Some Trends of Synchrotron Radiation X-ray Imaging

José BARUCHEL

European Synchrotron Radiation Facility

Grenoble, France

Outline

- **SR X-ray imaging**

- 1 – where from do we come?
- 2 – where are we?

  coherent beams ⇨ new science

  new aspects of microtomography
- 3 – where should we go?

  near future perspectives

- **1 – SR X-ray imaging: where from do we come?**
- 2 – SR X-ray imaging: where are we?

  # coherent beams ⇨ new science

  “poled” ferroelectric domains

  porosity and growth of quasicrystals

  # new aspects of microtomography
- 3 – where should we go?

  # near future perspectives

X-ray Imaging in the « Red book » of the ESRF

No actual imaging in the first phase of BLs construction

microfocus beamline ➪ microfluorescence or microdiffraction maps

Second phase:

- ➪ Medical imaging
- ➪ X-ray topography
- ➪ microscopy and microanalysis

Microtomography

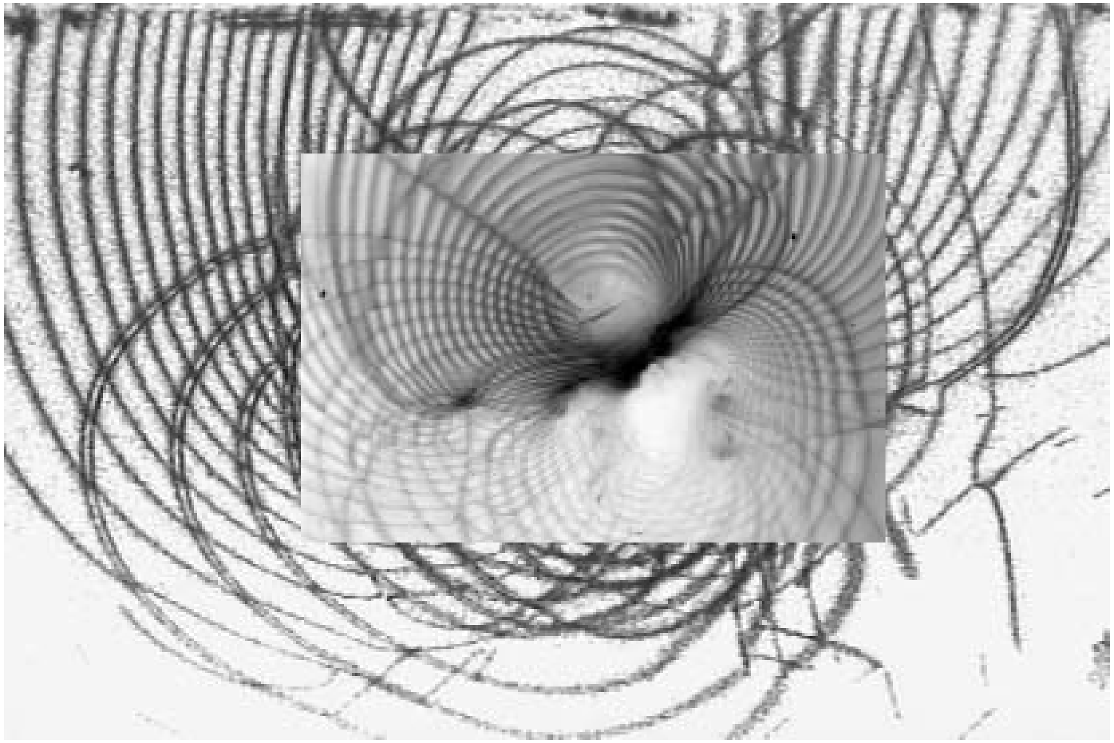
not foreseen in the « Red Book »

Reason

computer power

cluster of processors, 32 Mbyte of memory, 3 Gbytes of mass-storage

Diffraction imaging (“topography”)
Frank-Read dislocation sources in SiC



White beam topograph showing dislocations originating from several Frank-Read sources, not actually visible (distorted region)

« Weak beam » monochromatic topograph, which shows the location of the pinning centers (Frank-Read source)

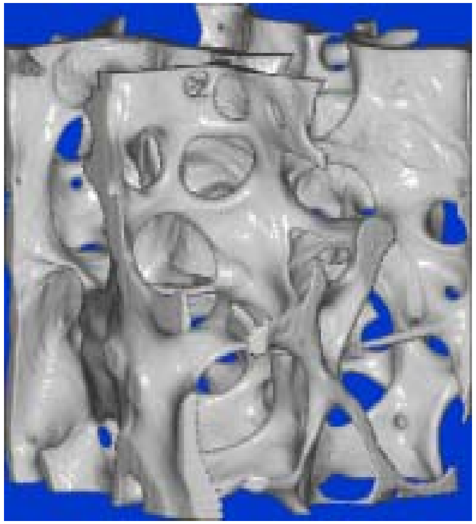
Eetu PRIEUR, PhD Thesis, Helsinki 1997

Microtomography

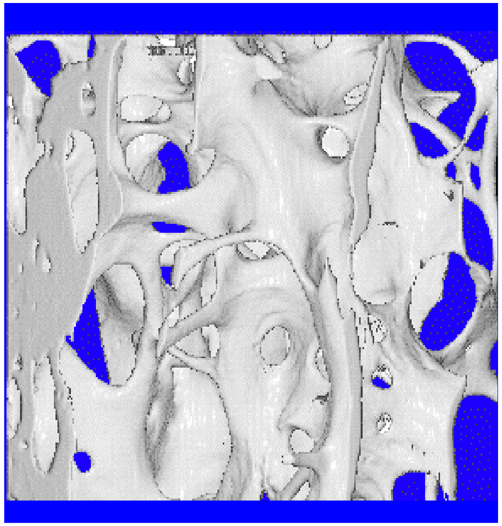
Osteoporosis disease

Human Vertebra Samples

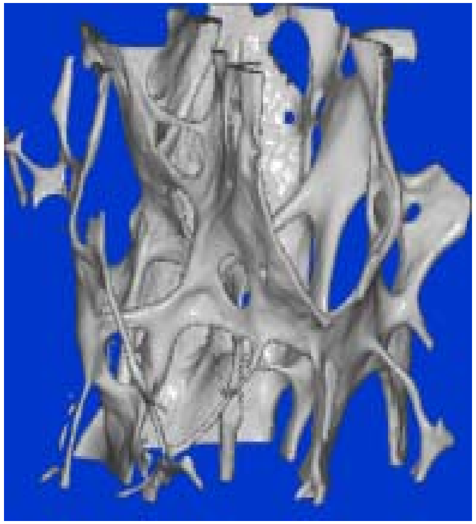
1mm



33 years



55 years



63 years

Images $(512)^3$, voxel size = 6.7 µm

M. Salomé, F. Peyrin et al., Medical Physics **26** (1999) 2194-2204

X-Ray Imaging Group

ESRF

- 1 – SR X-ray imaging: where from do we come?
- **2 – SR X-ray imaging: where are we?**

  # coherent beams ⇨ new science

  “poled” ferroelectric domains

  porosity and growth of quasicrystals

  # new aspects of microtomography
- 3 – where should we go?

  # near future perspectives

X-ray imaging at the ESRF

About 15% of the scientific activity at the ESRF

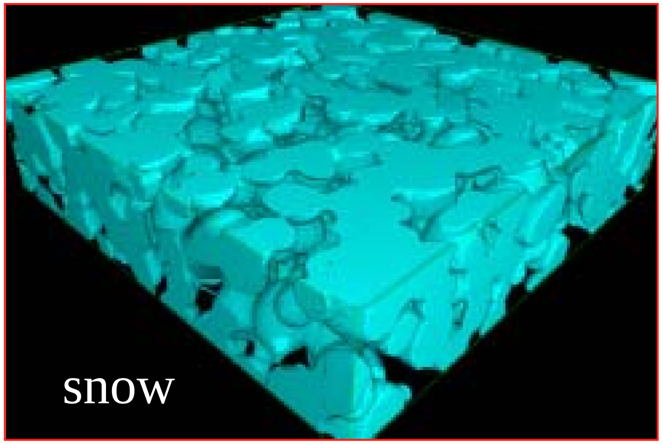
KEY WORDS: Medical imaging/ therapy, Use of Coherence, Microtomography and its applications, Combination of techniques, Microanalysis, Diffraction imaging

Increasing demand on microtomography and microanalysis

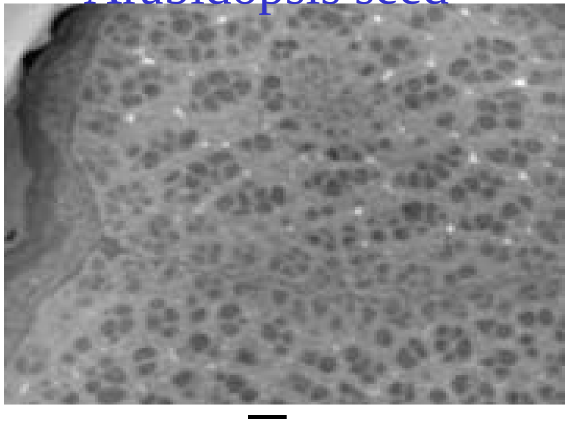
↕

enlargement of the number of scientific comunities concerned by these techniques

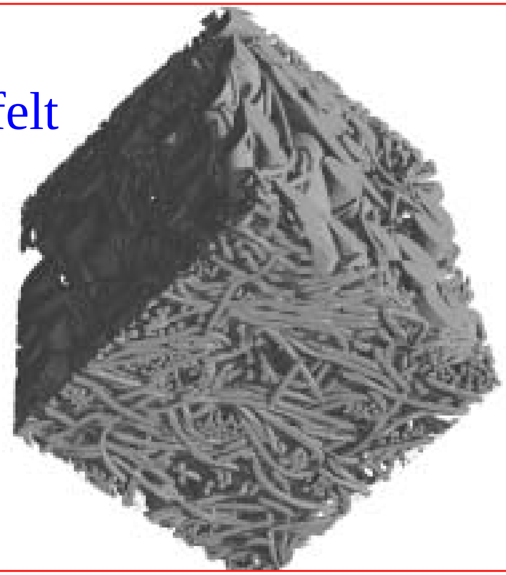
Microtomography: a multidisciplinary technique



Arabidopsis seed



10 µm



Beamlines in the X-ray Imaging Group

BM05
6-100 keV
optics & imaging
(microtomography)

ID17
10-60 keV
medical imaging
& beam therapy

ID21 (+ IR end station)
2-7 keV
imaging & microanalysis

ID19
6-60 keV
transmission & diffraction imaging

ID22+ID18F
6-50 keV
imaging & microanalysis

- 1 – SR X-ray imaging: where from do we come?
- **2 –** SR X-ray imaging: where are we?

 # coherent beams ⇨ new science

"poled" ferroelectric domains

growth and porosity of quasicrystals

new aspects of microtomography

- 3 – where should we go?

near future perspectives

X-ray transmission imaging

Interaction of X-rays with matter: complex refractive index

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

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

λ wavelength, μ linear absorption coefficient

Phase variation $\Delta\phi$ related to δ

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

Index of refraction

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

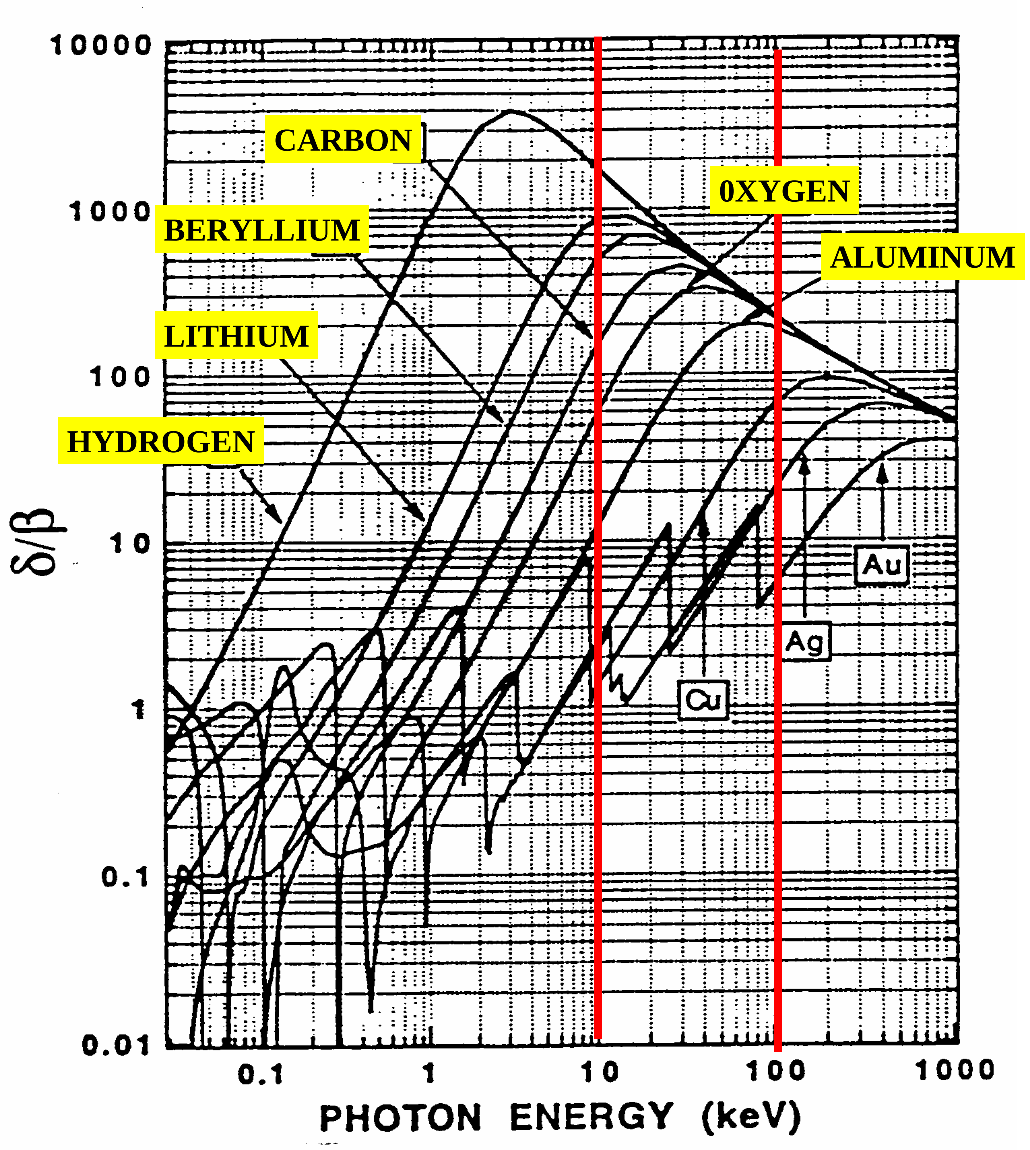
Ratio phase and amplitude (δ/β) as a function of the X-ray energy

bigger than 10 in the hard X-ray range

Either reduced dose

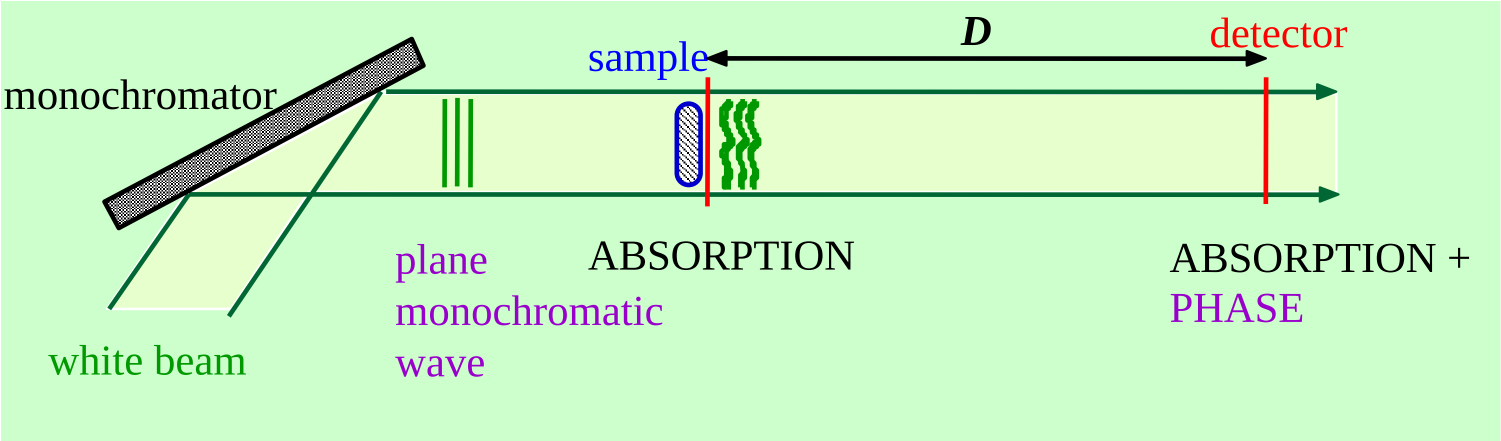
or

enhanced sensitivity



coherent beam ➡ **Phase contrast imaging**

the « propagation » technique



Two regimes: edge enhancement (only first fringes visible)

holograms

combined with high spatial resolution (0.5µm)
microtomography

Example: Damage in Composites

Scientific Case:

Quantification of *damage* in the *bulk* of composite materials

Plastic behaviour as a function of the hardness of the matrix

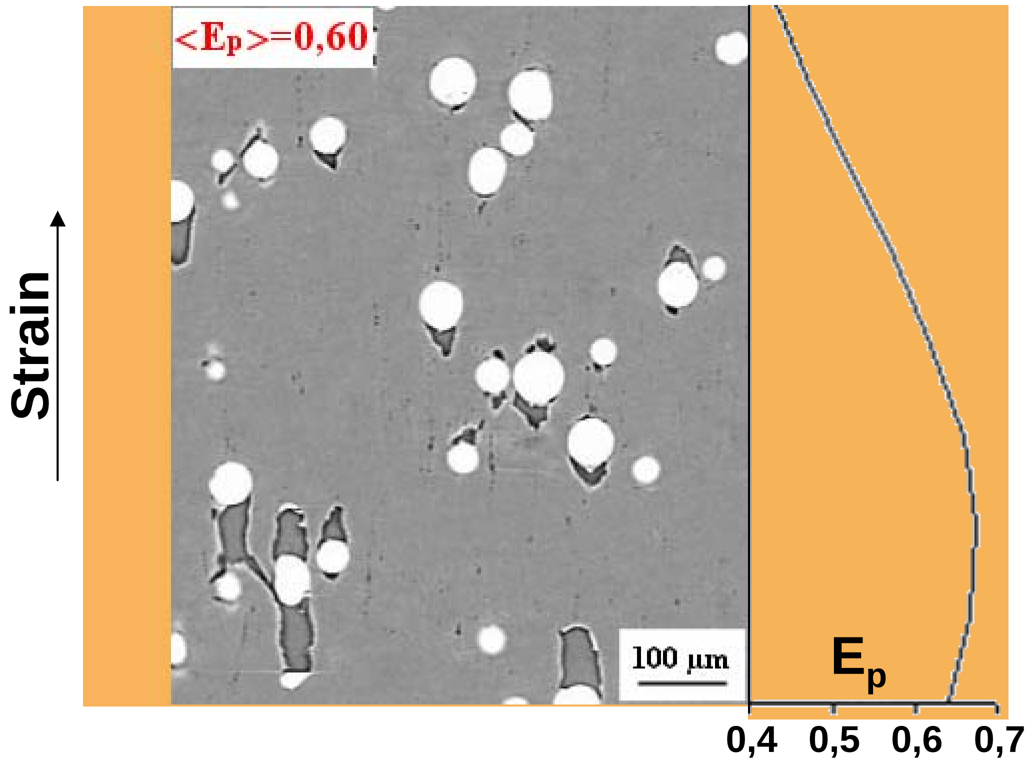
Authors:

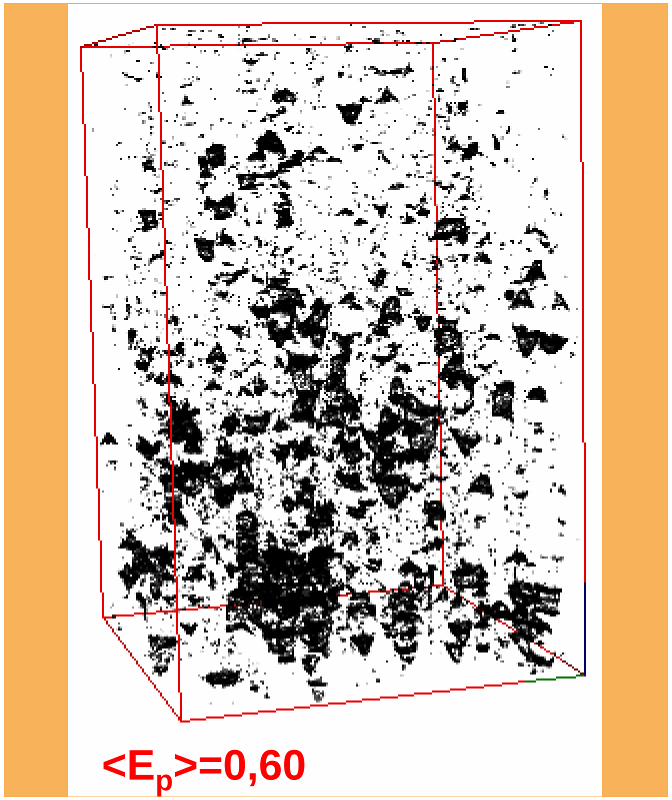
L. Babout, W. Ludwig, E. Maire, J.Y. Buffière, *INSA, Lyon*
P. Cloetens, E. Boller, *ESRF, Grenoble*

Damage in Composites

L. Babout, W. Ludwig, E Maire, J. Y. Buffière
NIM B 200, 303 (2003)

Example: soft matrix
Al + 4% ZrO_2SiO_2





Preferred damage initiation: Matrix-particle *decohesion*

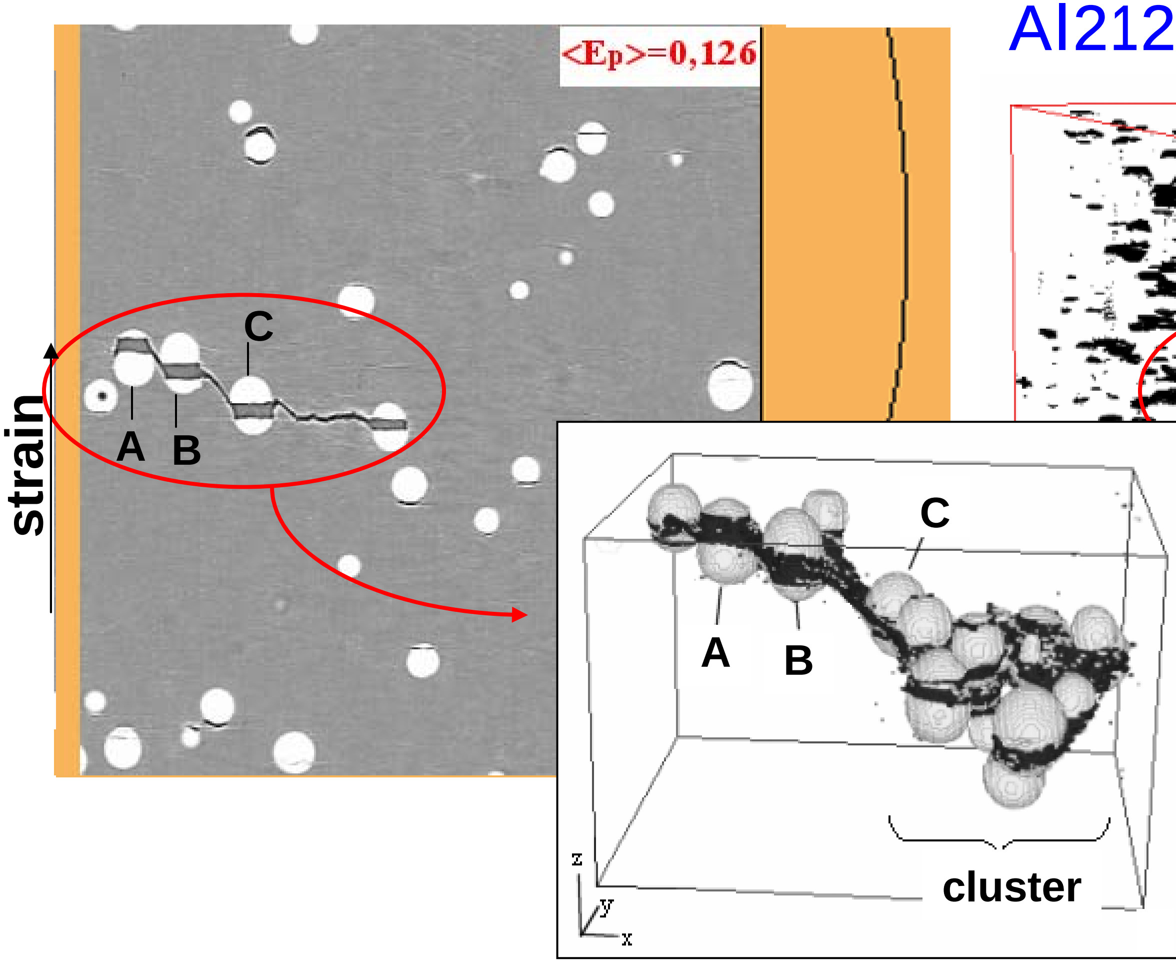
Conditions : in situ, E=30 keV abs/phase, 1 µm

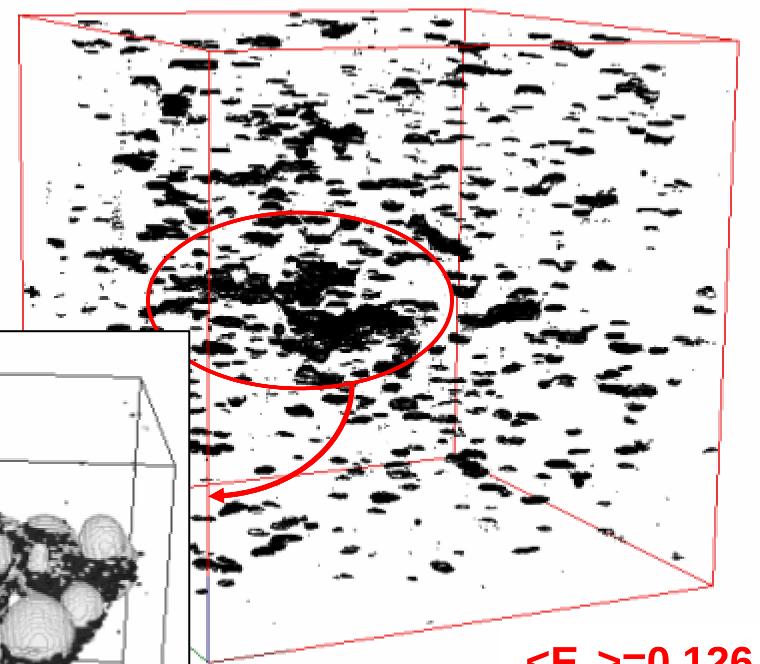
Damage in Composites

L. Babout, W. Ludwig, E Maire, J. Y. Buffière

NIM B 200, 303 (2003)

Example: hard matrix
Al2124(T6) + 4%ZrO_2SiO_2





Failure due to *coalescence* of cracks

Preferred damage initiation: particle rupture

Conditions : in situ, E=30 keV abs/phase, 1 µm

Phase imaging **propagation technique**

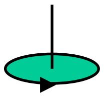
What is measured ⇨ $\nabla^2\phi(y,z)$, phase ϕ second derivative

Phase retrieval ⇨ integral (measurements at several distances)

holo-tomography

1) phase retrieval with 3-4 distances

2) repeated for 700 views



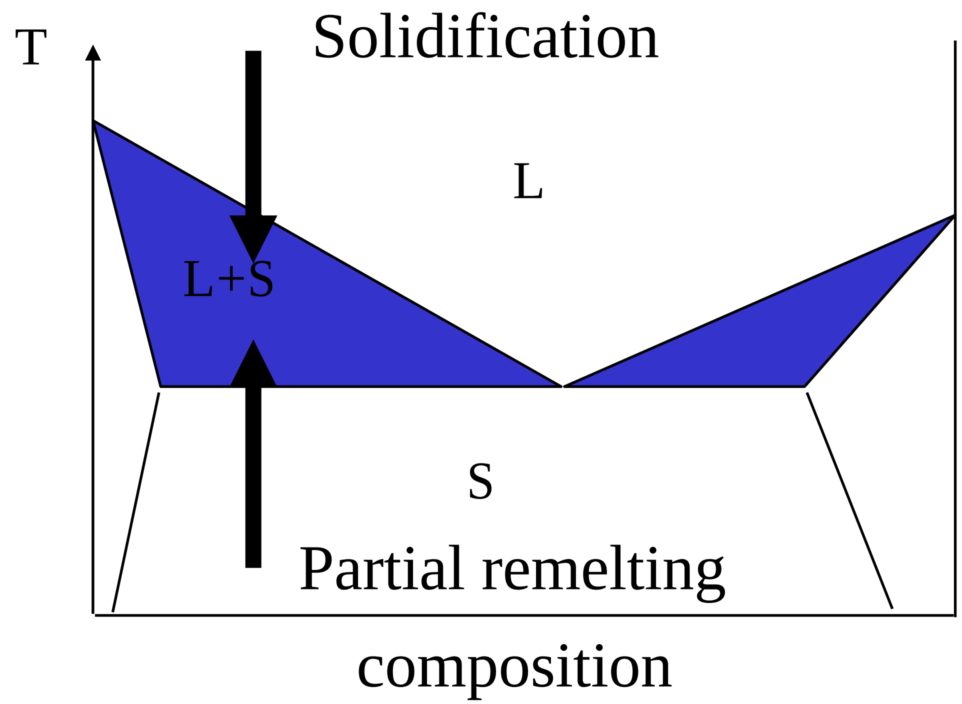
Experimental result ⇨ «local» ϕ (u,v)

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)

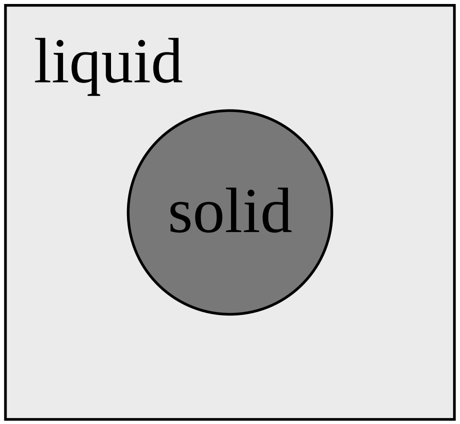
Rheology of aluminium alloys in the semi-solid state Al – Al/Si system



Microstructure

solid fraction
morphology
connectivity

Classical Models



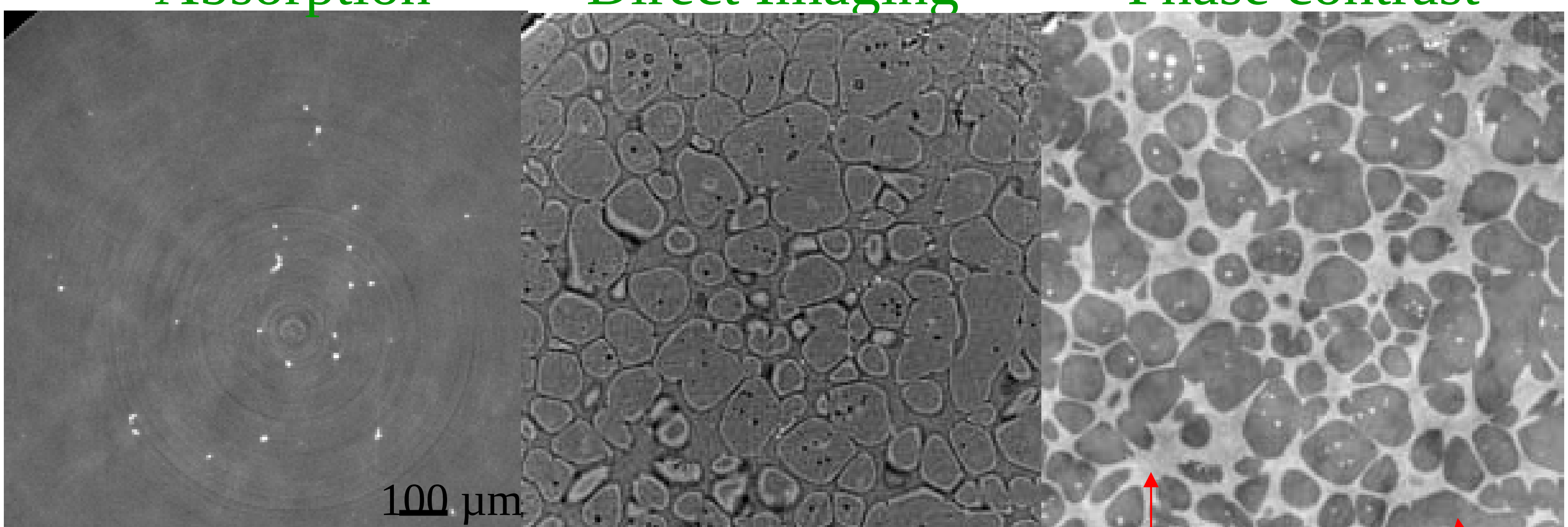
Holotomography on an Al - Al/Si system

Rheology of aluminium alloys in the semisolid state

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

E = 18 keV

Absorption | Direct Imaging | Phase contrast

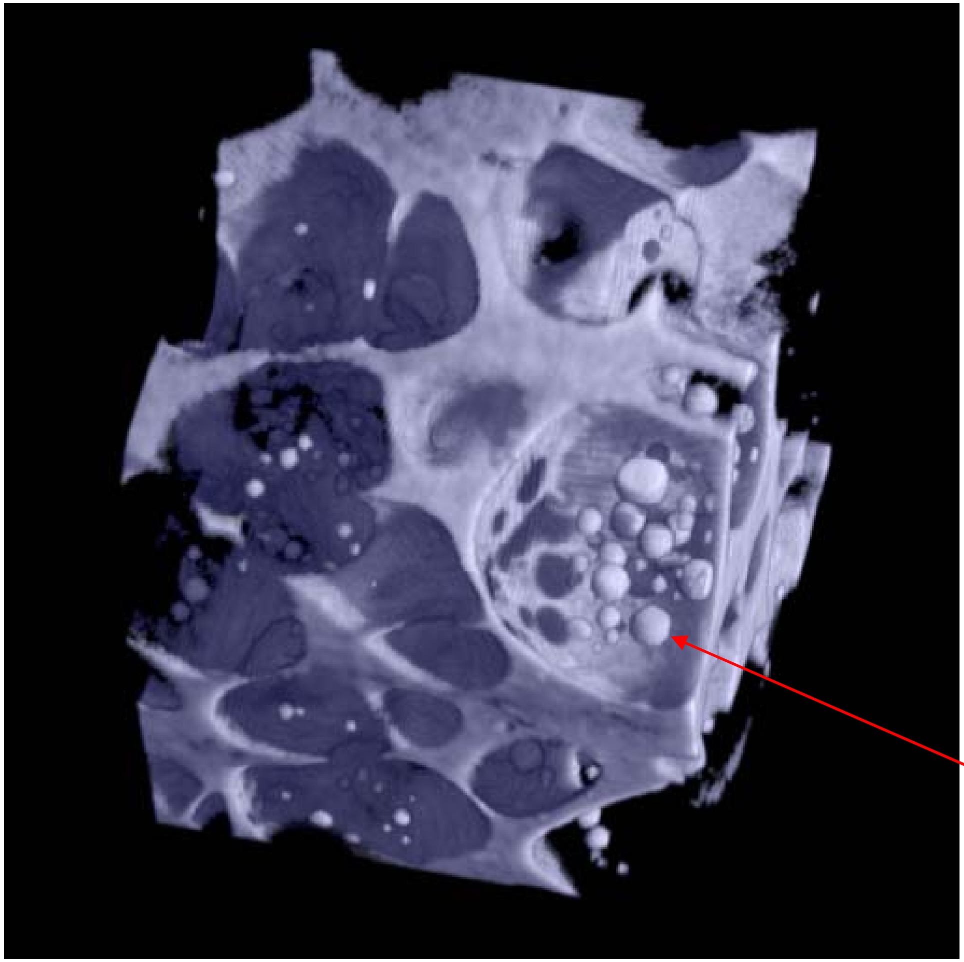


β-map | edge enhancement $D = 0.6$ m | Al/Si | δ-map | Al

L. Salvo, P. Cloetens

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 – SR X-ray imaging: where from do we come?
- **2 –** SR X-ray imaging: where are we?

 # coherent beams ⇨ new science

“poled” ferroelectric domains

growth and porosity of quasicrystals

new aspects of microtomography

- 3 – where should we go?

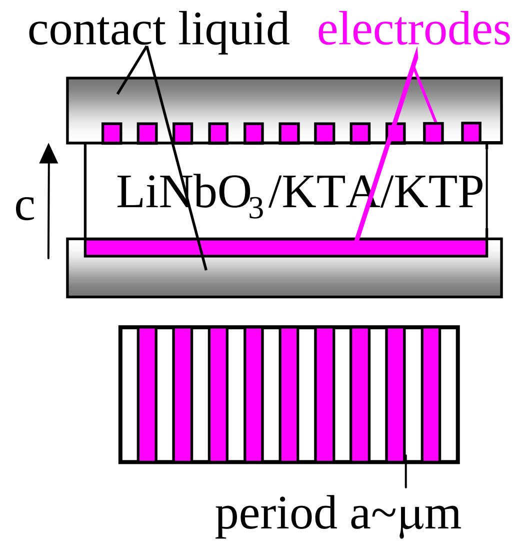
near future perspectives

PERIODIC FERROELECTRIC DOMAINS

P_S can be reversed by applying an electric field > $E_{COERCIVE\ FIELD}$

periodic electrodes defined on z+ face

high voltage pulses applied



⇨ Periodic ferroelectric domains

Diffraction by these domains:

domain I: F_{hkl}

domain II: F_{-h-k-l}

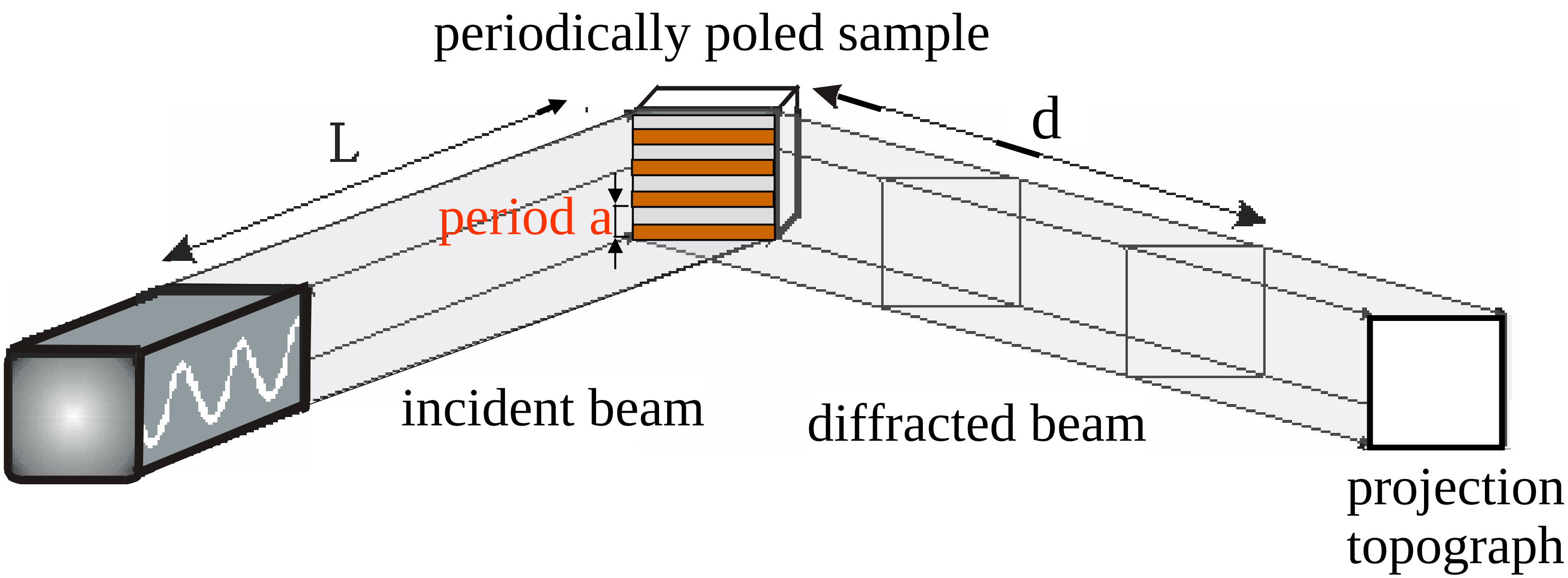
$|F_{hkl}| \sim |F_{-h-k-l}|$

but $\varphi_I = \varphi_{II}$

⇨ phase shift $\Delta\varphi = \varphi_I - \varphi_{II}$

BRAGG AND FRESNEL DIFFRACTION USED SIMULTANEOUSLY

$l_c > a$ ⇨ visualisation of the inverted ferroelectric domains



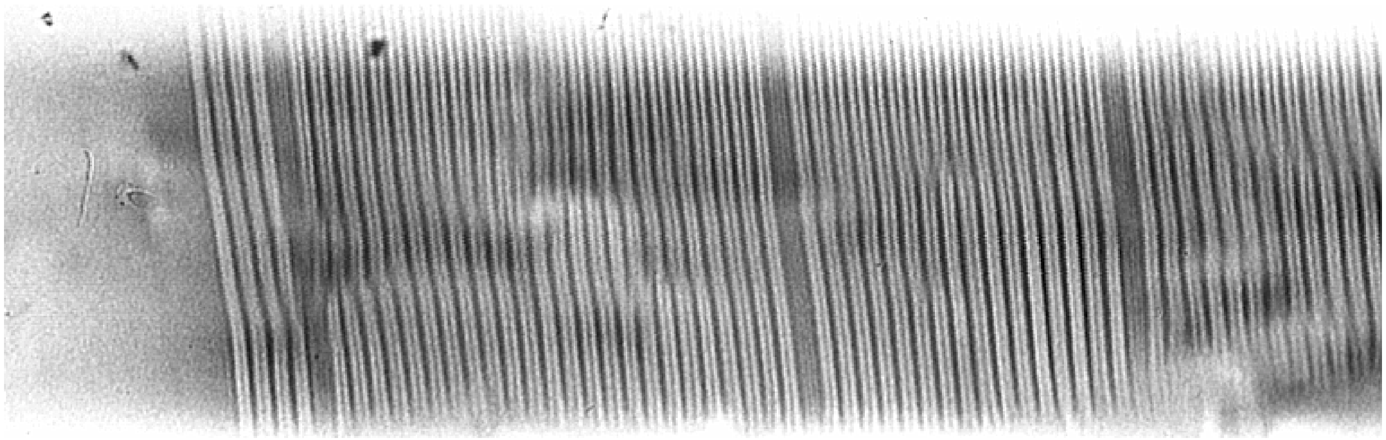
If wave diffracted by the adjacent domains exhibit a phase shift $\Delta\varphi$

possibility to retrieve it via free space propagation

Visualisation of the domains in the bulk

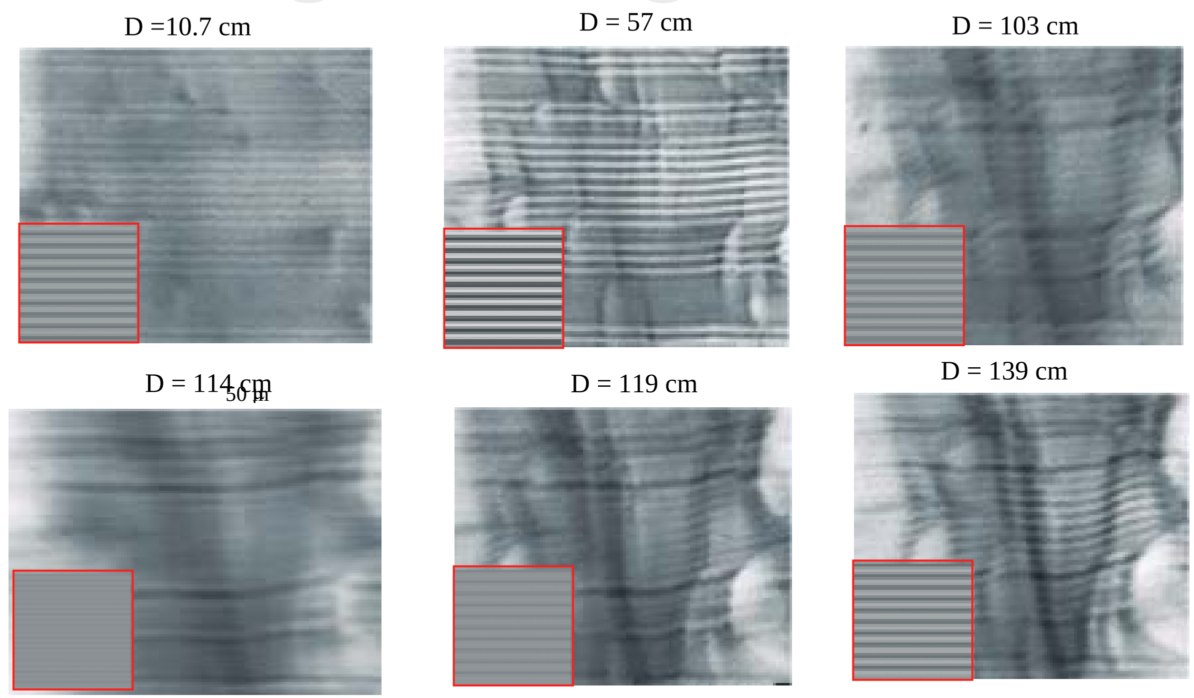
Section topograph, transmission mode, where Bragg and Fresnel diffraction are combined to reveal the domains

KTP, period of poling 9μm, Distance crystal-detector = 40 cm



Petra PERNOT, Pamela THOMAS, et al

The phase shift Δφ can be derived from the images through simulations



Δφ gives access to the domain matching schemes at the atomic level

- 1 – SR X-ray imaging: where from do we come?
- **2 –** SR X-ray imaging: where are we?

 # coherent beams ⇨ new science

"poled" ferroelectric domains

porosity and growth of quasicrystals

new aspects of microtomography

- 3 – where should we go?

near future perspectives

Quasicrystals

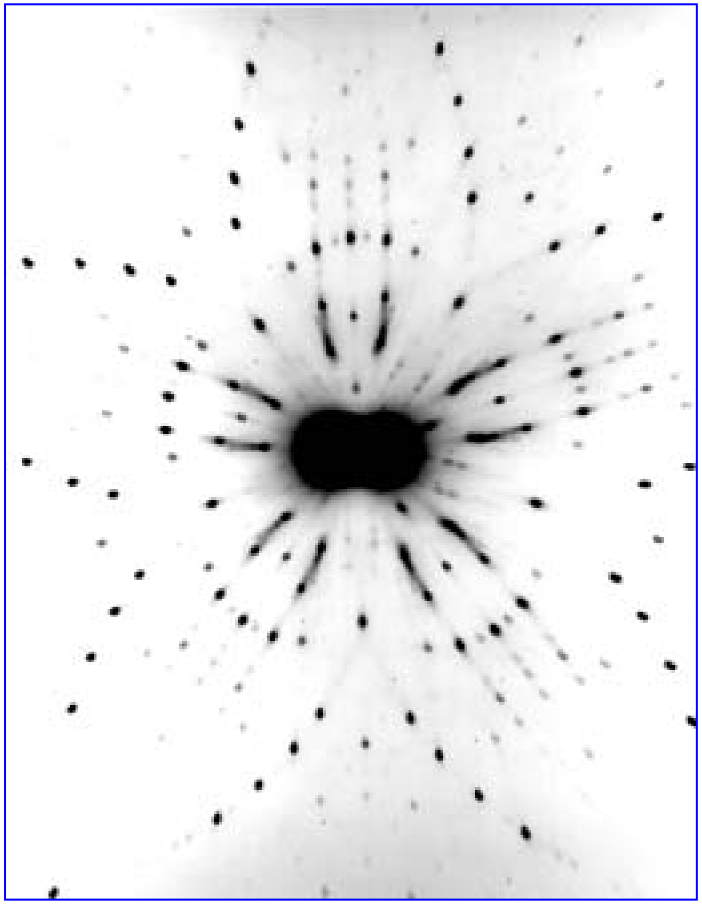
Quasicrystals (**QCs**) **are a new class of solids, discovered in 1984 (Shechtman et al.),** exhibiting:

→ a long range *quasiperiodic* translational order

→Orientational symmetries forbidden for crystals

(fivefold for instance)

→ sharp diffraction patterns



(J. Gastaldi)

Laue image of an AlPdMn quasicrystal showing five-fold symmetry

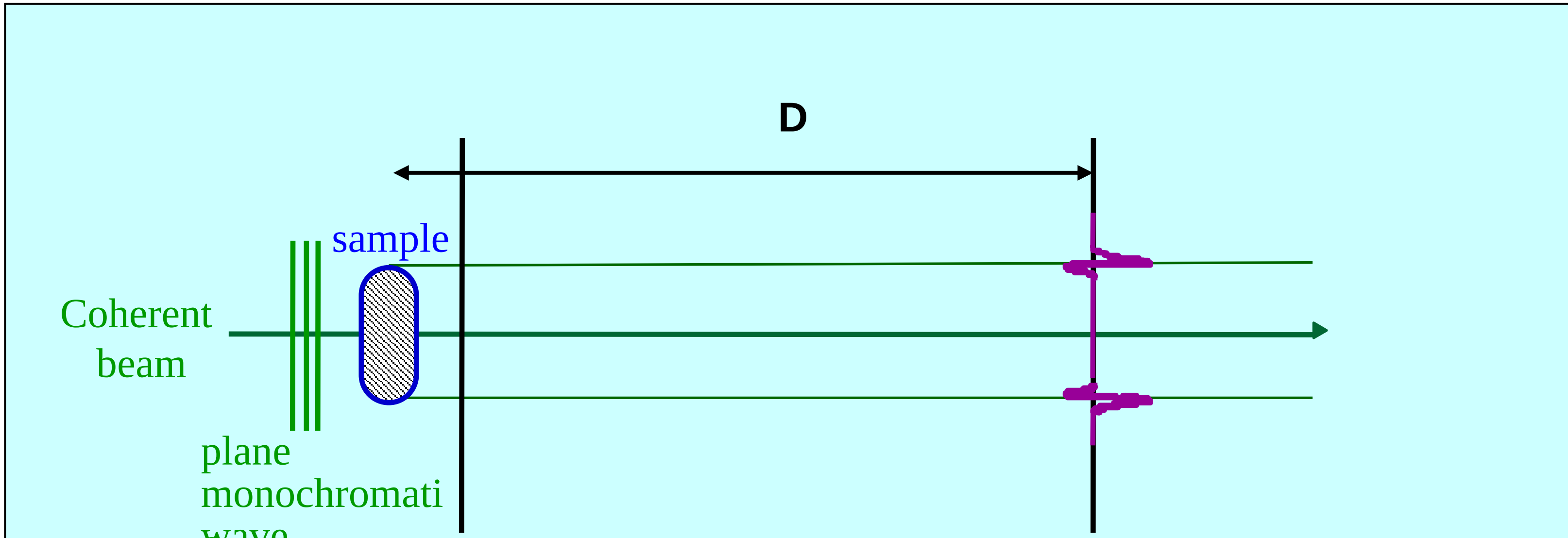
- The complex structure of quasicrystals results in peculiar physical properties (e.g. high resistivity).

- Defects can influence these properties.

- One may detect precipitates, (dislocations), pores, … .

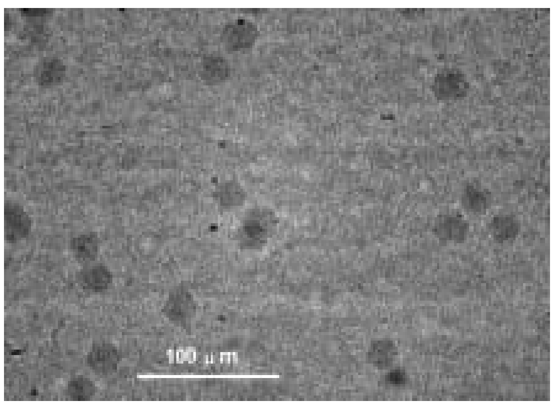
- Pores are defects commonly observed in quasicrystals.
  $\Rightarrow$ origin and formation of porosity ?

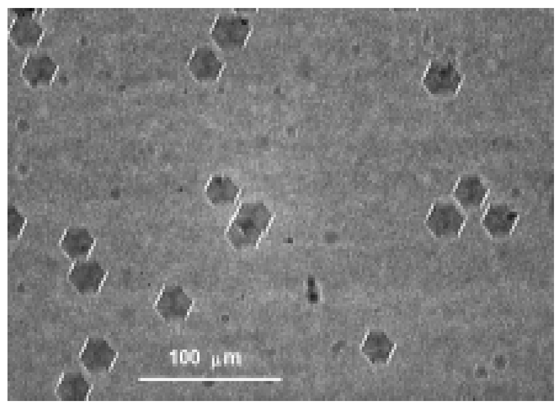
Phase Sensitive Radiography

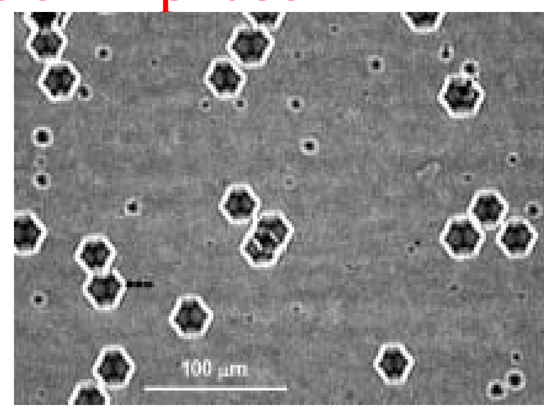


absorption

absorption + phase





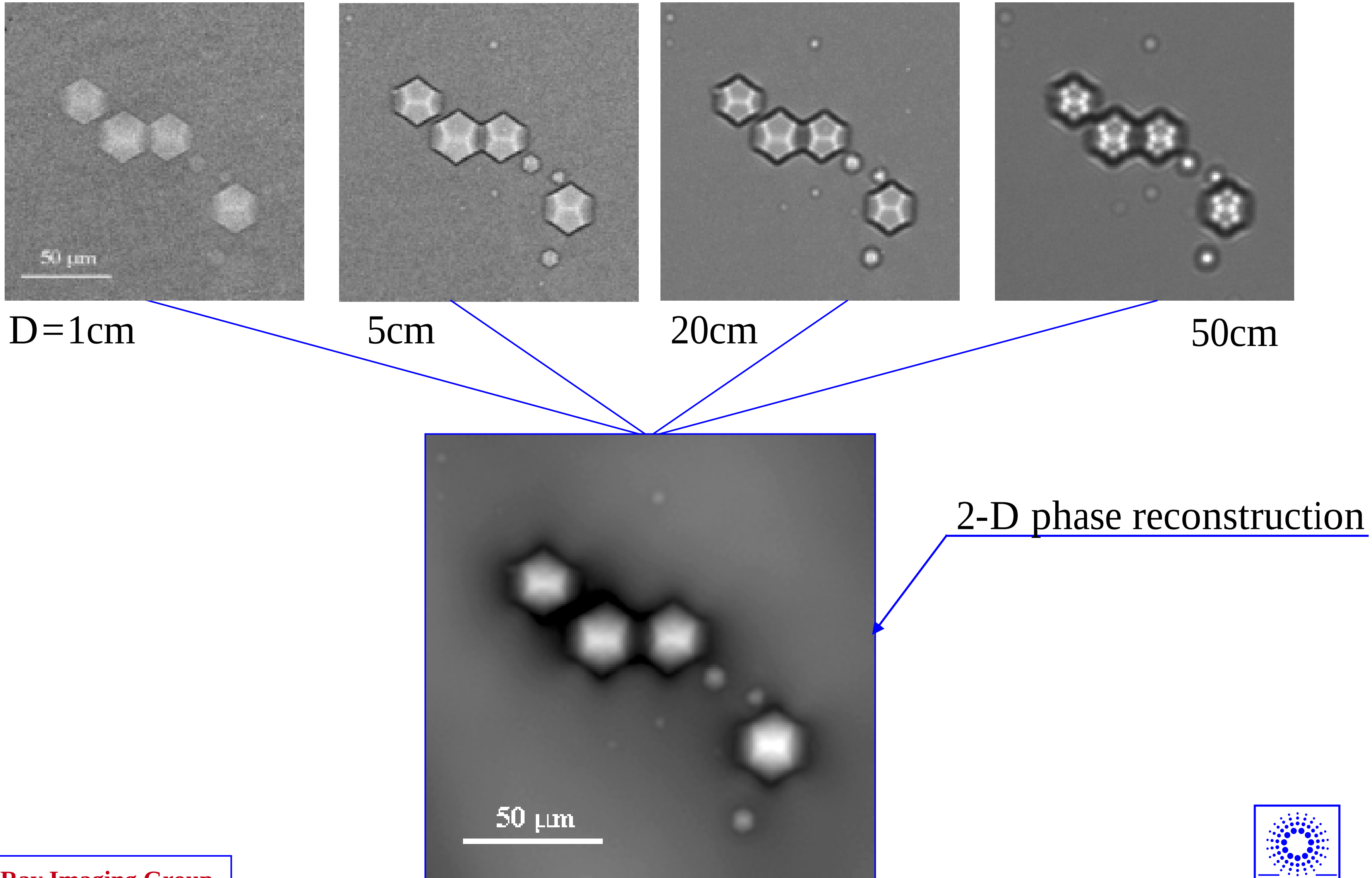


D = 1.3cm *D = 10cm* *D = 50cm*

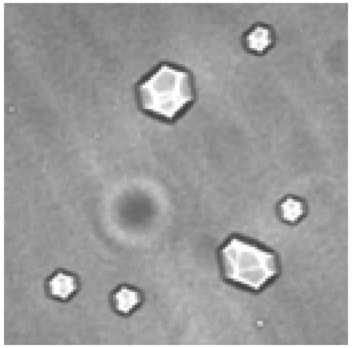
$\lambda = 0.35$Å

Determination of the real pore size

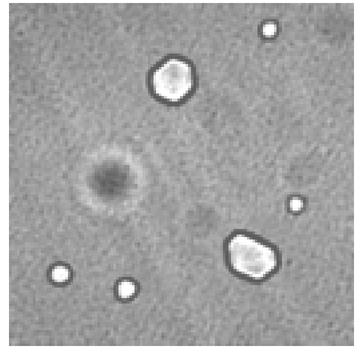
2-D phase reconstruction from recorded images at different distances



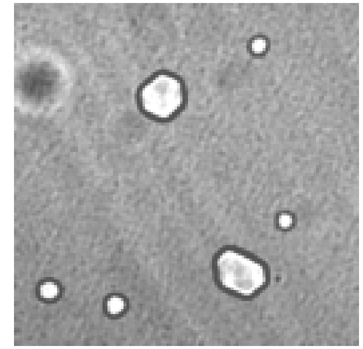
1st Annealing



a) RT ↗

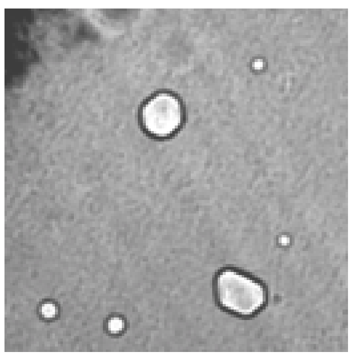


b) 800°C 3h25

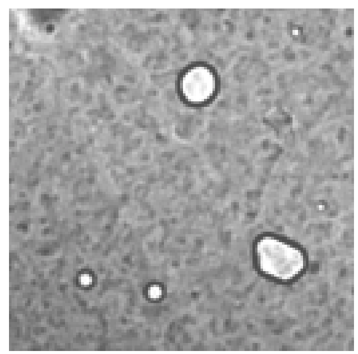


c) 800°C 4h55

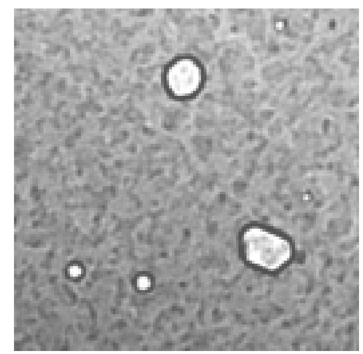
2nd Annealing



d) 800°C 4h50



e) 800°C 27h 10



f) RT ↘

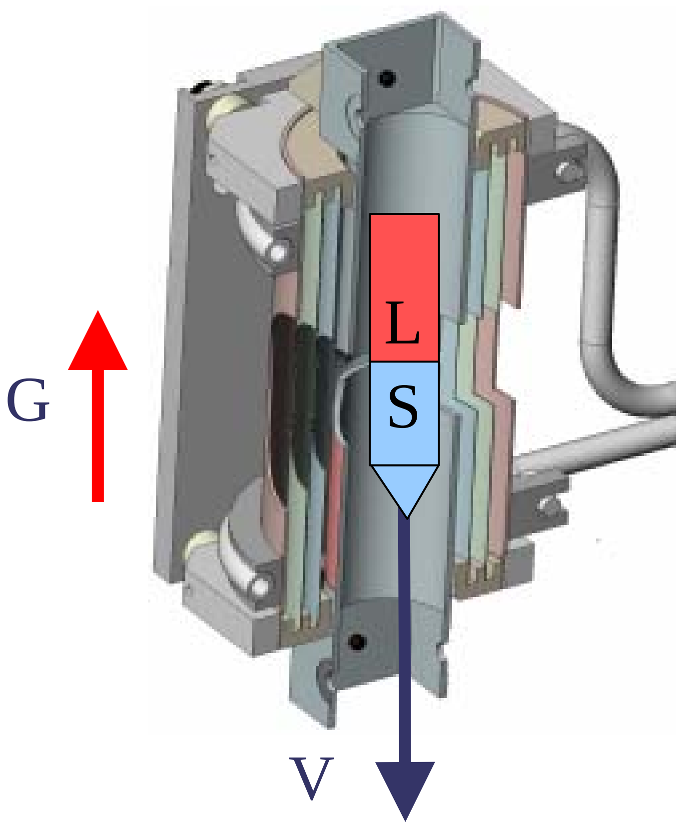
- Increase of the global perfection after annealing
- Most of pores reduce their sizes.
- Small pores are not anymore detectable
- Pores at surfaces did not shrink

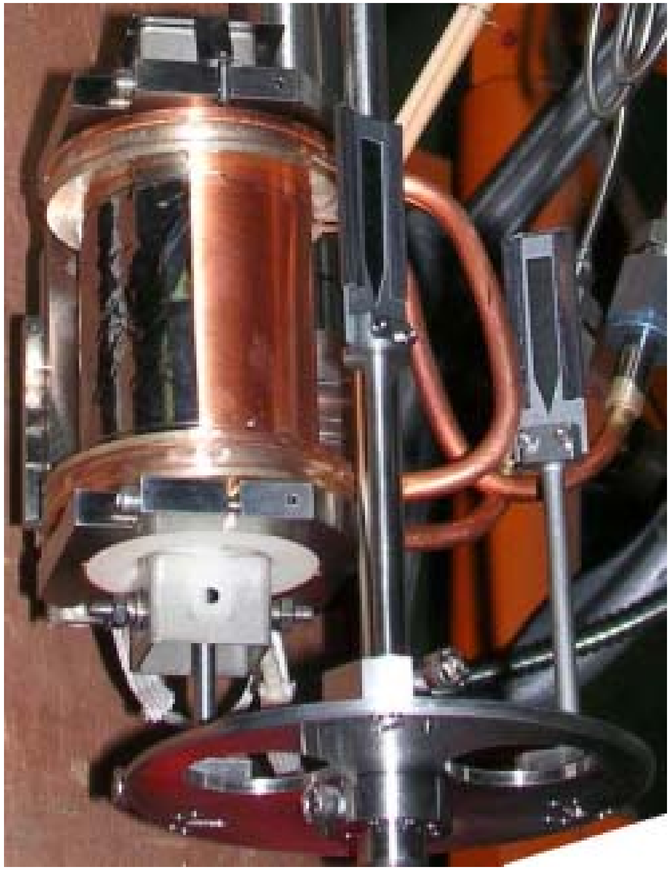
results support the hypothesis of vacancy diffusion

Experimental device

Experimental device

Bridgman furnace with two independent heating elements
Sample size: 37 x 6.0 x 0.20 mm; Ultra vacuum ($10^{-7/-8}$ mbar)

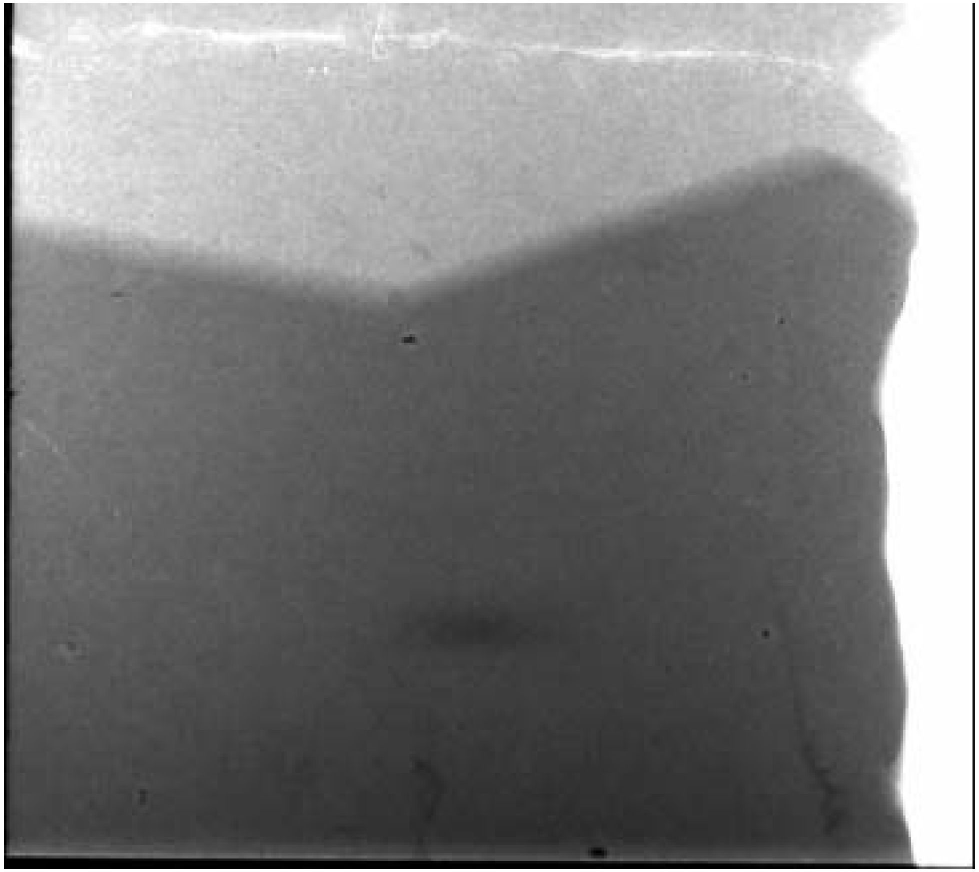




Real-time observation of the growth of a AlPdMn quasicrystal

$Al_{68.8} Pd_{24.0} Mn_{7.2}$, G = 20 K/cm, V = 3.6 µm/s

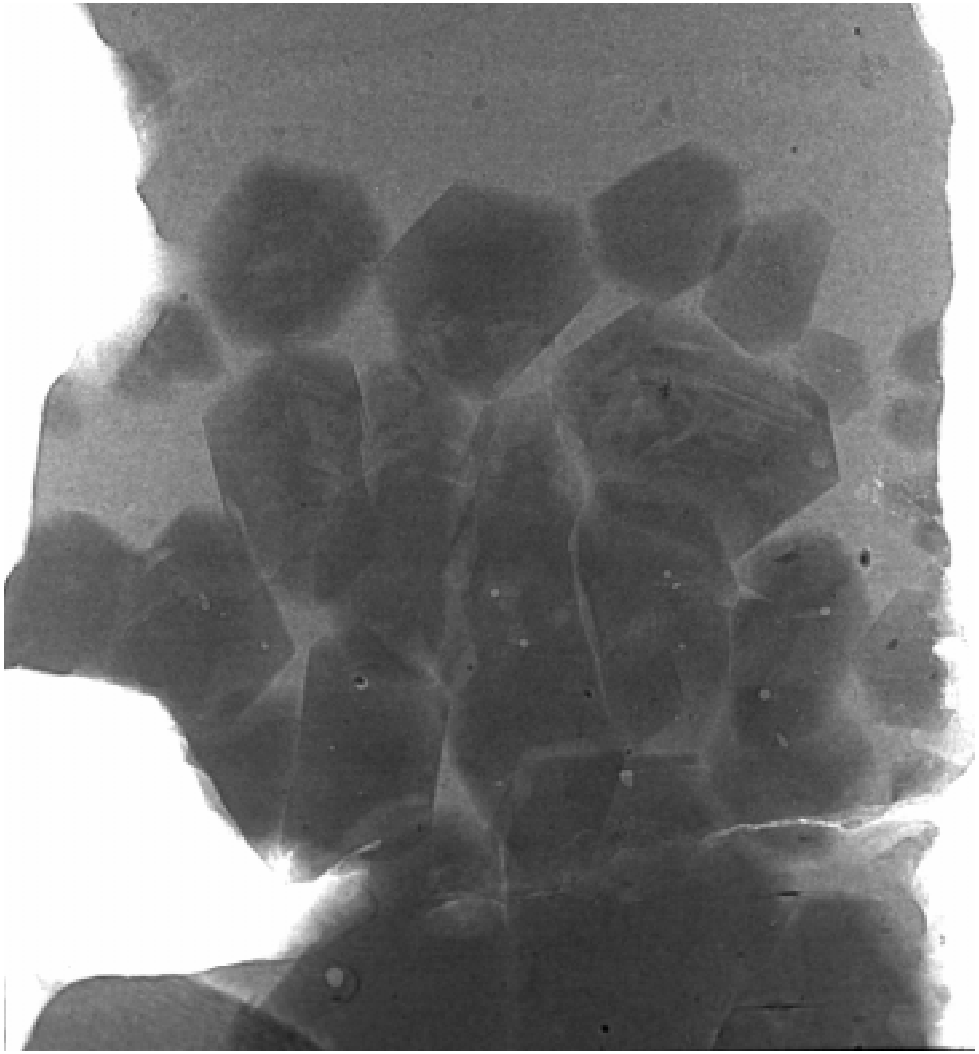
Real time length : 0h27



- Nucleation of multiple grains ahead of SL interface
- Step-like marks in each grain: macrosteps,segregations?

H. Nguyen Thi, T. Schenk, J. Gastaldi, J. Härtwig, H. Klein, J. Baruchel (J. Cryst. Growth, in press)

Real time observation of the growth of a Al-Pd-Mn quasicrystal



H. Nguyen Thi,

J. Gastaldi,

T. Schenk,

H. Klein,

J. Härtwig

(2004)

X-Ray Imaging Group

ESRF

- 1 – SR X-ray imaging: where from do we come?
- **2 –** SR X-ray imaging: where are we?

  # not only imaging, but therapy

  # coherent beams ⇨ new science

  “poled” ferroelectric domains

  growth and porosity of quasicrystals

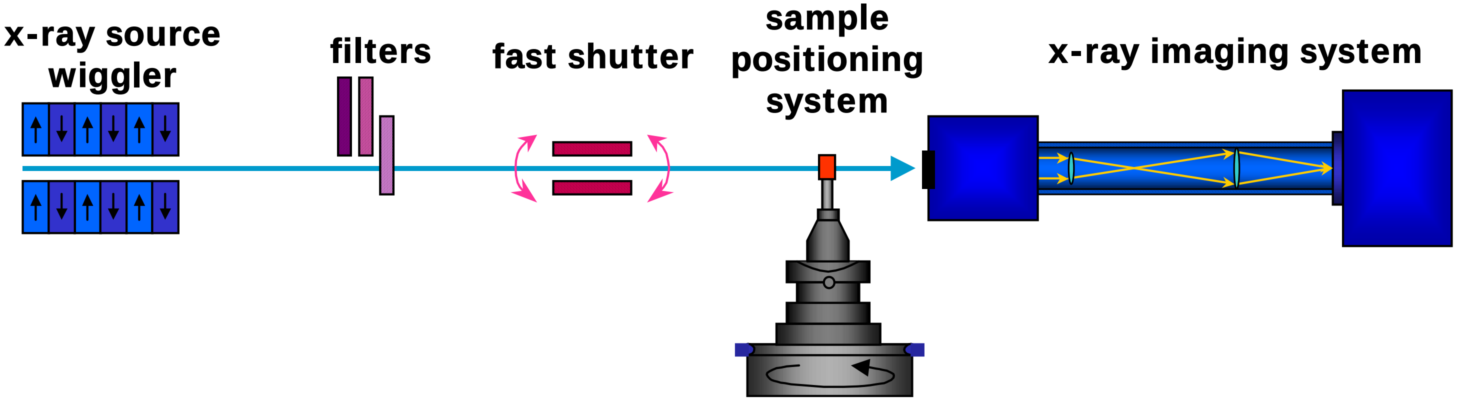
  # **new aspects of microtomography**
- 3 – where should we go?

  # near future perspectives

FAST TOMOGRAPHY

ID15 Beam line at ESRF

Experimental device

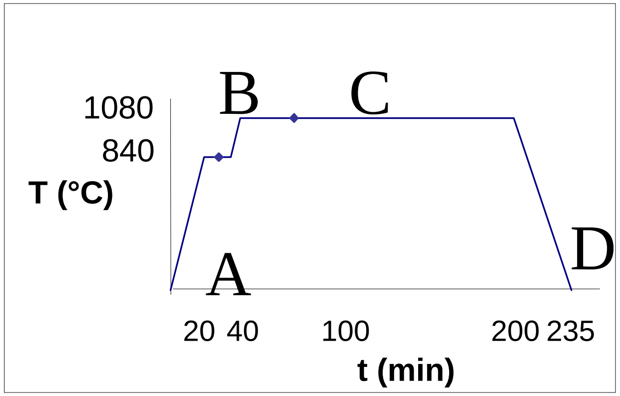


By adjusting the wiggler gap and choosing the proper filter the white beam spectrum is optimized in order to get the best signal-to-noise ratio

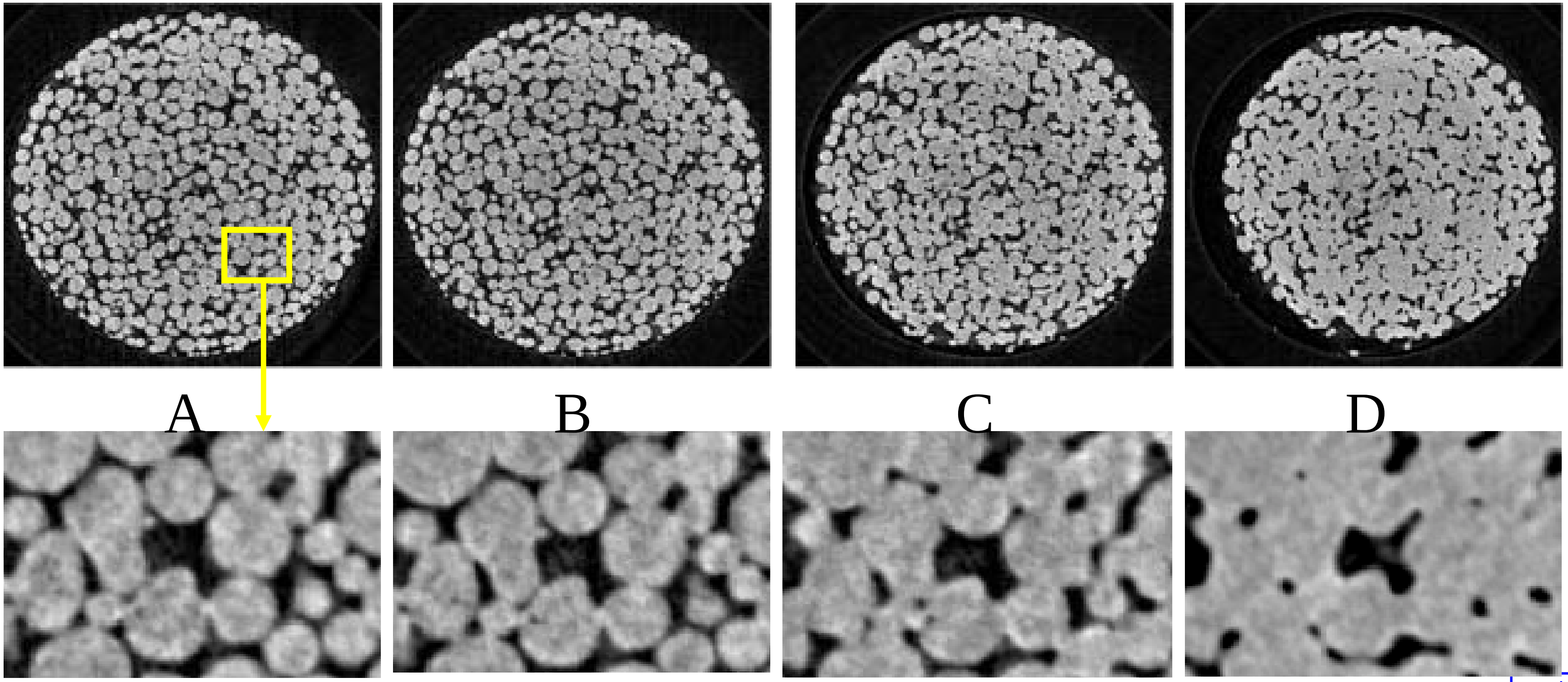
Exposure time per image: from 10 to 50 ms

In-situ follow of Cu powder sintering at 1050°C

O. Lame, D. Bellet (INPG, Grenoble),
M. di Michiel (ESRF)

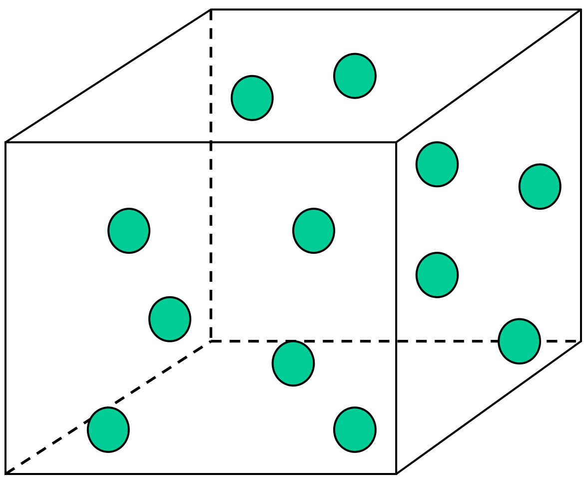


300 μm

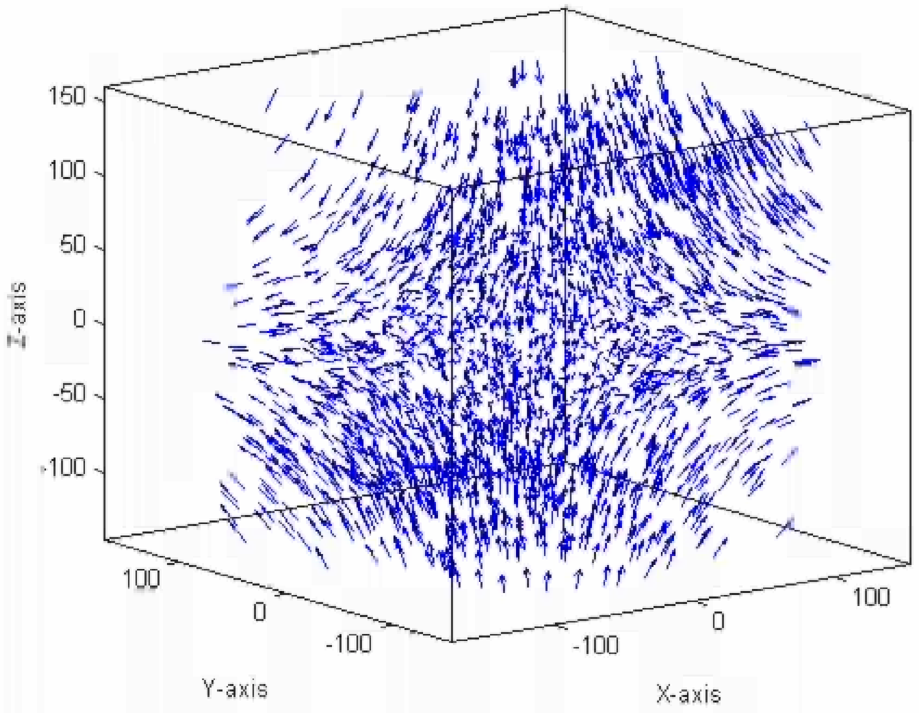


Tomography can reveal plastic strain field

Markers:



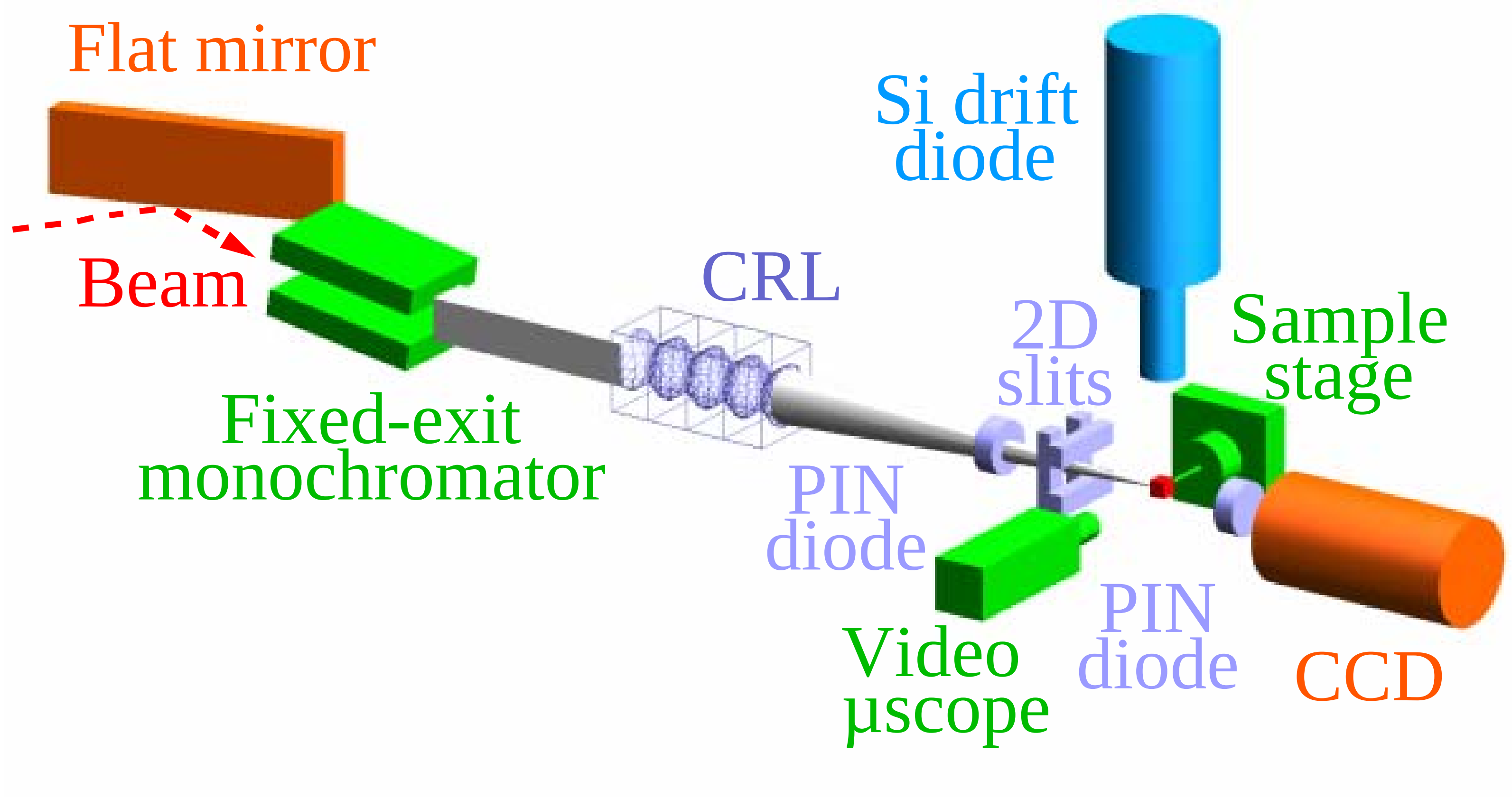
Compression of Al/W sample:



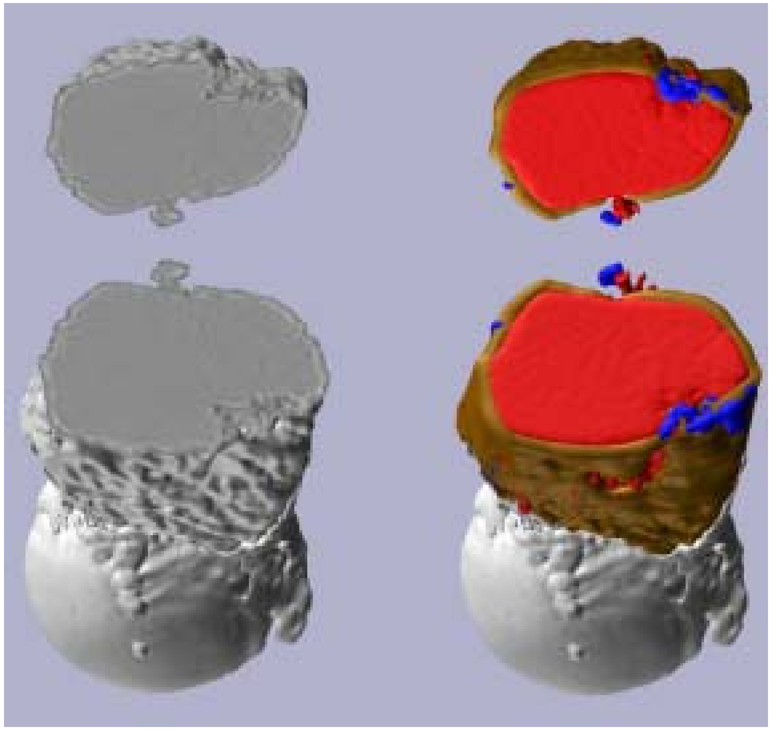
Work performed at HASYLAB (Germany)

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

Fluorescence tomography: towards 3D imaging



Combination with fluotomography



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*, in press,

- 1 – SR X-ray imaging: where from do we come?
- 2 – **SR X-ray imaging: where are we?**

coherent beams ⇨ new science

“poled” ferroelectric domains

growth and porosity of quasicrystals

new aspects of microtomography

- 3 – **where should we go?**

 # near future perspectives

Scientific Case

Nano-technology/fabrication: 50 nm scale and below

Materials Science: relevant scale 50 nm - 50 μm

Bio-materials: implants, …

Soft condensed matter: soils, …

Micro-structure, composition, plasticity

3D imaging, with improved temporal and spatial resolution

In-situ experiments with sample environment

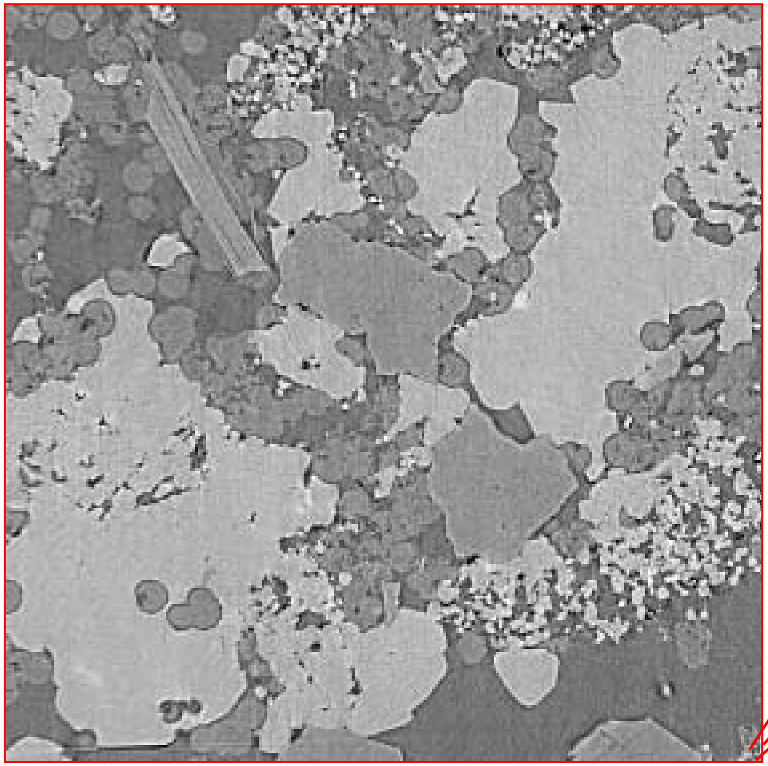
Micro-analysis possibilities

The resolution gap (1)

<u>X-ray micro-tomography</u>

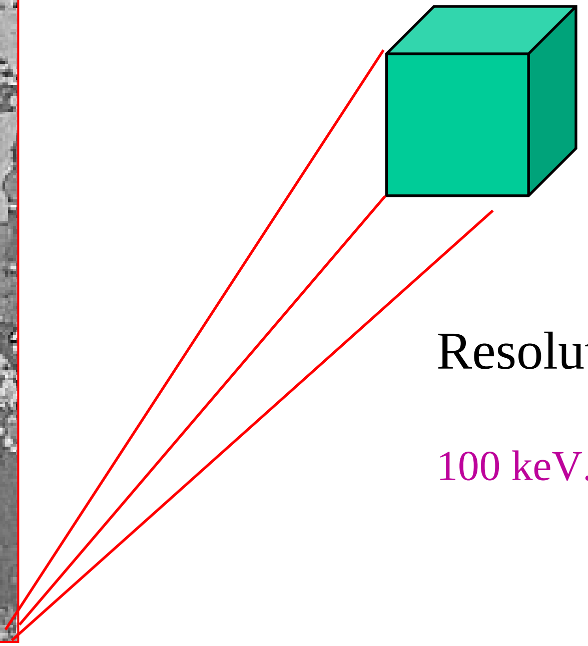
O Rozenbaum, Univ. d'Orléans / CNRS-ISTO

240 µm



1 voxel = 280 nm

<u>Tomography in STEM with 1 nm diameter electron probe.</u>



Resolution about 1nm.

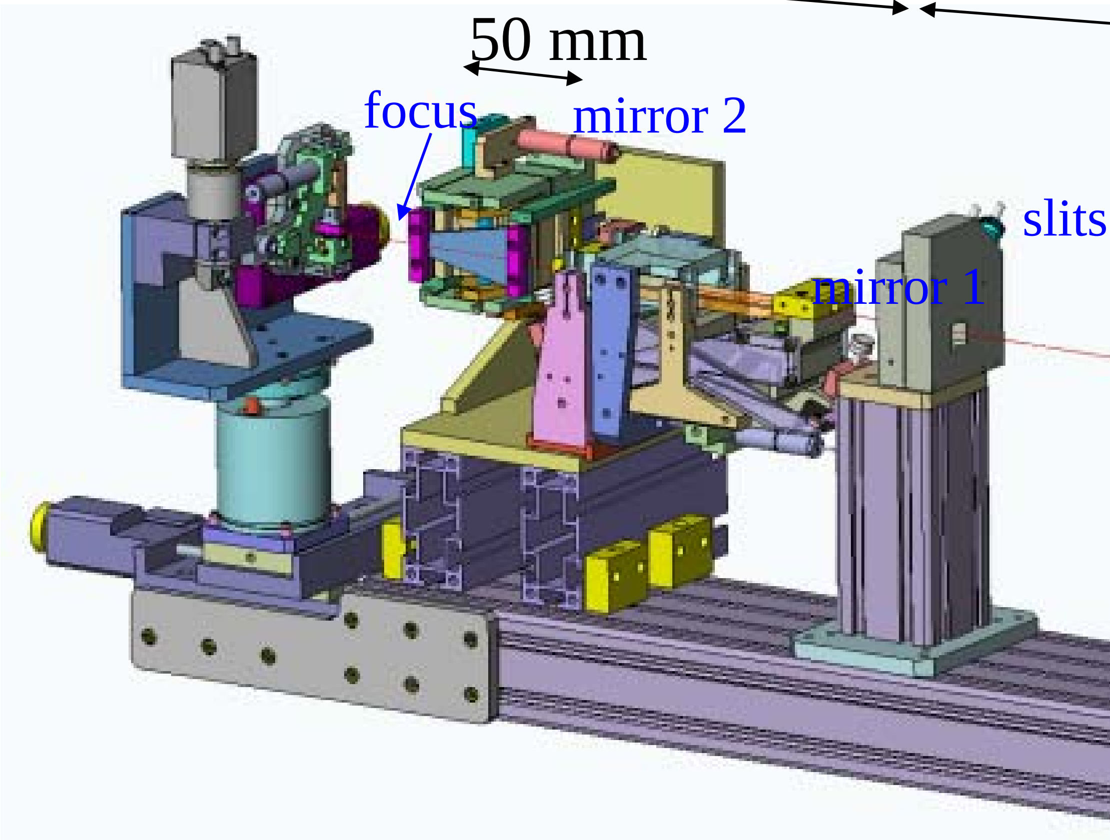
100 keV. P.Midgely et al, 2002

Kirkpatrick-Baez focusing (O. Hignette, P. Cloetens)

F_2< 300 mm

F_1=150 m

50 mm

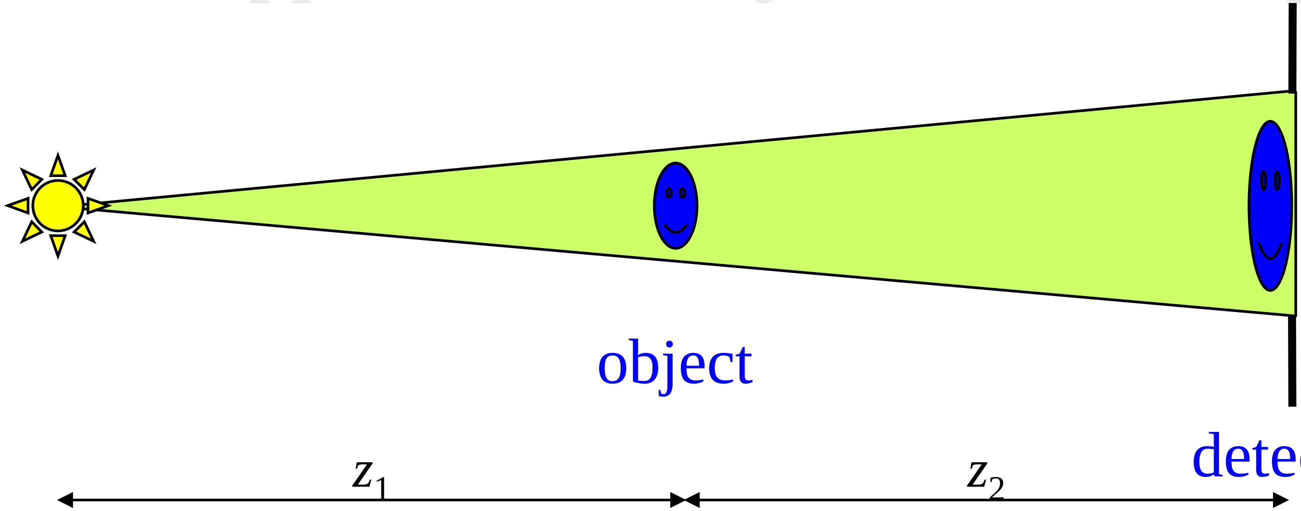


Source size
Mirror quality
Diffraction

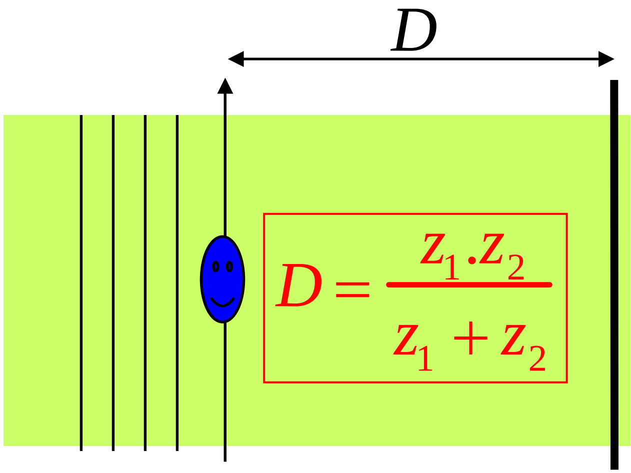
focus = demagnified image of the source; demagnification = F_1/F_2

Source size: 125 μm(h) * 25 μm (v); **experimental result 70 nm x 90 nm**

Applications: Projection Microscopy



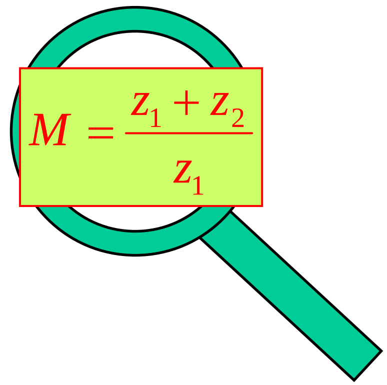
equivalent to



$$D = \frac{z_1 . z_2}{z_1 + z_2}$$

Plane wave illumination

+



$$M = \frac{z_1 + z_2}{z_1}$$

Magnification

Limit cases

$z_1 >> z_2$

$D = z_2$; $M = 1$

$z_1 << z_2$

$D = z_1$; $M = z_2/z_1$

Projection Microscopy

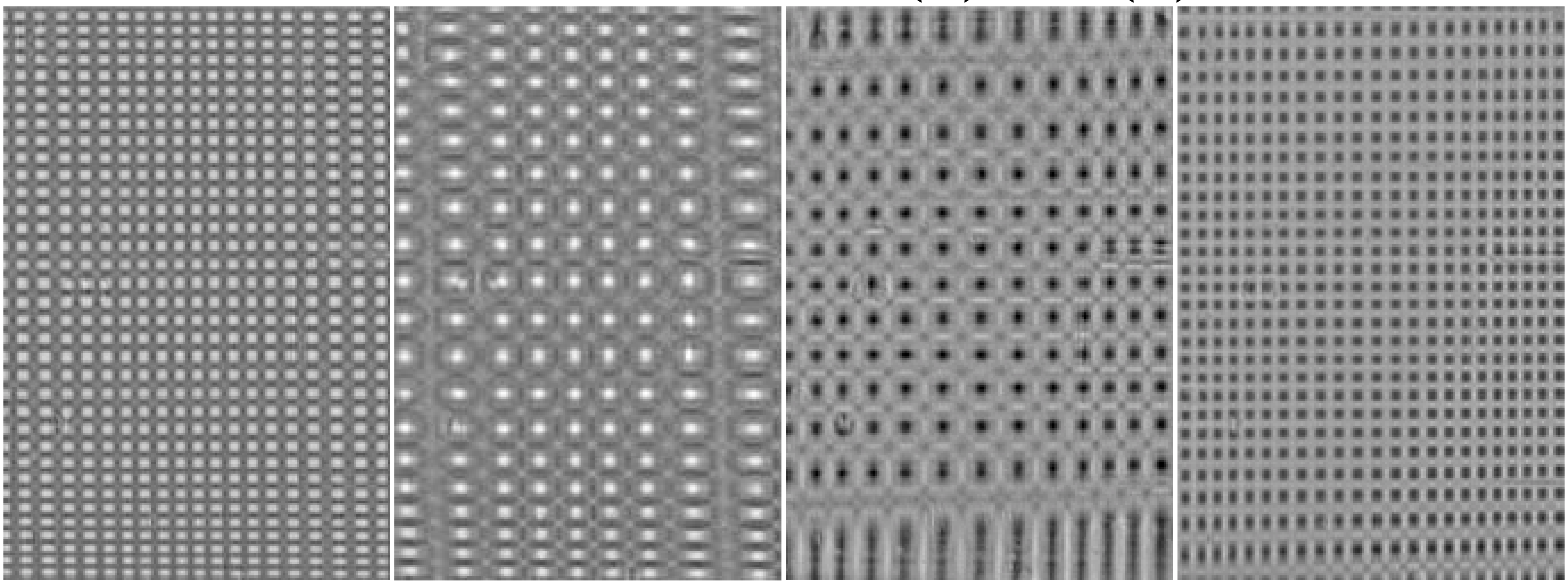
2 μm pitch grating (Si)

C. David, PSI

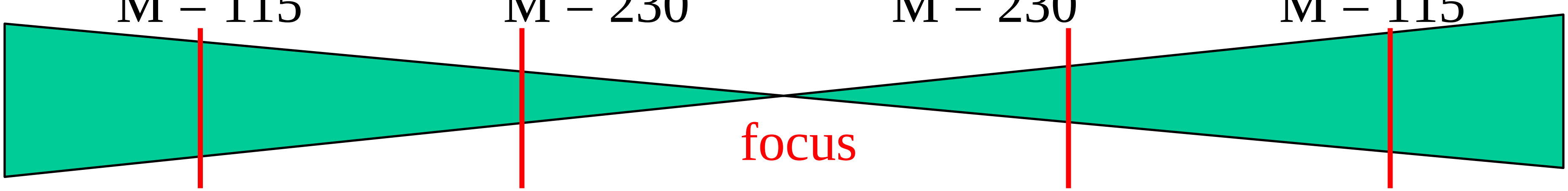
Mirrors fully illuminated

0.2 (H) x 1.1 (V) mm, *E*=20.5 keV

10 μm



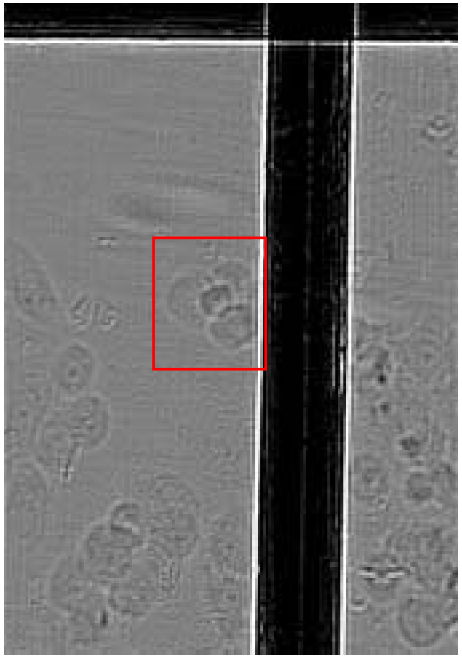
D = -20 mm	D = -10 mm	D = 10 mm	D = 20 mm
M = 115	M = 230	M = 230	M = 115



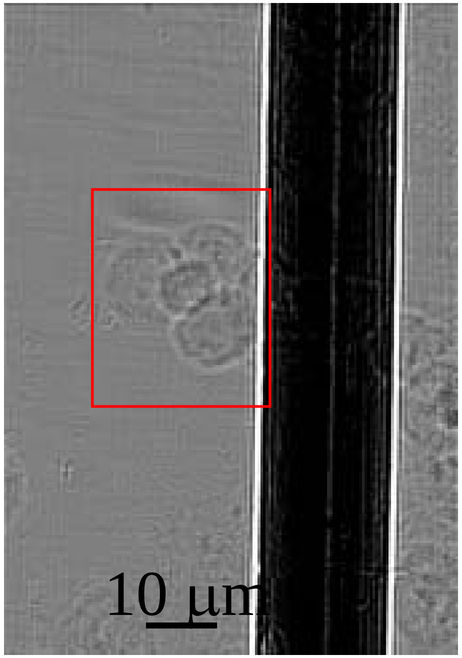
Exposure time = 0.4 s ! (16-bunch, saturation CCD)

P. Cloetens, O. Hignette

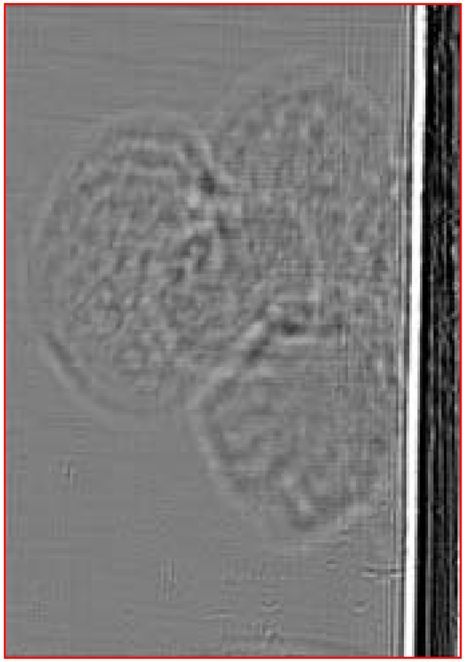
Projection Microscopy: cancerous cells



D = 50 mm
M = 45



D = 30 mm
M = 75



D = 10 mm
M = 230

E = 20.5 keV; exposure time = 0.3 s ! (16-bunch)

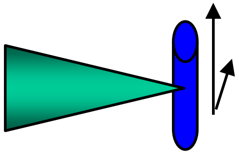
Problems: aberrations, instabilities, refraction by the sample itself, ….

S. Bohic, P. Cloetens, W. Ludwig

Applications: Fluorescence

Cancerous cells

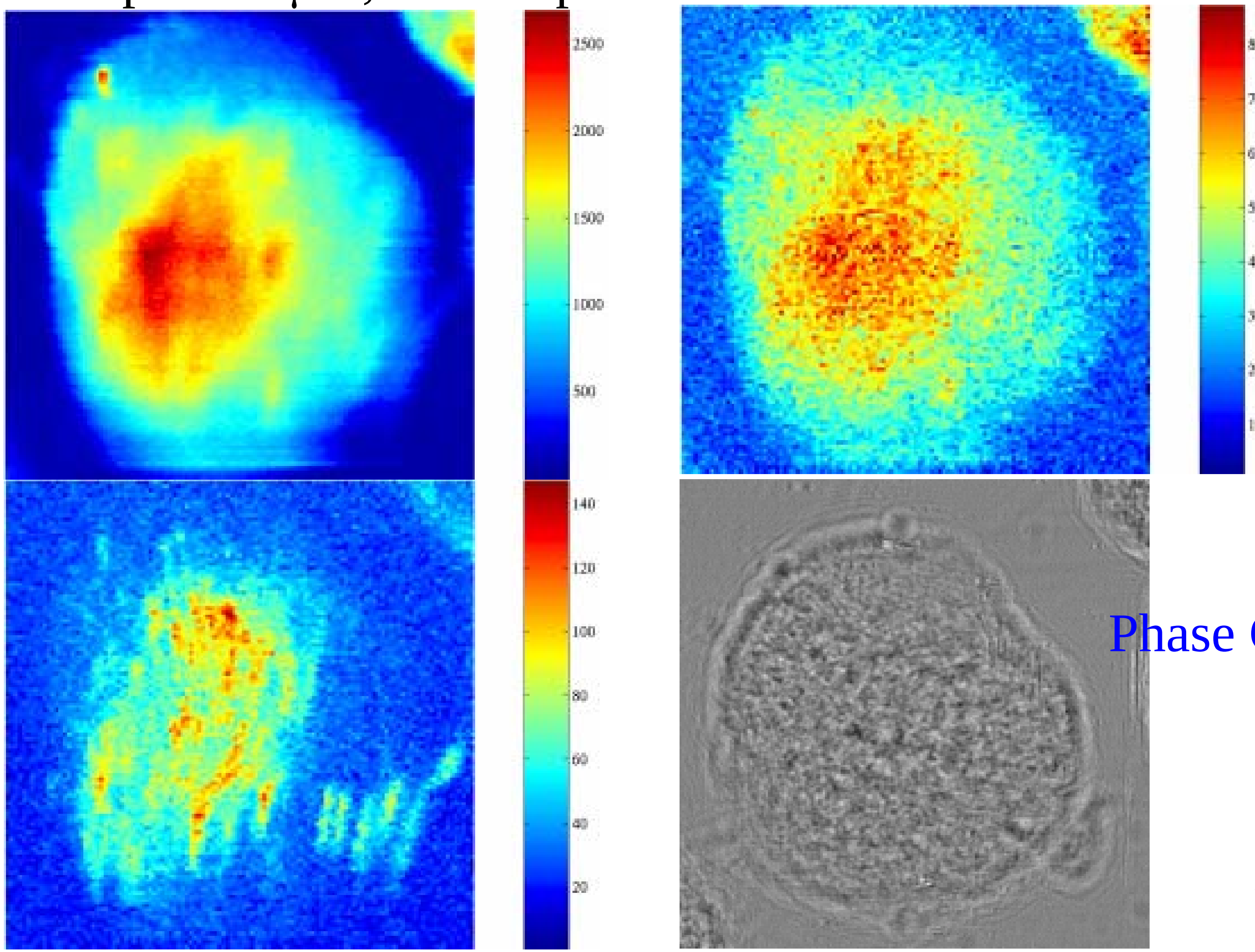
Step = 0.3 µm, 0.9 sec/pt



40 µm

K

Pt



Fe

Phase Contrast

P. Cloetens,
W. Ludwig,
S. Bohic

The resolution gap (2)

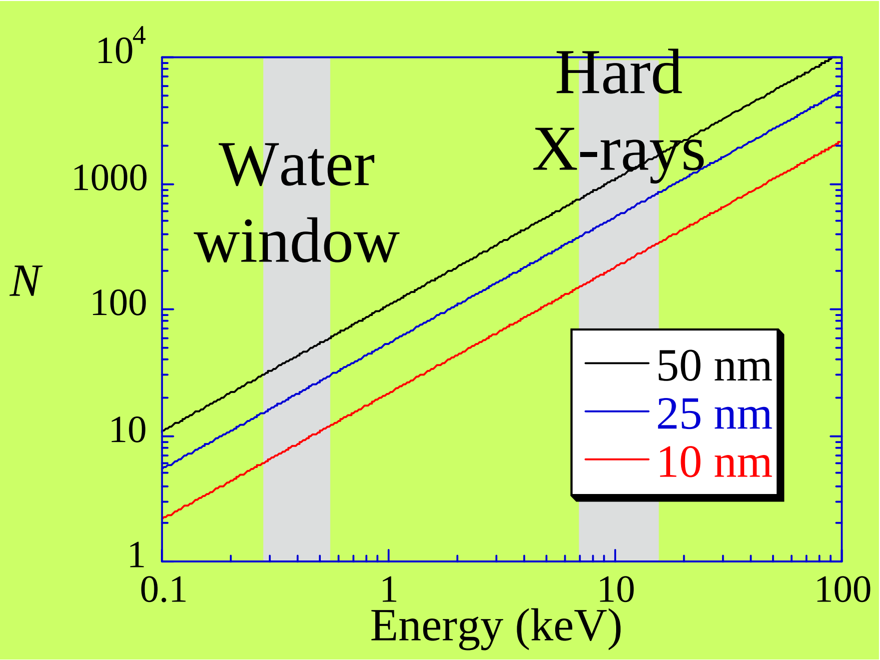
Can X-ray imaging become a true 3D ultra-microscopy technique?

$$N = \frac{\text{max. sample thickness}}{\text{resolution element}}$$

max. sample thickness →
- Absorption
- Depth of focus
- Multiple scattering

resolution element →
- Optics
- Interaction strength
- Photon statistics

Depth of focus



50 nm resolution

water window ▶ $N = 60$ (=3µm)

10 keV ▶ $N = 1000$ (= 50µm)

⇨ Soft X-ray microscopy remains 2D technique

⇨ 'Hard' X-rays will have to fill the gap

↘

"nanotomography project" (Peter CLOETENS)

Conclusions

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

The coherence of the beam adds new possibilities

We must improve the spatial and time resolution to be useful for many of the problems encountered in modern science and technology

Images contain a lot of information: the scientific use this information implies an accurate knowledge of the contrast mechanisms, and (often) image processing