3D Imaging using Coherent Synchrotron Radiation

P. Cloetens

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ESRF, Grenoble

[1] GEMPPM, INSA, Lyon

X-ray Imaging Group

ID17 Medical Beamline

ID19 Micro-Tomography

ID21 Microscopy / Micro-analysis

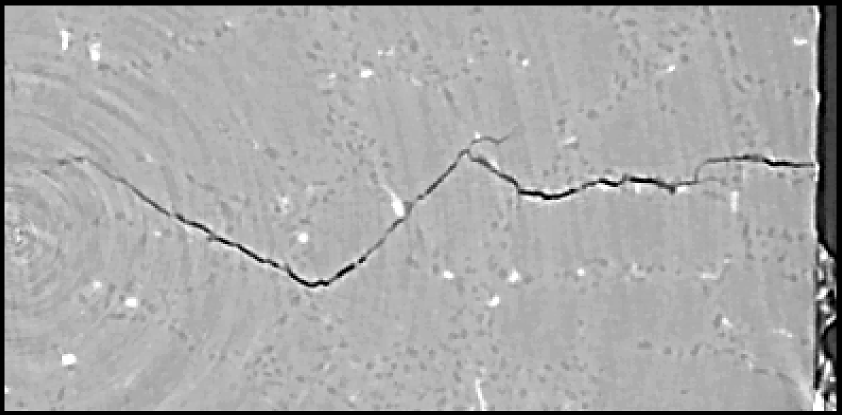
ID22/ID18F Micro-Fluorescence

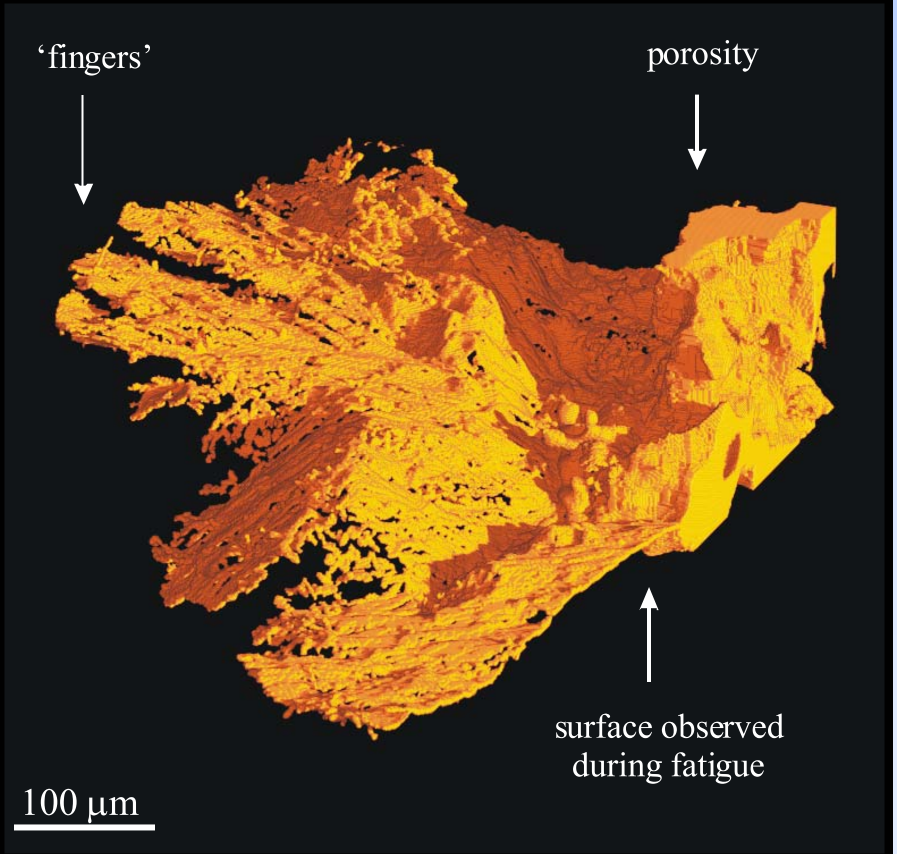
150 of 900 ESRF proposals

2D vs 3D Imaging

- Microscopist:
  knows the answer to his problem
  looks for the image that confirms his idea

- Tomographist
  tries to get the whole picture





Microtomography using synchrotron radiation

High flux, monochromatic, parallel, coherent beam:

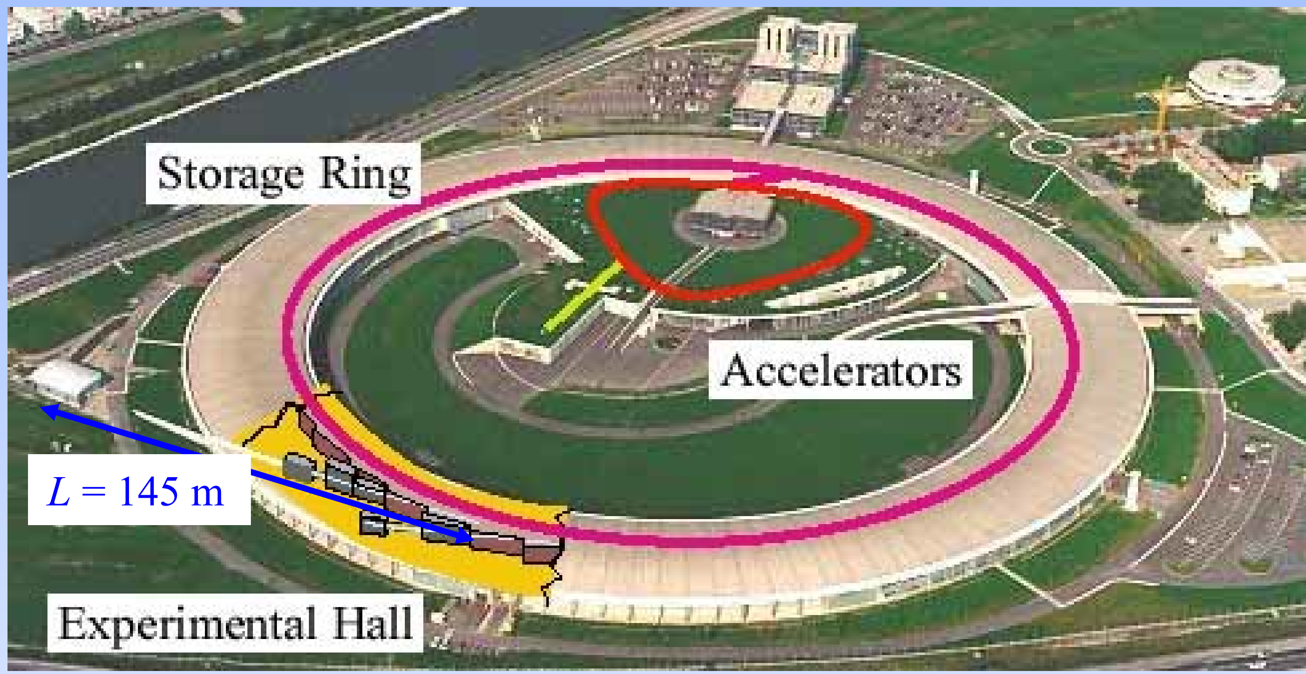
- High spatial resolution (~ micron)
- Quantitative and 'exact' tomography
- Fast Tomography (*in-situ*)

- Phase Contrast Tomography

Trends:

- Combined techniques: Fluo-Tomography
- Multidisciplinary Technique
- Nano-tomography

3-D Imaging: Better X-ray Sources



- High photon flux
- Monochromatic and parallel beam
- Partially Coherent beam

ESRF, ID19 Beamline:

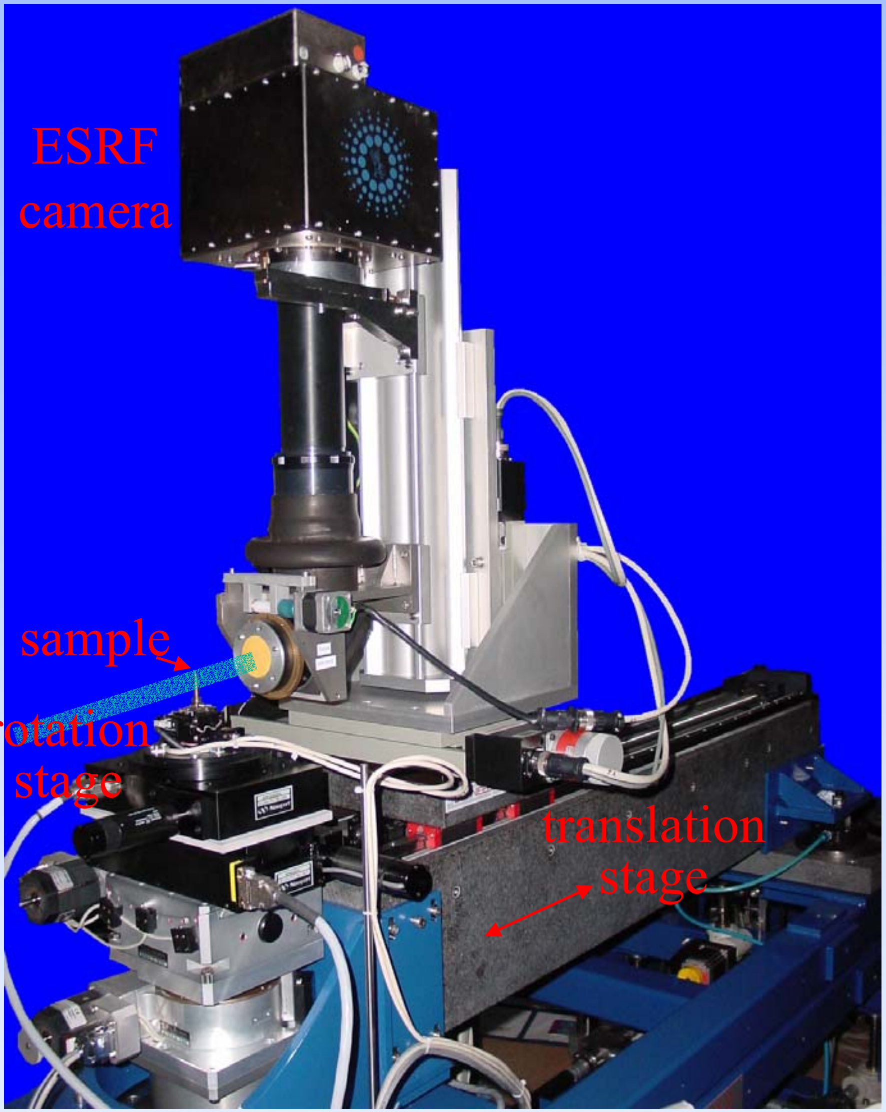
source

$L = 145$ m
$s_v \approx 25$ µm
$s_h \approx 125$ µm

$\alpha \approx 0.2$ µrad
and horizontally < 1 µrad

→ $l_{coh} = \lambda / 2\alpha \approx 250$ µm

3-D Imaging: Digital Detectors



Implemented on ID19 / ESRF

Dedicated microtomograph (P. Bernard)

Monochromator:

double Si crystal ($\Delta\lambda/\lambda=10^{-4}$)

or multilayer ($\Delta\lambda/\lambda=10^{-2}$)

Sample stage

rotation stage (tomography)

Detector

CCD-based (FReLoN)

on translation (propagation)

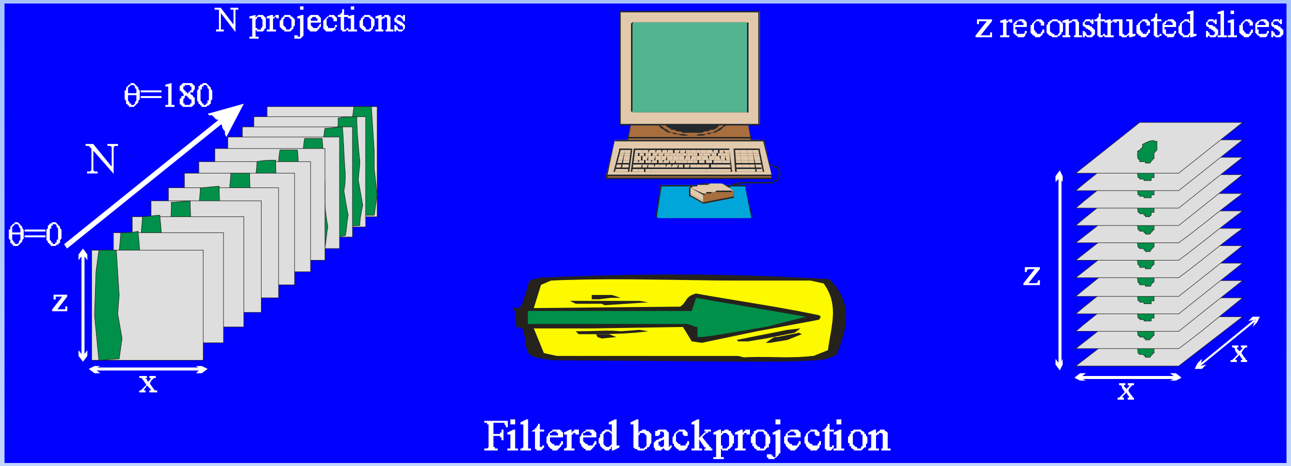
soon FReLoN2 (frame transfer)

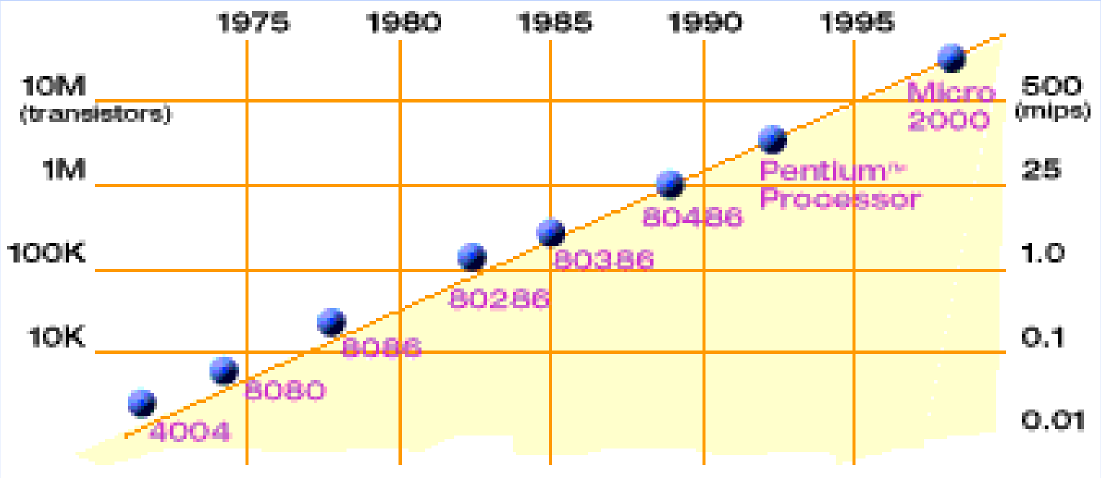
Scan time

1024^2 * 900 proj.: 5 minutes

2048^2 * 1500 proj.: 15 minutes

3-D Imaging: Faster Computers





Computing power grows exponentially

1024^3 volume reconstructed in 15 minutes

In-situ Tomography

Follow 3D structure and composition as function of

- time (fast tomography)
- temperature (furnace, cryostat)
- strain (tensile stage)
- ...

Sequential Tomography

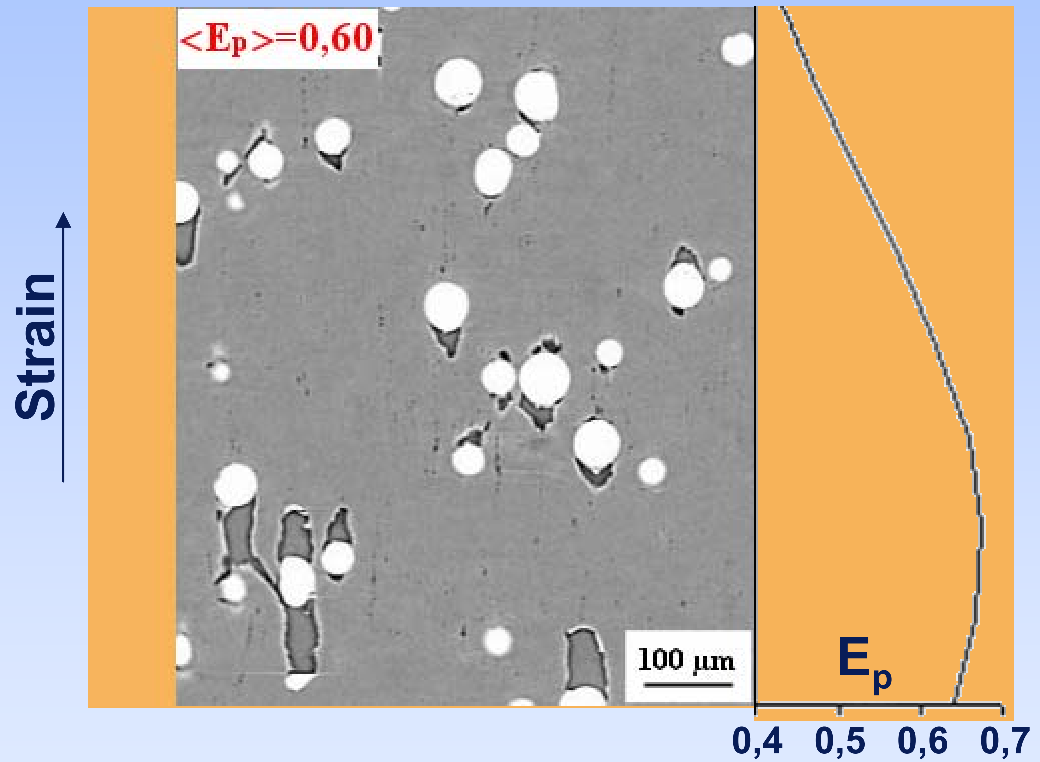
- interrupt evolution between successive scans

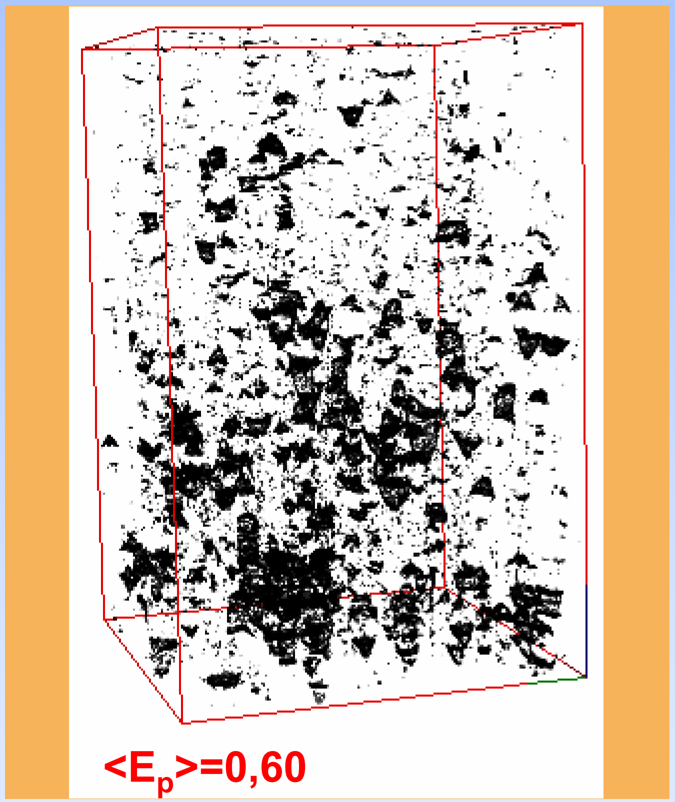
Real-time Tomography

- scan time << evolution time
- High Energy Beamline

Damage in composites

Example: soft matrix
Al + 4%ZrO_2SiO_2





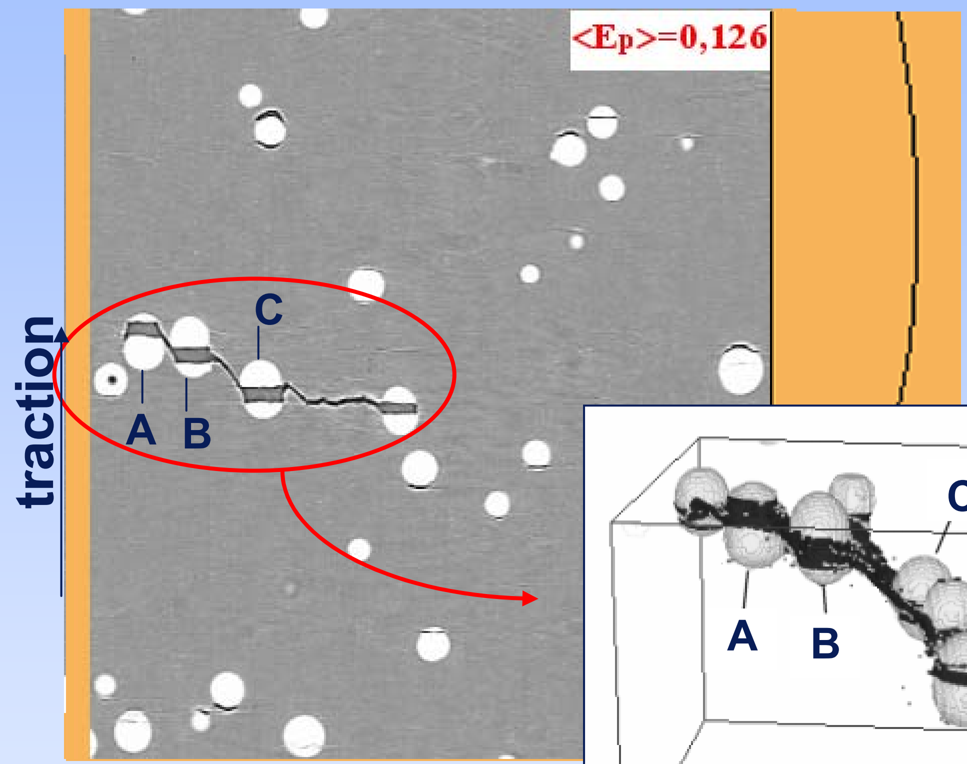
Preferred damage initiation: Matrix-particle *decohesion*

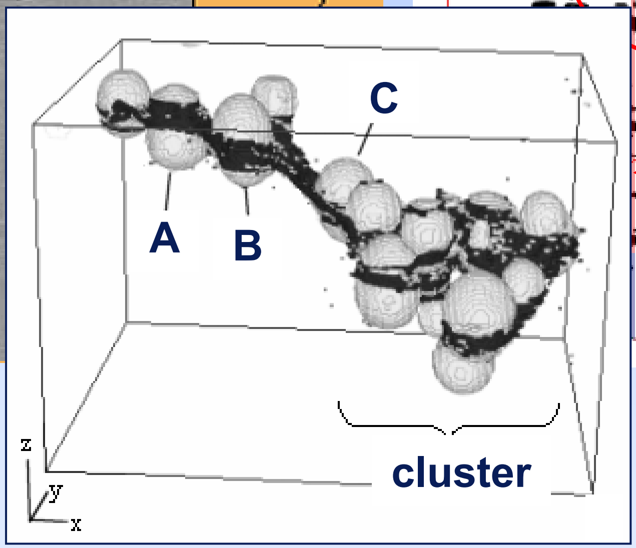
Conditions : in situ, E=30 keV abs/phase, 2 µm

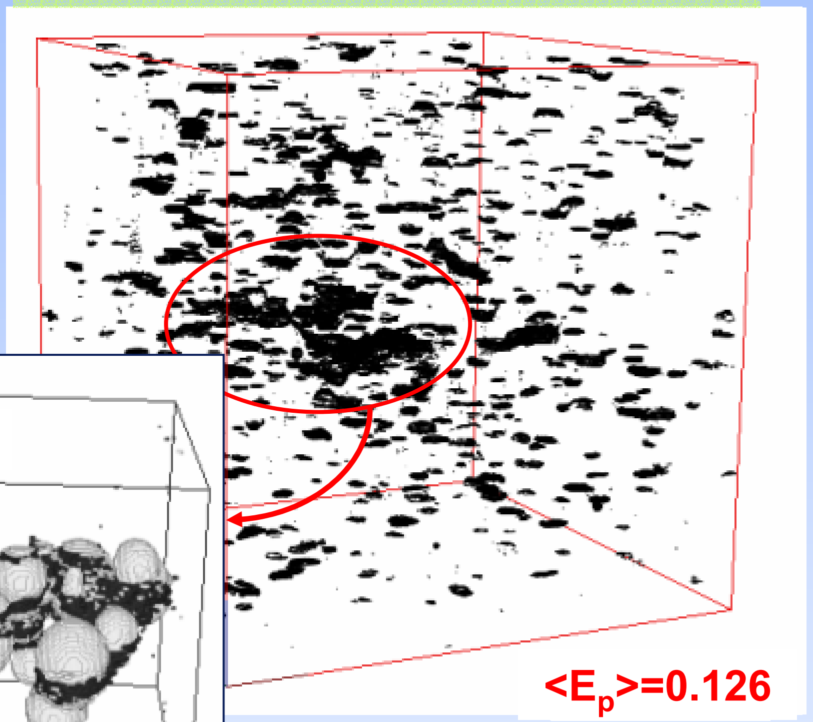
L. Babout, E Maire (INSA-Lyon)

Damage in composites

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







Failure due to *coalescence* of cracks and *local* configuration

Preferred damage initiation: particle rupture in mode I

Conditions : in situ, E=30 keV abs/phase, 2 µm

L. Babout, E Maire

Damage in composites

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

Damage initiation

mechanism

criteria (normal stress / stored elastic energy)

link to finite-element simulations

Damage growth: discrepancy with classical models

Failure: coalescence of cracks *under investigation*

Authors:

L. Babout, E. Maire, J.Y. Buffière

GEMPPM, INSA, Lyon

Why Phase Contrast?

- Dream 1: **Sensitivity**

  Absorption contrast too low

  high spatial resolution

  light materials

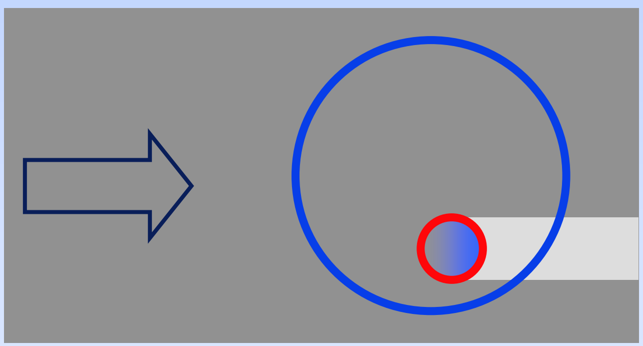
  similar attenuation: C-C, Al-Si, Al-Al_2O_3

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

$$\delta >> \beta$$

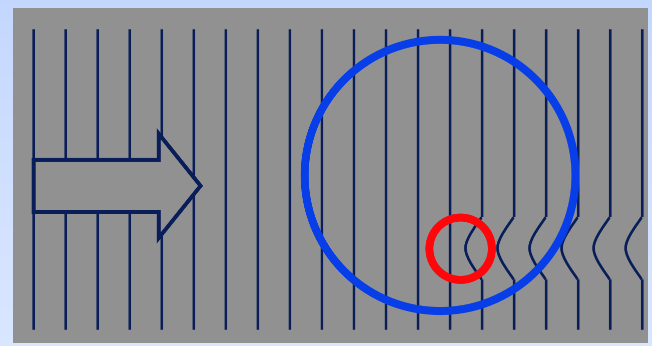
- Dream 2: **Zero Dose**

Absorption



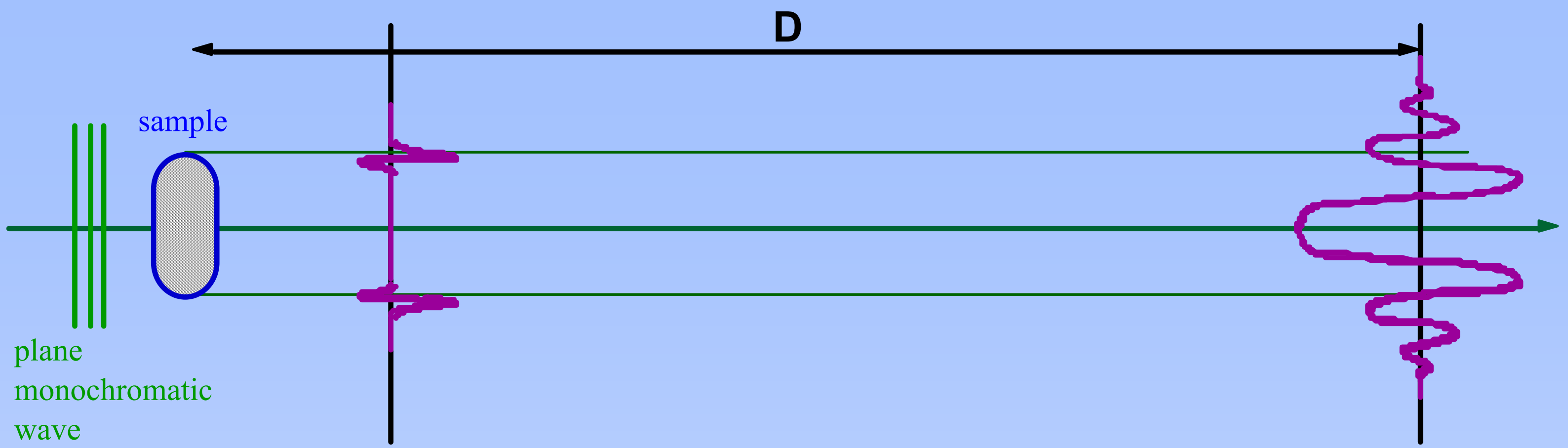
Sample

Phase

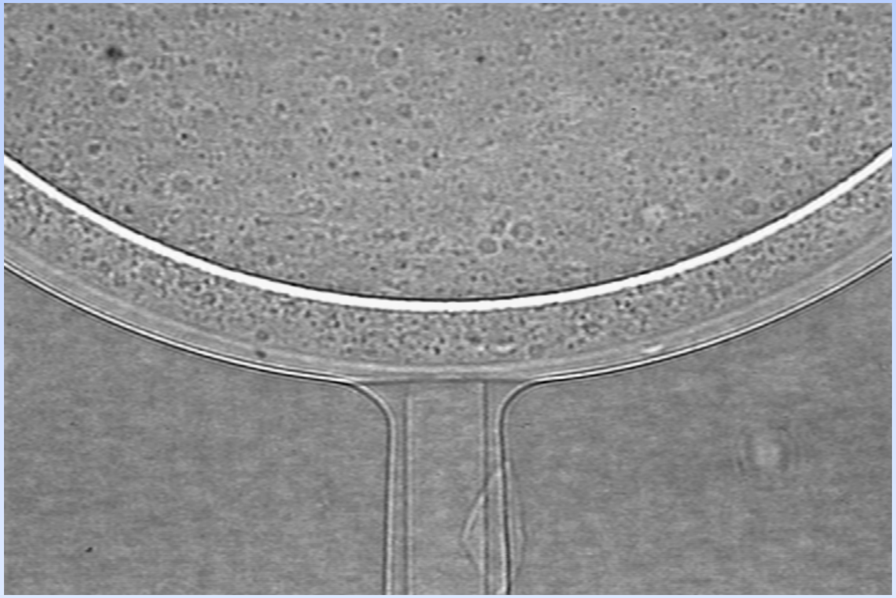


Sample

0.1% shrinkage $\Rightarrow$ 2 voxels motion (N=2048)



edge detection versus holography (Fresnel diffraction)



D = 15 cm

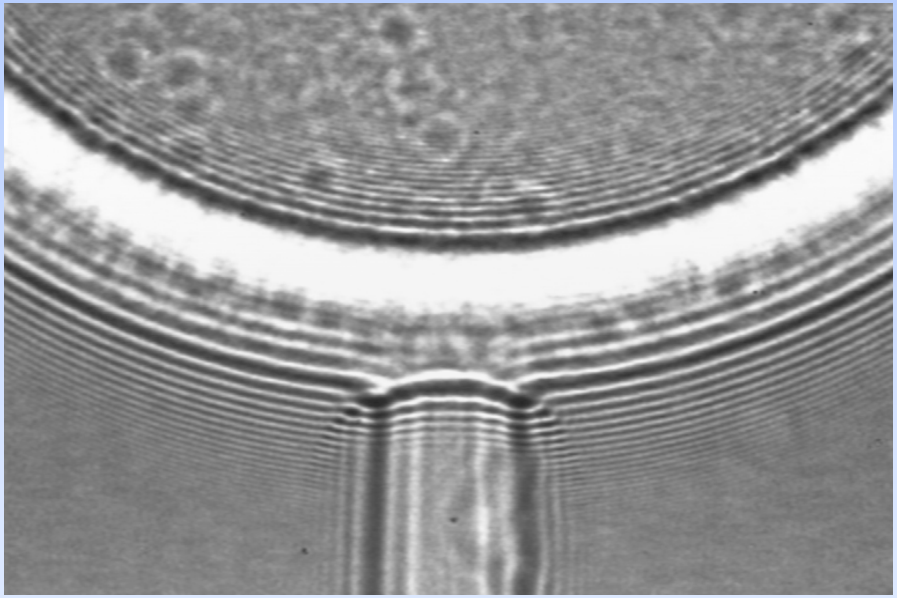
each edge imaged independently

no access to phase, only to border

$$\sqrt{\lambda D} << a$$

λ = 0.7 Å

50 µm



D = 310 cm

deformed *image* of whole object

Phase retrieval needed

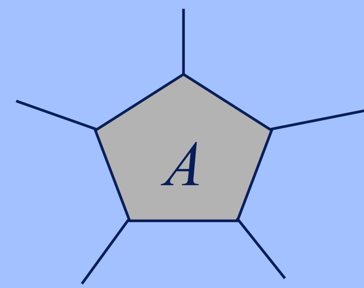
$$\sqrt{\lambda D} \approx a$$

Phase Contrast: Liquid Foams

Scientific Case:

Evolution (coarsening) of *liquid* foams in 3D

vs. in 2D



Bubble with surface A and n sides

$$\frac{dA}{dt} \propto (n-6)$$

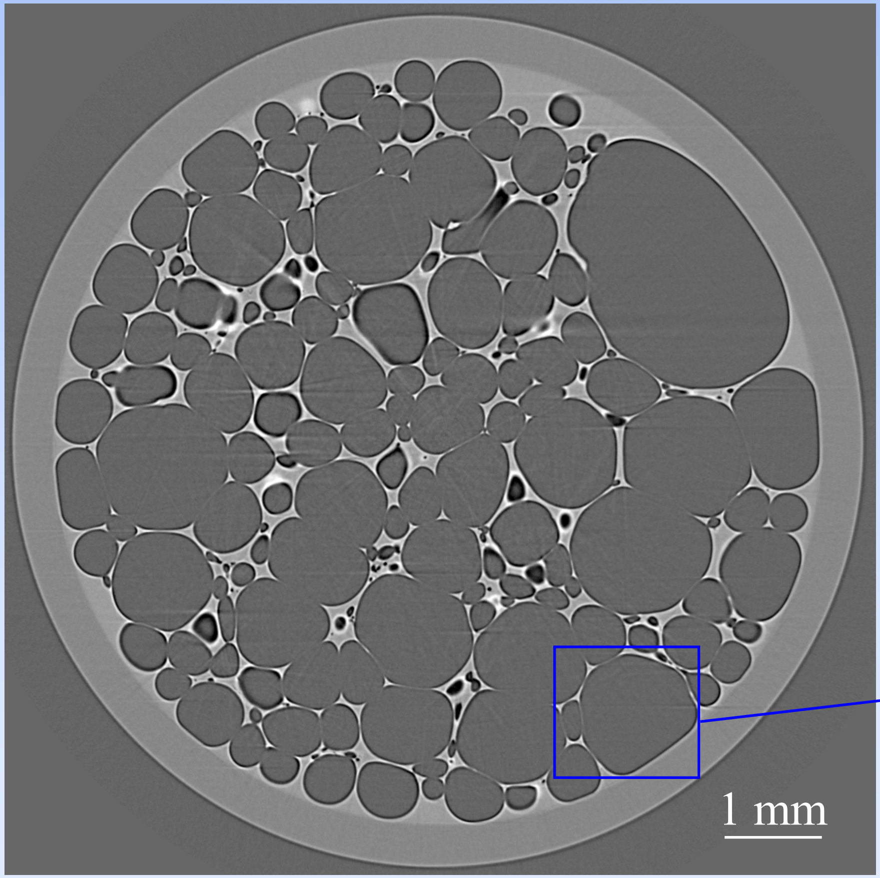
Dry foam reaches *scaling state*
average size grows with power law
normalized distributions are constant

F. Graner (UJF), J. Lambert, P. Cloetens

Phase Contrast: Liquid Foams

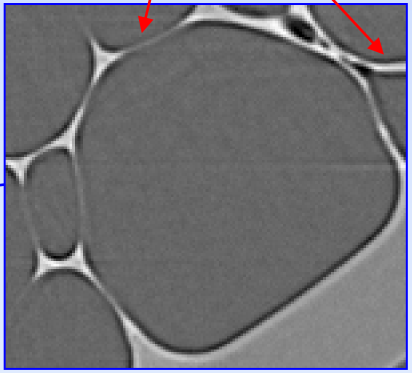
Scientific Case:

Evolution (coarsening) of liquid foams in 3D



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 (UJF), P. Cloetens

Phase Contrast: Liquid Foams

Coarsening: pressure driven growth or disappearance of bubbles

QuickTime™ and a Sorenson Video 3 decompressor are needed to see this picture.

2 minutes/scan (2GB data)

⇒ 3D Growth Law

volume individual bubbles in time

(cfr. grain growth, sintering)

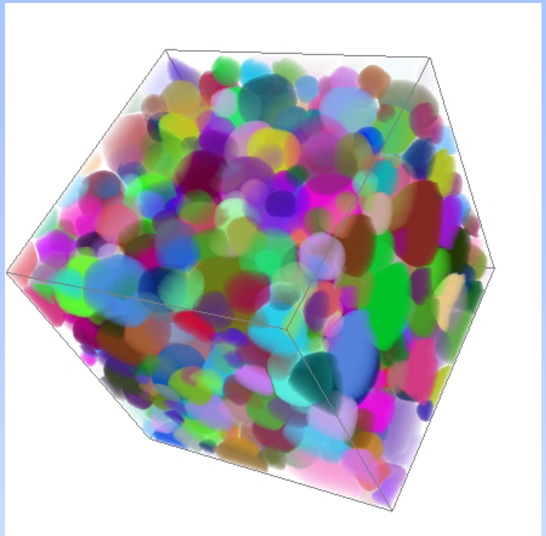
F. Graner (UJF), J. Lambert, P. Cloetens

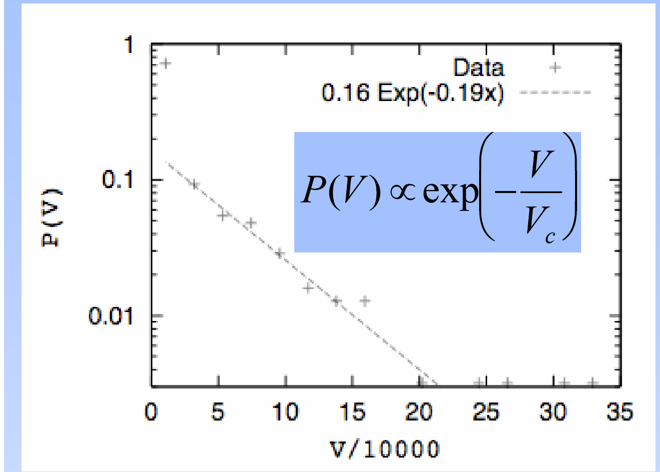
Liquid Foams

Model Foams: Dry Foam (liquid fraction → 0)

Individual growth rate

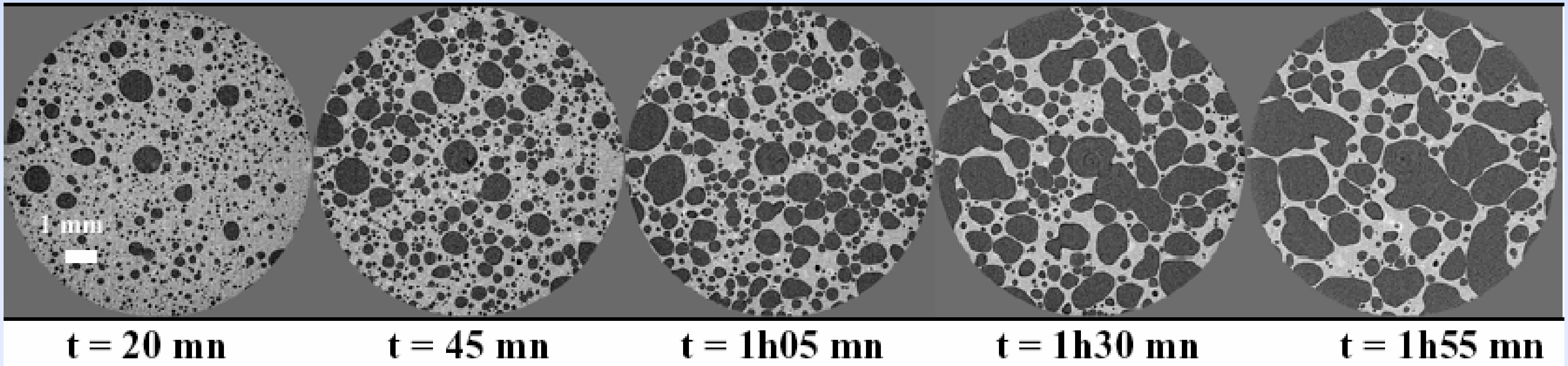
Scaling state ?





F. Graner (UJF), J. Lambert (Univ. Rennes)

Real foams: proteins, bread dough, baking

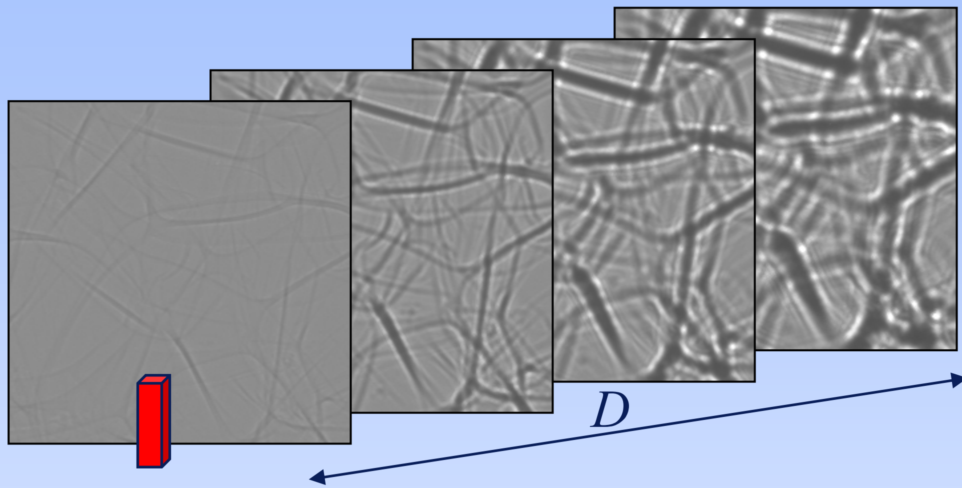


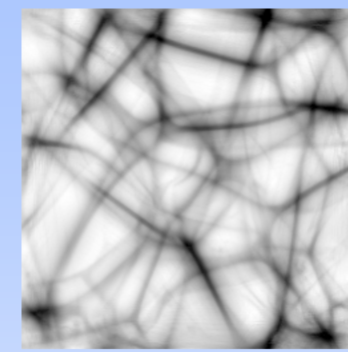
28 s / scan

P. Babin (INPG), H. Chiron (INRA, Nantes)

Holo-tomography

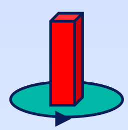
1) phase retrieval with images at different distances



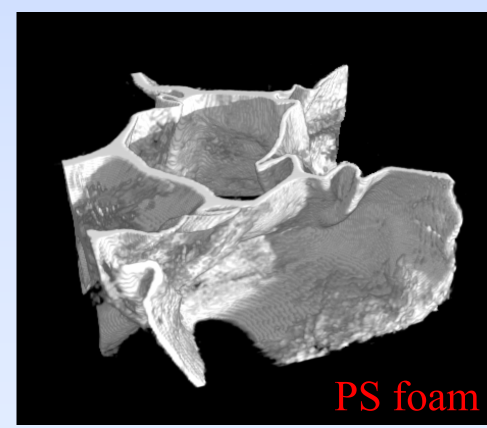


Phase map

2) tomography: repeated for ≈ 1000 angular positions



3D distribution of δ or the electron-density
improved resolution
straightforward interpretation
processing



P.Cloetens et al., Appl. Phys. Lett. 75, 2912 (1999)

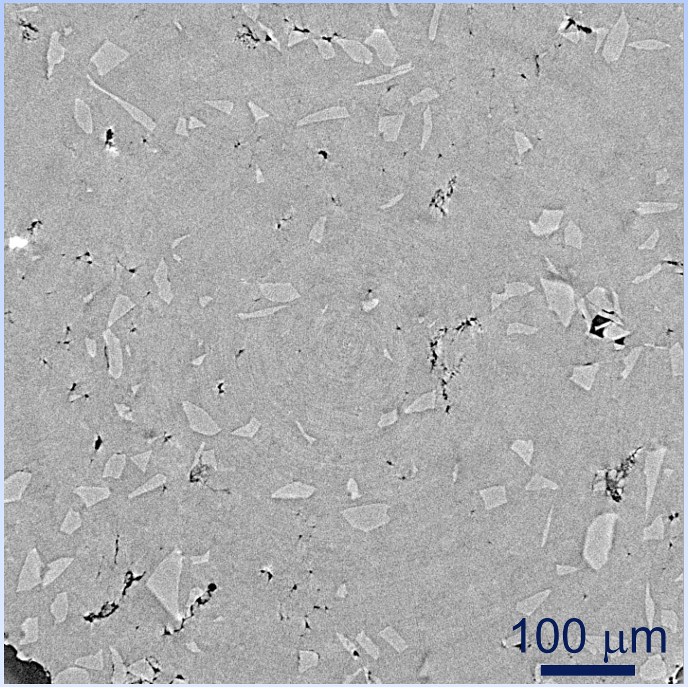
Holotomography of composite materials

Al-SiC composite material

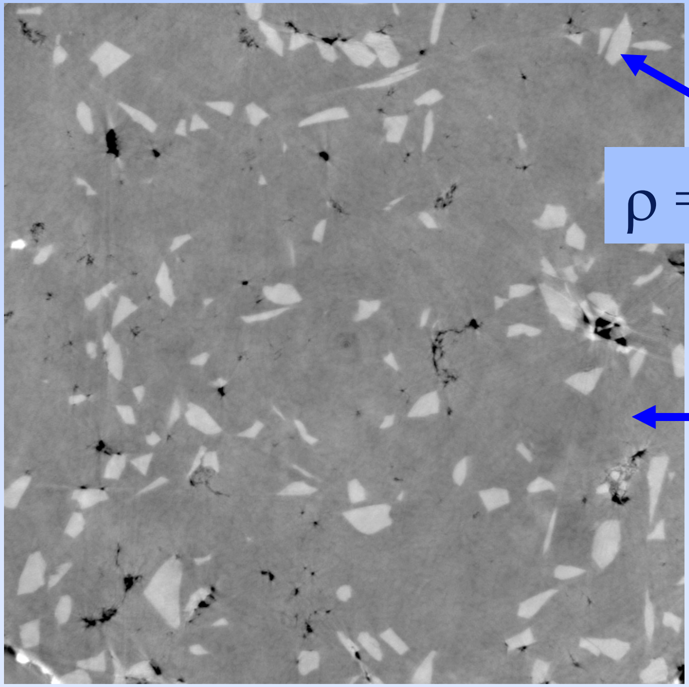
$\Delta\delta = 3 \cdot 10^{-7}$ et $\Delta\beta = 3.3 \cdot 10^{-10}$ gain ≈ 900!

E = 20.5 keV

Phase retrieval with 3 distances



≈ Absorption
(D=7mm)



Holotomography
density-map

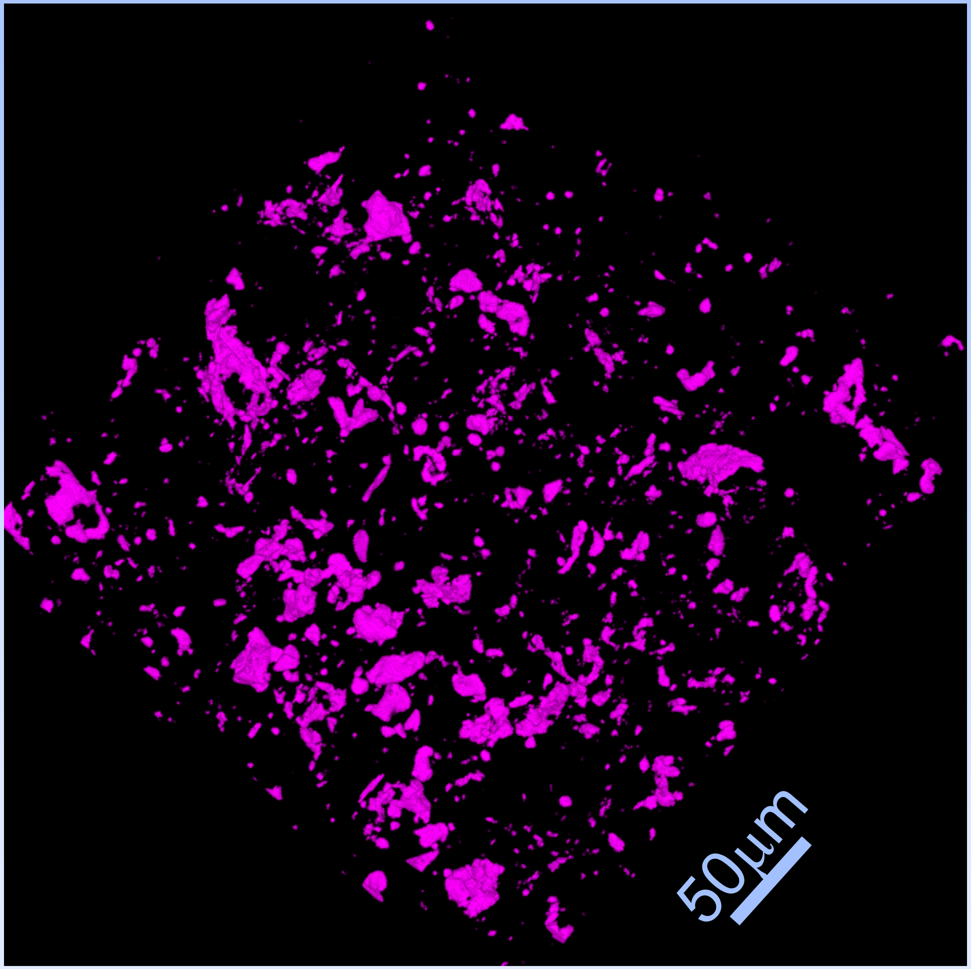
A. Velhinho, B. Fernandes, Universidade Nova de Lisboa

Composite materials

Al-SiC composite material

E = 20.5 keV
3 distances

SiC particles



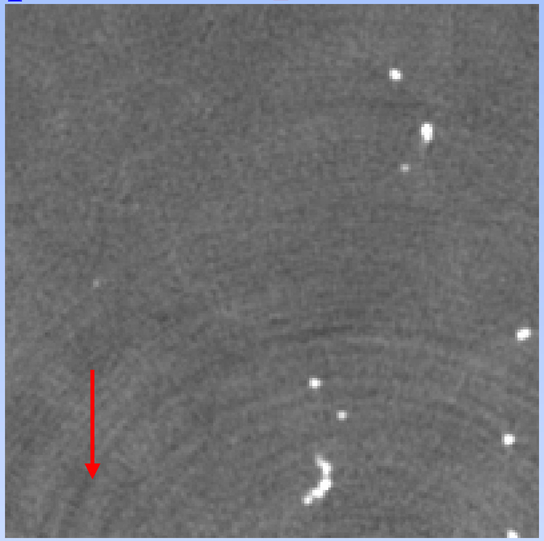
holes

A. Velhinho, B. Fernandes, Universidade Nova de Lisboa

Density Resolution

Semi-solid
Al / Al+Si

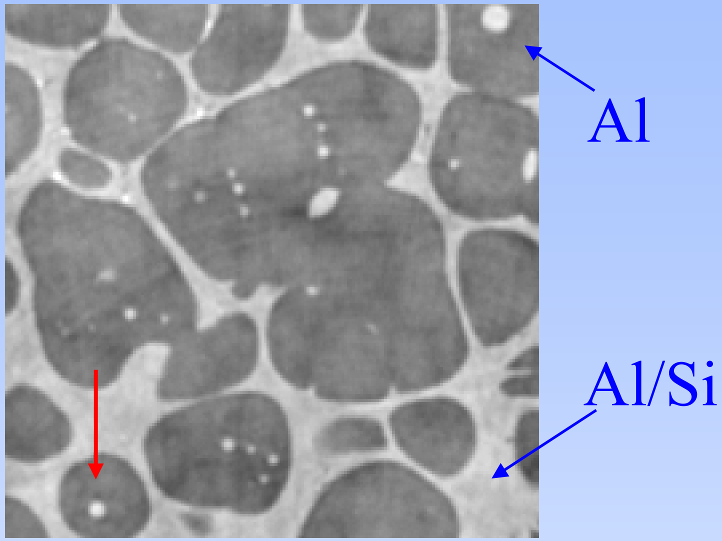
Absorption



β-map

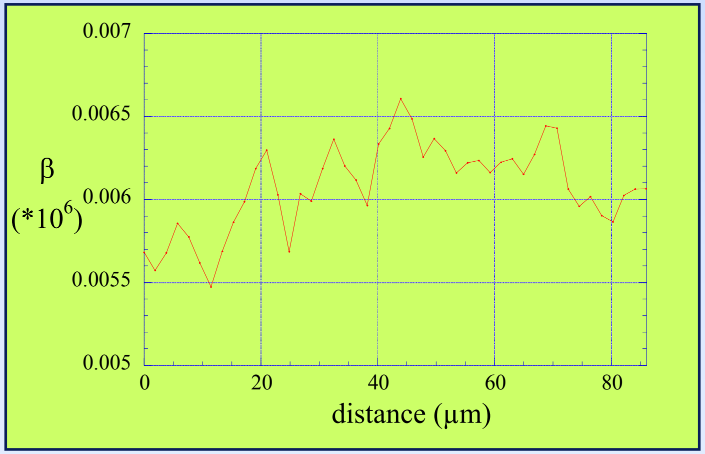
E = 18 keV

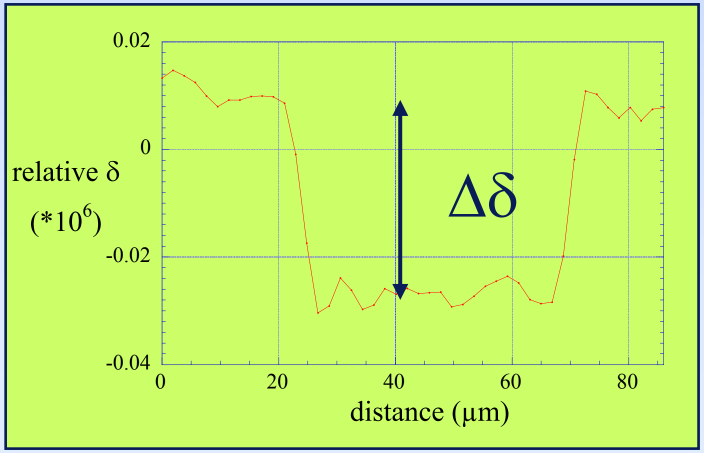
Phase contrast



50 μm

δ-map



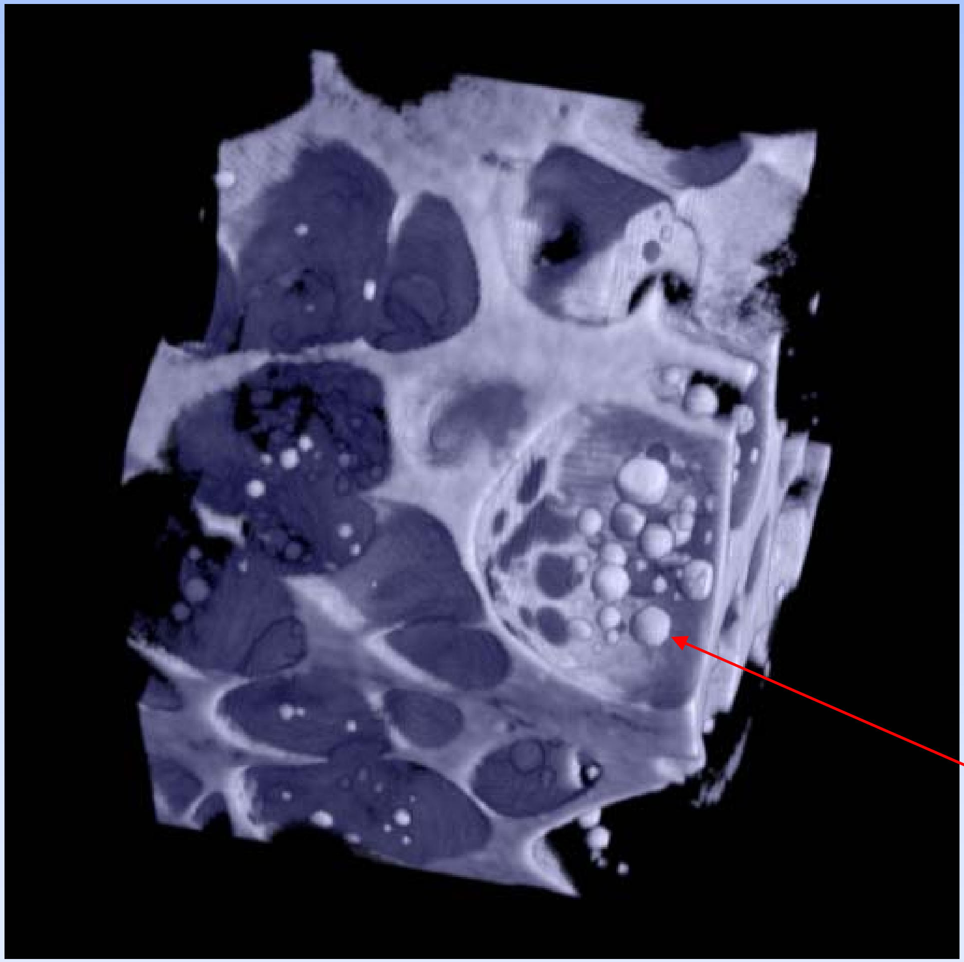


$\Delta\delta \approx 3.5\ 10^{-8} \Rightarrow \Delta\rho \approx 0.05\ g/cm^3$ 15 % of Si

L. Salvo (GPM2, Grenoble)

Holotomography of *semi-solids*

Volume Rendering (solid transparent)



Connectivity:

Liquid phase:

total

\+ trapped liquid

Solid phase:

very strong

trapped liquid

→ input for permeability calculations

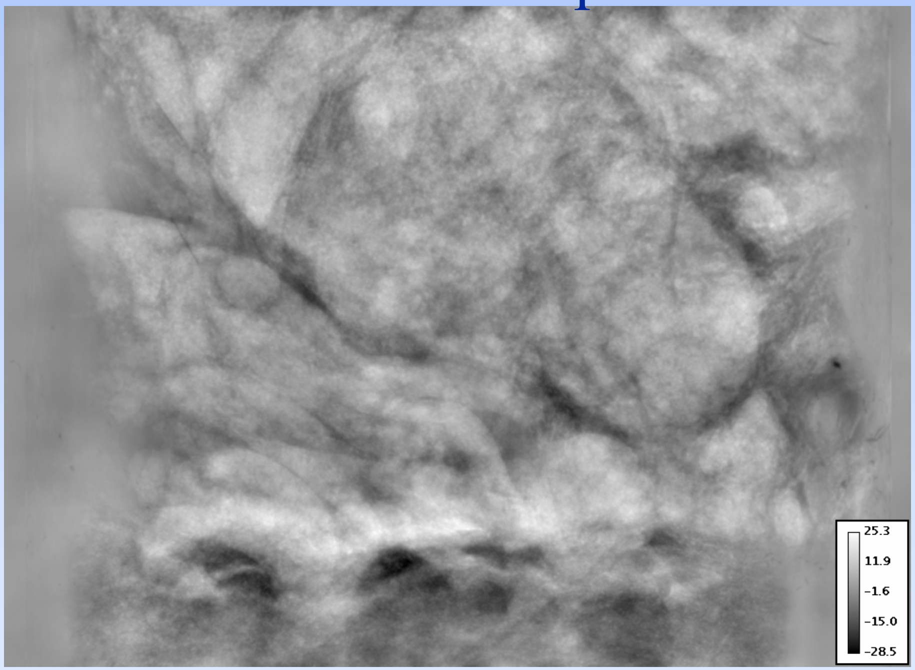
Larger field of view

Breast Biopsy $\text{Ø} \approx 15$ mm ; pixel size = 7.5 µm

⇒ Huge distances: 0.03,1, 4.3 and 8.8 m !

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

Phase Map



rad

13 mm

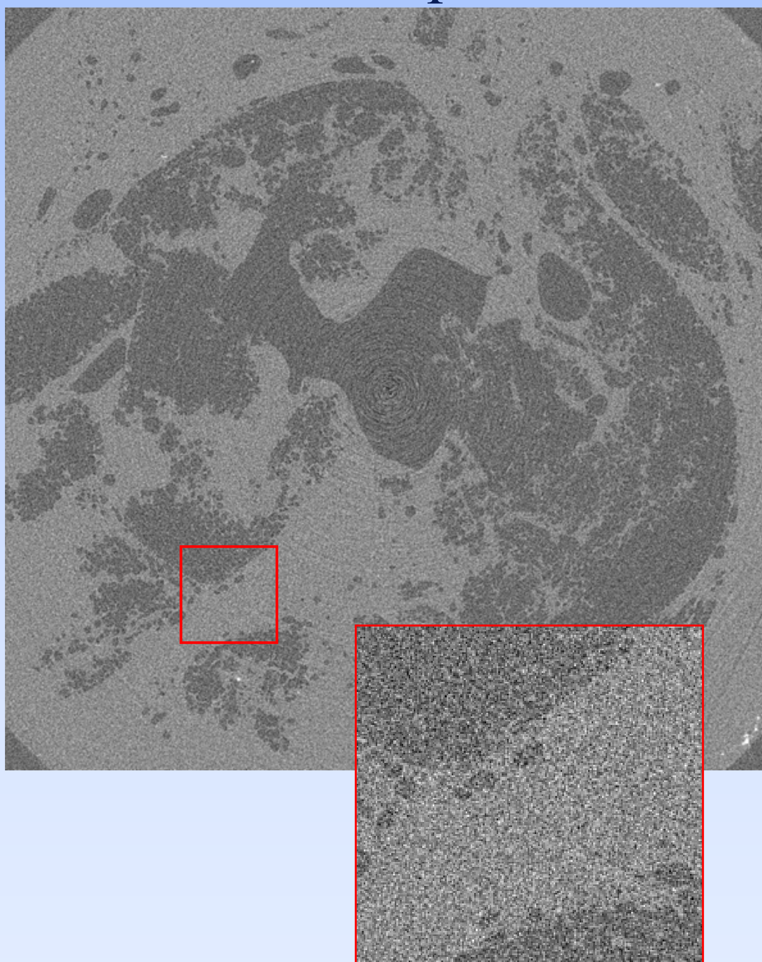
Energy = 25 keV

E. Pagot, K. Fezzaa

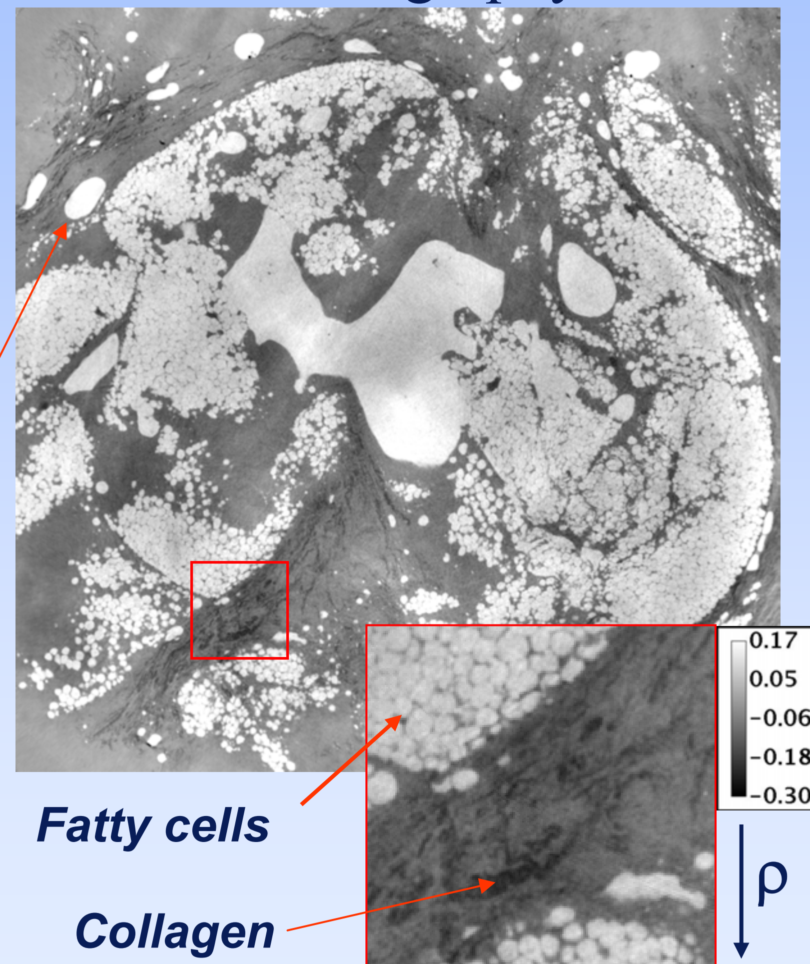
Larger field of view

Breast Biopsy Ø ≈ 15 mm ; pixel size = 7.5 µm

Absorption



Holotomography



P. Cloetens, E. Pagot

Sub-micron Tomography

- Improve the spatial resolution

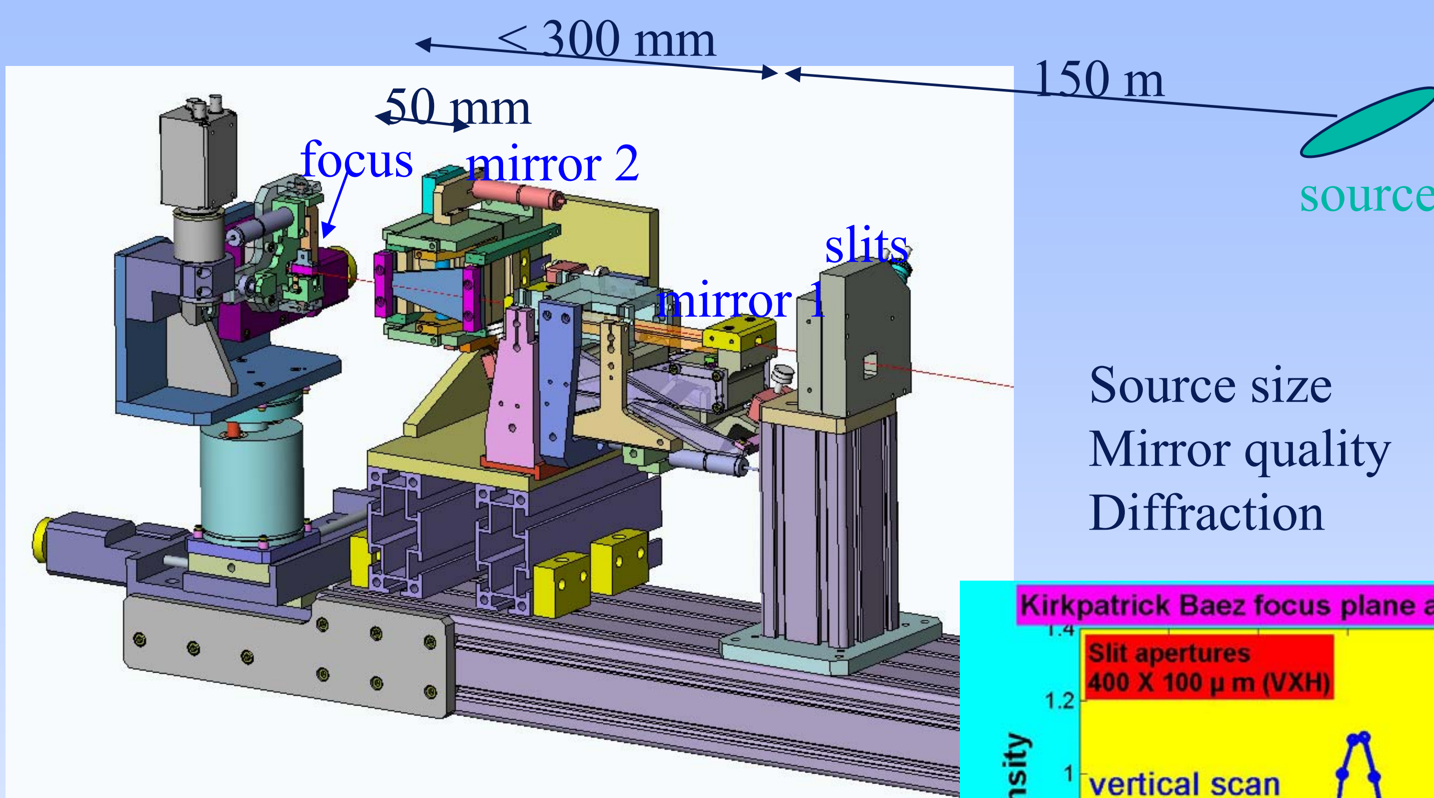
$\lambda < 1$ Å

resolution ≈ 0.5 μm
with scintillator based detectors
parallel beam

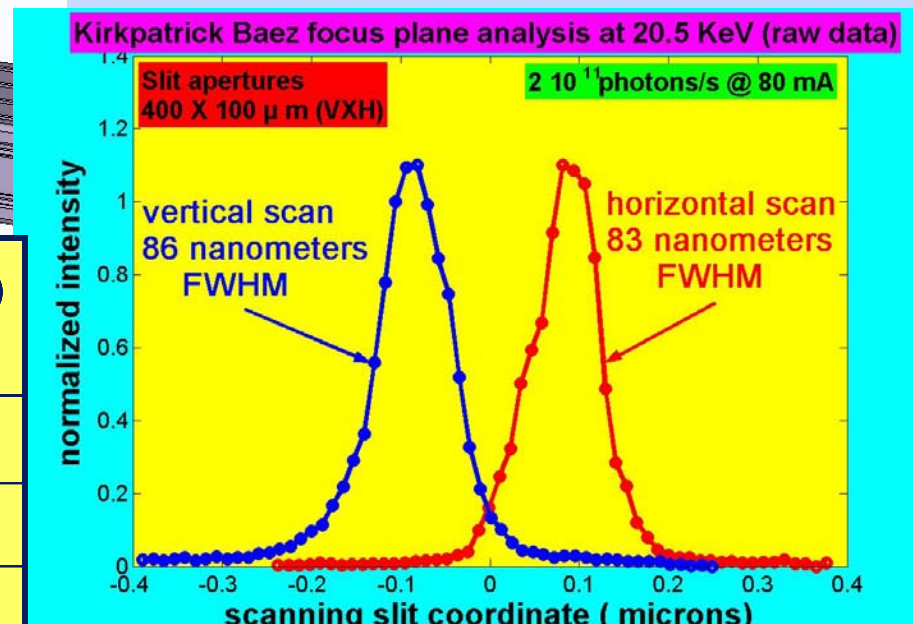
Gabor's Microscopic principle (Nature, 1948)

The object is illuminated by an electron beam brought to a fine focus… The object is a small distance behind (or in front) of the point focus, followed by a photographic plate at a large multiple of this distance…

KB focusing at 20.5 keV



Source size
Mirror quality
Diffraction



Aperture V*H (μm)	Spot fwhm V*H (nm)	Flux ($\Delta E/E=10^{-2}$) ph/s @ 80 mA
200 x 50	118 x 109	$5\ 10^{10}$
400 x 100	86 x 83	$2\ 10^{11}$
600 x 160	116 x 90	$4.5\ 10^{11}$

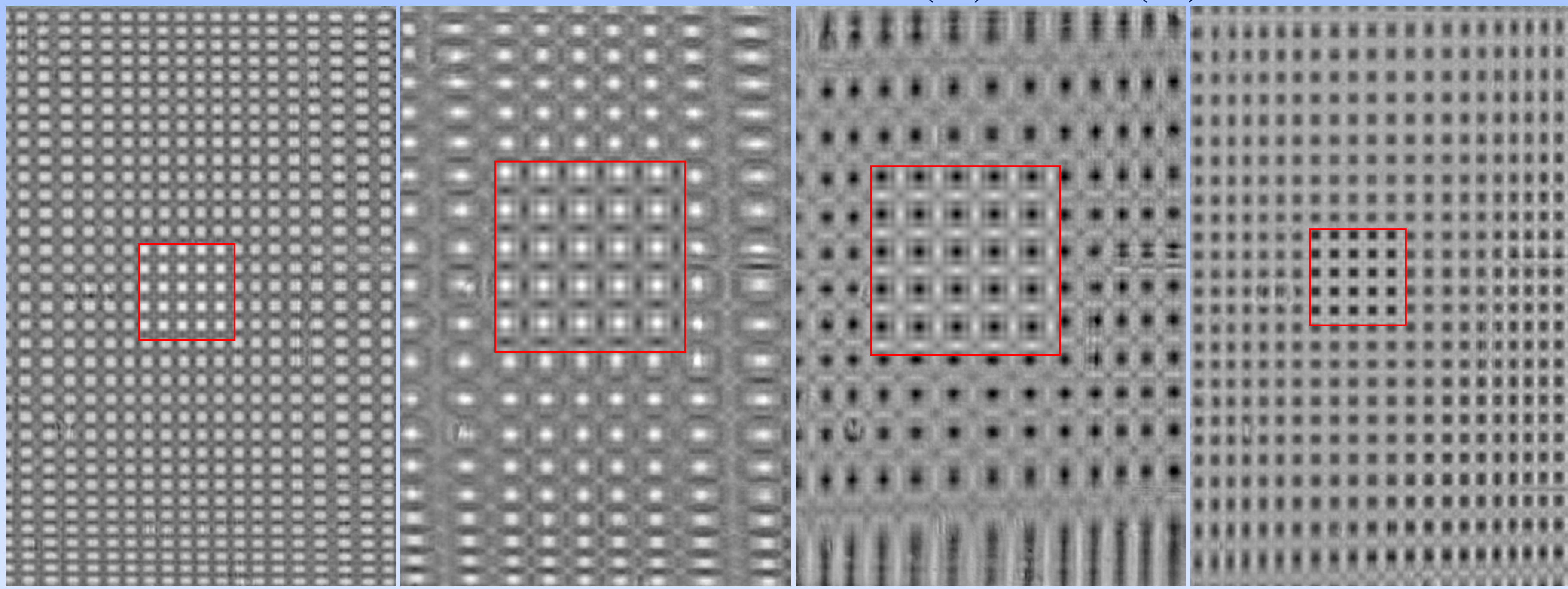
O. Hignette, P. Cloetens

Projection Microscopy

2 μm pitch grating (Si)
C. David, PSI

10 μm

Mirrors fully illuminated
0.2 (H) x 1.1 (V) mm, E=20.5 keV

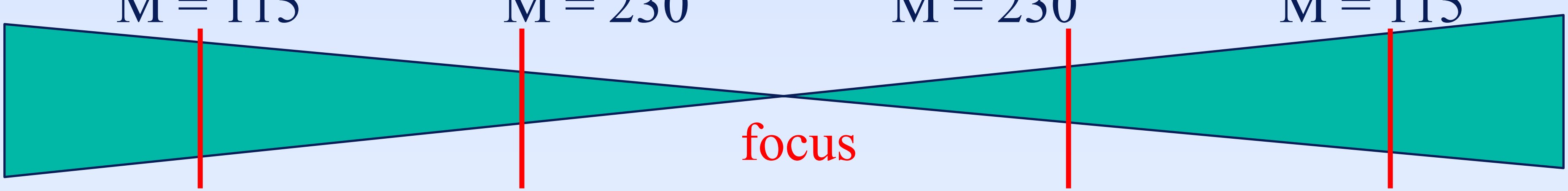


D = -20 mm
M = 115

D = -10 mm
M = 230

D = 10 mm
M = 230

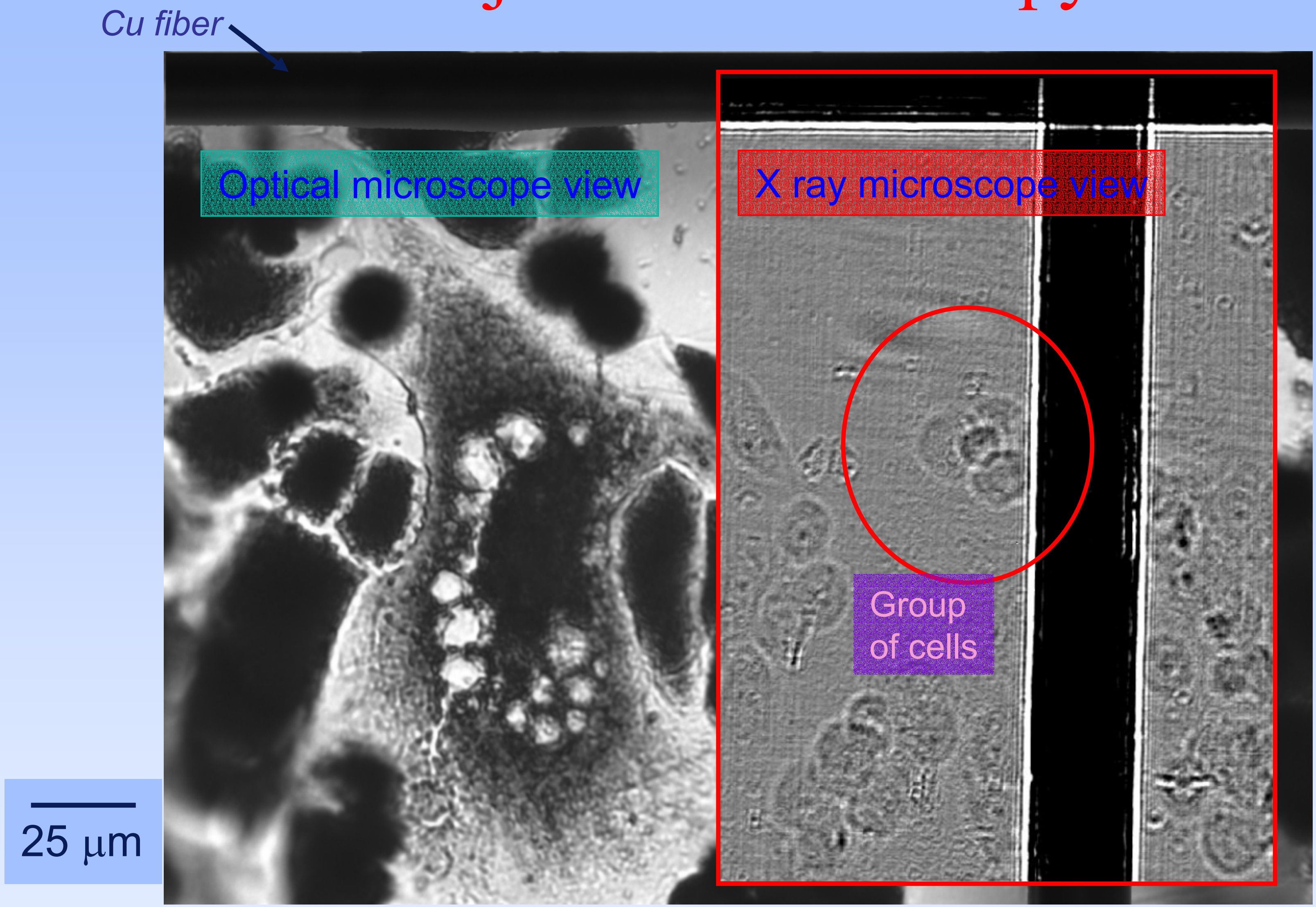
D = 20 mm
M = 115



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

P. Cloetens, O. Hignette

Projection Microscopy



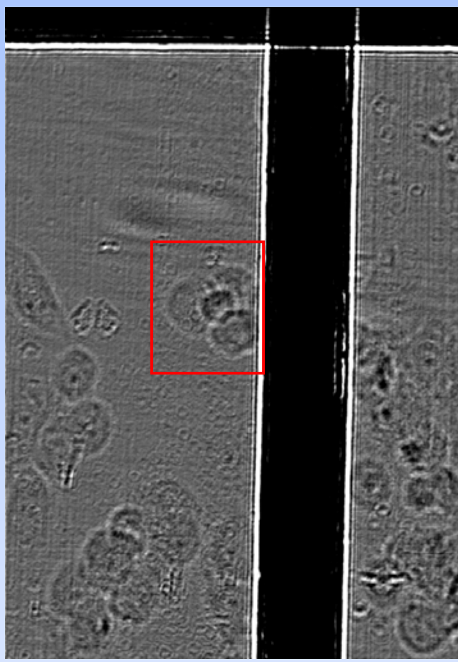
Cellular imaging

Cancerous cells
S. Bohic

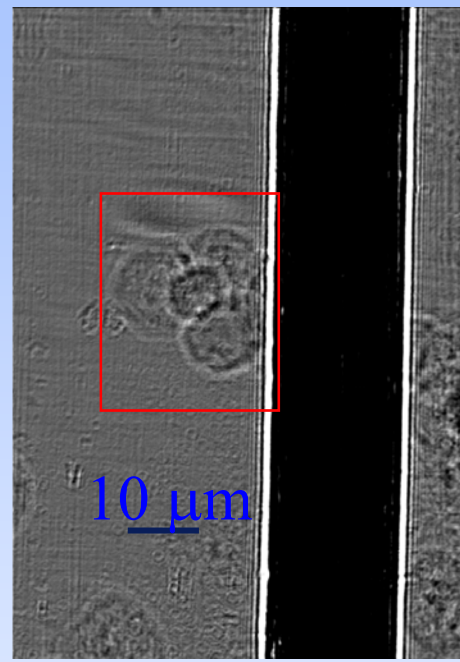
Projection Microscopy

Cancerous cells
S. Bohic

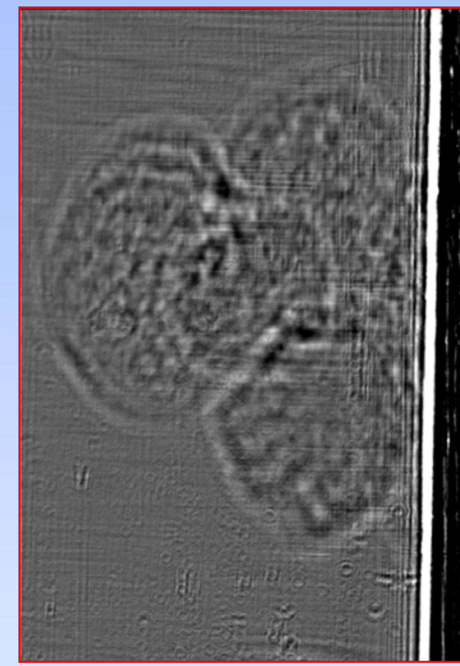
Towards focus



D = 50 mm
M = 45



D = 30 mm
M = 75



D = 10 mm
M = 230

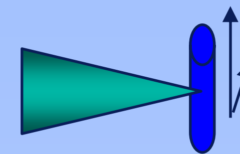
E = 20.5 keV
Exposure time = 0.3 s ! (16-bunch)

No X-ray optics behind the sample $\Rightarrow$ dose efficient

P. Cloetens, W. Ludwig

Applications: Fluorescence

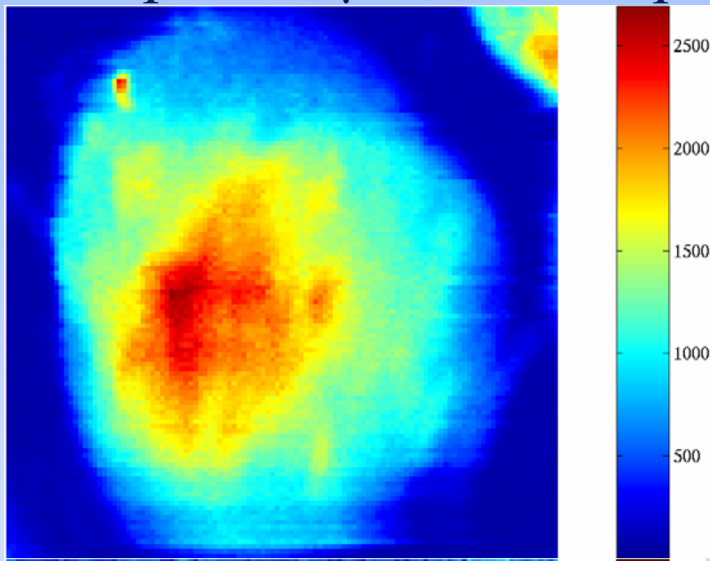
Cancerous cells treated with anti-cancer drug (Cisplatin)

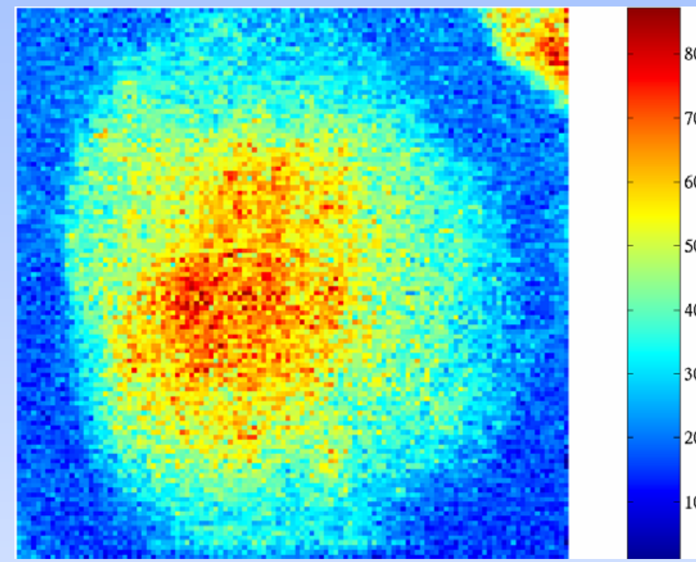


Step = 0.3 μm, 0.9 sec/pt

40 μm

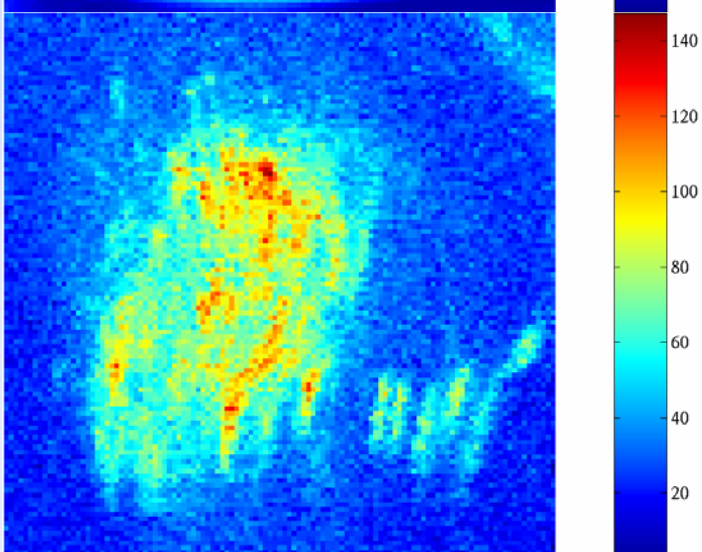
K

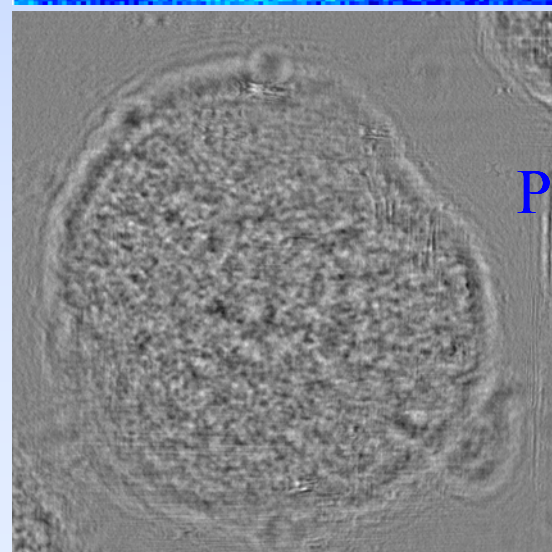




Pt

Fe

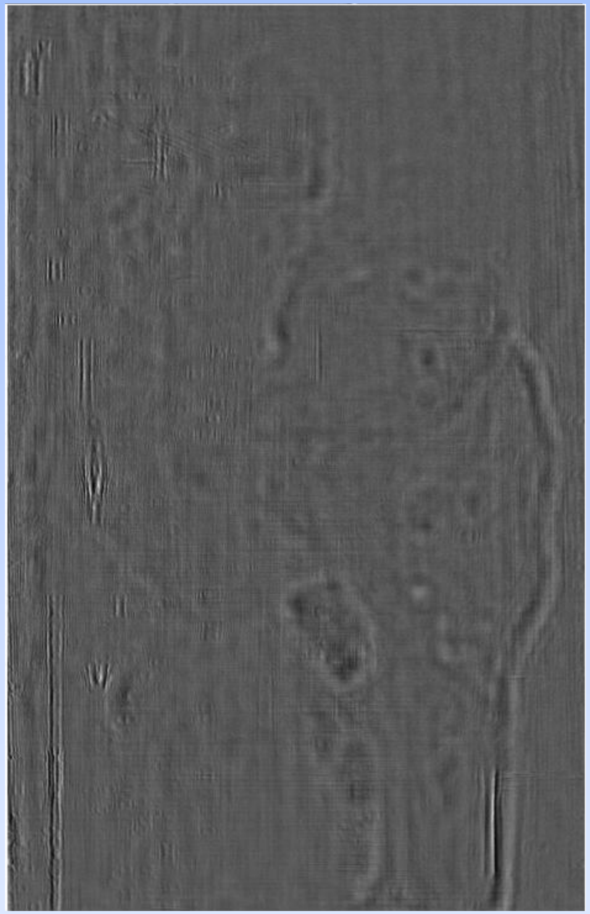




Phase Contrast

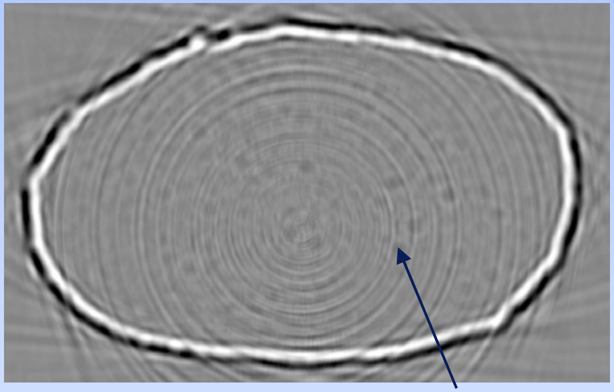
P. Cloetens,
W. Ludwig,
S. Bohic

Projection Microscopy: 3D



Human hair: 80 μm diam
Distances:
270, 120, 50 mm from source

Tomographic slice



Localised structures
porosity

D ~ 60 mm   M=70

E = 20.5 keV
Exposure time = 1 s (16-bunch)

Improvement X-ray optics for coherent imaging

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Nano-Tomography Project

Motivation:

Materials Science: relevant scale 0.1-10 µm

Nano-technology/fabrication: 50 nm scale and below

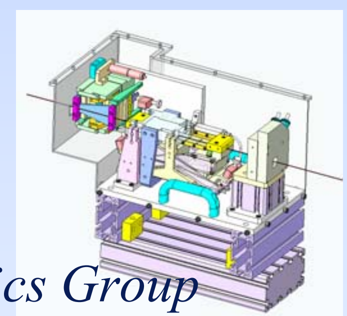
Soft condensed matter

Goal: *routine* 3D microscopy with < *50 nm* spatial resolution with *in-situ* and *micro-analysis possibilities* (7 - 15 keV)

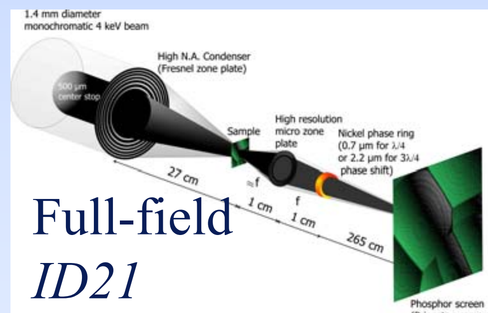
Strategy:

Multiple-mode 3D microscope

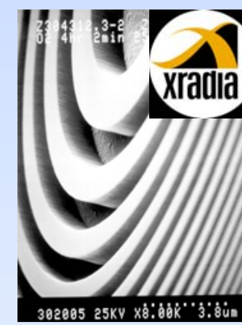
Optics



KB
Optics Group



Full-field
ID21



Coherent / Incoherent Illumination
Full-field / Mapping

Conclusions

- Real space imaging often required
- Tomography more powerful than ever: absorption, phase contrast, holotomography, fluo-tomography
- *Quantitative* mapping in 2D and 3D
- *In-situ* experiments
- Multidisciplinary approach

- Improve the spatial resolution

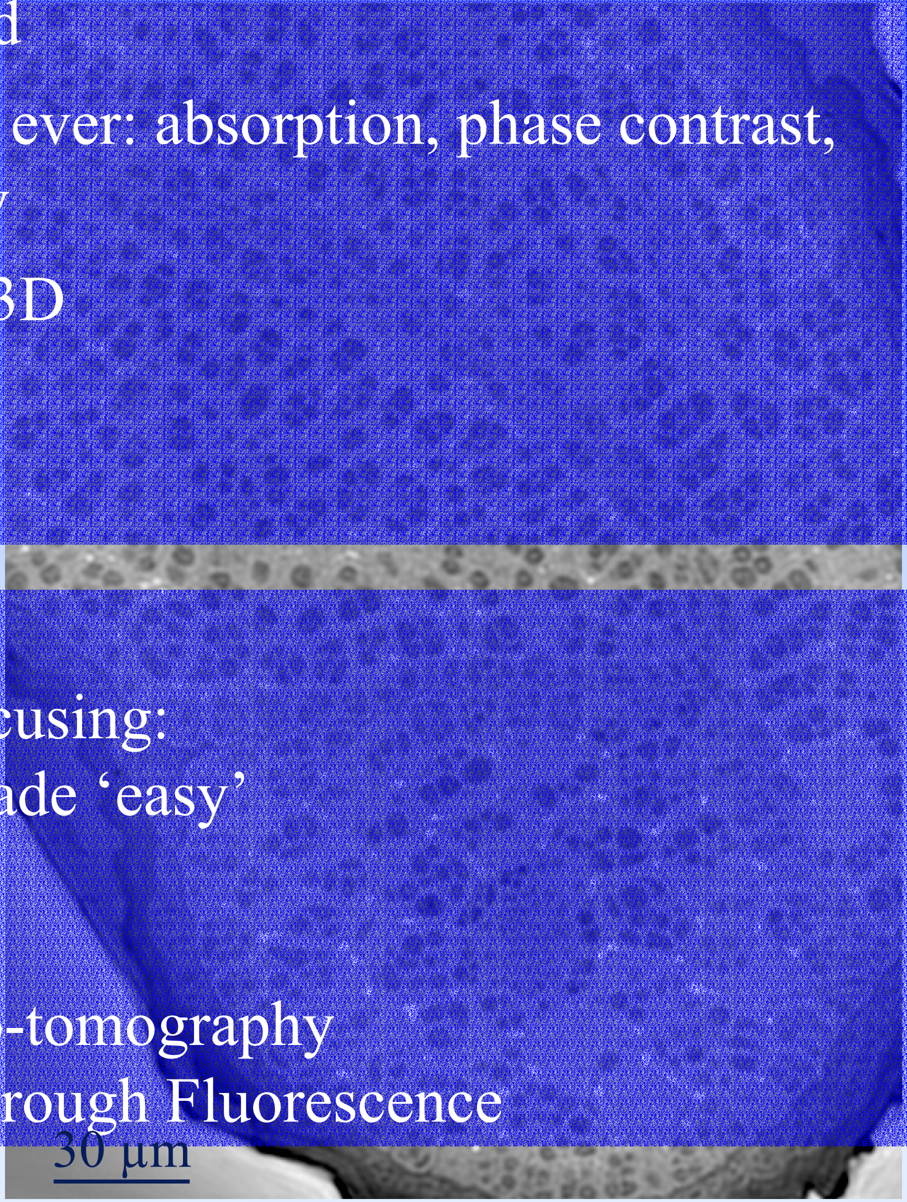
  Projection microscopy by KB focusing:

  Sub $(100\ nm)^2$ focusing made 'easy'

  High flux (10^{12} ph/s)

Imaging: Micro-structure via nano-tomography

Nanoprobe: Element specificity through Fluorescence



Seed of Arabidopsis

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  - Breast imaging