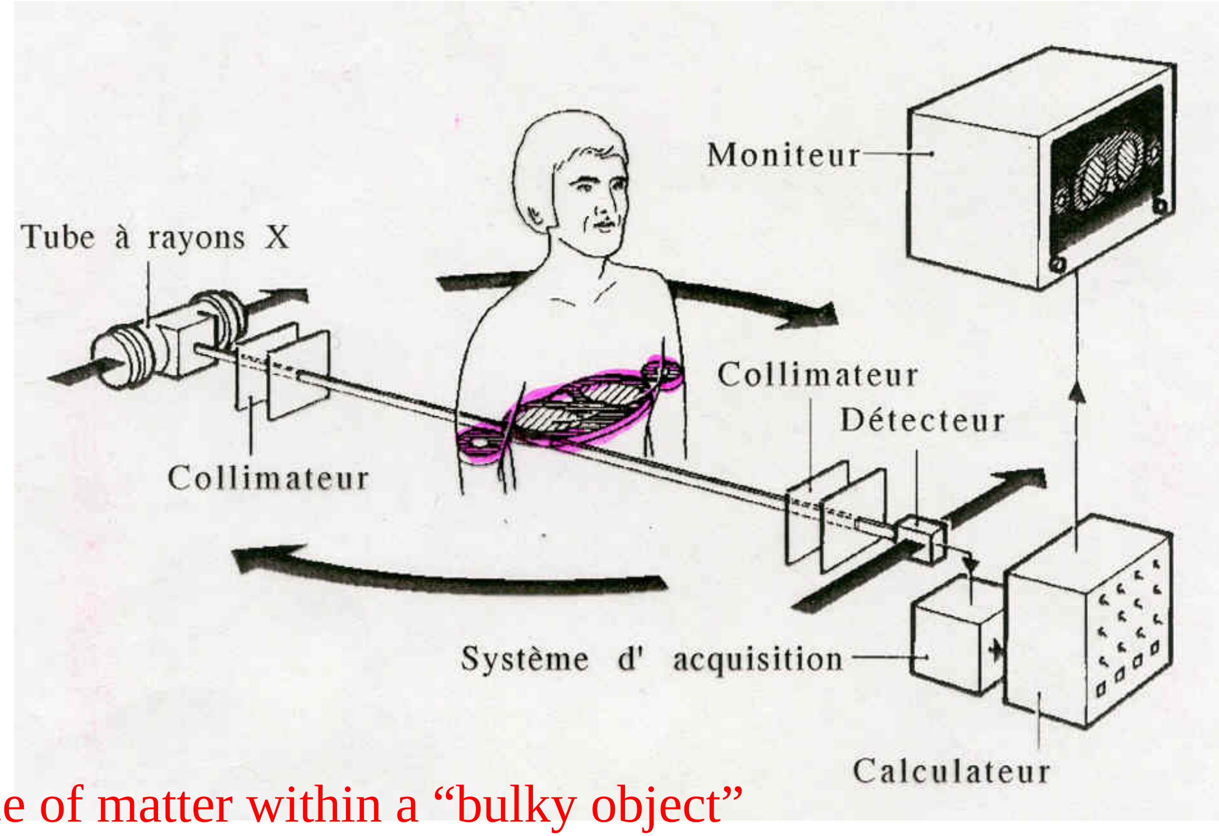
Medical Scanner

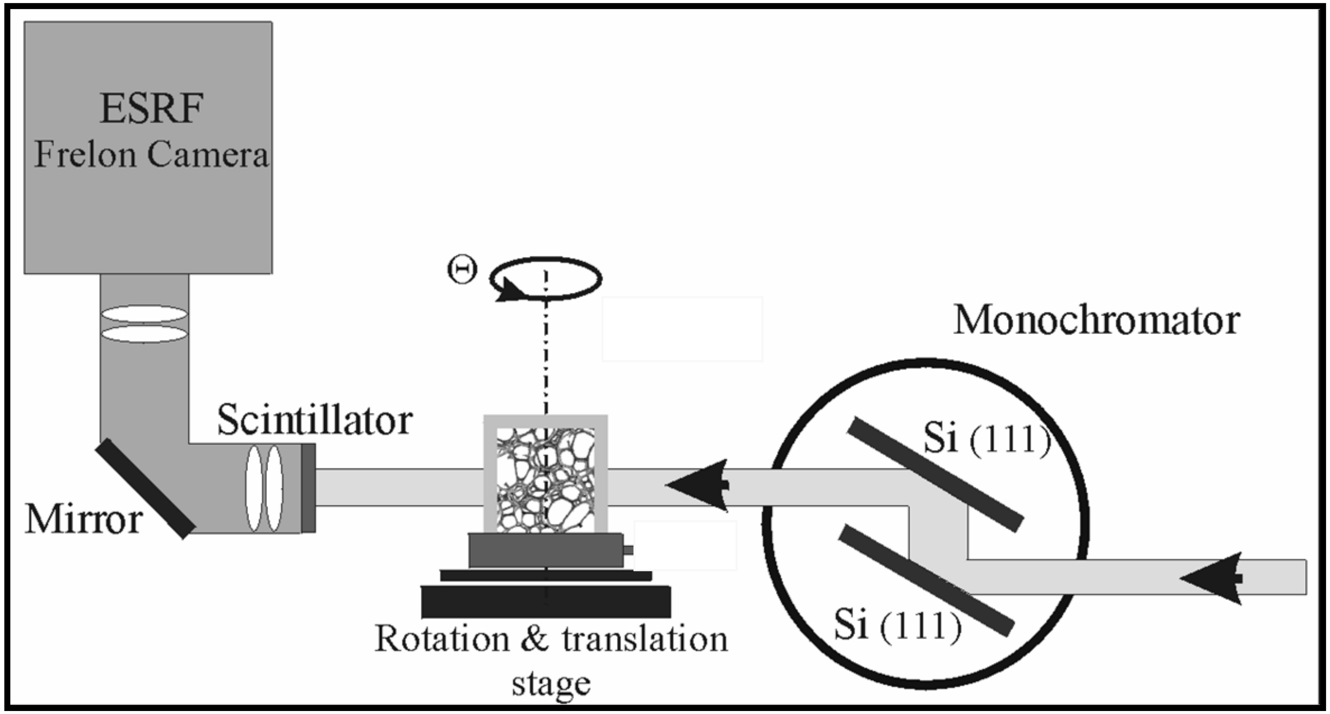


- slice of matter within a “bulky object”
- based on radiographs collected at various angles

Specificity of SR Microtomography

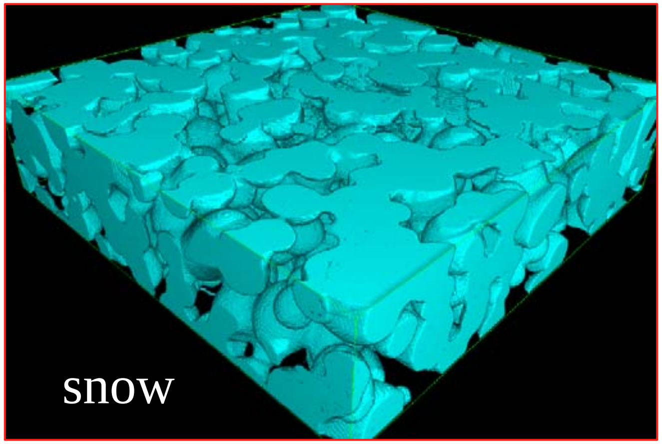
- parallel, coherent, monochromatic, high flux beam

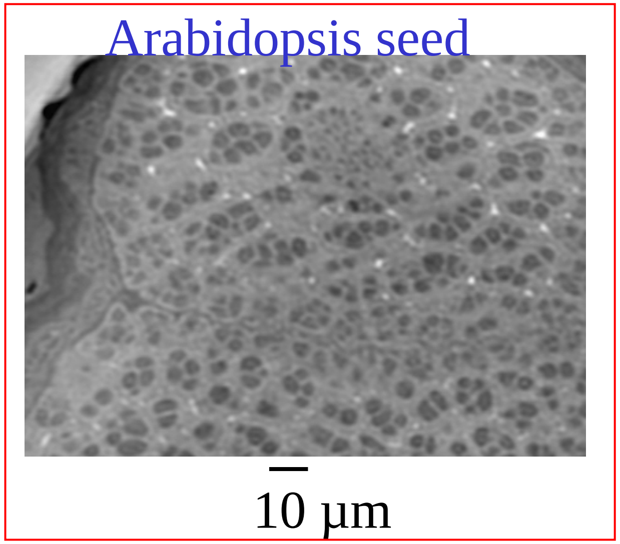
⇨ High spatial (0.5 μm) and temporal resolution, quantitative measurements, in-situ, real-time, images

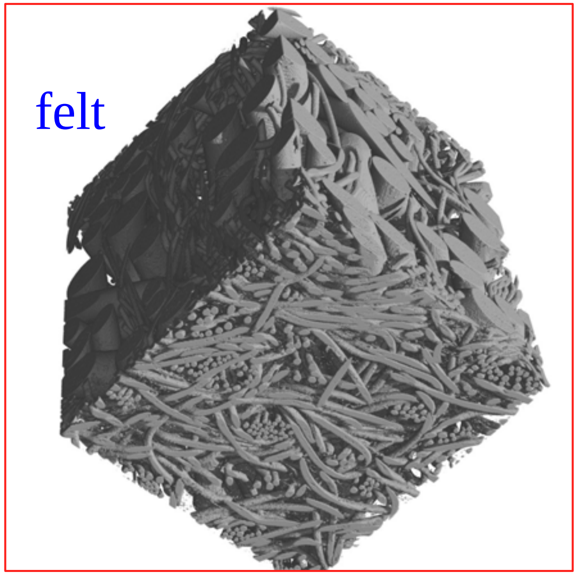


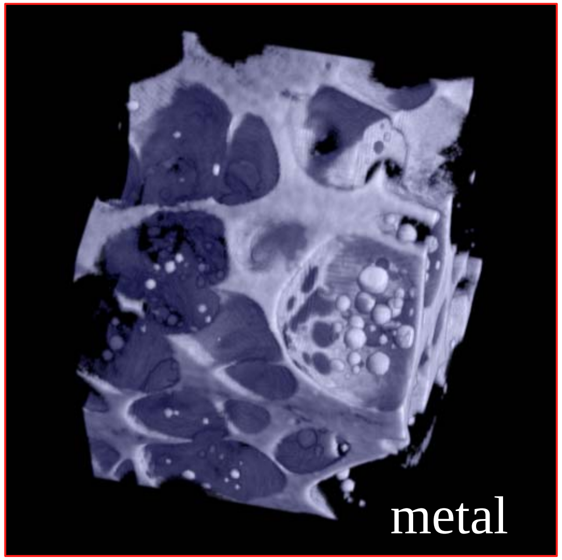
Rotation from 0 to 180 °, ~ 900 radiographs

Microtomography: a multidisciplinary technique



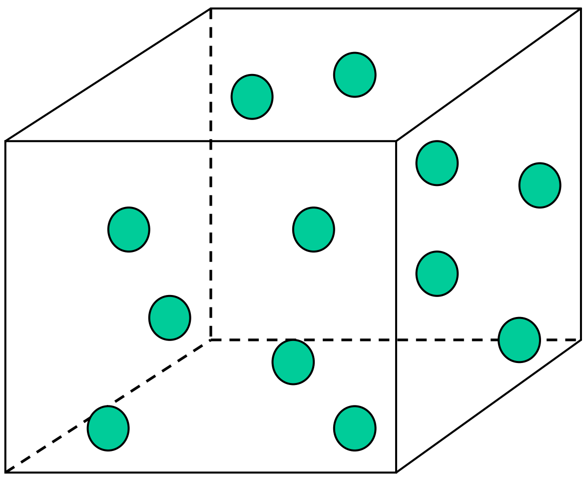




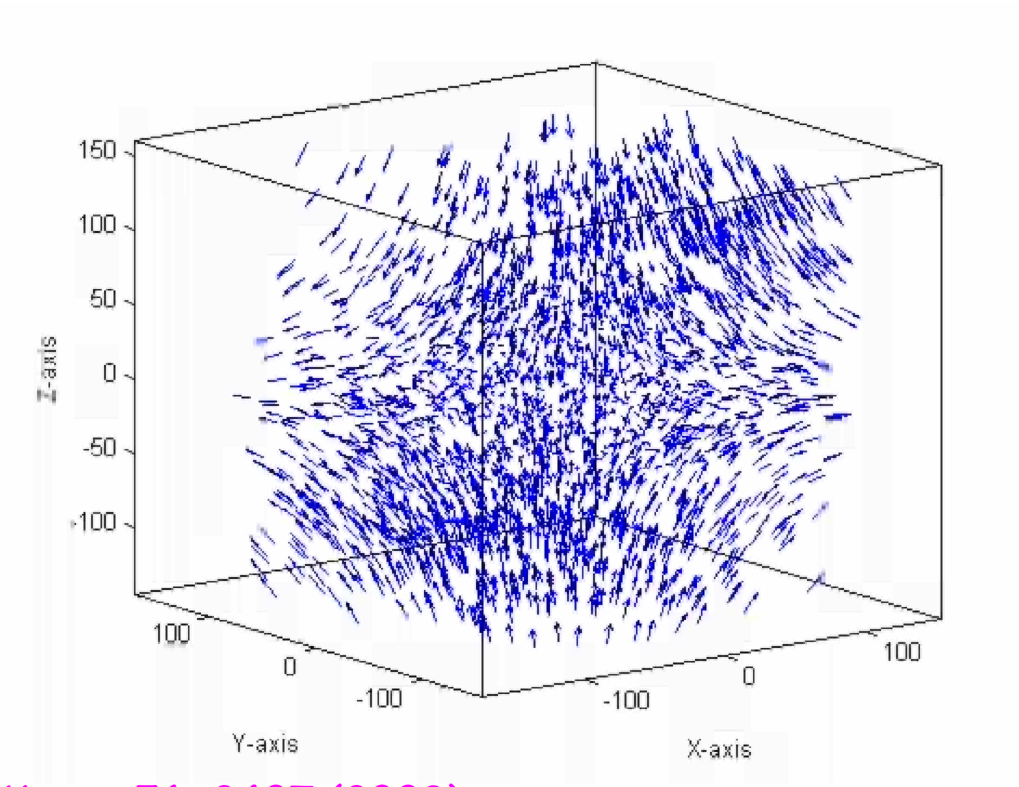


Tomography can reveal plastic strain field

Markers:



Compression of Al/W sample:

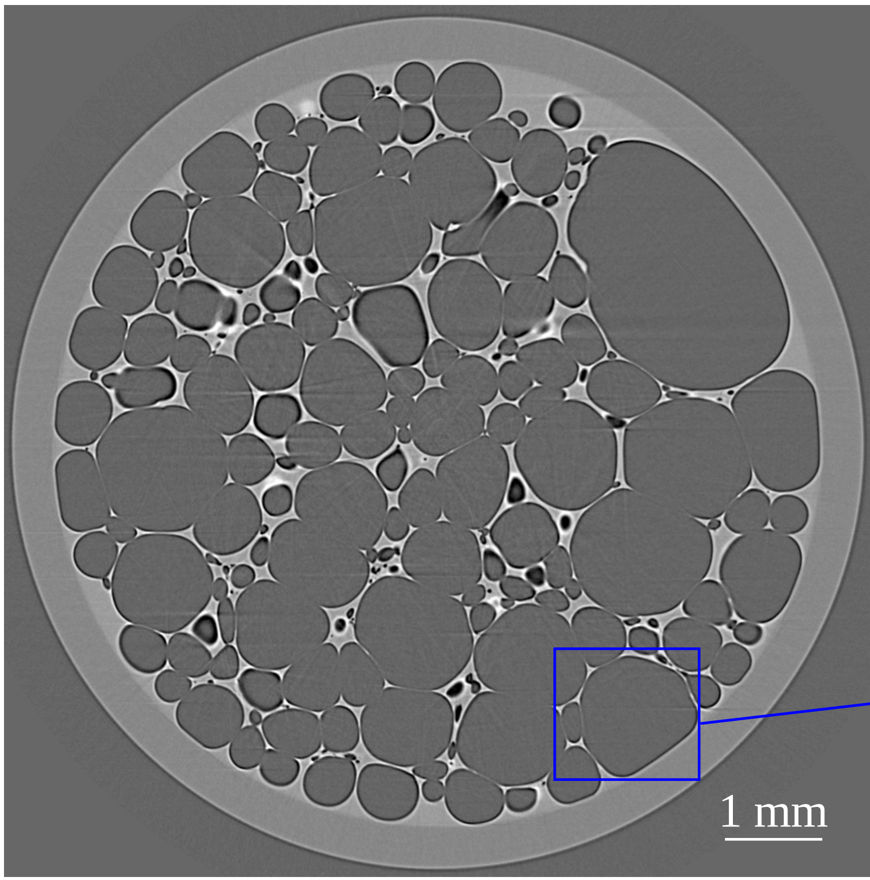


Work performed at HASYLAB (Germany)

S.F. Nielsen, F. Beckmann *et al. Acta Mater.* **51**, 2407 (2003)

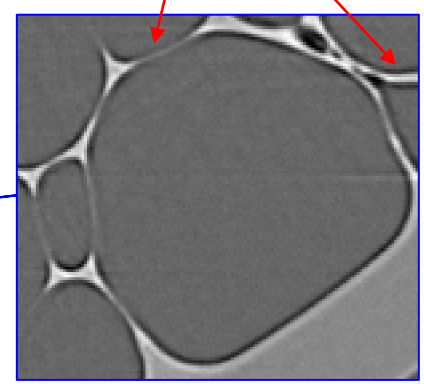
Phase Contrast: Liquid Foams

Growth or disappearance of bubbles in a liquid foam in 3D
(the “mousse au chocolat is an example of “liquid foam”)



Phase enhancement to visualise
liquid films separating bubbles:
Film thickness << voxel size

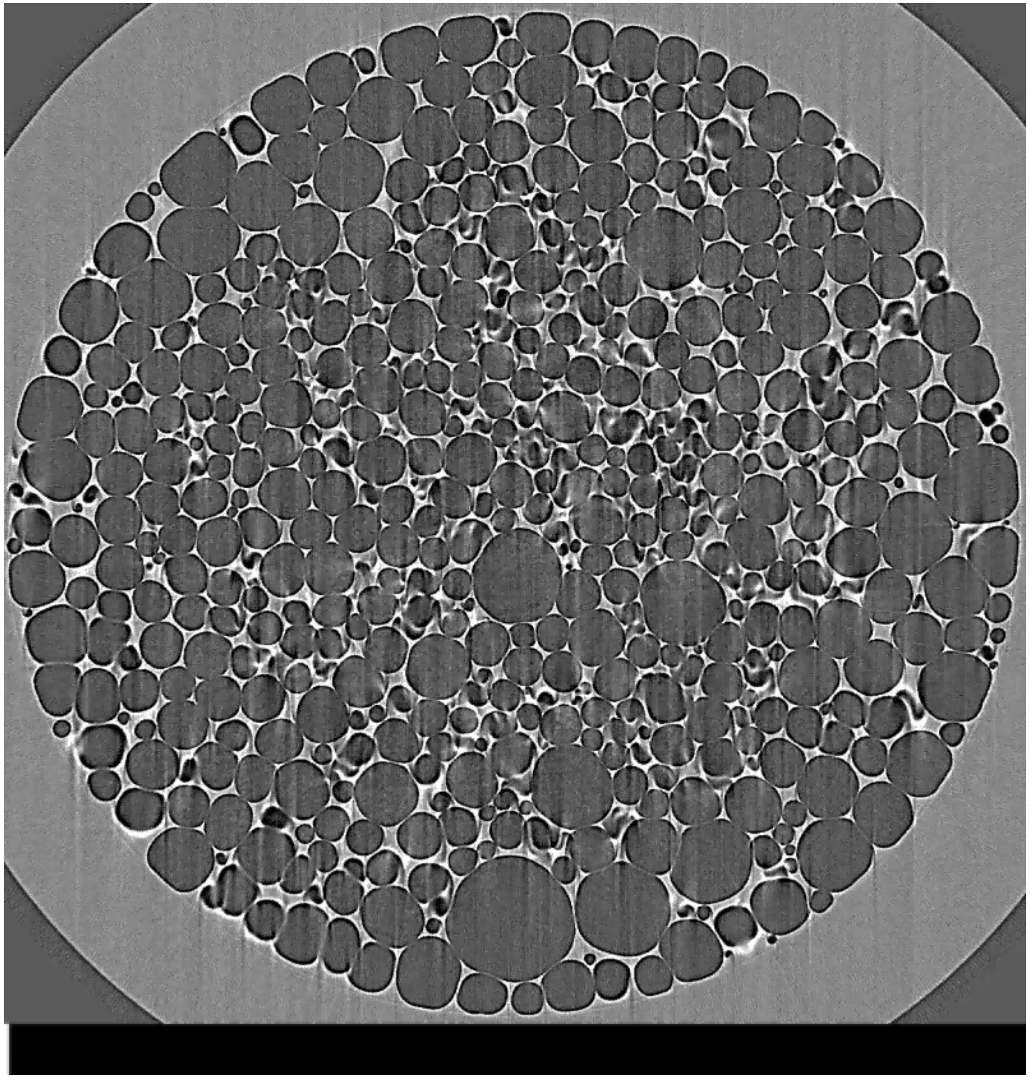
thin films



E = 15 keV, Sample-detector distance: 0.15 m

F. Graner, P. Cloetens

Temporal evolution of a liquid foam



F. Graner, P. Cloetens

Non destructive investigation of Cretaceous enigmatic tiny eggs from Thailand

Dinosaur or bird ?

PALEONTOLOGY

P. Tafforeau, E. Buffetaut

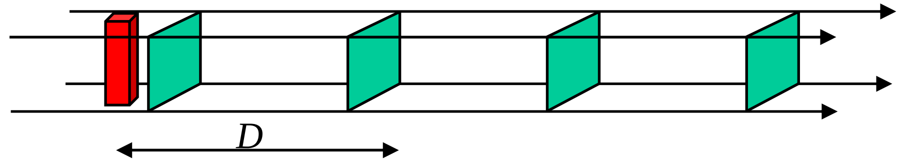
[redacted]

Phase imaging propagation technique

What is measured ⇨ $\nabla^2\phi(y,z)$, phase ϕ second derivative

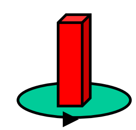
Phase retrieval ⇨ integral (measurements at several distances)

holo-tomography



1) phase retrieval with 3-4 distances

2) repeated for 700 views



(P. Cloetens *et al*, APL 75, (1999) 2912)

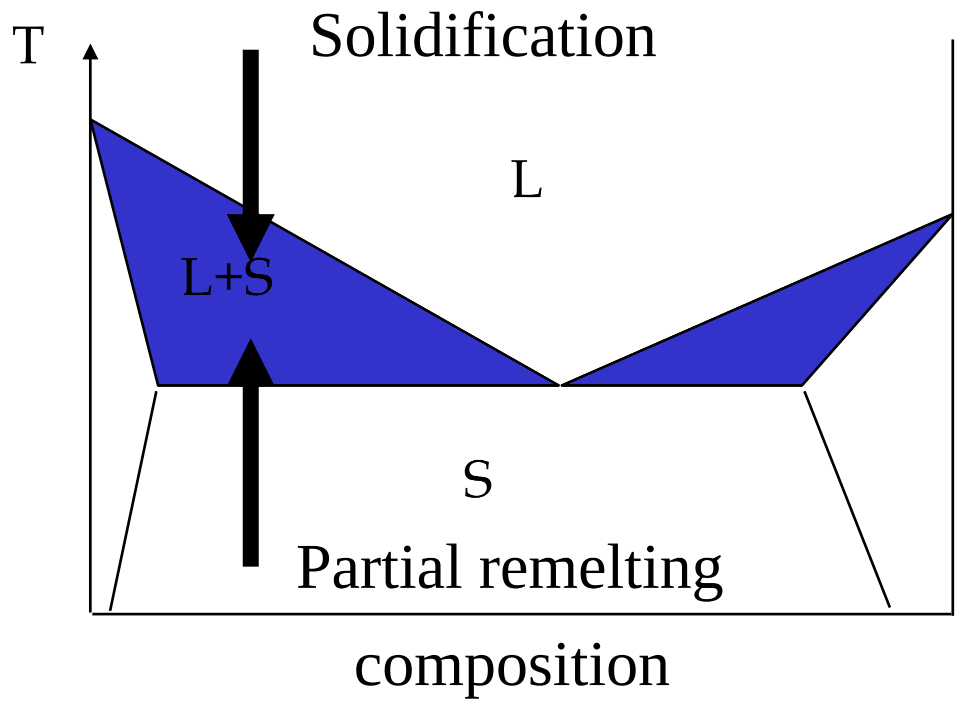
Experimental result ⇨ «local» ϕ (u,v)

It can be shown that $\Delta\,\phi$ with respect to a propagation in vacuum is proportional to the local electronic density ρ_e ϕ ⇨ ρ_e

Holotomography of *semi-solids*

P. Cloetens (ESRF), L. Salvo (GPM2, Grenoble)

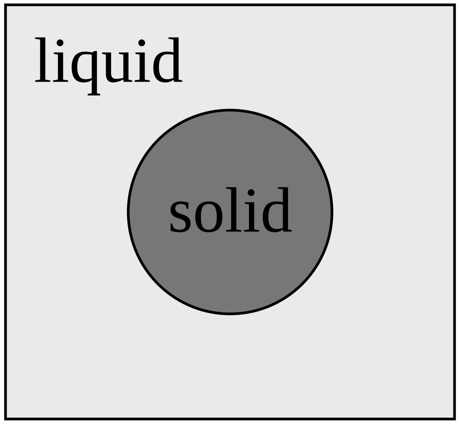
Rheology of aluminium alloys in the semi-solid state Al – Al/Si system



Microstructure

solid fraction
morphology
connectivity

Classical Models



Holotomography on an Al - Al/Si system

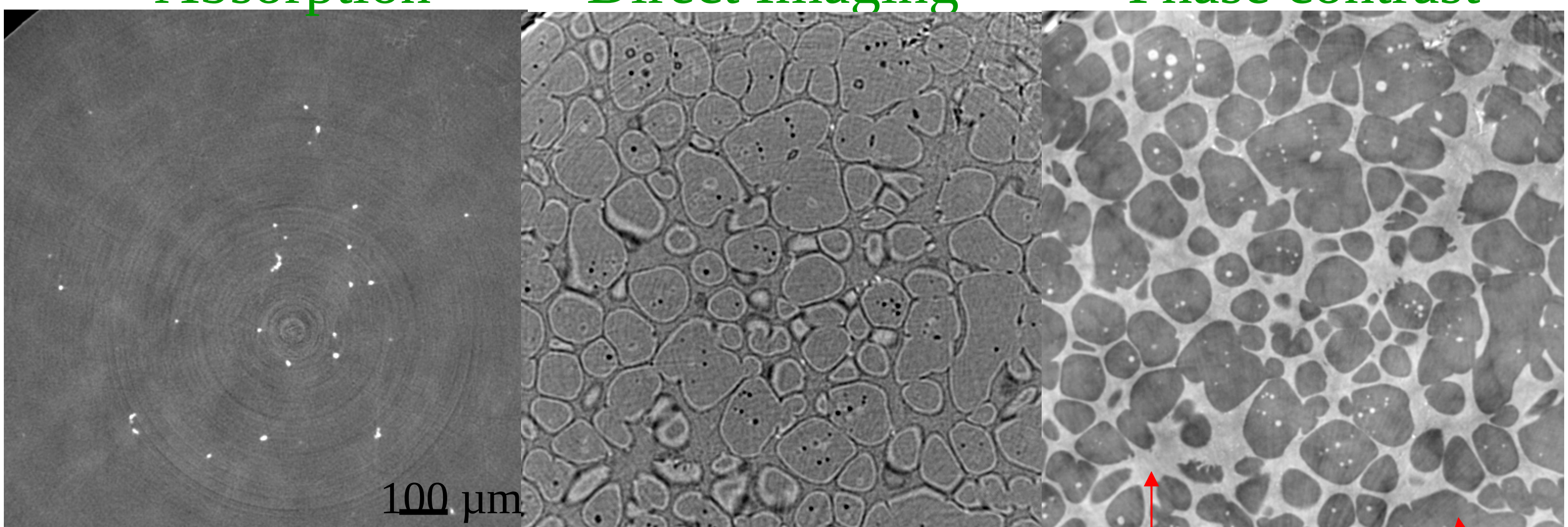
Rheology of aluminium alloys in the semisolid state

E = 18 keV

4 distances: absorption + 0.2 m, 0.5 m and 0.9 m
800 angular positions
multilayer as monochromator: total time ≈ 40 minutes

$$\frac{\Delta\rho}{\rho} \approx 2\%$$

Absorption | Direct Imaging | Phase contrast



β-map | edge enhancement $D = 0.6$ m | δ-map

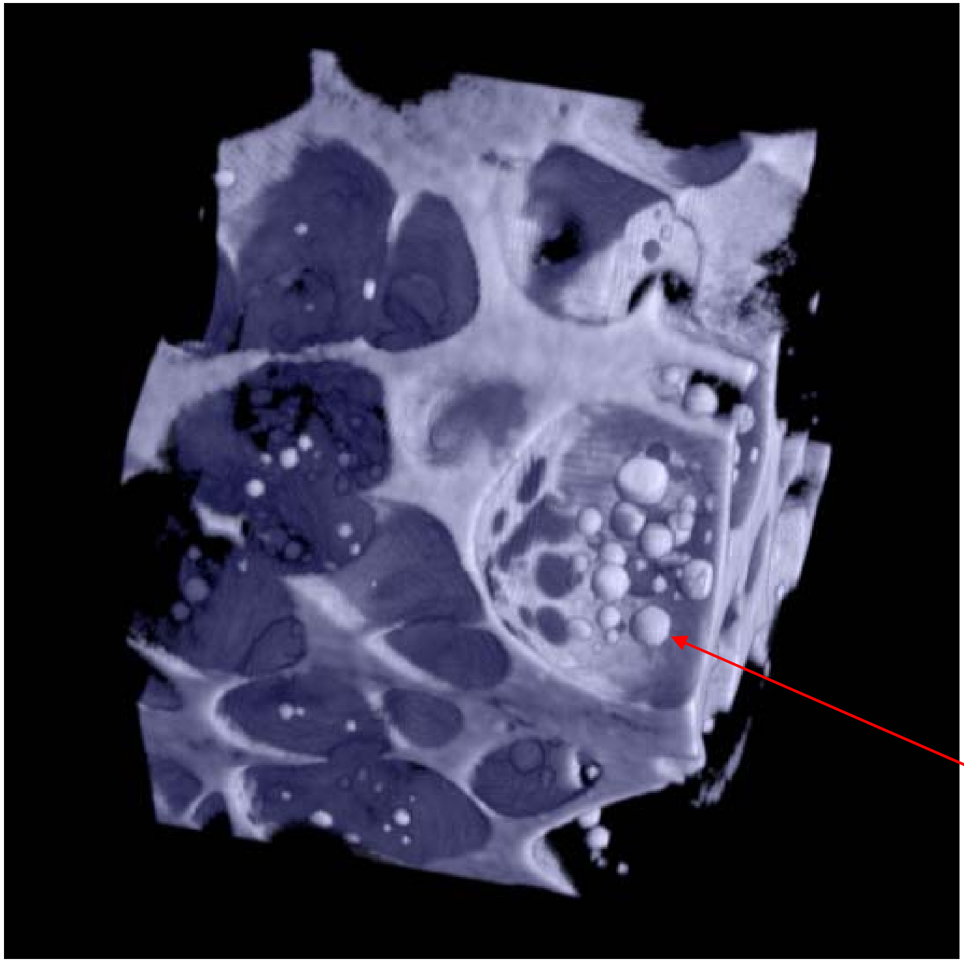
Al/Si

Al

L. Salvo (GPM2, Grenoble) P. Cloetens (ESRF)

Holotomography of *semi-solids*

Volume Rendering (solid transparent)



Connectivity:

Liquid phase:

total

+ trapped liquid

Solid phase:

very strong

trapped liquid

L. Salvo (GPM2, Grenoble) P. Cloetens (ESRF)

- 1 – Introduction: synchrotron radiation and imaging
- 2 – Interactions and detectors
- 3 – Radiography: absorption, phase
- 4 – Microtomography
- **5 – Microbeam-based techniques**
- 6 – Bragg diffraction based techniques

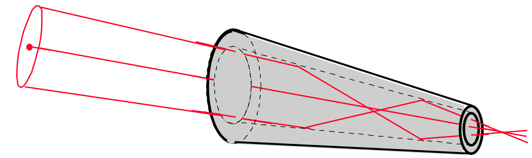
  (DEI, “topography”, diffraction tomography)

Micro-Focussing Devices

Focussing devices have dramatically progressed over the last years.

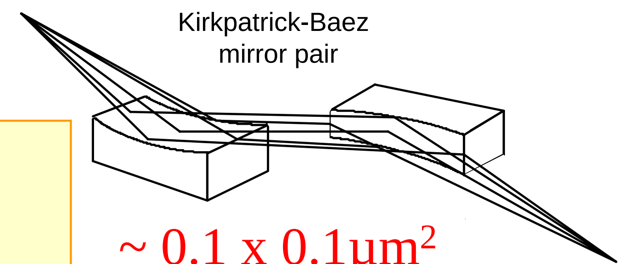
Coupling with the modern SR sources
⇨ focus the beam to the sub-micron size range

Capillaries



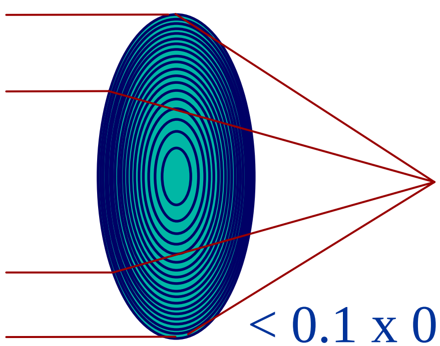
~ 1 x 1μm^2

Elliptically bent mirrors (Kirkpatrick-Baez, KB)



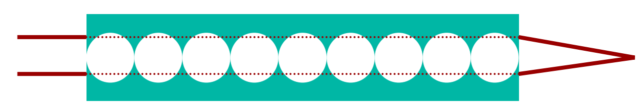
~ 0.1 x 0.1μm^2

Fresnel lens (zone-plate)



< 0.1 x 0.1μm^2

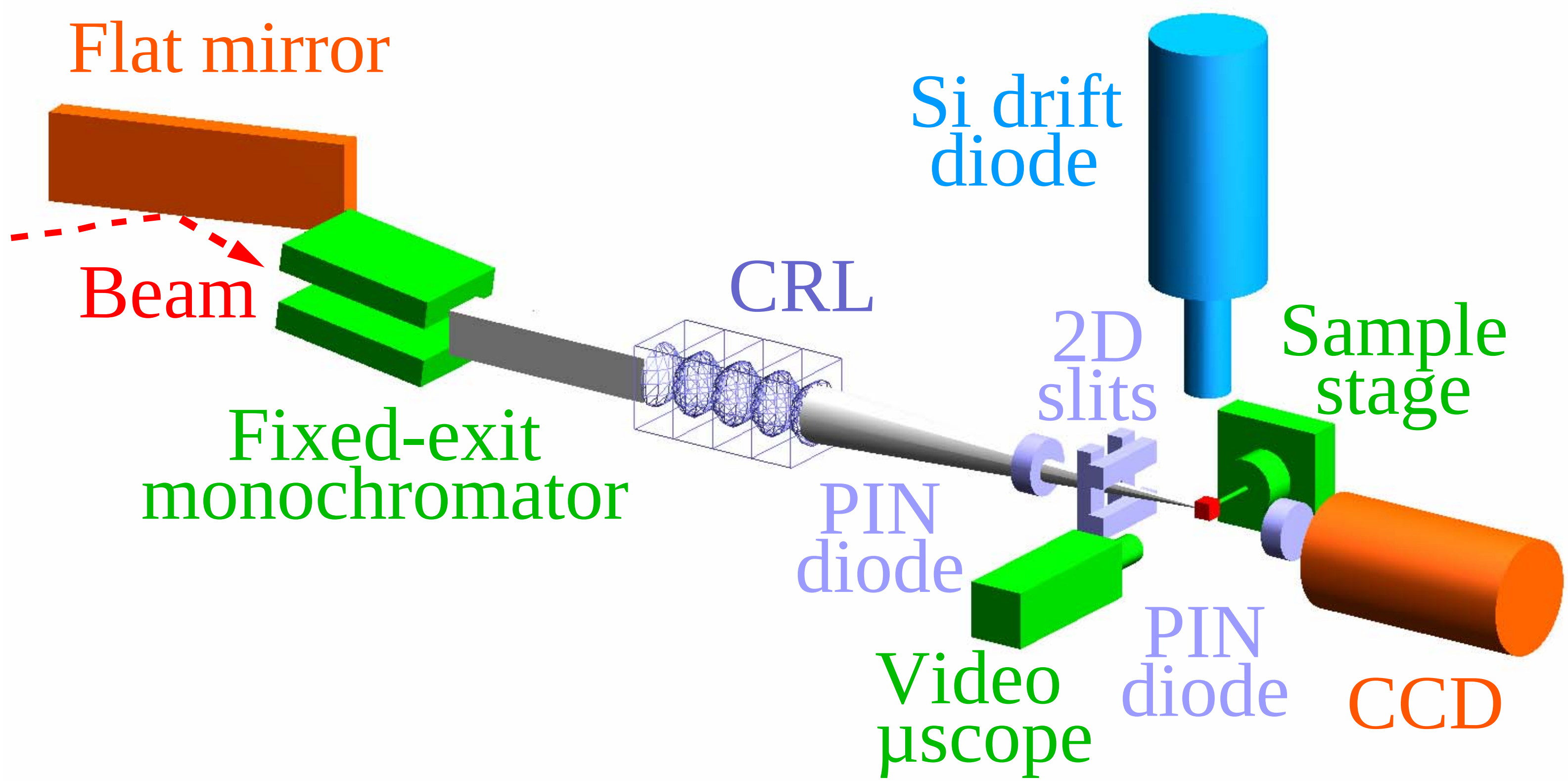
Refractive lens



~ 2 x 5 μm^2

Fluorescence tomography

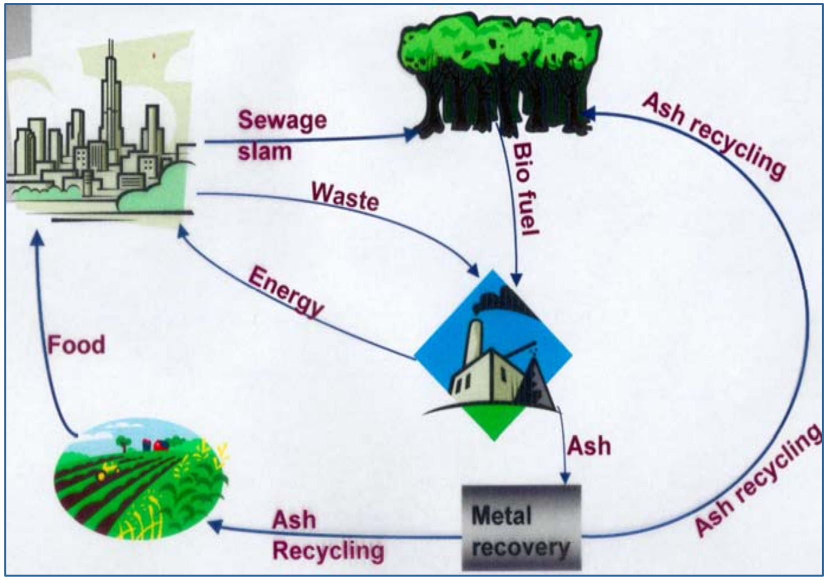
Each element present in the sample, when irradiated by the synchrotron beam, re-emit X-rays with an energy that is a signature of the element



Contamination in fly ashes

Content of heavy metals (i.e. Cd, Pb, Ni, Cu) makes **fly ashes** ecologically harmful

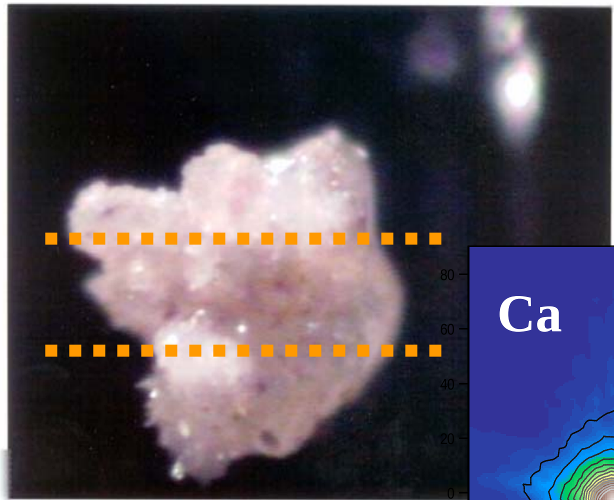
The environmental risk of Cd in fly ashes depends on its *speciation* (oxidation state, association with other organic or inorganic compounds, …)



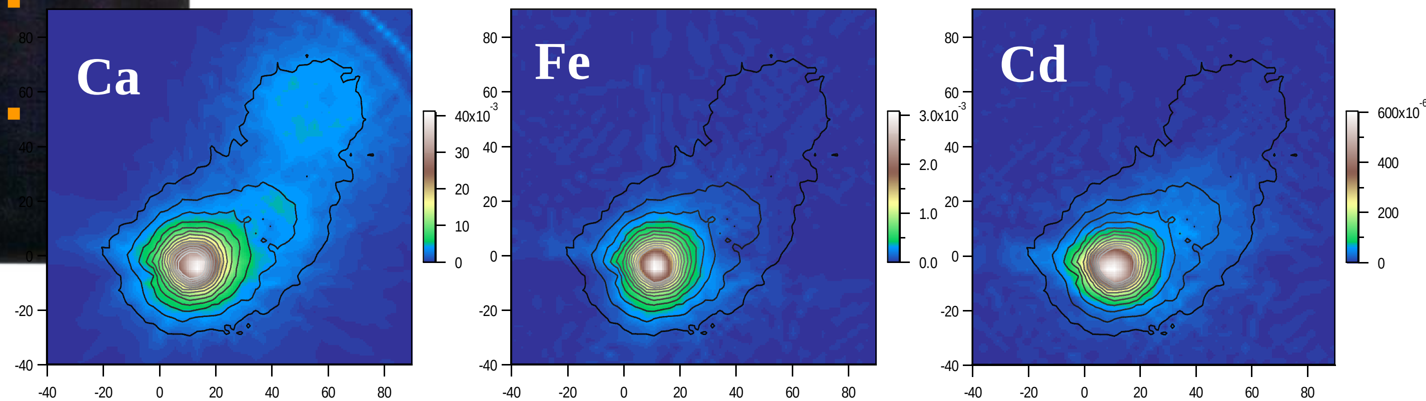
MC. Camerani Pinzani, A. Somogyi, A. Simionovici, S. Ansell, B.M. Steenari and O. Lindquist, *Environmental Science and Technology (2002),*

B. Golosio, A. Simionovici, A. Somogyi, MC. Camerani , B.M. Steenari *J. Phys IV France (2003)*

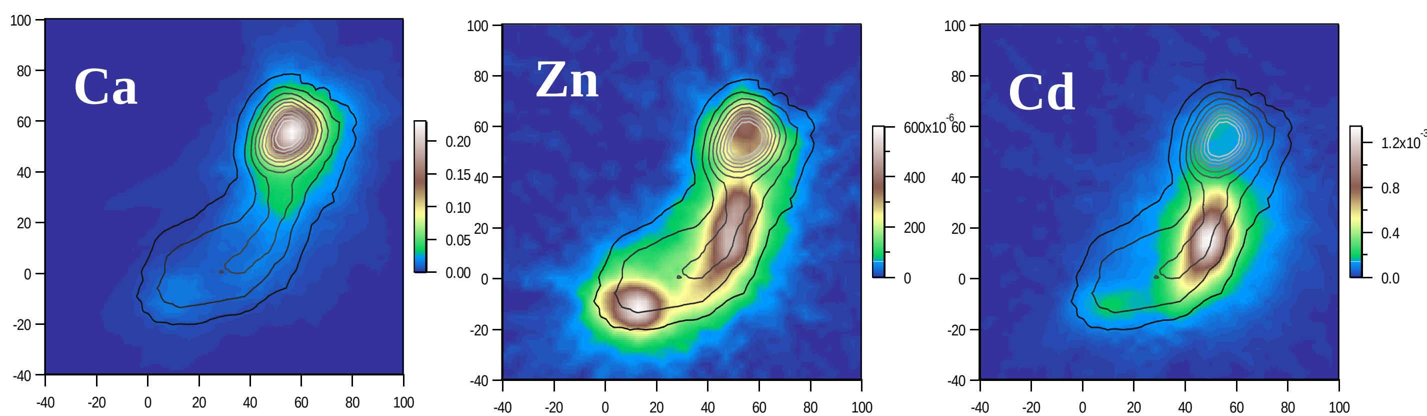
Investigation of individual fly ash particles: Fluo-tomography



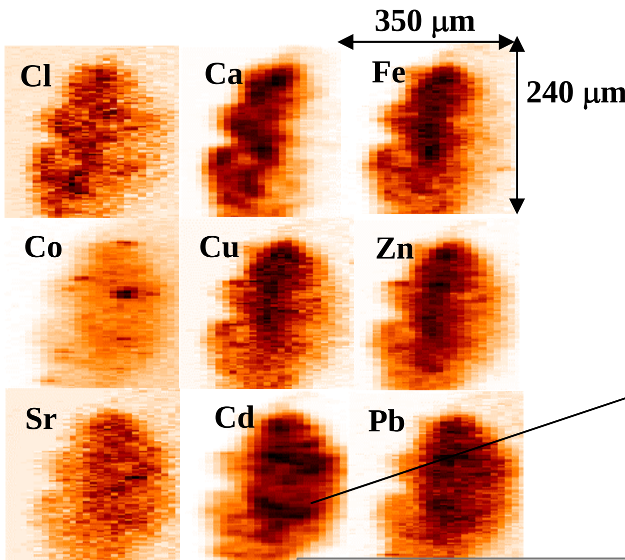
elemental distribution within Slice2

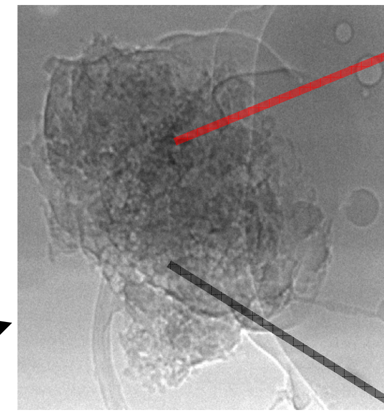


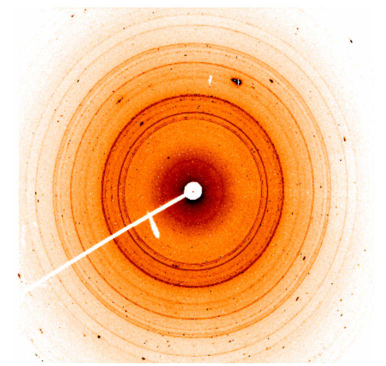
elemental distribution within Slice1

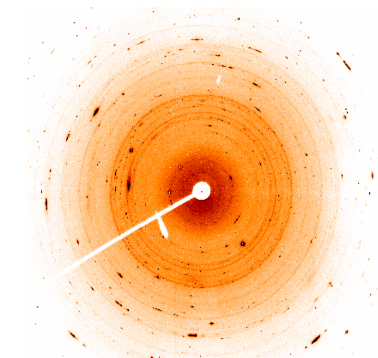


Investigation of individual fly ash particles: µXRD and µXAS

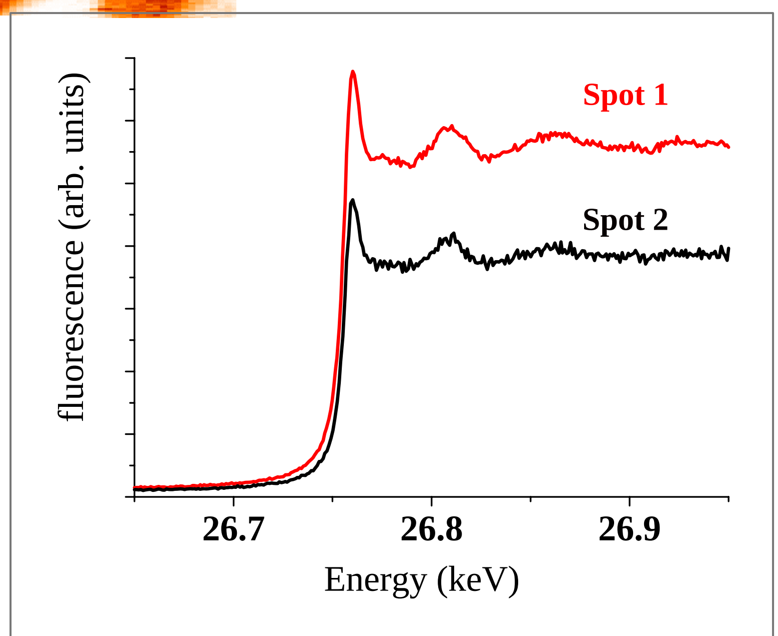






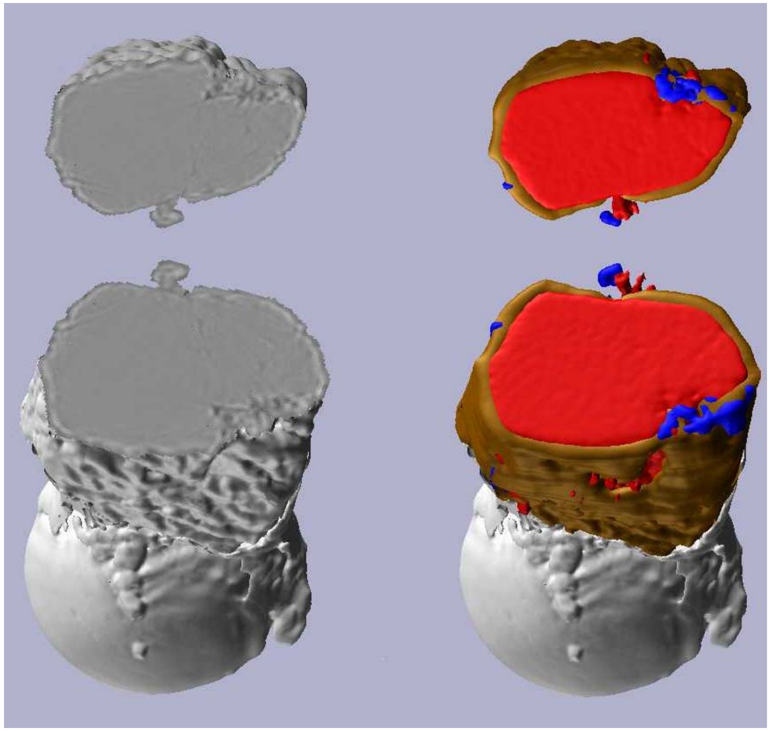


- *spot: 3 x 3 µm²*
- *E = 27 keV*
- *dwell time:6 s/pt*



- Complex Cd-Ca
- Cd distribution independent from particle sizes
- Cd in grain structure
- Cd in soluble form (Cd^{2+})

Fluotomography 3D rendering of a fly ash



3D rendering, fly ash particle

transmission tomography

distribution of Rubidium (red), Manganese (brown) and Iron (blue)

.
Voxel size 3×3×3 μm^3.

B. Golosio, et al., *J. Appl. Phys.* **94**, 145-156 (2003) and *Appl. Physics Letters*, (2004),

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(DEI, “topography”, diffraction tomography)

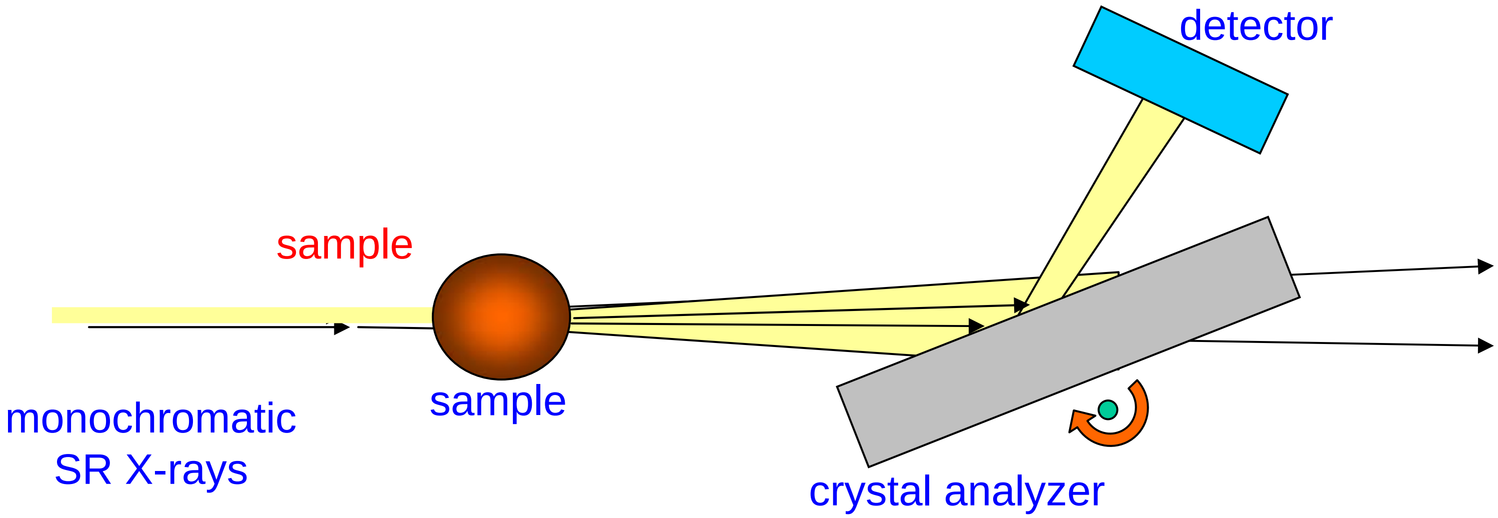
Imaging techniques involving Bragg diffraction

Diffraction Enhanced imaging, where an « analyzer » perfect crystal is used to map the amount of refraction of the beam transmitted through a sample (more tomorrow in Alberto Bravin's talk)

Bragg diffraction imaging, which shows defects or domains in single crystals (more tomorrow in José Baruchel's talk)

Diffraction contrast tomography, which provides the shape and orientation of crystallites within a polycrystal (more tomorrow in Wolfgang Ludwig's talk)

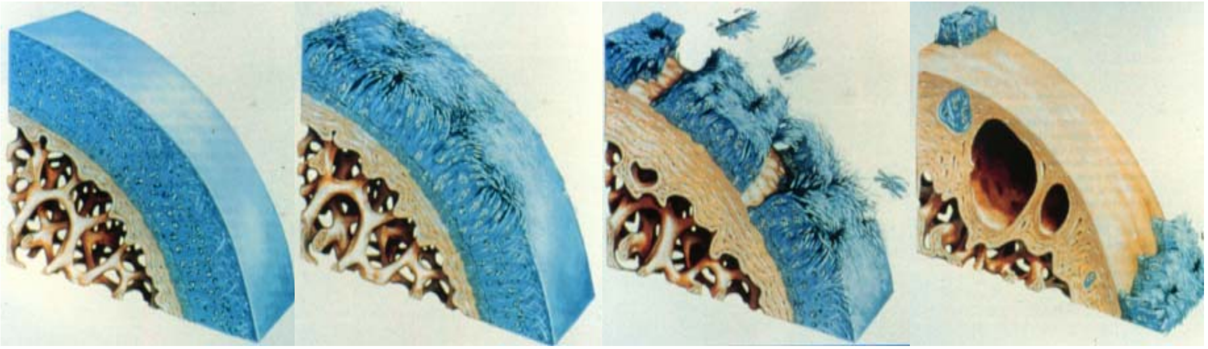
Diffraction Enhanced Imaging Technique



Crystal acts as an angular filter of the radiation coming from the sample

Why focusing on cartilage joint diseases (in particular osteoarthritis) ?

Cartilage degeneration process - grading system



0&1 2 3 4

Osteoarthritis (OA) is one of the leading causes of disability;
people with OA usually have joint **pain** and **limited movement** (stiffness)

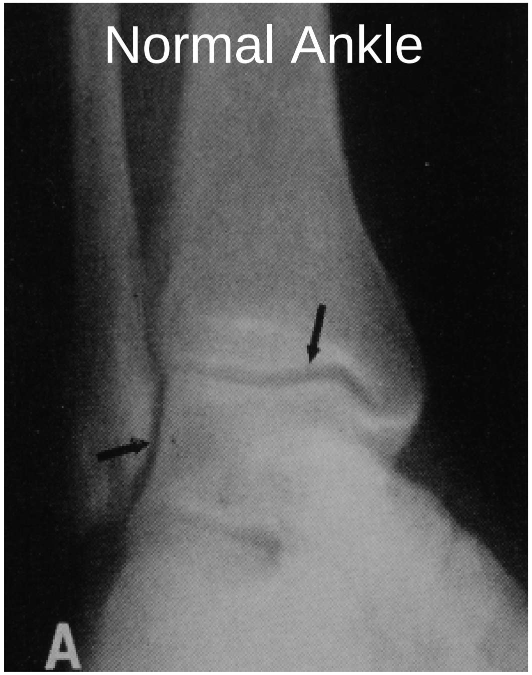
OA affects approximately 12% of the population

(in USA, Japan, France, Germany, Italy, Spain, and the UK)

As OA presently has no cure, treatment strategies focus on treating the disease symptoms with minimal side effects.

Conventional radiology limitations

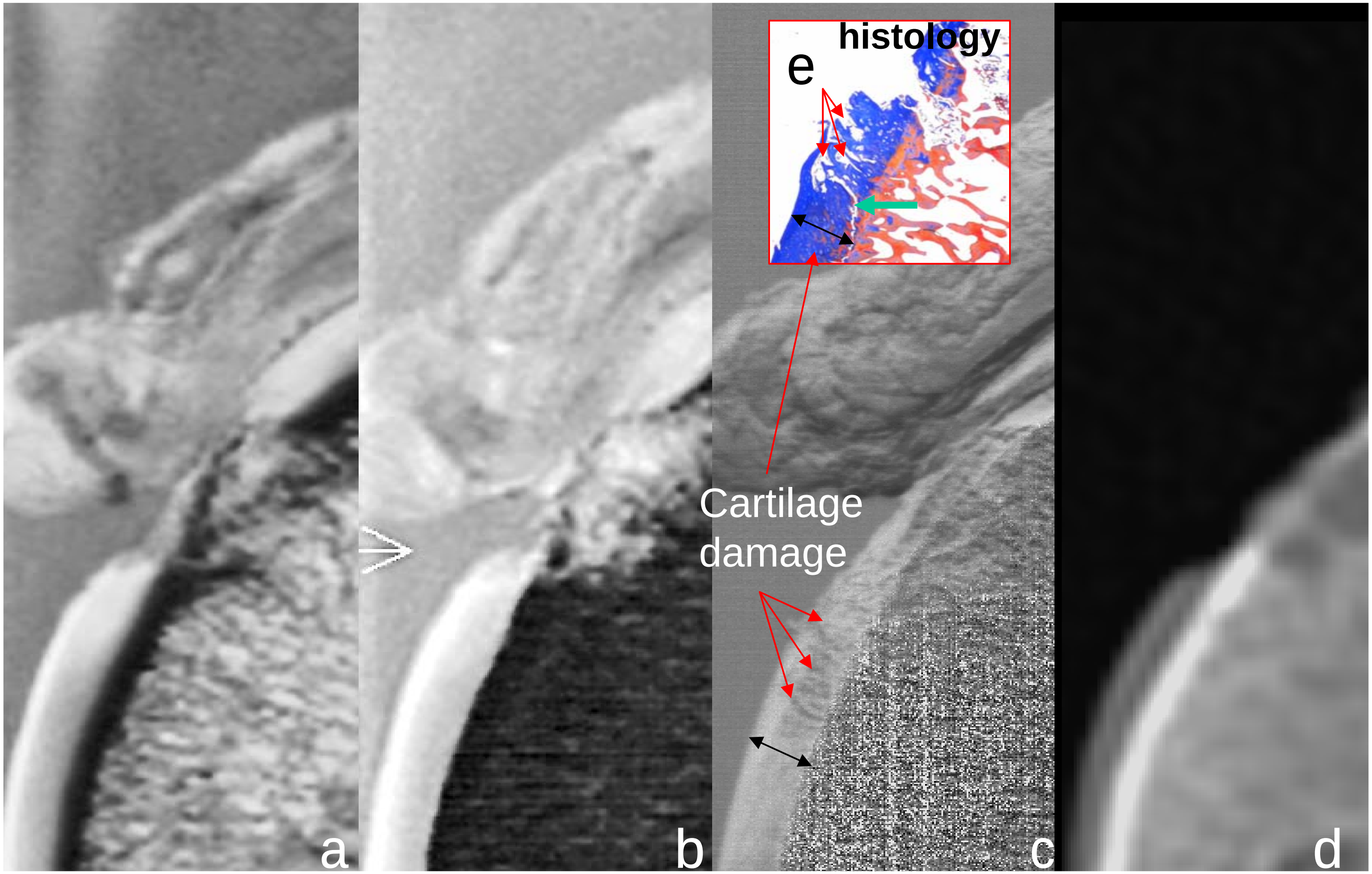
Conventional radiology limitations



Conventional radiographs do not show cartilage;
joint space narrowing is evaluated by proximity of bones

DEI vs conventional techniques

DEI vs conventional techniques



MRI (T1-TFE) >5h | MRI (T1-FFE) >5h | **DEI** | Conventional CT

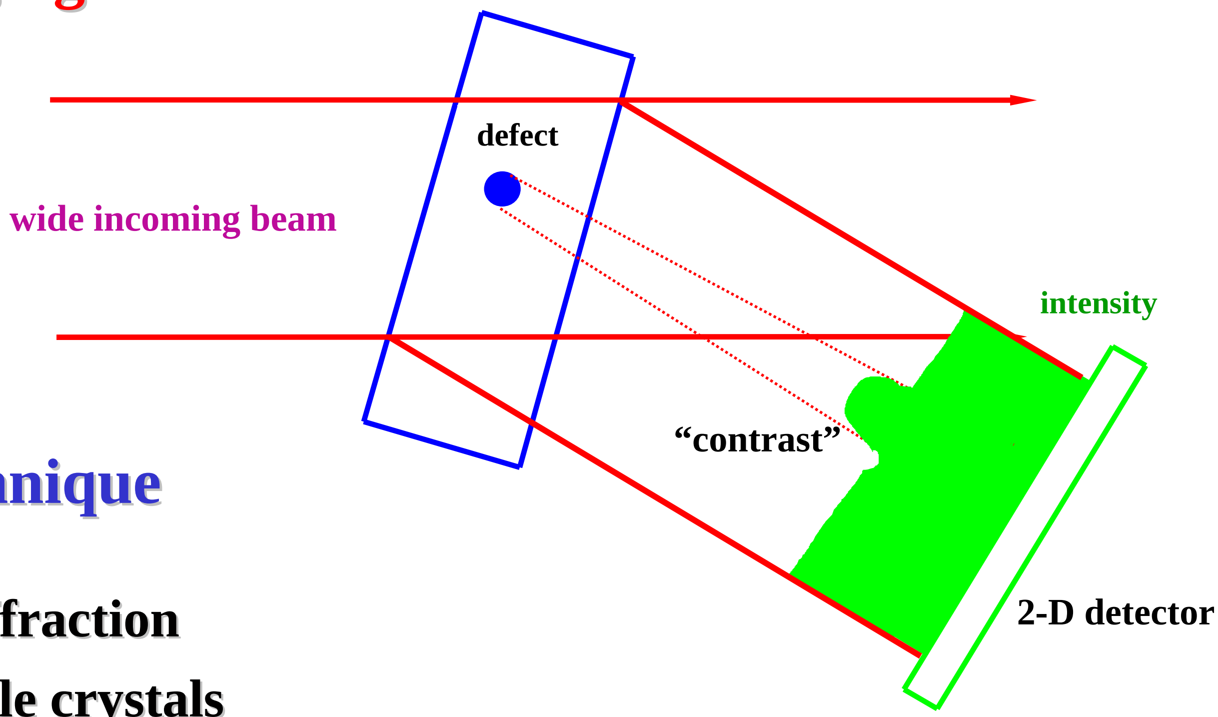
DEI shows structural damage of cartilage invisible in MRI and CT

Wagner, …, Coan, Bravin, Mollenhauer Nucl. Instr. Meth. A **548**, 47-53 (2005)

X-ray diffraction imaging

historically called

X-ray topography



is an imaging technique

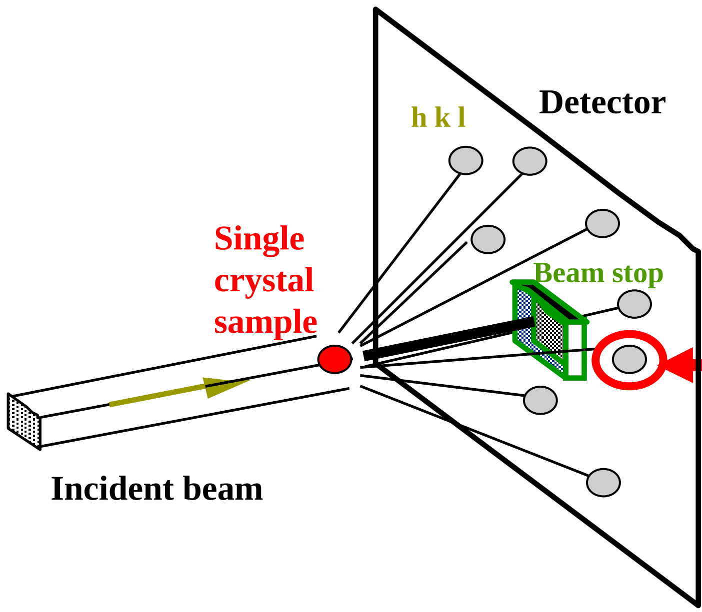
- **based on Bragg diffraction**
- **that applies to single crystals**
- **shows the inhomogeneities within the crystal**

Defects, distortions: dislocations, twins, domain walls, inclusions, impurity distribution, …bending, acoustic waves…

Usual setup for Laue technique, but incoming "white" beam is

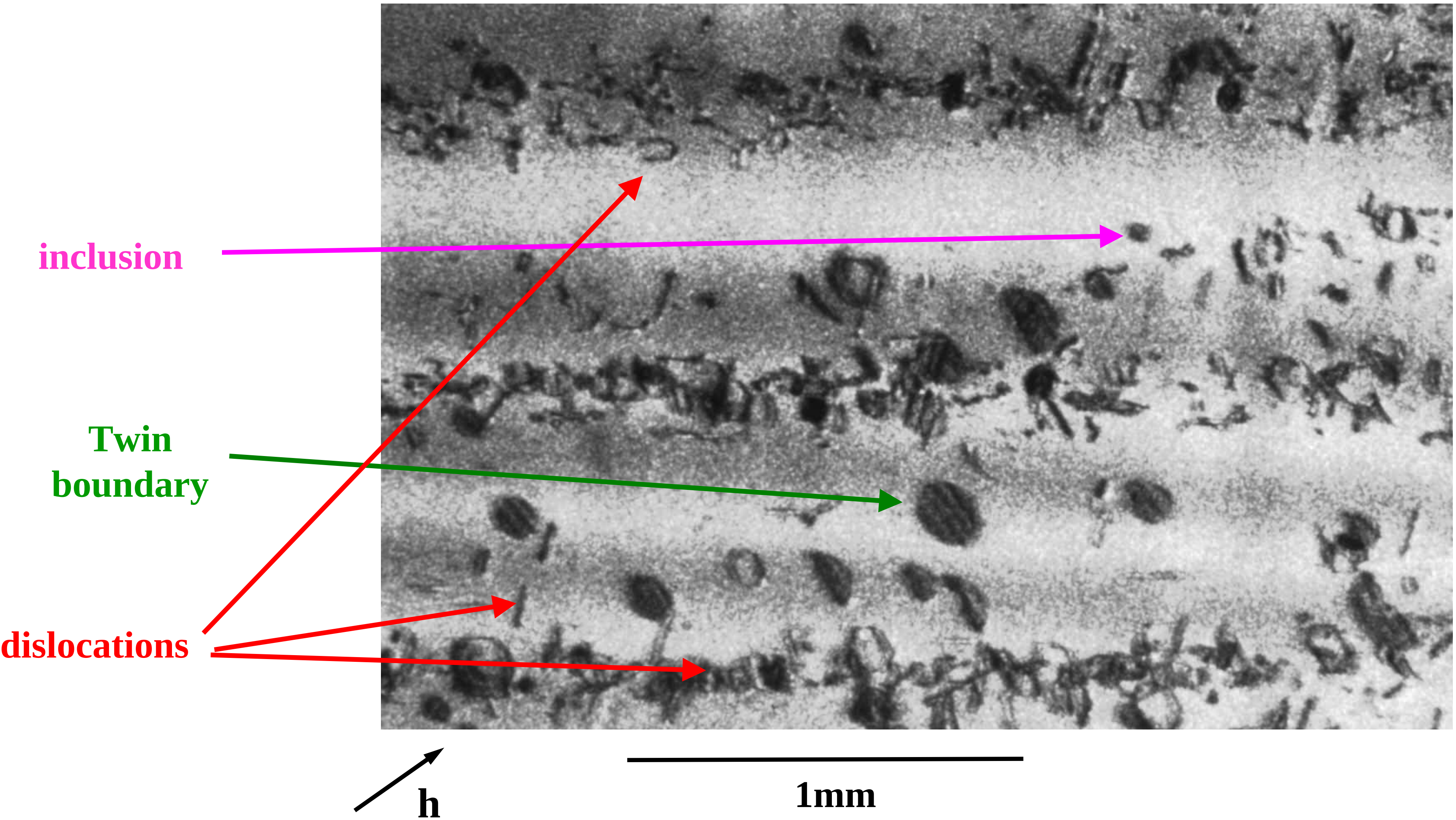
- ⇨ **wide, illuminates all or a substantial fraction of the sample**
- ⇨ **low divergence , small angular size of the source seen from the sample**

Laue imaging ("white beam topography")



each Laue spot is a Bragg diffraction image that allows observing, when looking inside the spot, inhomogeneities like defects, domains, phases…

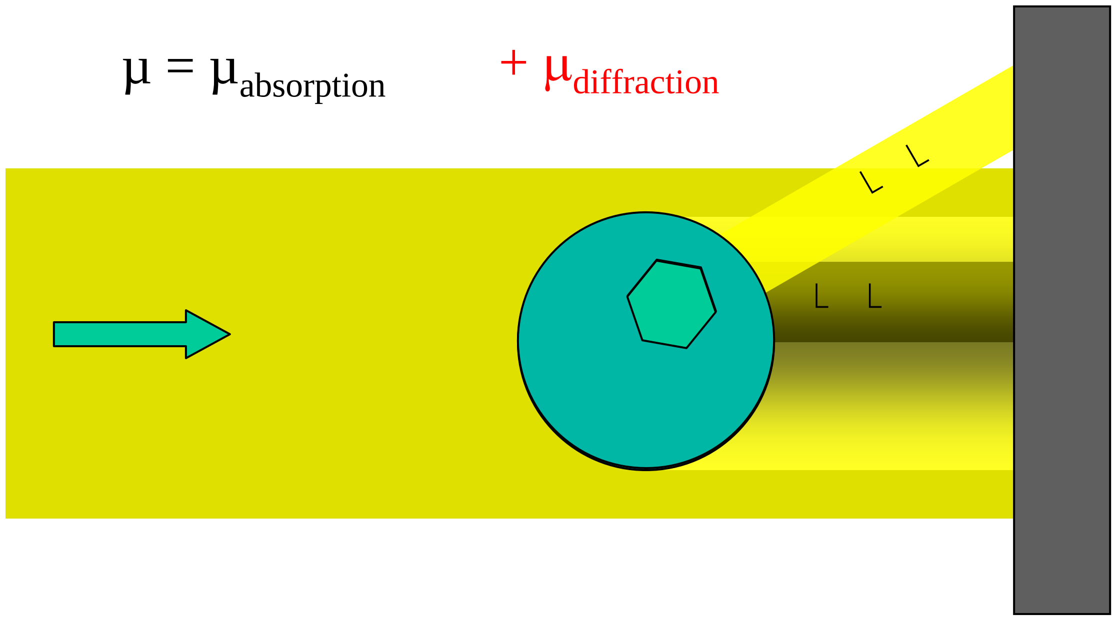
How a X-ray topograph may look like:



White beam topograph of a Si crystal containing oxygen, after a thermal treatment inducing precipitates, dislocations and twin boundaries

New 3D approach: "topo-tomography"

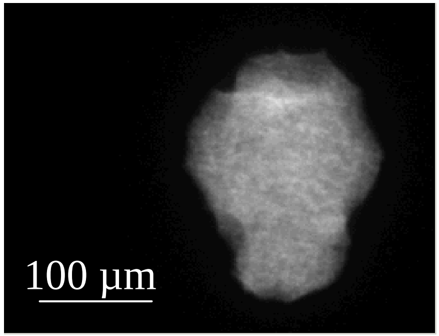
Basic idea: $\mu = \mu_{absorption} + \mu_{diffraction}$

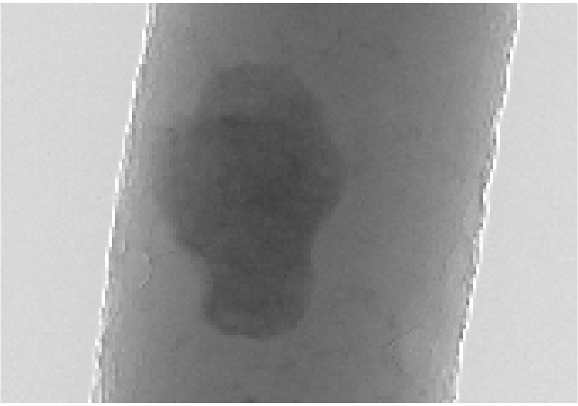


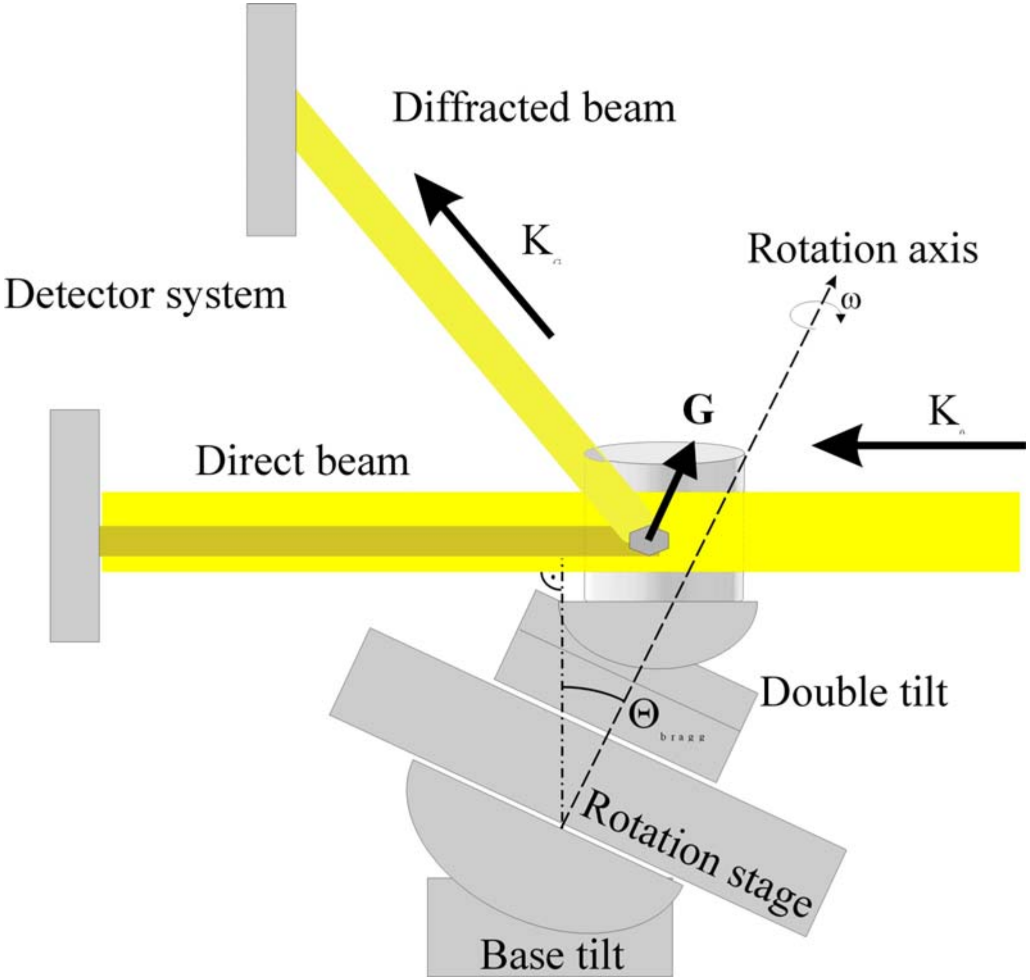
$$I = I_0 e^{-\int \mu t}$$

Ludwig, Poulsen, Schmidt, Lauridsen et al.

Topo-Tomography applied to Polycrystals







Topo-Tomography: data acquisition

QuickTime™ and a
decompressor
are needed to see this picture.

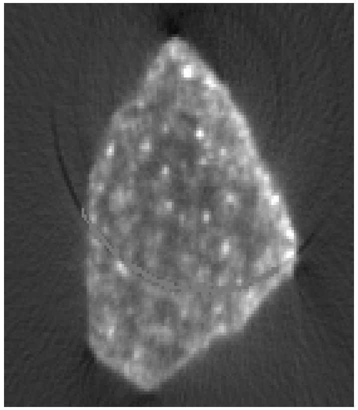
diffracted beam

100 µm

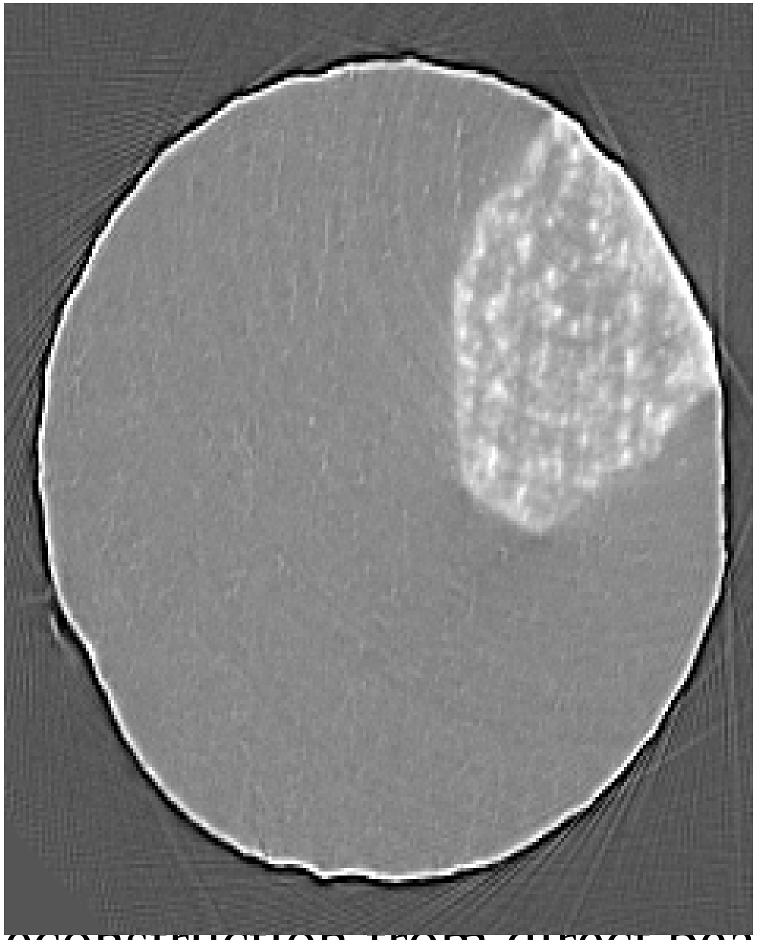
QuickTime™ and a
decompressor
are needed to see this picture.

direct beam

Topotomography

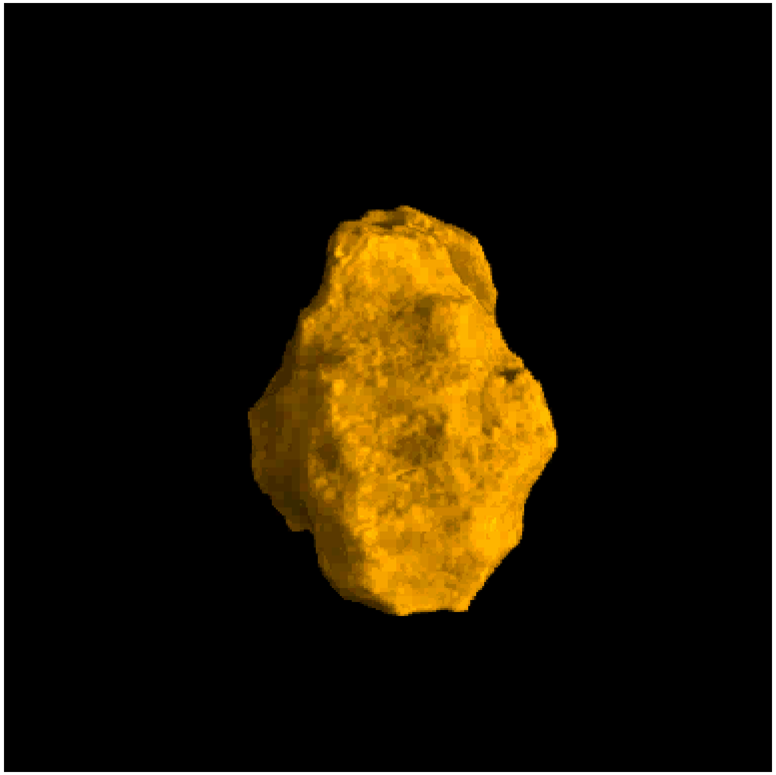


Reconstruction from diffracted beam (180 projections)

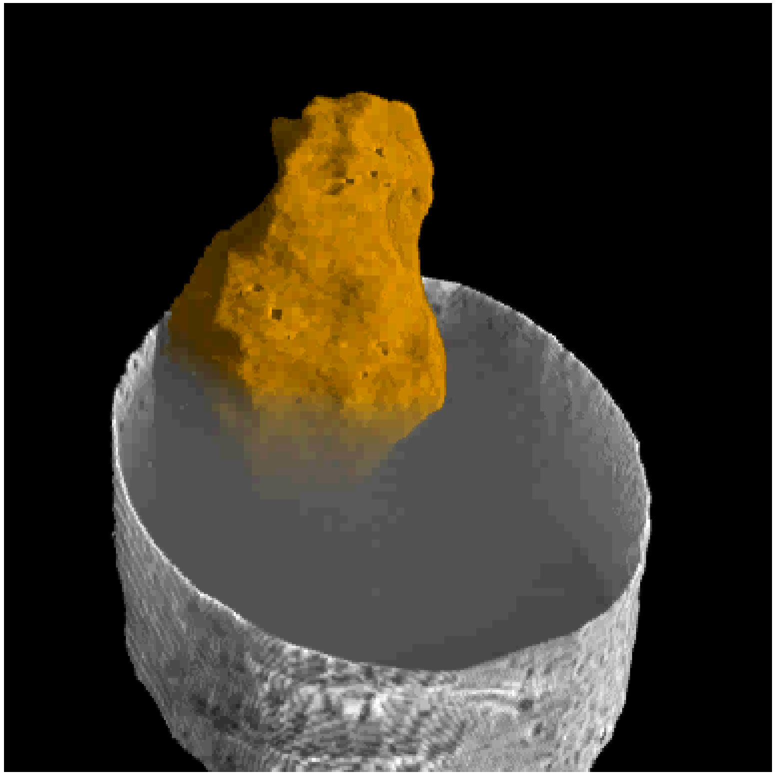


Reconstruction from direct beam (720 projections)

3D reconstruction
(cone beam algorithm)

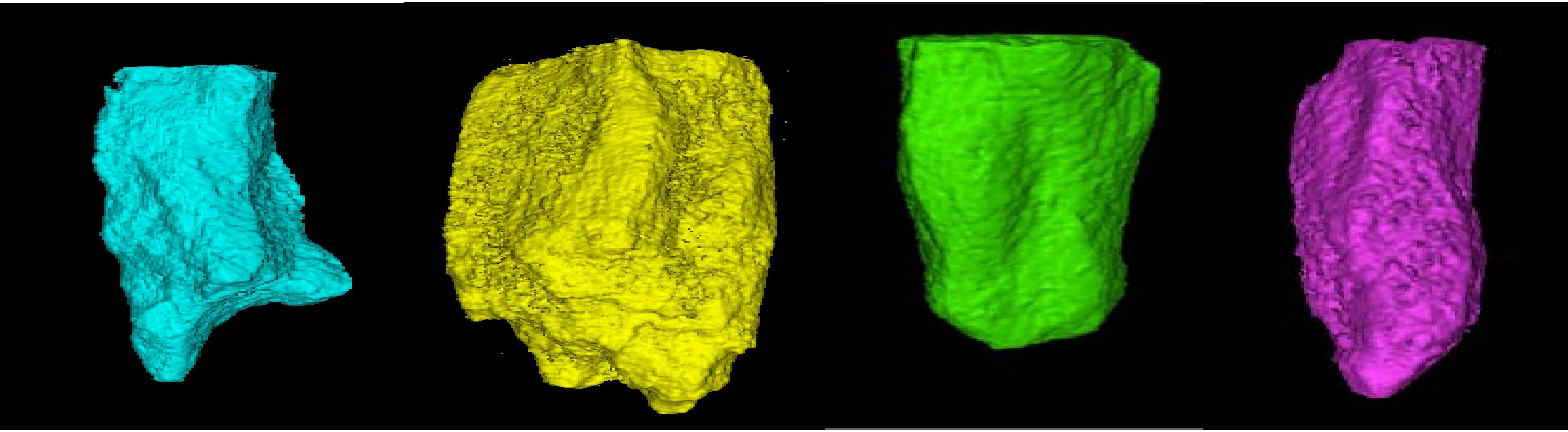


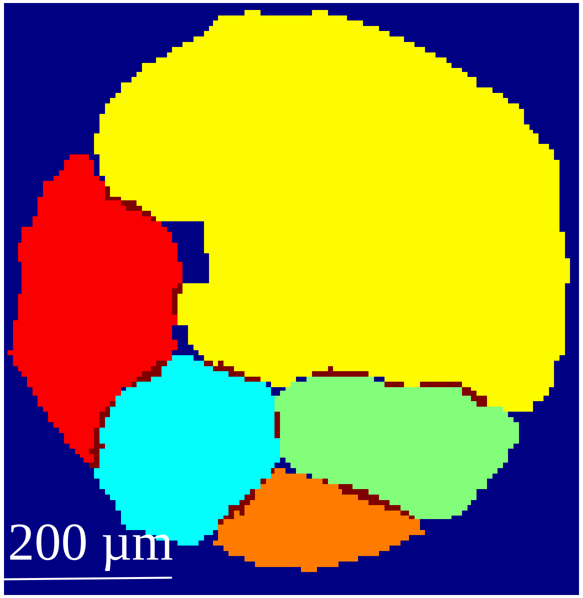
Diffracted beam



Direct beam

Grain by grain reconstruction





Summary and **Conclusion**

« X-ray Imaging » , series of (rapidly evolving) techniques which apply to a large variety of topics

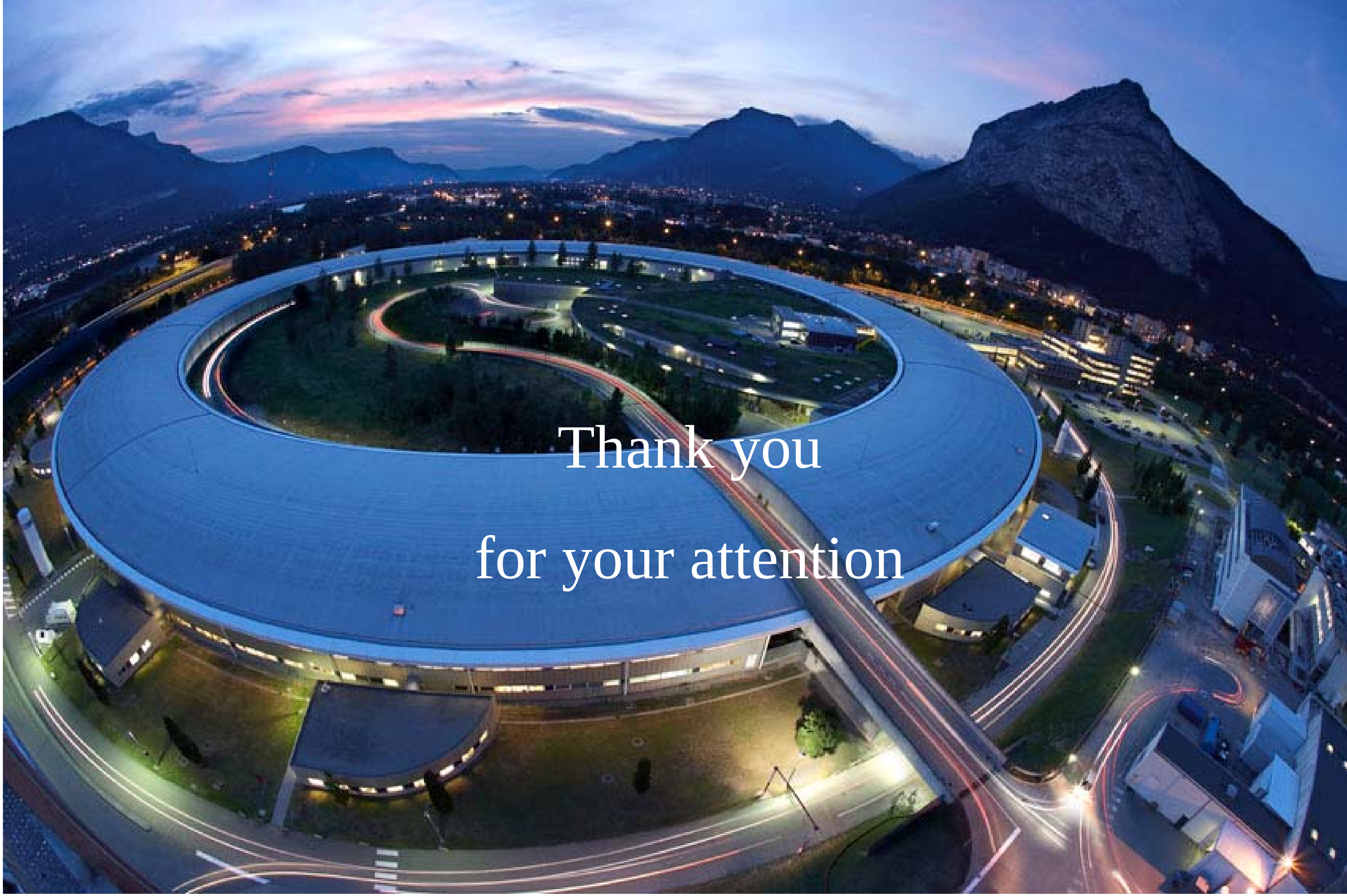
In particular in its 3D version, has biomedical and material science applications at a large variety of scales

The coherence of the beam adds new possibilities

High spatial and time resolution are useful for many of the problems encountered

Images contain a lot of information

The scientific use this information implies an accurate knowledge of the contrast mechanisms, and (often) image processing


Thank you
for your attention