Synchrotron Imaging Techniques

Applications in materials science

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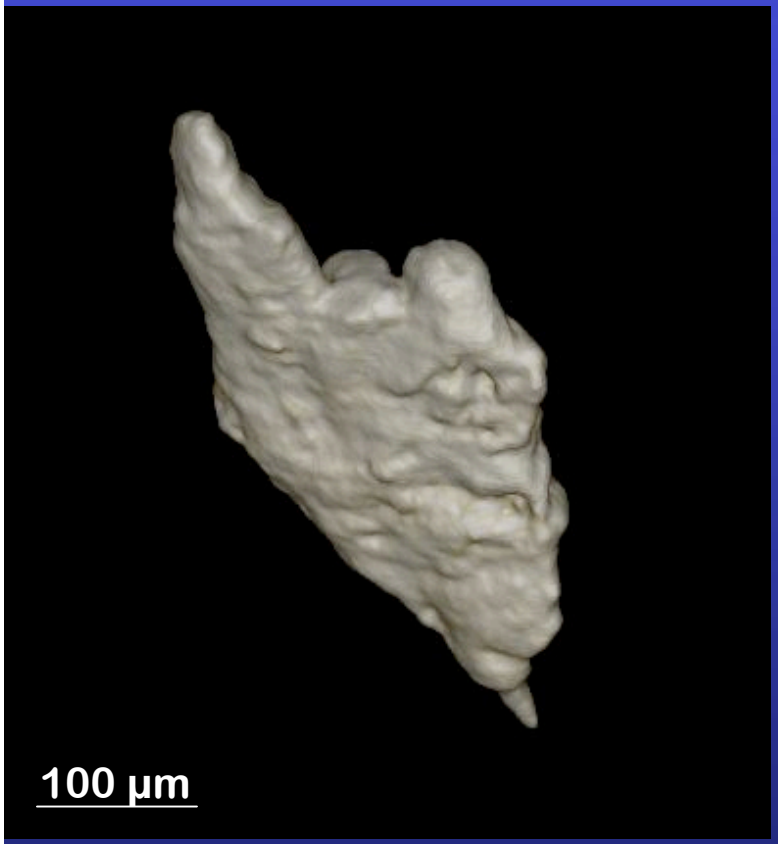
2 Mateis, INSA de Lyon, France



Topotomographic reconstruction
of Al grain during recrystallization

Outline:

- Tomography endstations
- Sample environment
- Combination of techniques
- Fast tomography
- Diffraction contrast tomography

Available setups / specifications

ID19 : - high resolution 0.28 …30 µm pixelsize
- coherence (145 m)
- diffraction contrast tomography

ID15 : - high energy
- fast tomography (few seconds)
- simultaneous diffraction (strain measurement)

ID17 : - extra large field of view

BM5 : - medium resolution (5-10 µm)

ID22 : - fluorescence tomography
- diffraction tomography

ID22NI - projection microscopy
- sub-micron resolution

Experimental setup

Example: ID19 (ESRF)

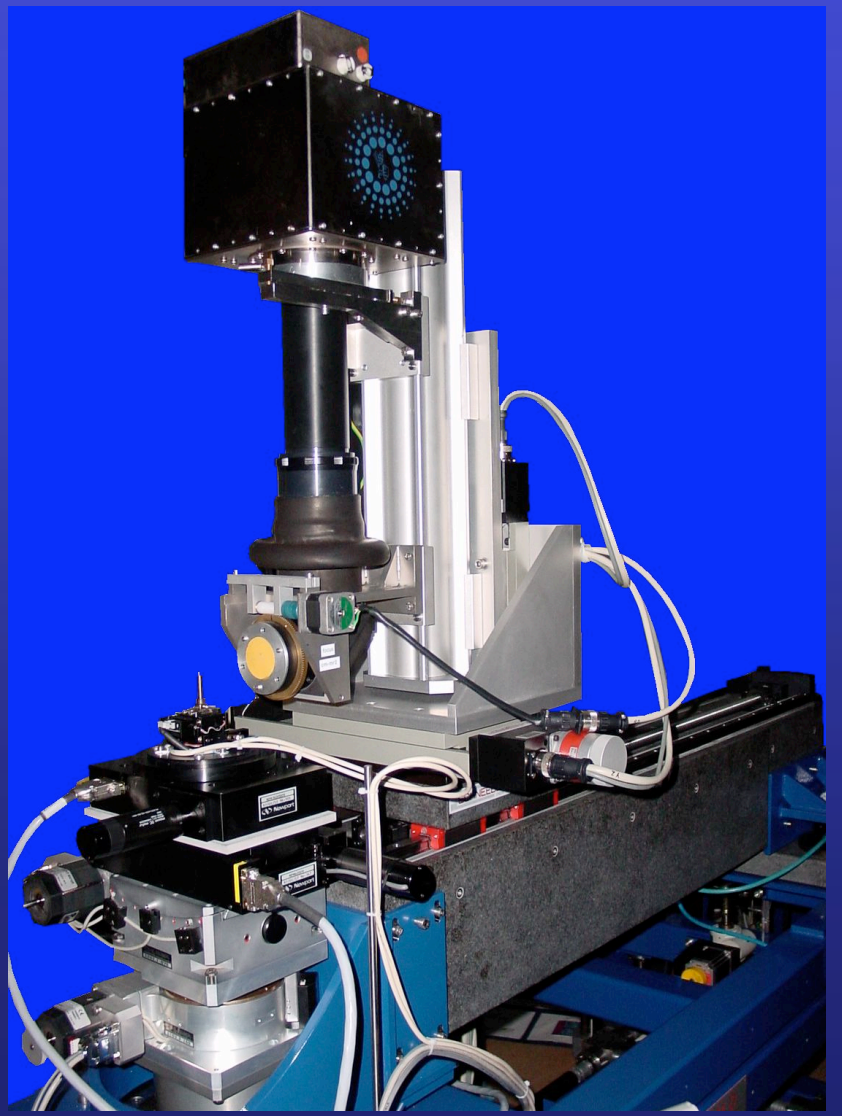
Long distance (145 m): coherence

Multilayer monochromator: $\Delta\lambda/\lambda=10^{-2}$

High resolution detector system: 1 µm,
14 bit, 20482 CCD, 60 ms readout

Dedicated µ-tomography set-up

Sample environment: fatigue machine,
cold cell, furnace, …



Absorption vs. Phase

Weak interaction with matter

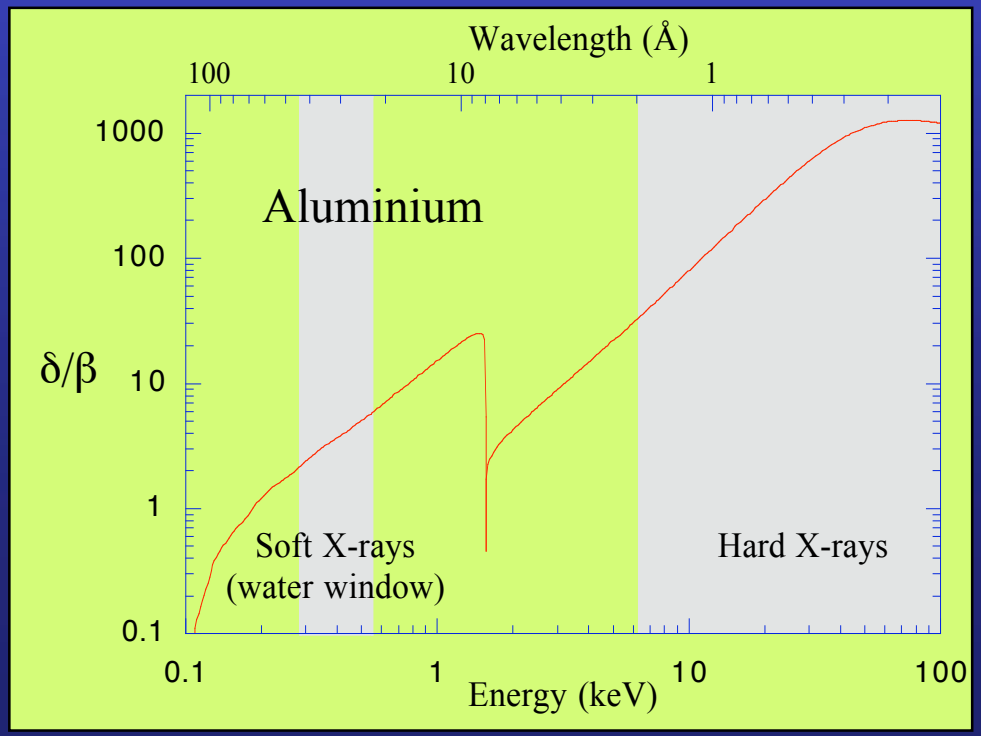
Refractive index n

$n \approx 0.999999$

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

$\delta >> \beta$

10^{-6} 10^{-9}



δ $\propto$ electron density $\Leftrightarrow$ phase

β $= (\lambda / 4\pi).\mu$ $\Leftrightarrow$ amplitude

Smallest detectable hole at 25 keV in an up to 4 mm thick sample:

Absorption: 20 µm

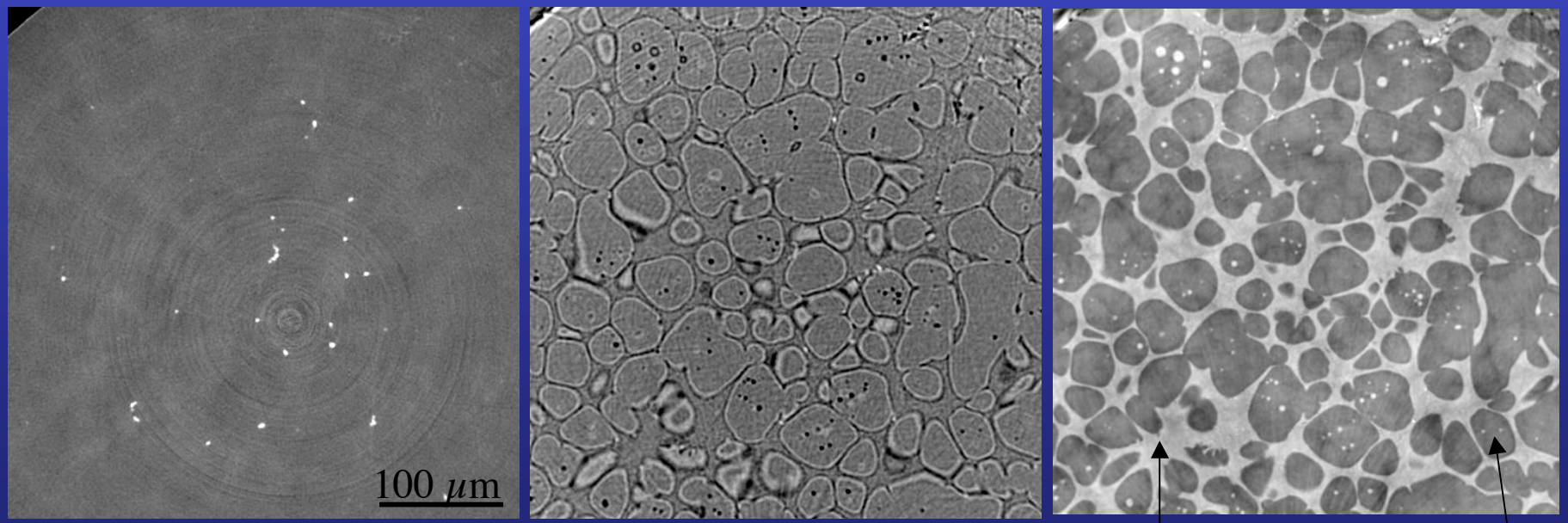
Phase: 0.05 µm

Holotomography of semisolid Al/Si alloy

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

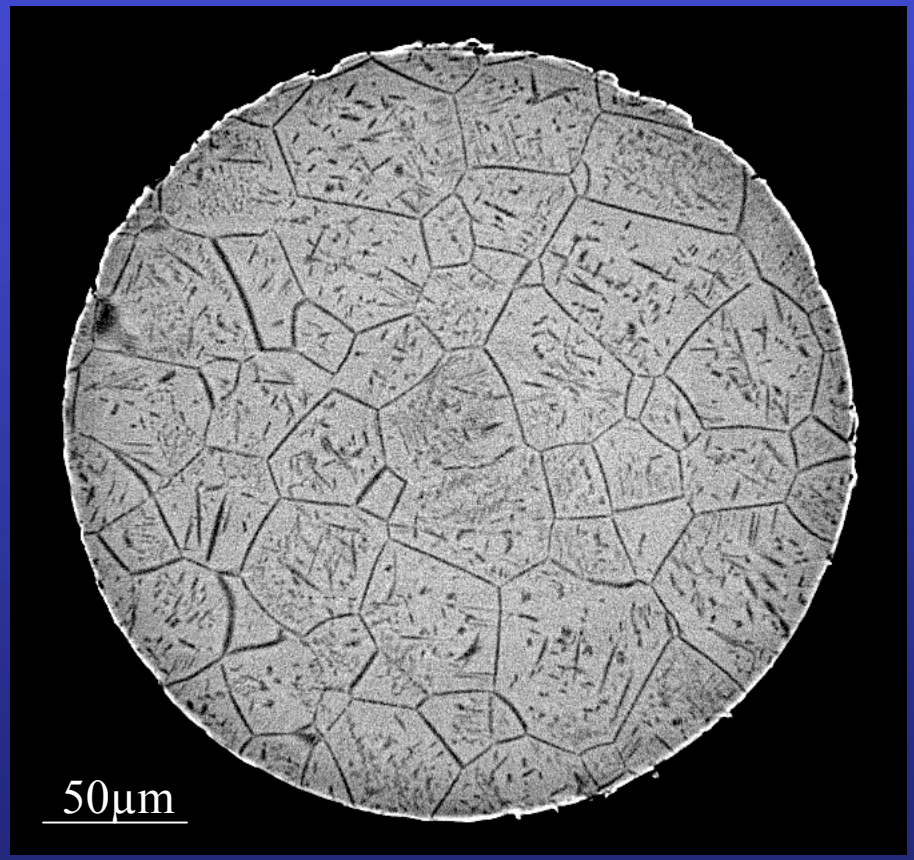
E = 18 keV

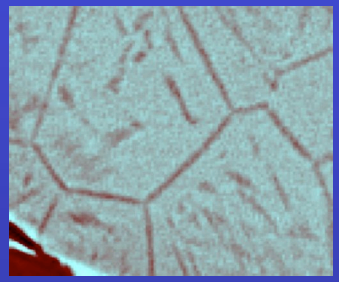
Absorption | Phase sensitive | Holotomography



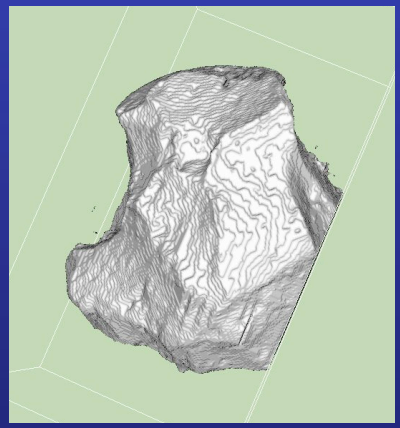
β-map | edge enhancement | δ-map

3D microstructure of Ti β21S alloy





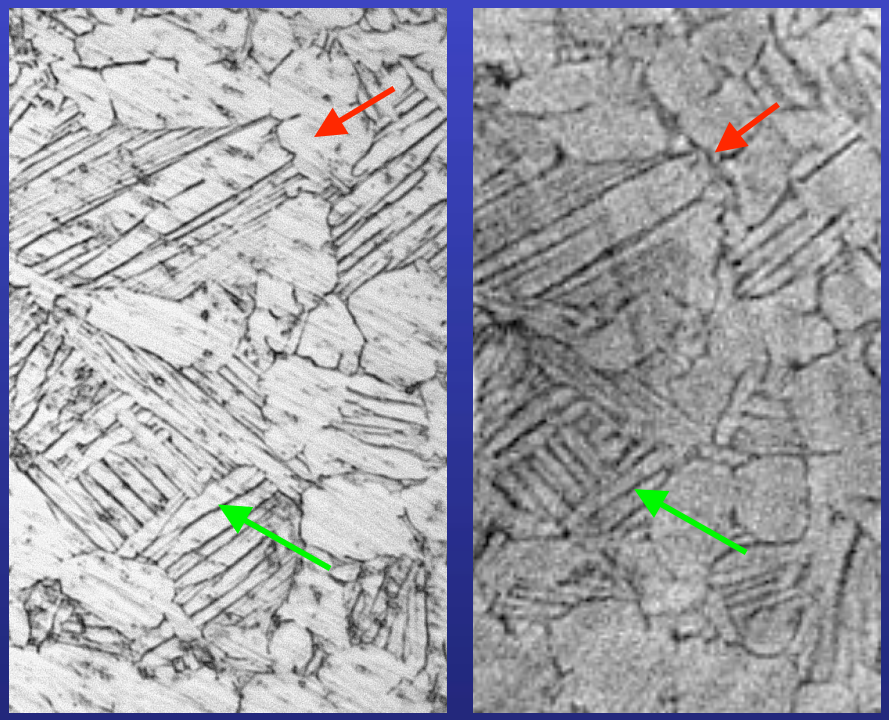
30μm



E.M. Lauridsen, R. Fonda, W. Ludwig et al.

Comparison: visible vs. X-ray

Ti-6Al-4V



Optical microscopy
2D

μ-tomography
3D !

Today:

Measurement of displacement fields in 2D

(e.g. Roux, Hilde, et al.)

Tomorrow:

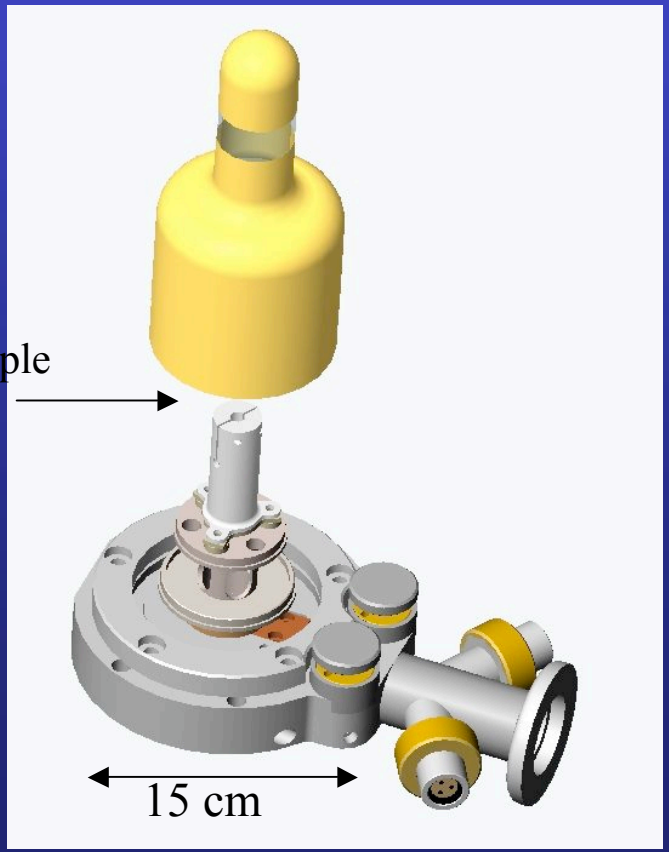
Measurement of displacement fields in 3D

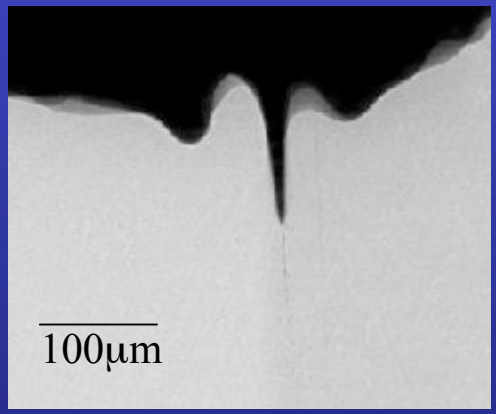
Sample environment

- Furnace(s) (ESRF)
- Cold cell (ESRF)
- Stress rig (INSA -Lyon)
- in-situ Fatigue machine (INSA-Lyon)
- hydraulic INSTRON stress rig (ESRF, ex-situ)
- … your initiative !

Furnace with 360° visibility (quartz cell)
Up to 1000 °C under vacuum

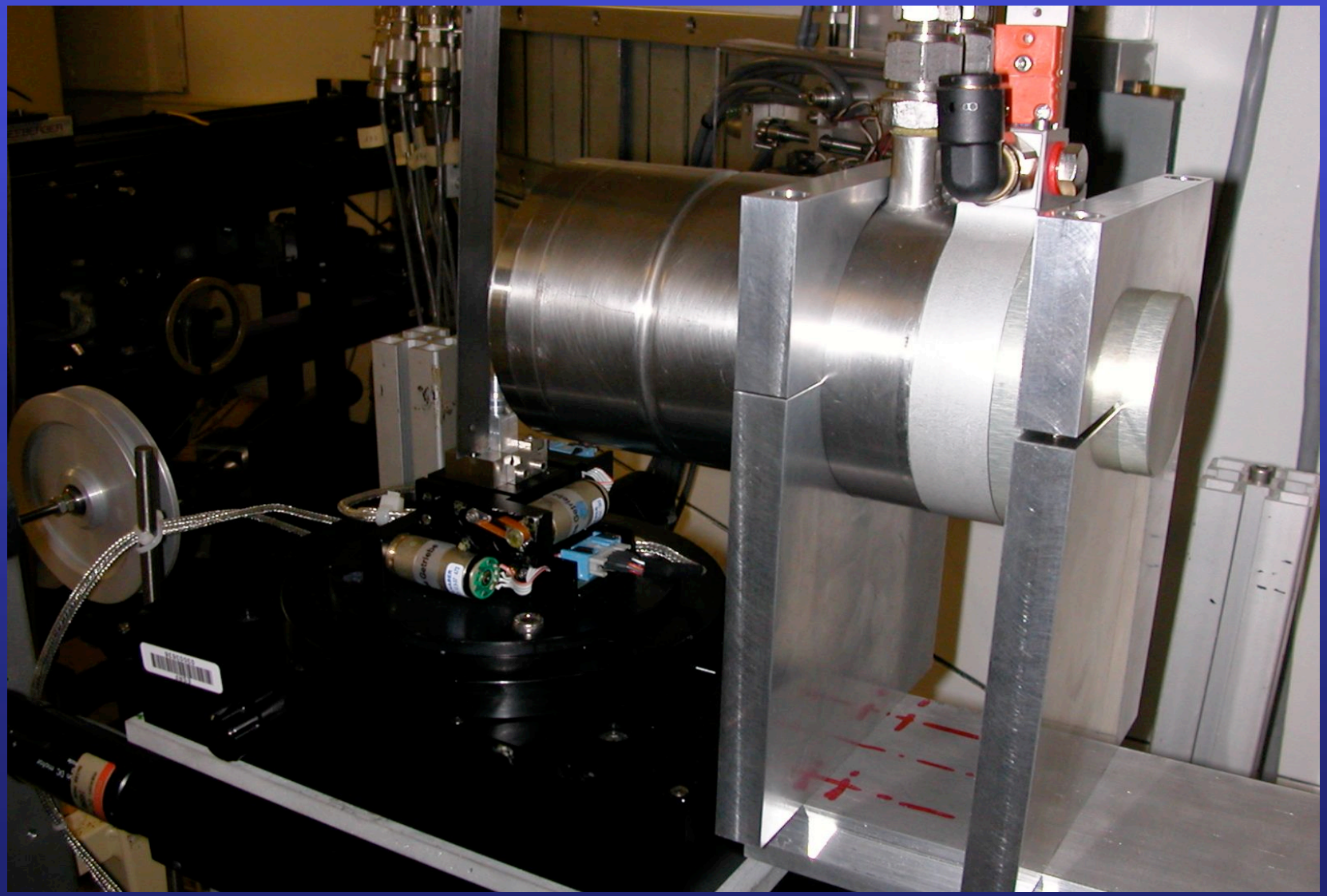
+ vacuum or controlled atmosphere
camera can be set at 20 mm from the sample





X-ray microradiograph
(ID19, 18 keV)
of Al bicrystal after 1h
in contact with Sn melt at 620°C

Gas-blower furnace (ESRF)



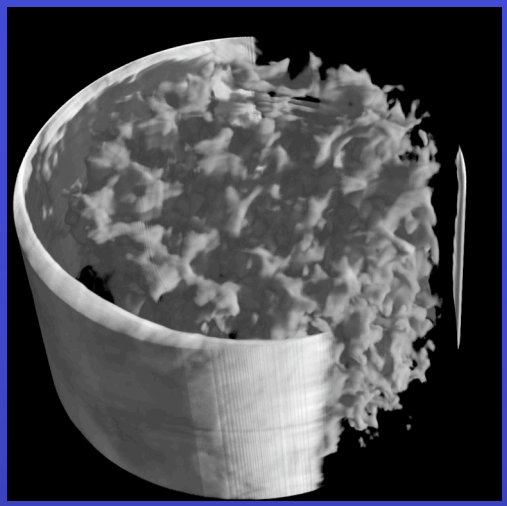
Sample environment

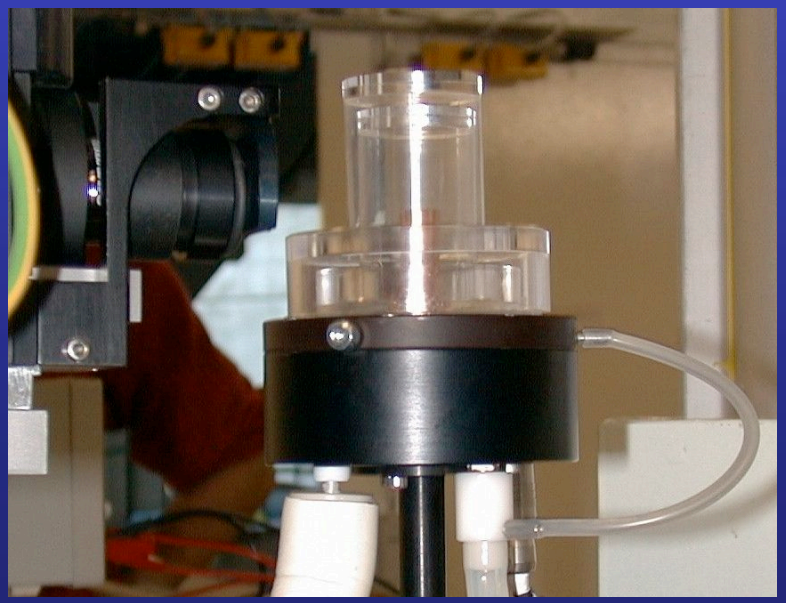
Cold!

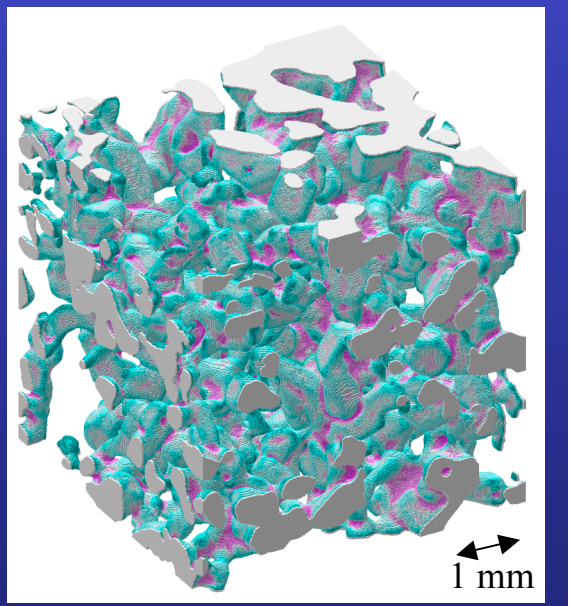
- ✓ Refrigerated cell
  120 K-320 K

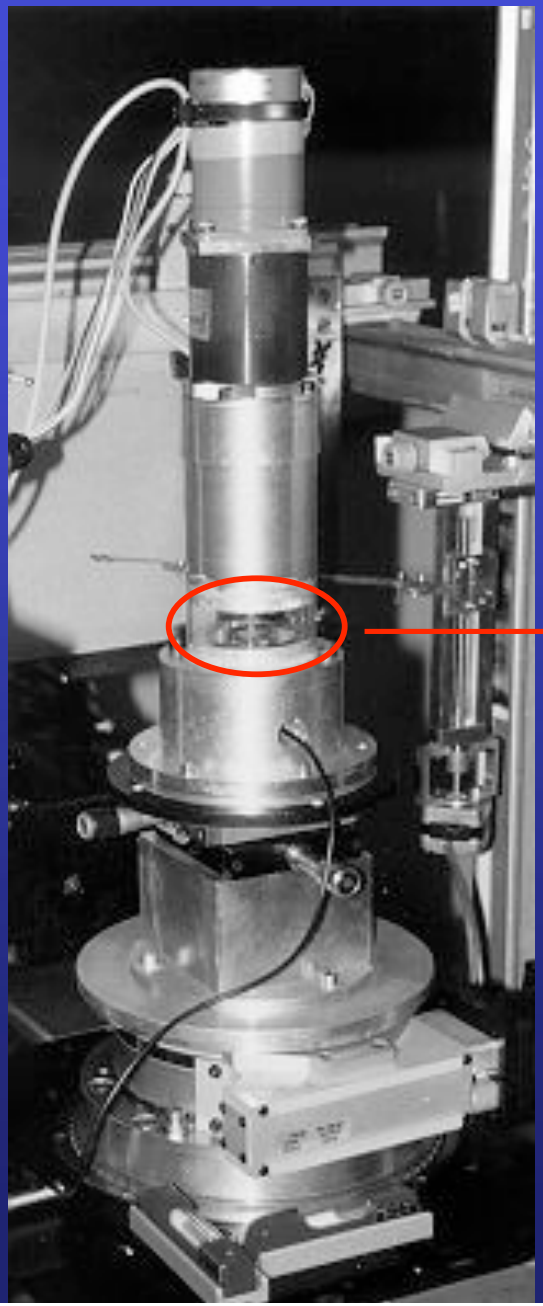
CEN, METEO FRANCE

Laboratoire de Glaciologie et Géophysique de l'Environnement

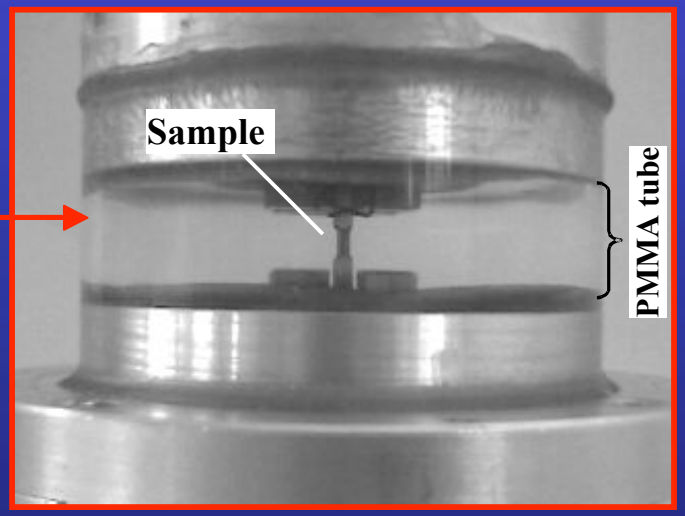




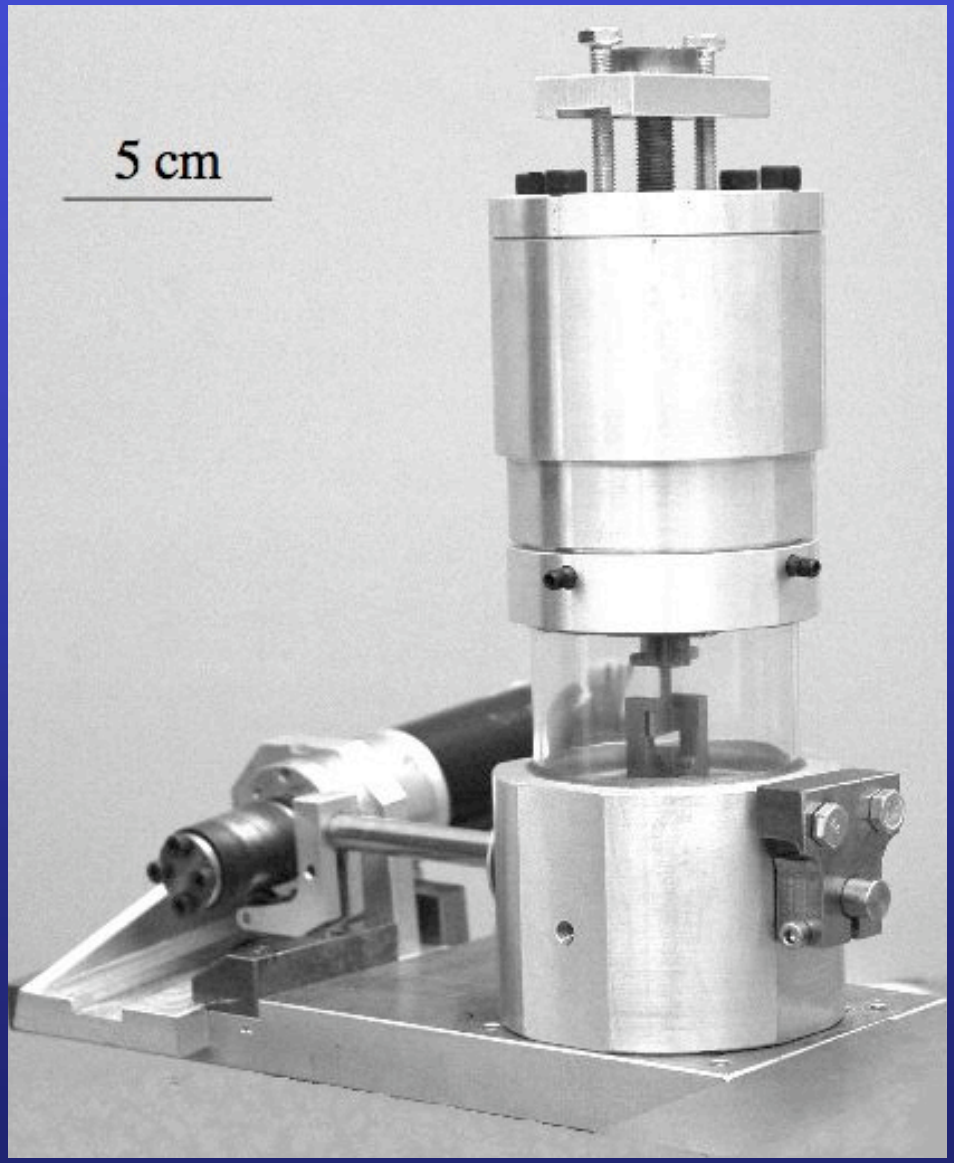




- ✓ Tension/compression stage (displacemement controled)
  - +/- 2500N
  - translation speed: from 0.1 µm/s to few mm/s
  - max displacement between 5 mm and 1 cm

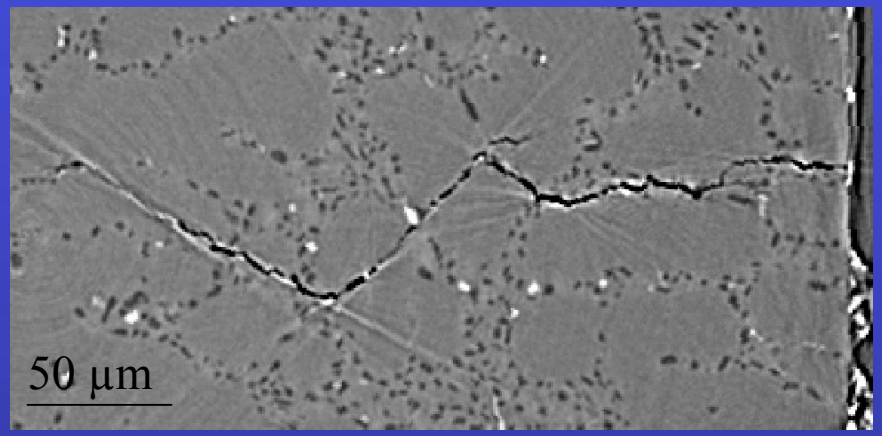


Fatigue machine (INSA-Lyon)

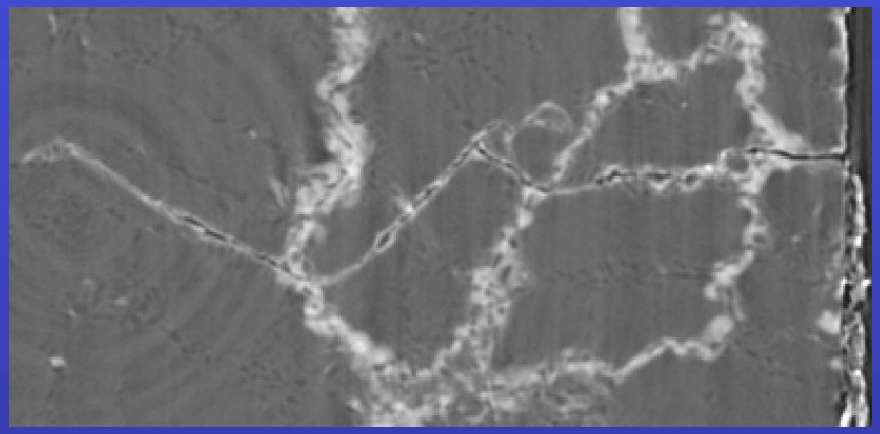


- Mounts directly on sample stage
- Polymer tube
- Maximum load 1000 N
- Tension / Tension
- up to 50 Hz

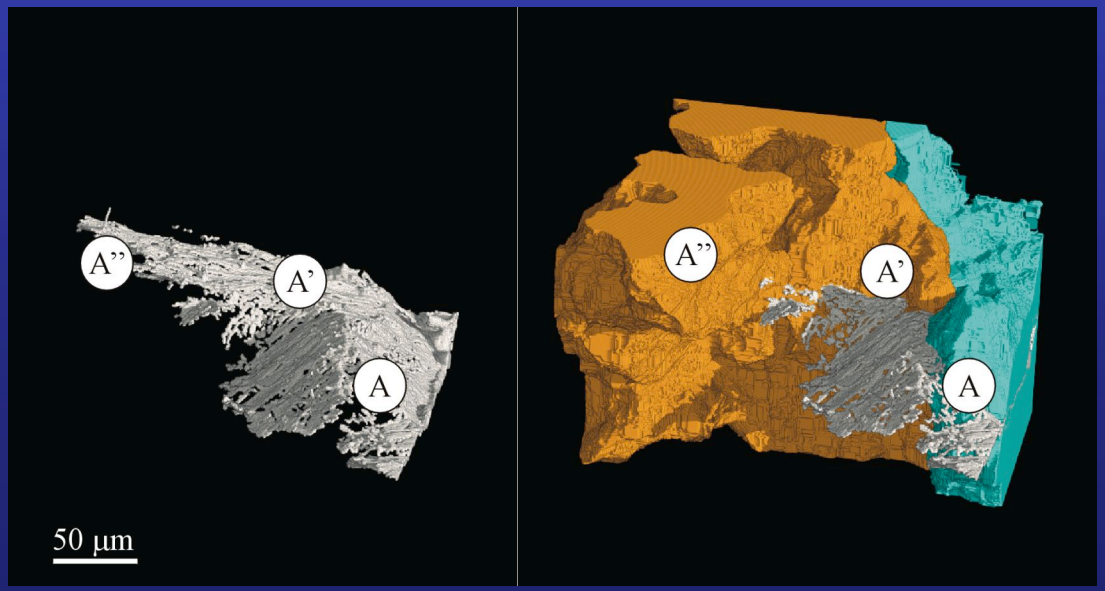
Example: characterization of fatigue crack



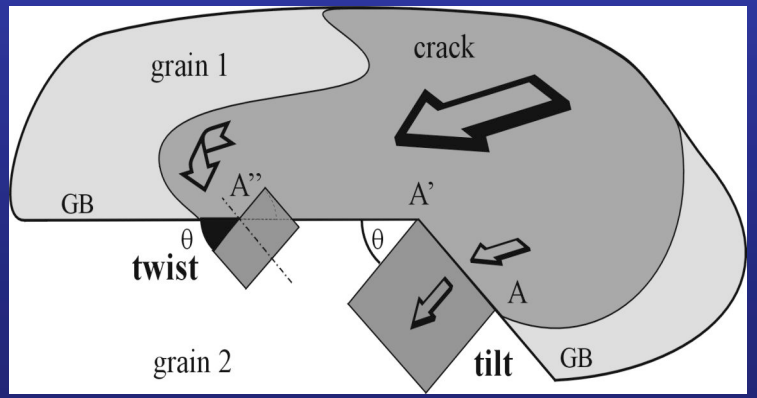
short fatigue crack (phase contrast)



crack and grain boundaries (Ga decoration)



3D rendering after morphological segmentation

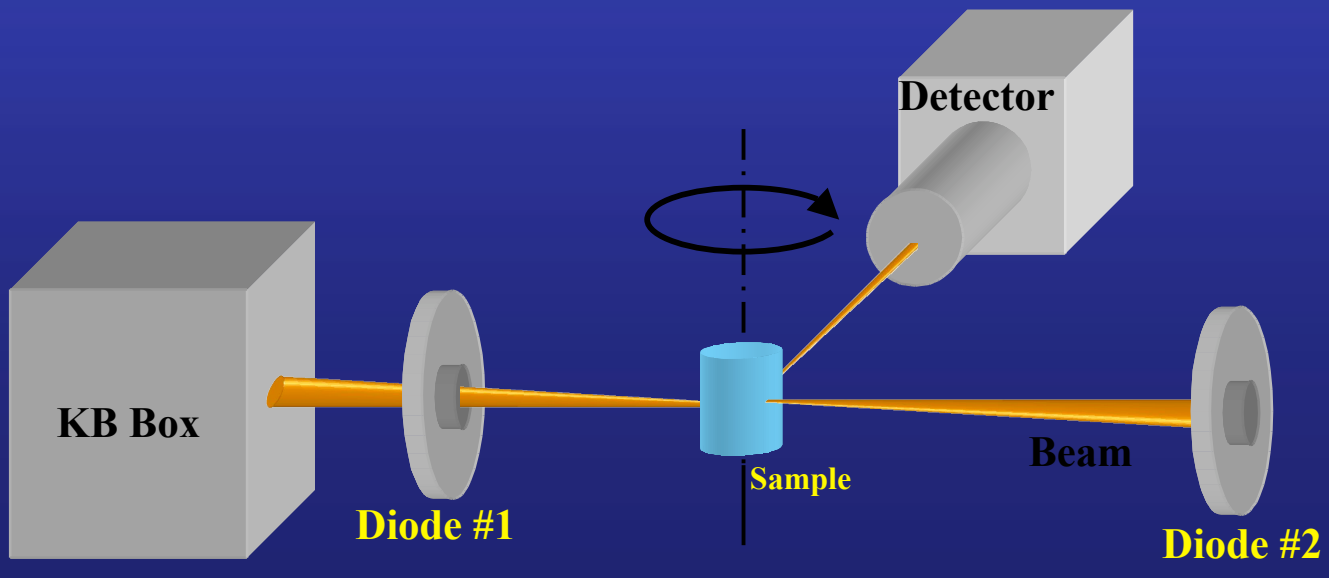


“Special” tomographic imaging techniques

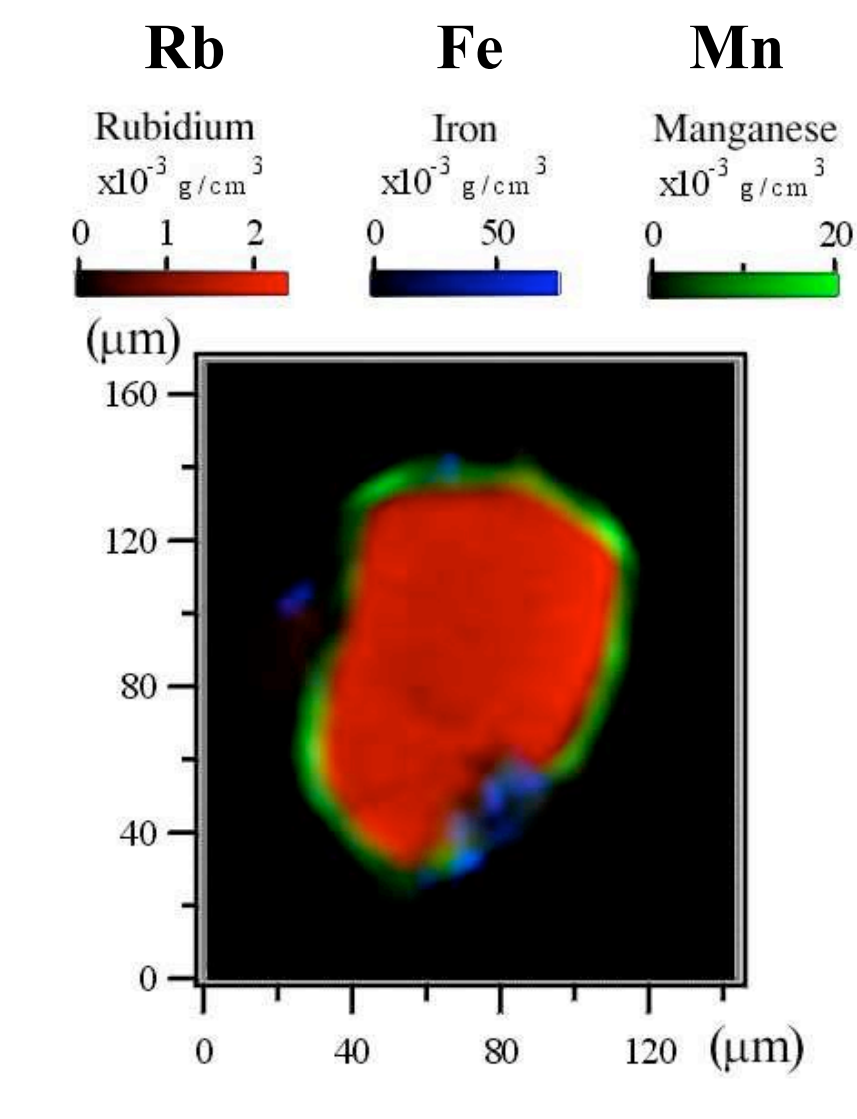
- **Fluorescence tomography (ID22)**
  - map 3D element distribution
- **Laminography (ID19)**
  - tomography on flat samples
- **Projection Microscopy (ID22 NI)**
  - down to 200 nm, holotomography
- **Topo-Tomogaphy (ID19)**
  - map 3D defect distribution inside one grain
- **Diffraction contrast tomography (ID19)**
  - map 3D grain structure of undeformed polycrystals

X-ray Fluorescence Tomography

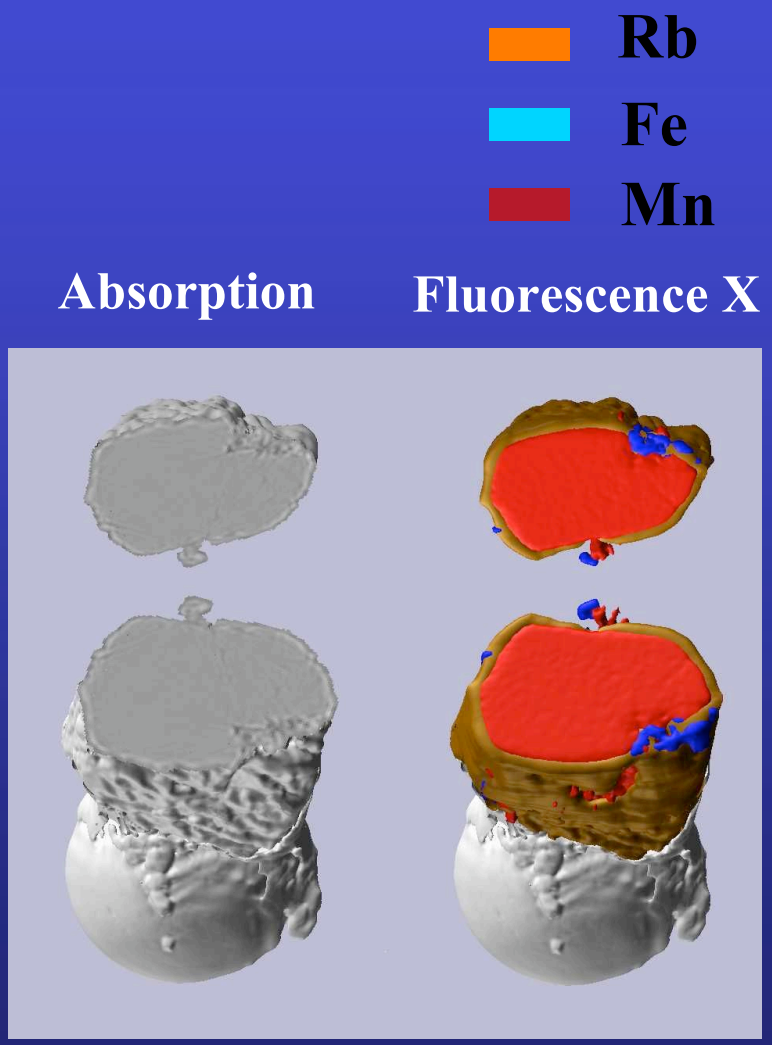
+ 3D distribution / concentration map of chemical elements
- slow (scanning technique)
- Restrictions on size / type of materials



Environmental science example



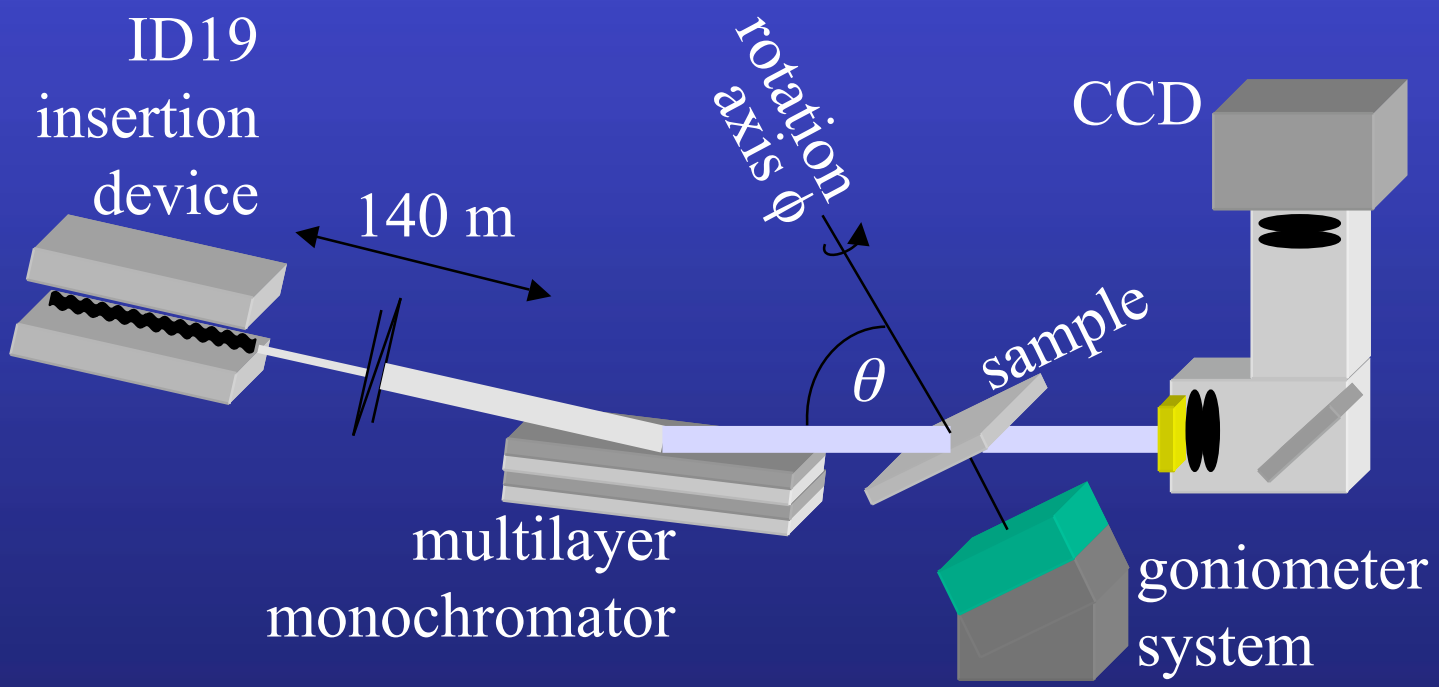
Golosio et al., App. Phys. Lett. 2004



Single waste Fly ash particle

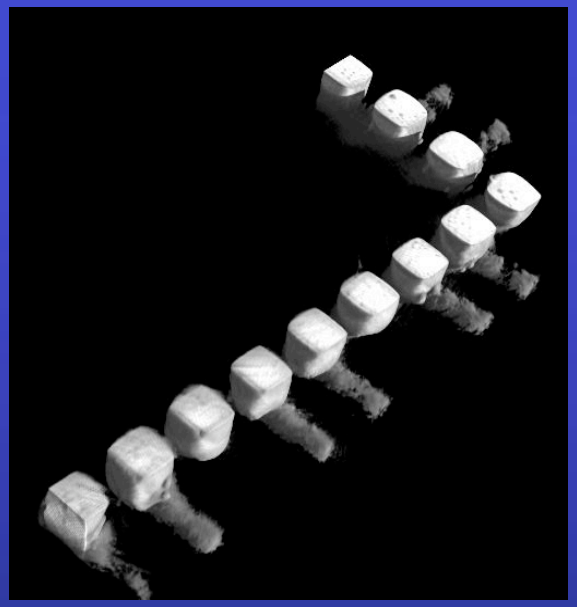
X-ray Laminography

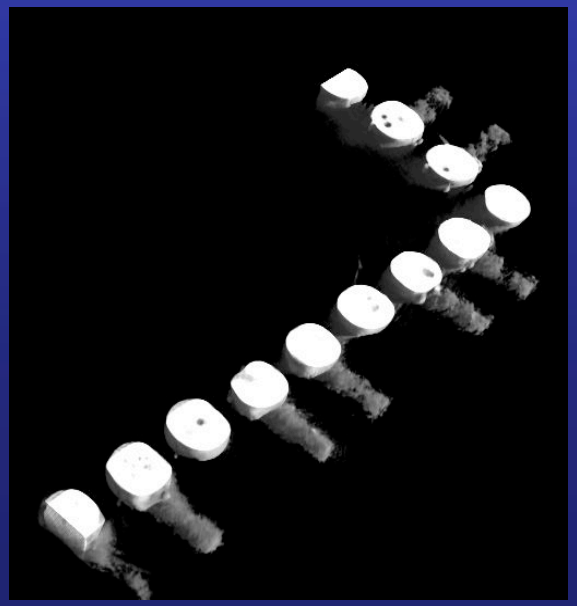
Imaging flat samples: -> microelectronics



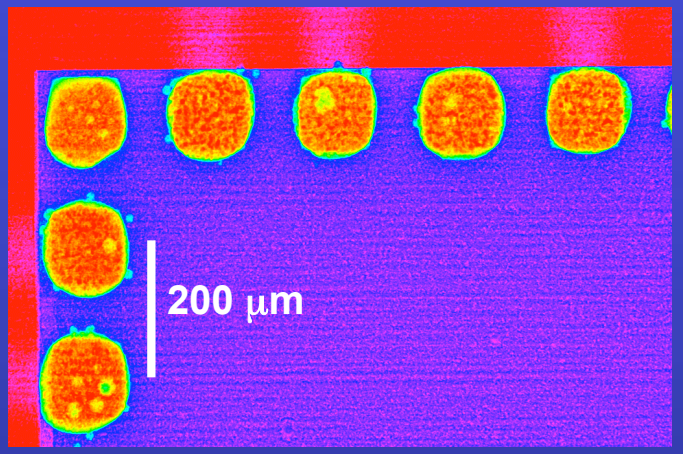
L. Helfen et al.

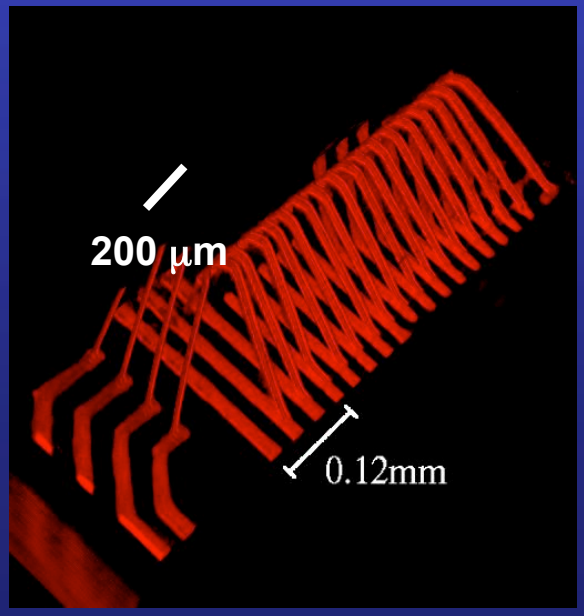
Imaging of Flip-chip interconnects





Flip-chip interconnections: 180 μm Pitch

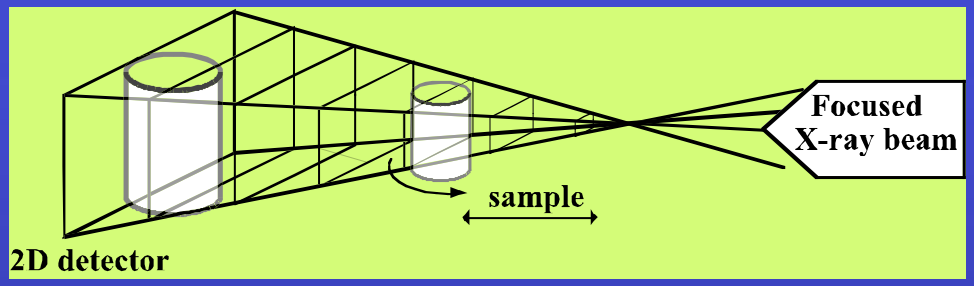




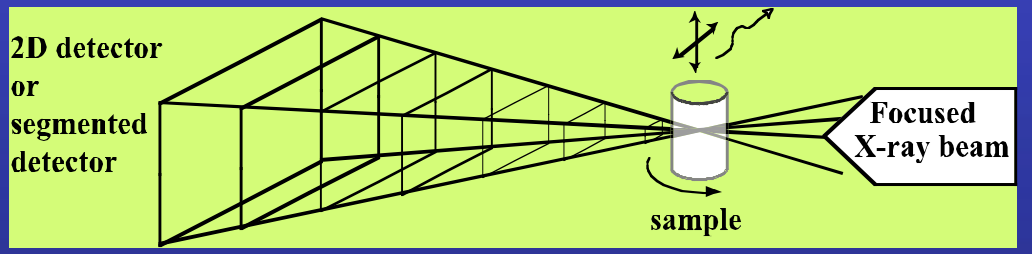
Helfen et al.

X-ray microscopy techniques

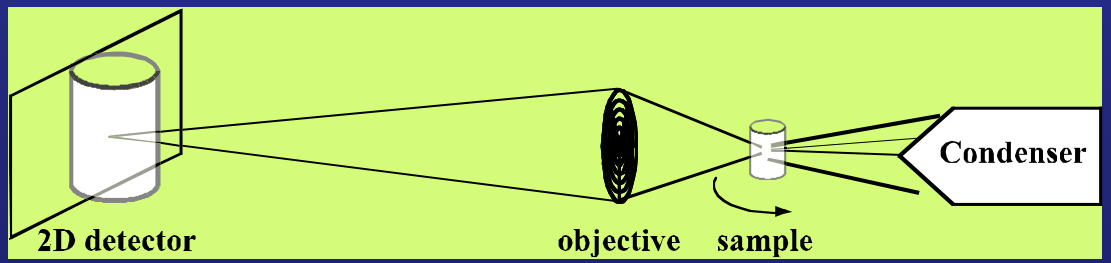
Projection Microscopy:
Structure
Dose efficient, fast
Phase contrast



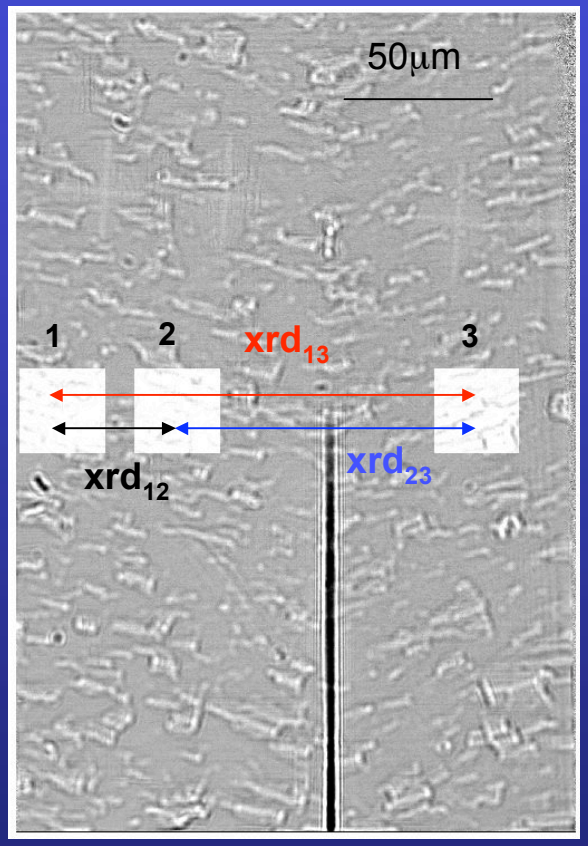
Scanning Microscopy
Fluorescence, Spectroscopy
Slow
Rich, trace elements
Phase contrast



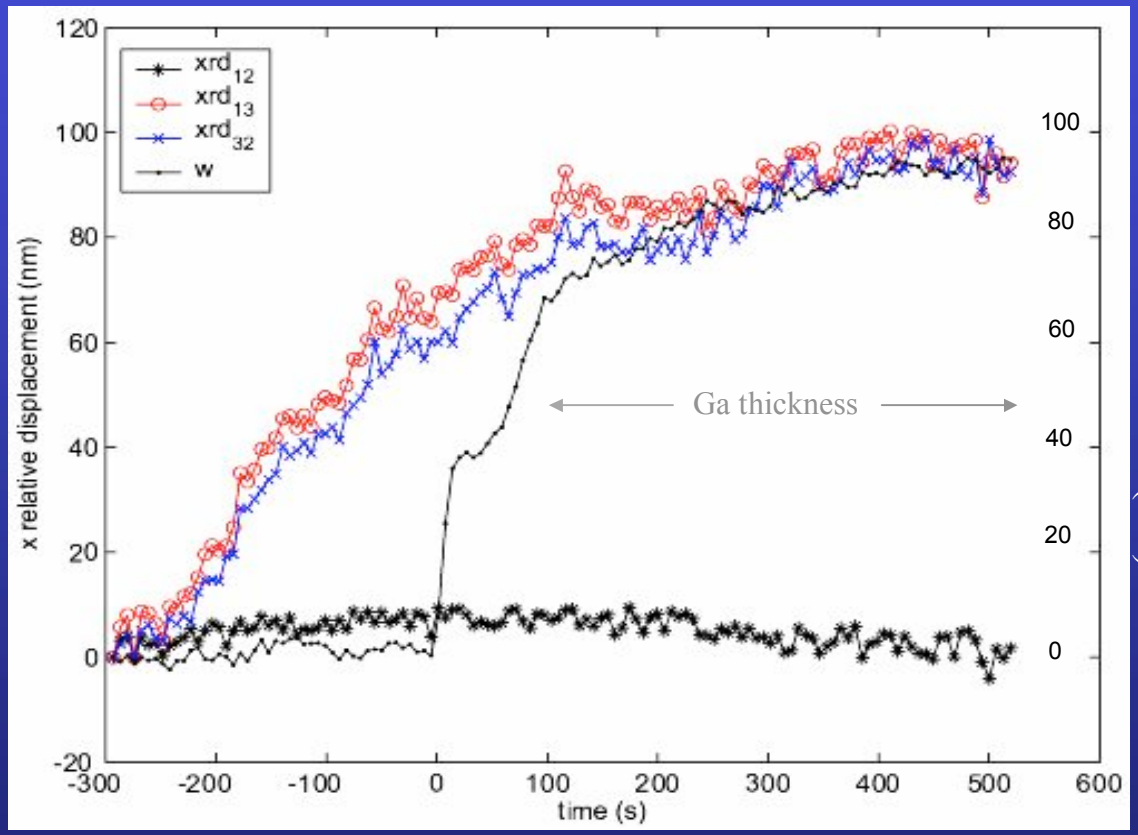
Full-field microscopy
Structure
Dose inefficient, fast
Absorption + phase



Measurement of grain displacements at the 10 nm level



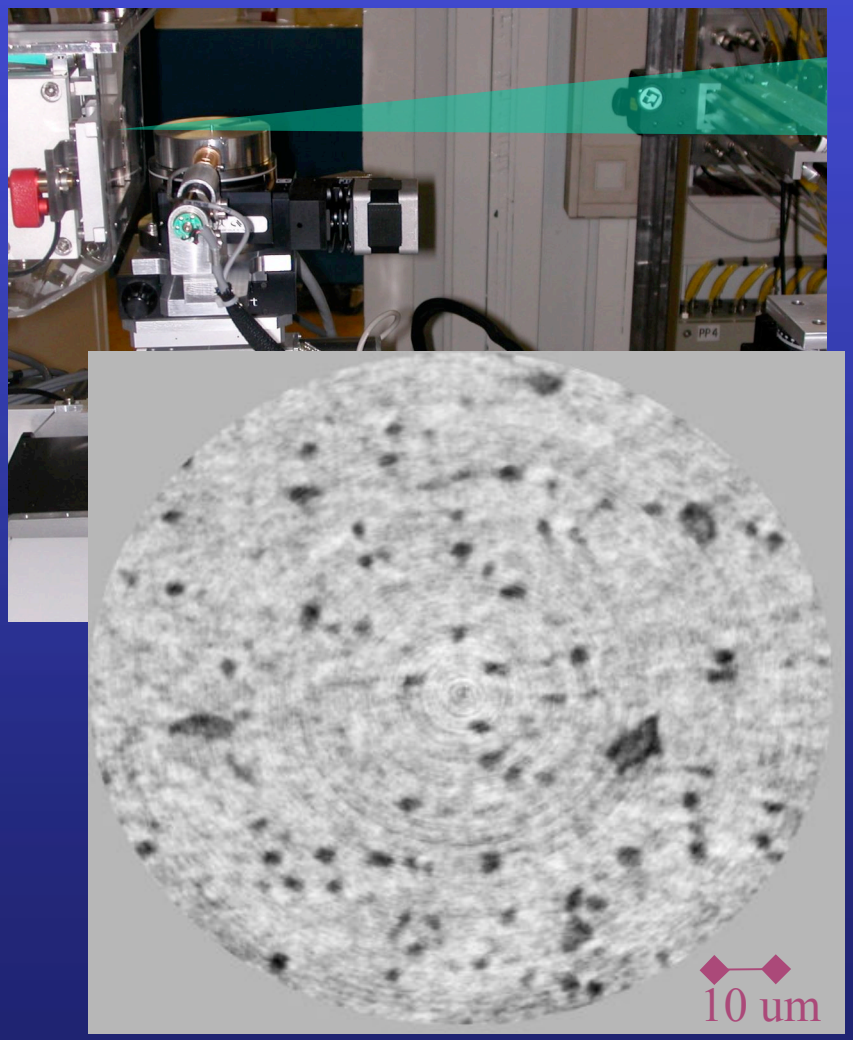
X-ray beam direction ⊗



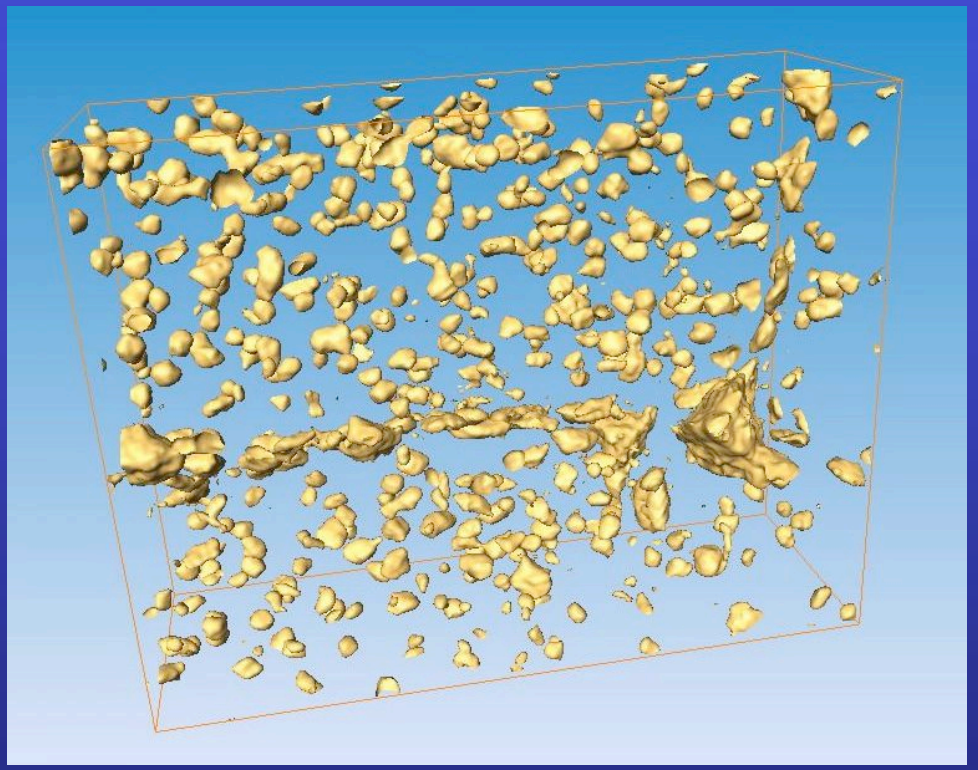
t = 0 s: arrival of liquid Ga at the level of the ROIs

E. Pereiro-Lopez, W. Ludwig et al. PRL 95 219501 (2005)

Magnified Tomography



$CuAl_2$ particles in Al matrix



Inside $\phi = 1$ mm sample

$E = 20.5$ keV

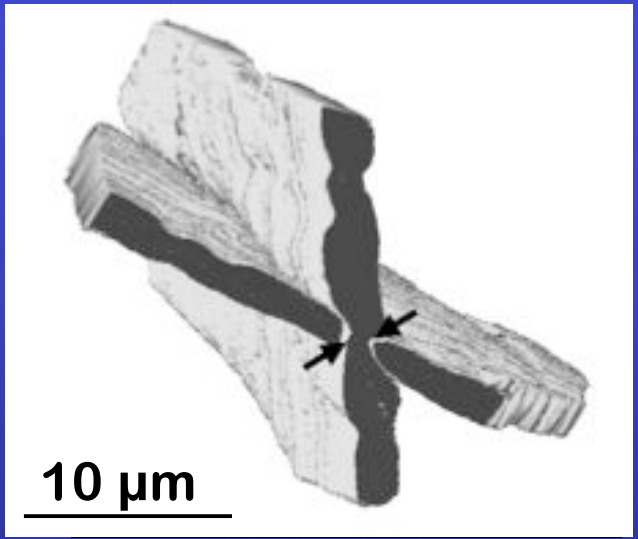
X-ray magnification = 55

R. Mokso, et al. submitted

Mapping Polycrystals in 3D

Conventional techniques:

- Serial sectionning + EBSD
- Focused Ion Beam (FIB)

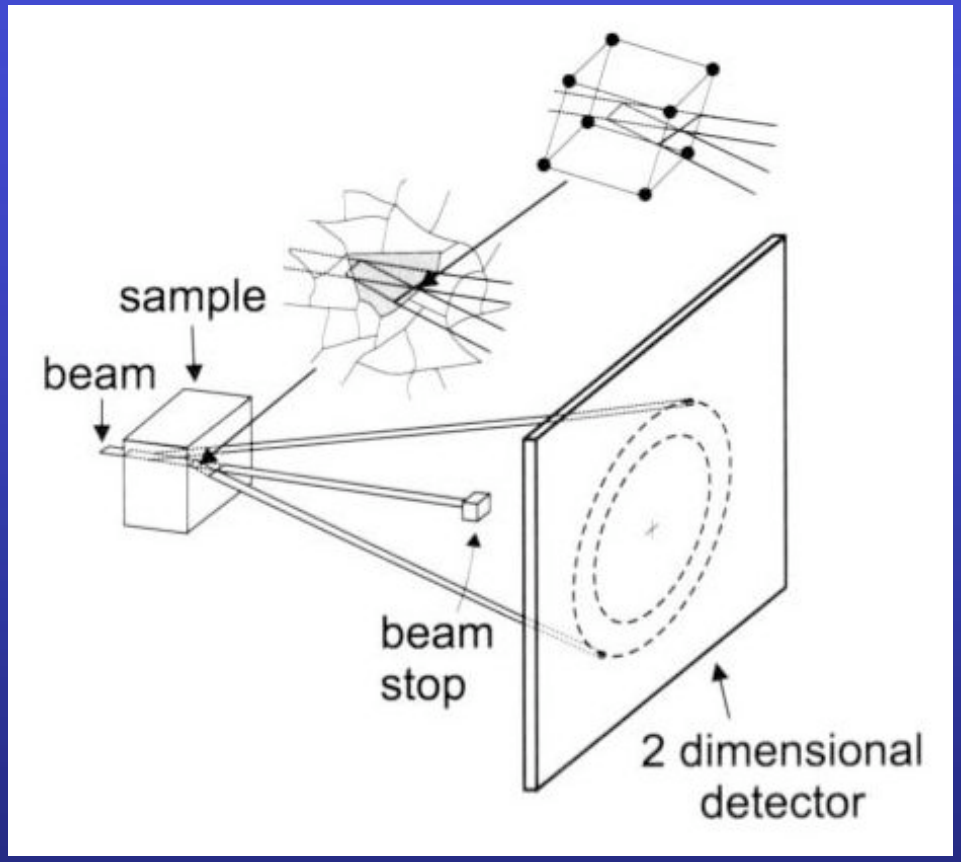


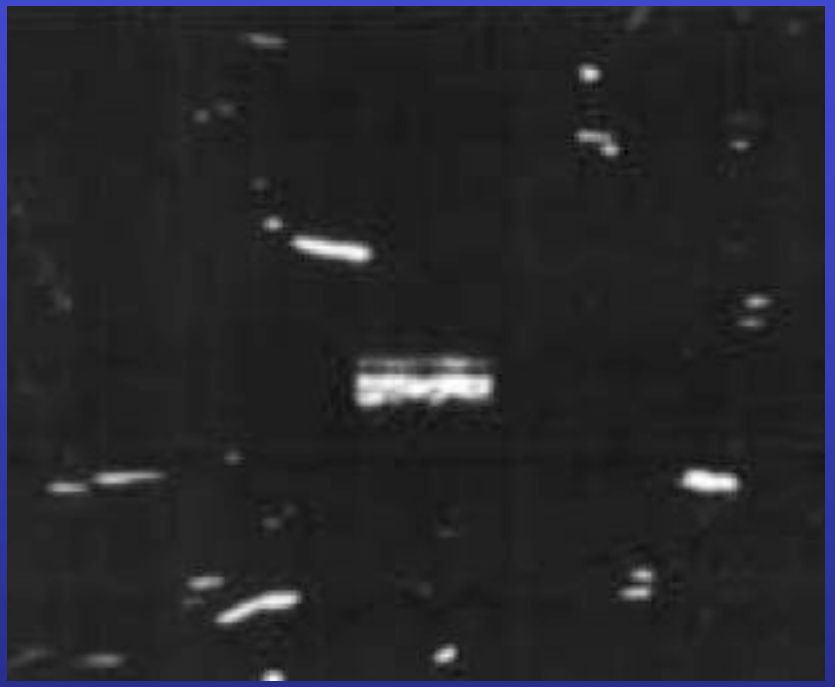
Advantages: + works on deformed material
+ fully automated imaging mode
+ high in-plane resolution

Problems: - characterization time…
- destructive

3D X-ray Diffraction Microscopy

Poulsen, Lauridsen, Schmidt et al. (Risoe)



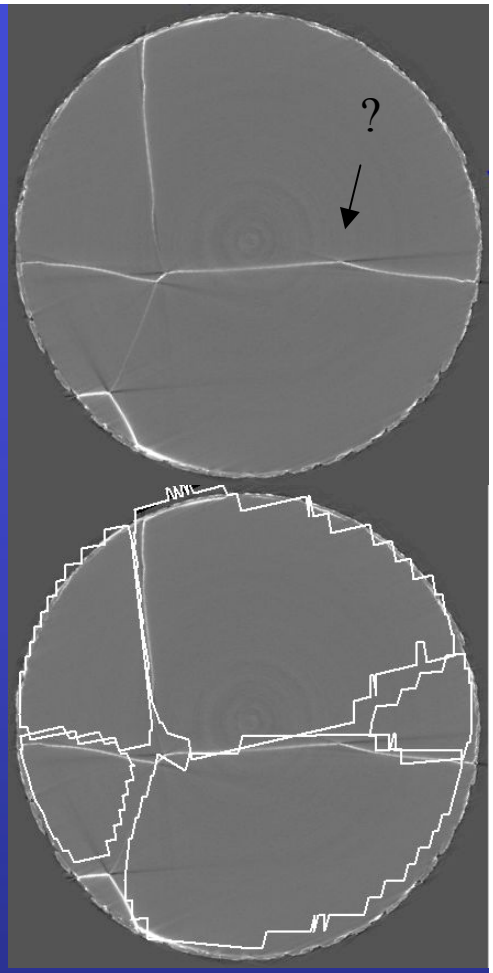


Two basic reconstruction strategies:

1) solve the inverse problem (ART)

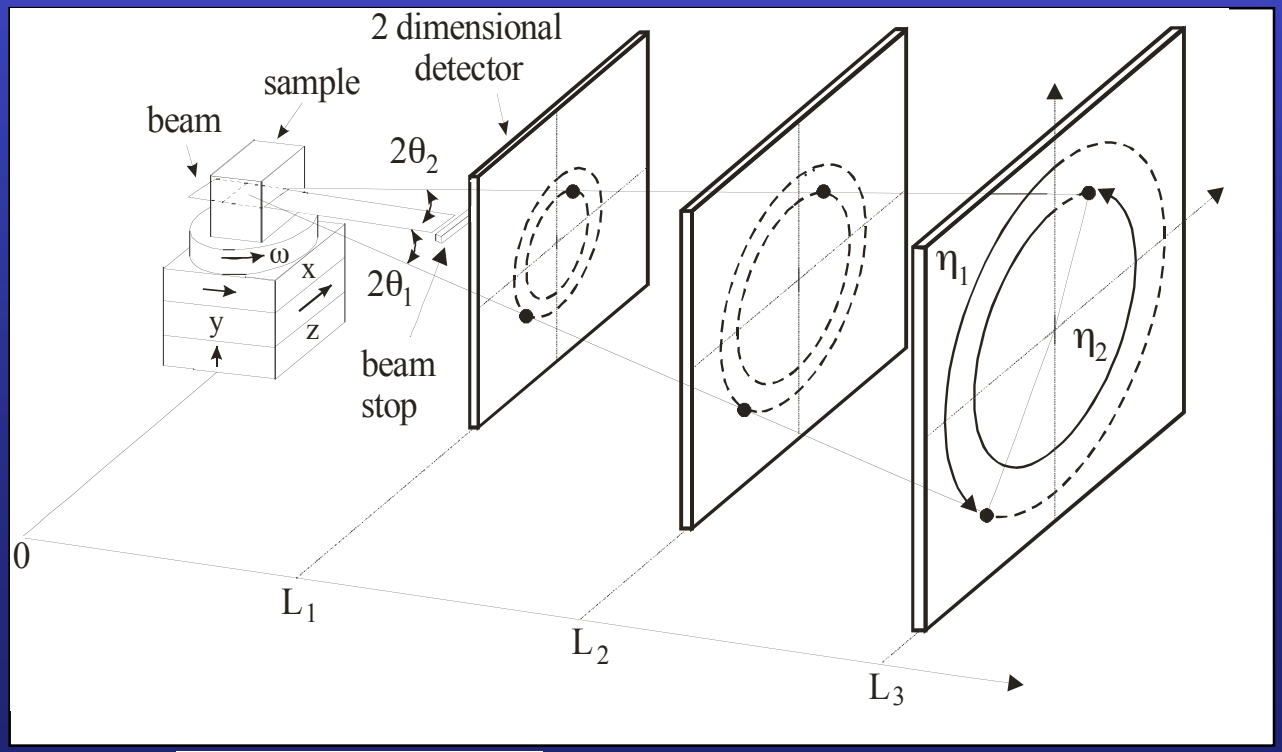
2) solve the forward problem

Combination of 3D imaging and 3D diffraction



"X-ray tracking"

- Non- destructive diffraction technique
- Crystallographic orientation & 3D grain contour



Principle of the X-ray tracking technique (H.F. Poulsen et al., Risoe)

‘Diffraction’ contrast tomography

Goal

Characterize *shape and orientatation* of all grains simultaneously with the *absorption* microstructure

Principle:

(1) Run ‘optimised’ tomography scan in conventional alignment

(2) Extract grain ‘projections’

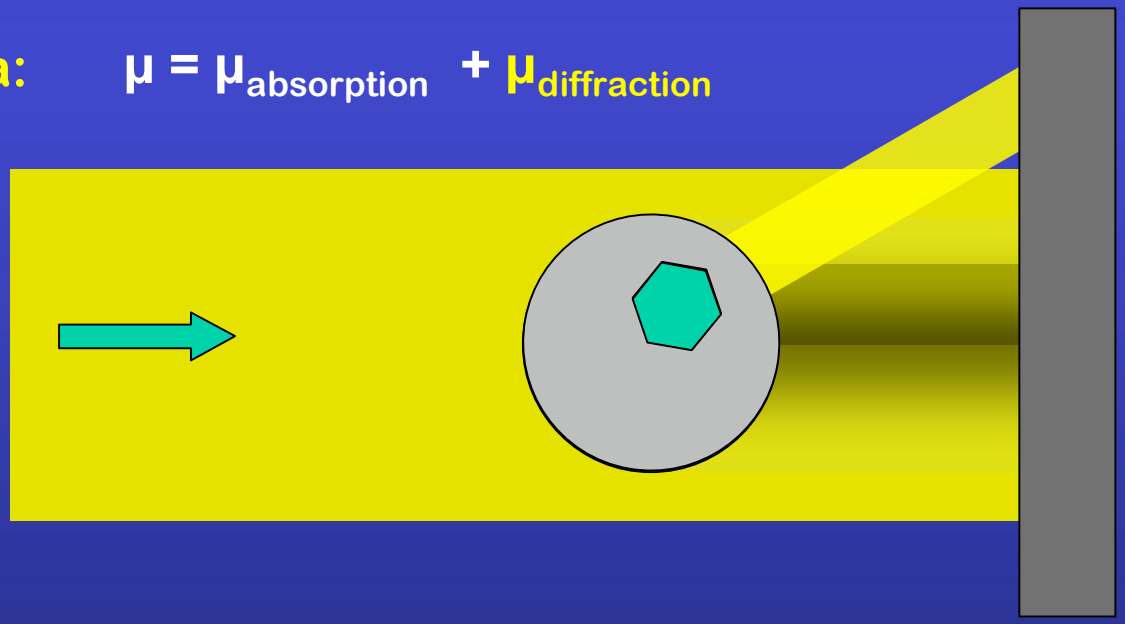
(3) Sort diffraction spots with respect to grains of origin

(4) Grain by grain tomographic reconstruction (2D-ART)

New tomographic approaches

Ludwig, Poulsen, Schmidt, Lauridsen et al.

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



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

Two data acquisition strategies:

1) Topotomography

2) Diffraction contrast tomography

Diffraction contrast tomography

Extraction of grain projections

Measured projection

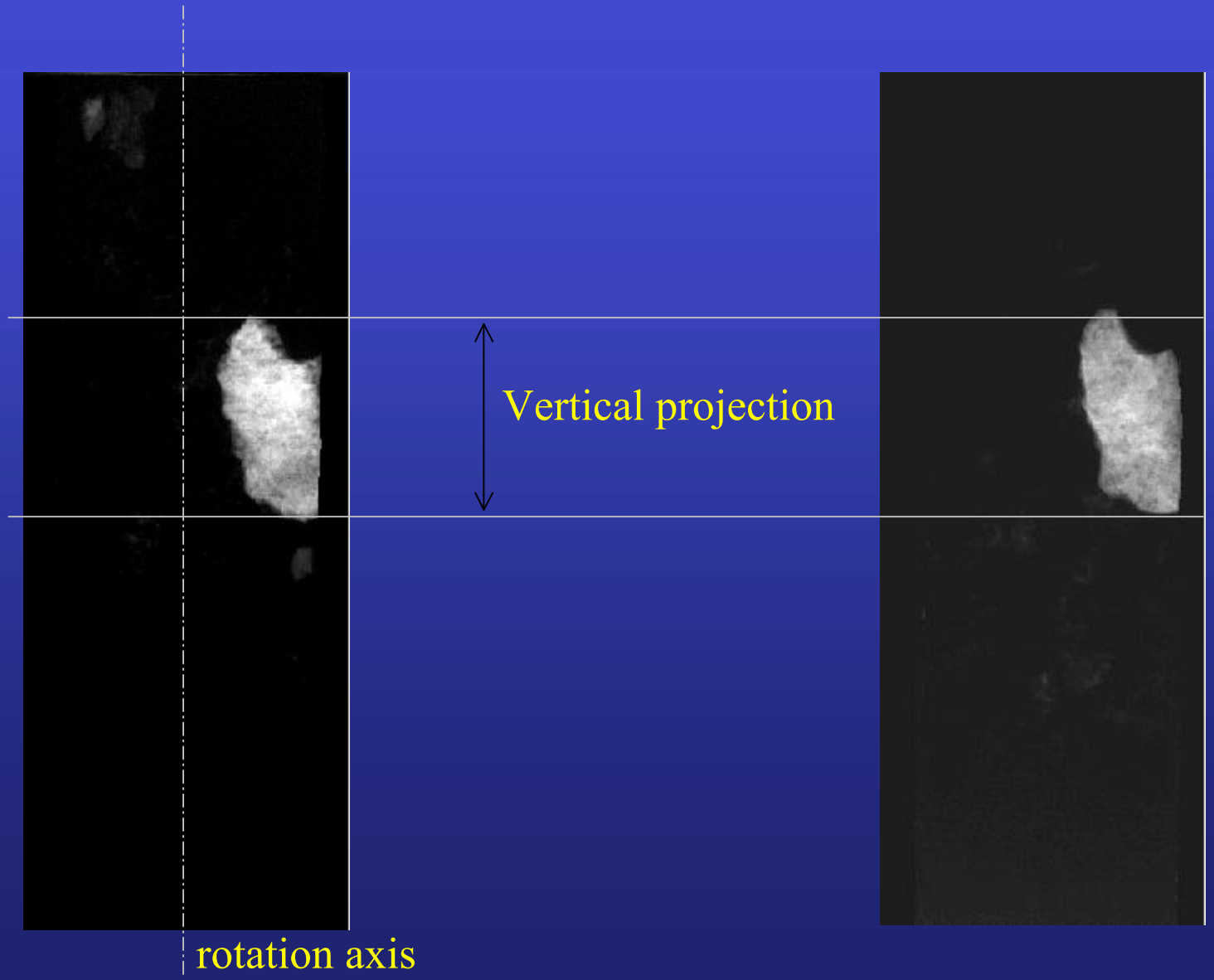
-

Calculated absorption

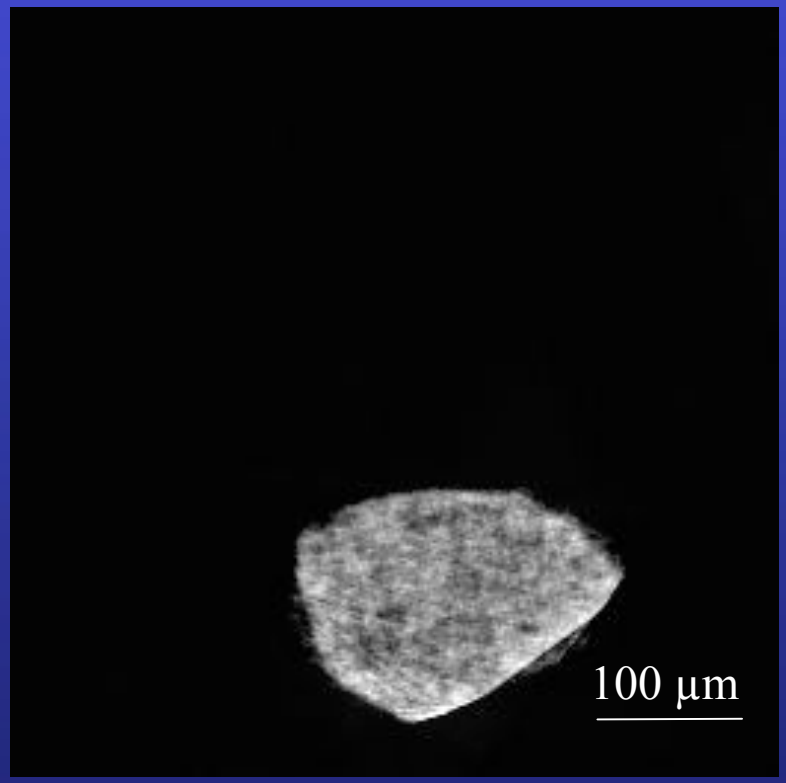
=

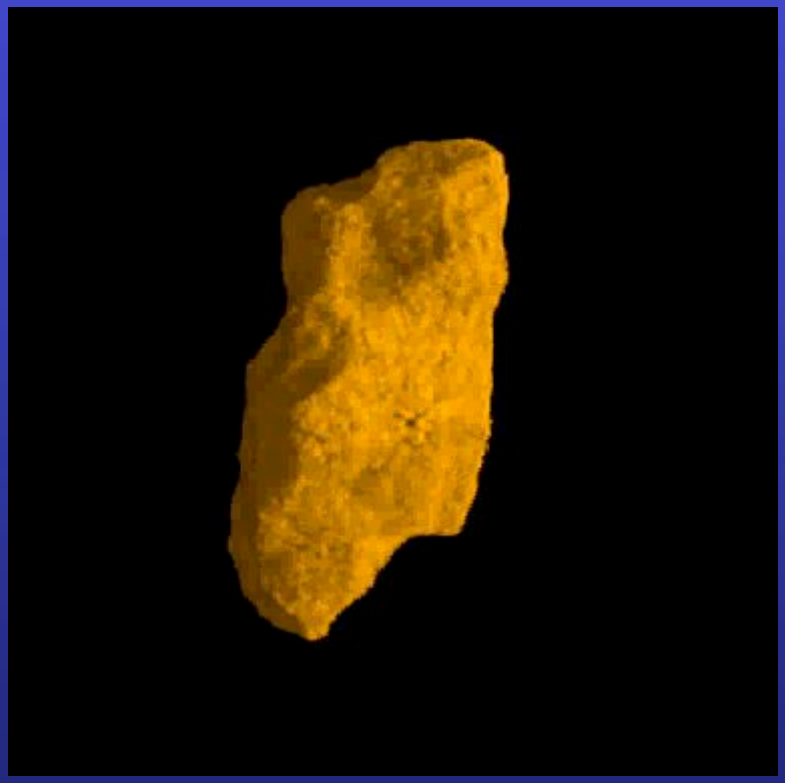
Grain projection

Sort diffraction spots with respect to grain of origin

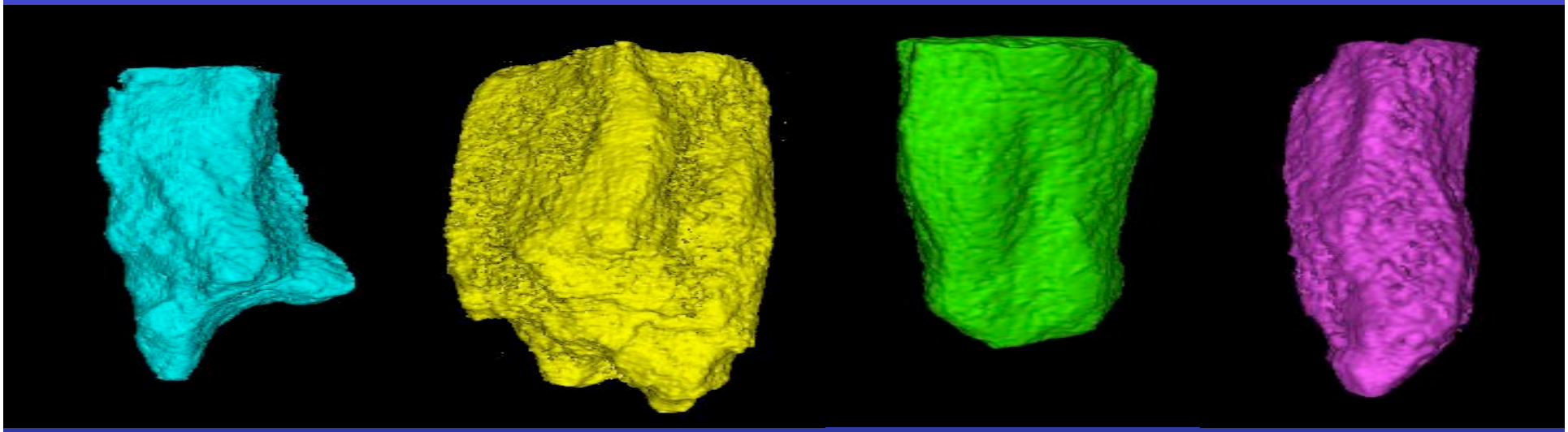


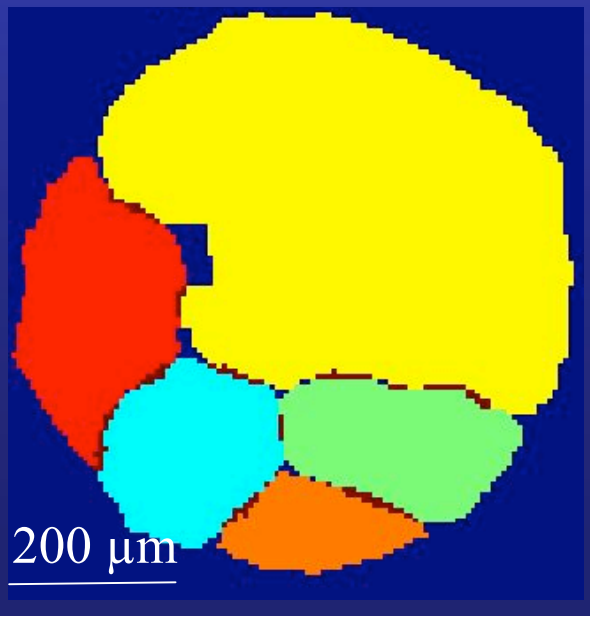
2D ART reconstruction



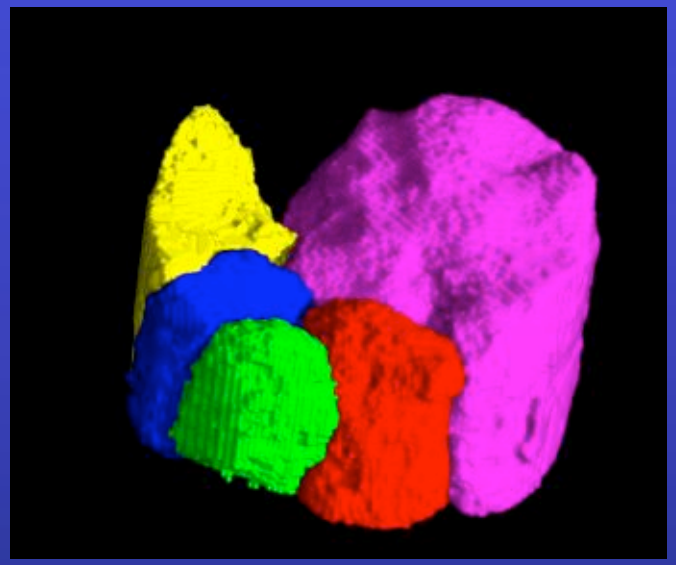


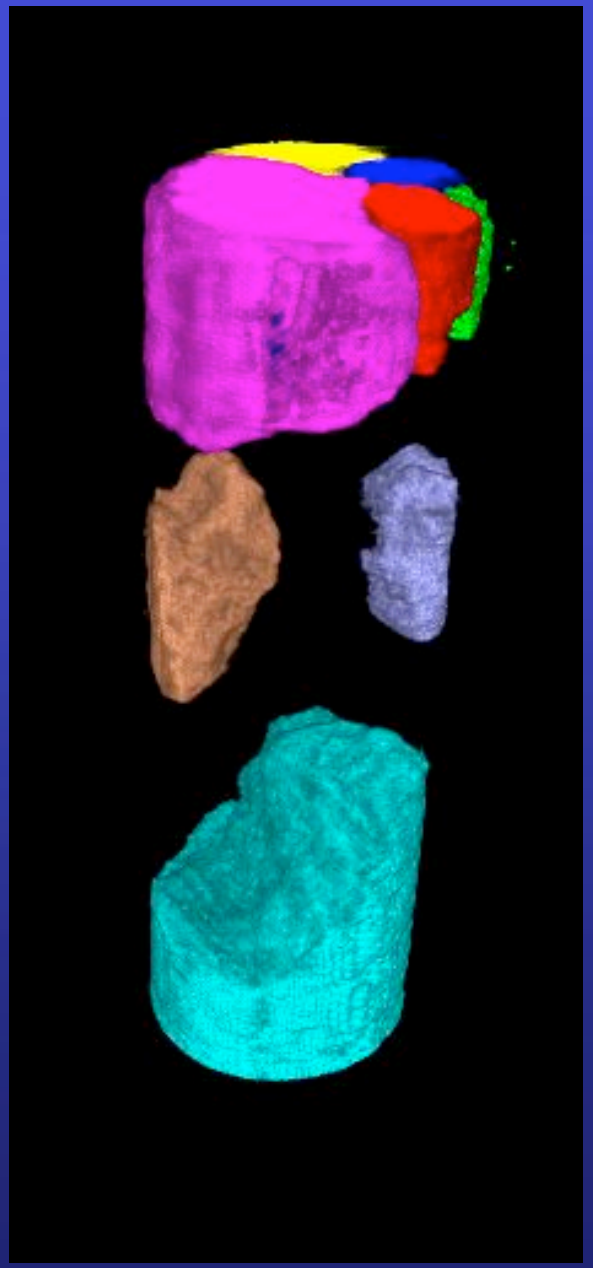
Grain by grain reconstruction





Known limitations...





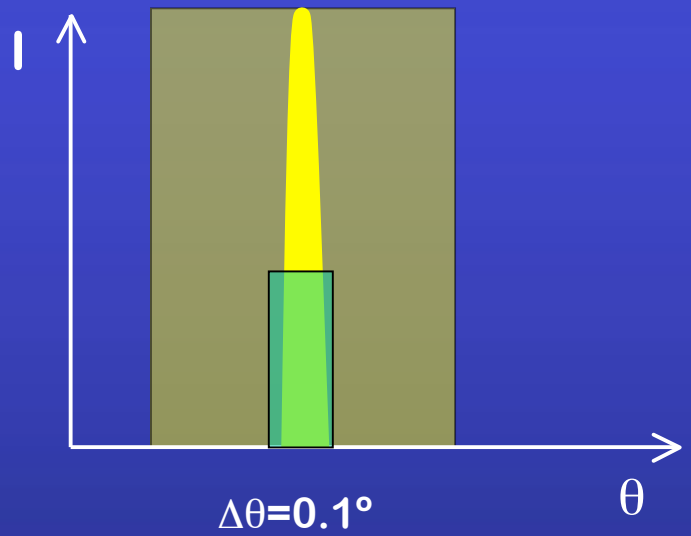
«missing grains» :

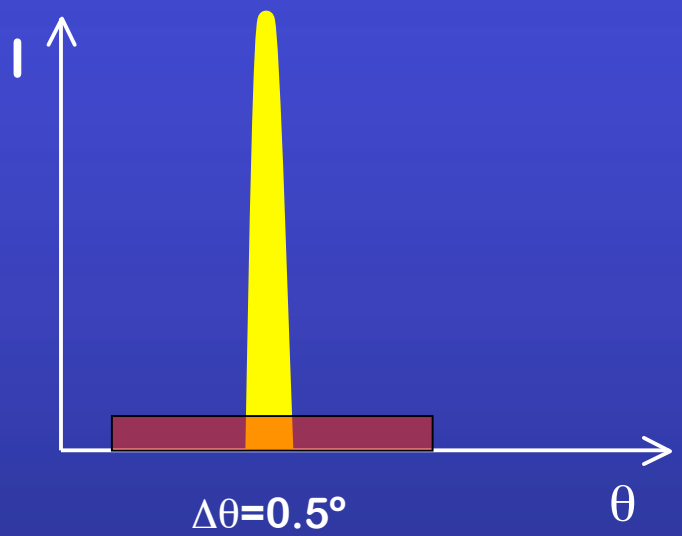
Can’t handle orientation gradients

spot overlap :

< 50-100 grains per cross-section

Mosaicity & Visibility



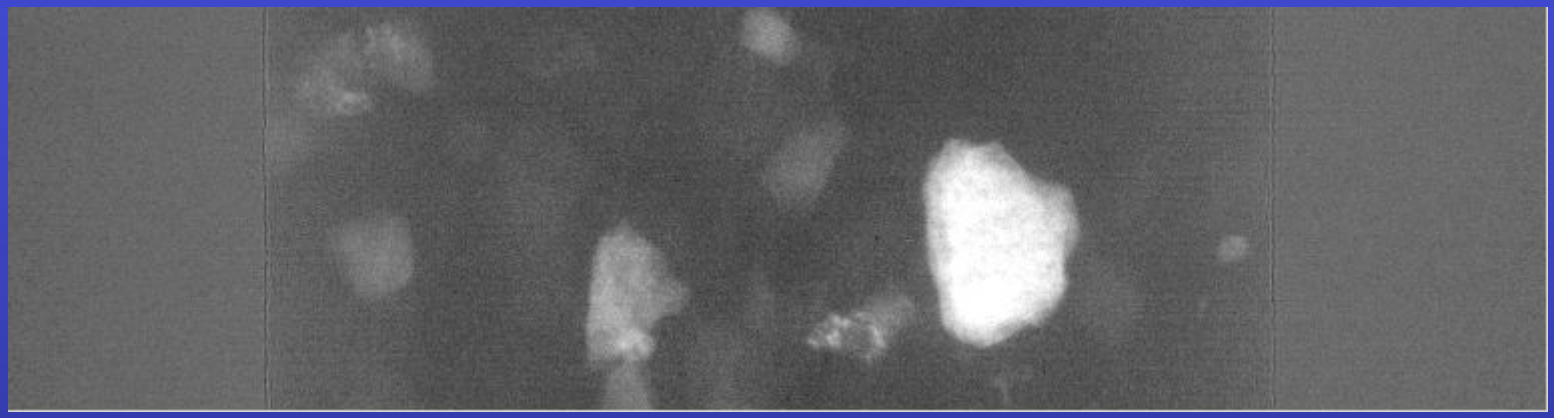


undeformed grain

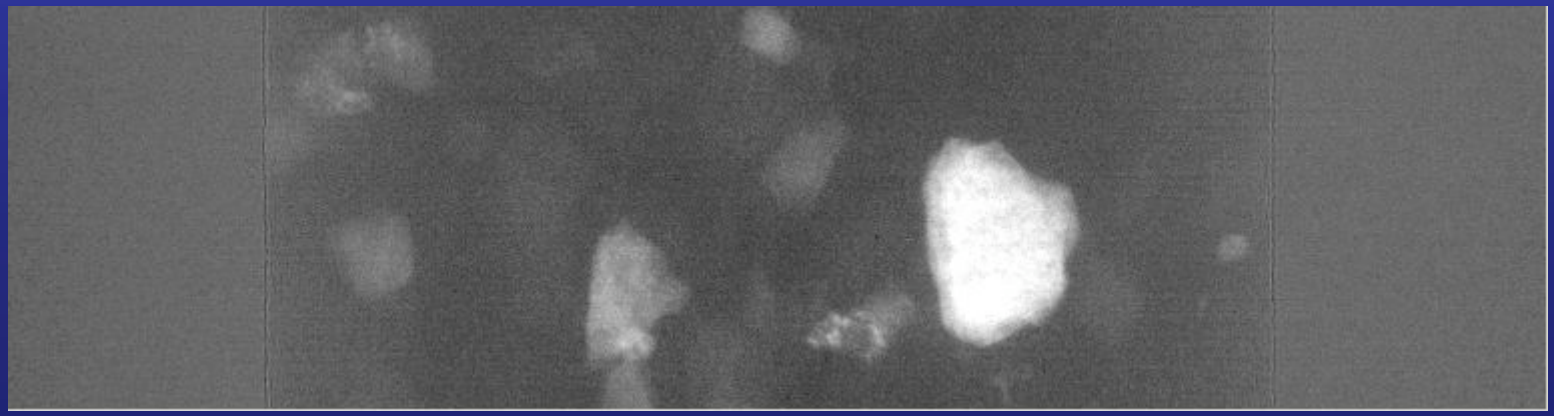
deformed grain

Crystal monochromator and low mosaicity required !

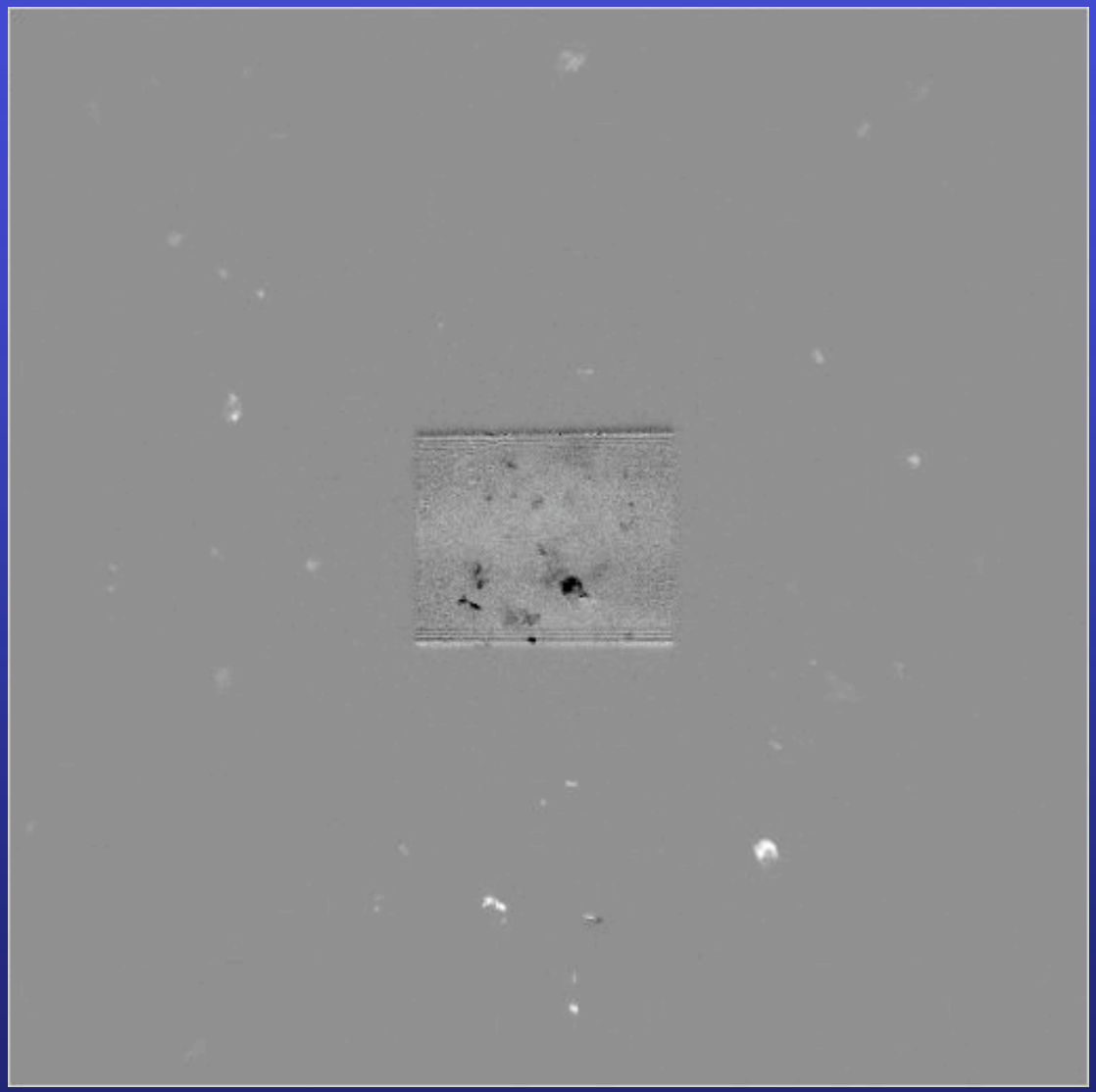
Filtering of diffraction spots



Filter: Vertical projection & sine movement



Combined acquisition



Conclusions

- Synchrotron Imaging: 3D microscope with 1µm resolution
- Dynamic 3D Imaging possible (few seconds … minutes)
- Contrast mechanisms: absorption, phase, fluorescence, diffraction, etc…
- X-ray Topography allows to image defects and distorsions

…under development

- Nanotomography : down to 50 nm resolution
- Characterization of 3D grain structure in polycrystals

Acknowledgements

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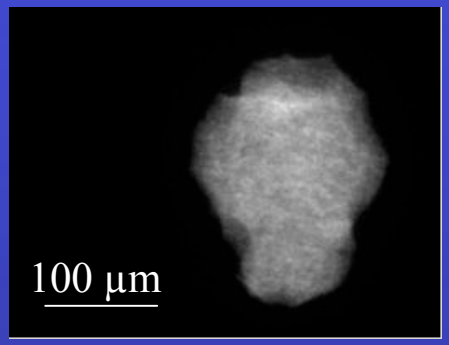
S. Schmidt, D.J. Jensen

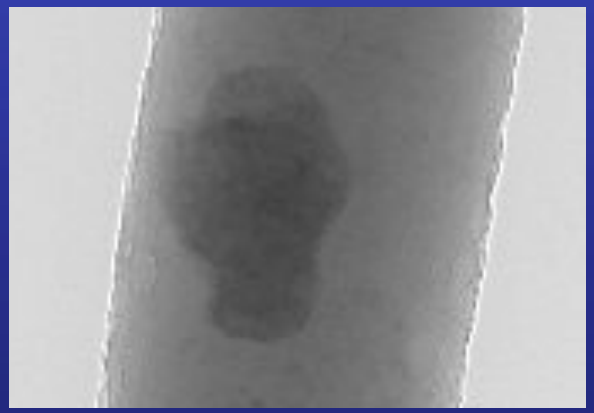
LGGE, Grenoble

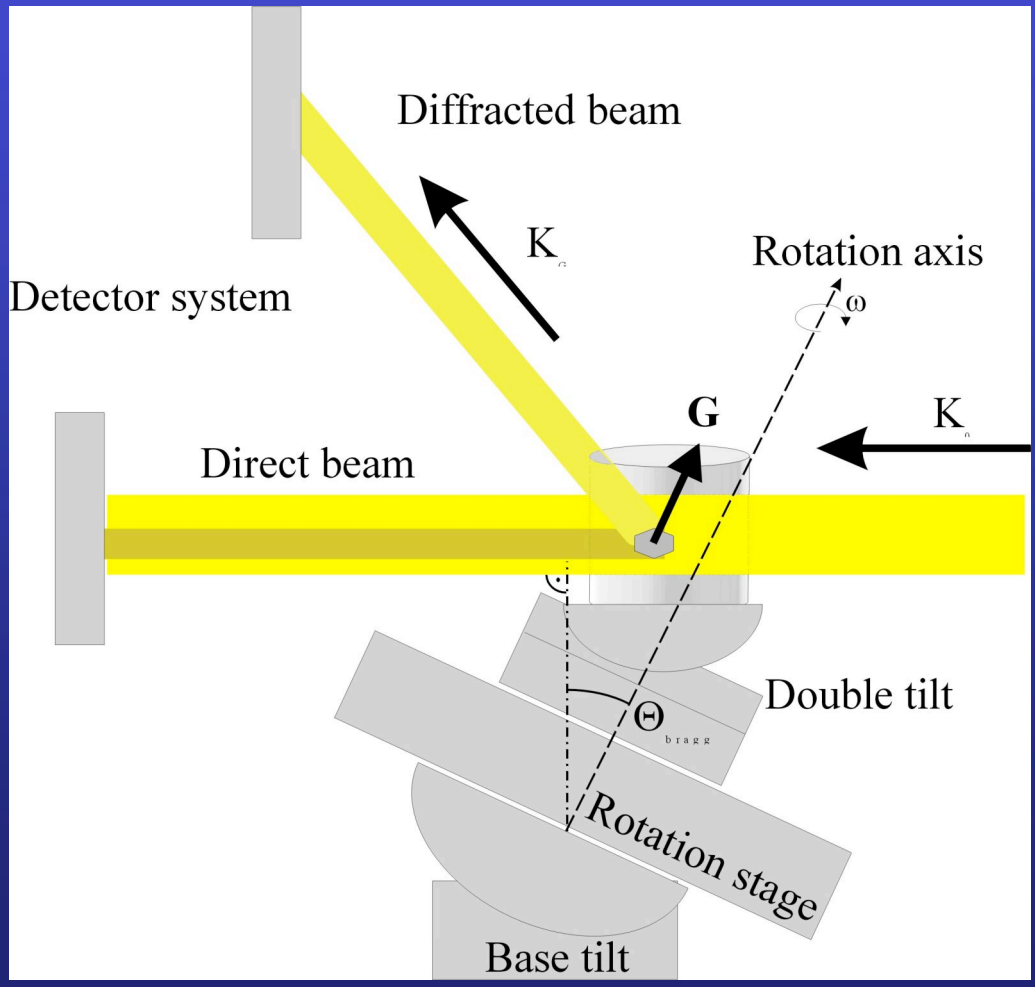
A. Philip, J. Meysonnier,

…

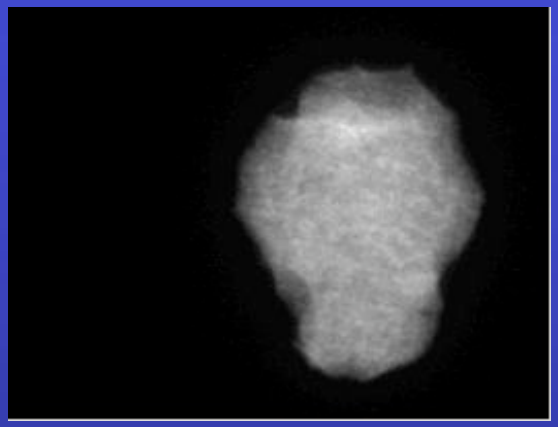
Topo-Tomography applied to Polycrystals





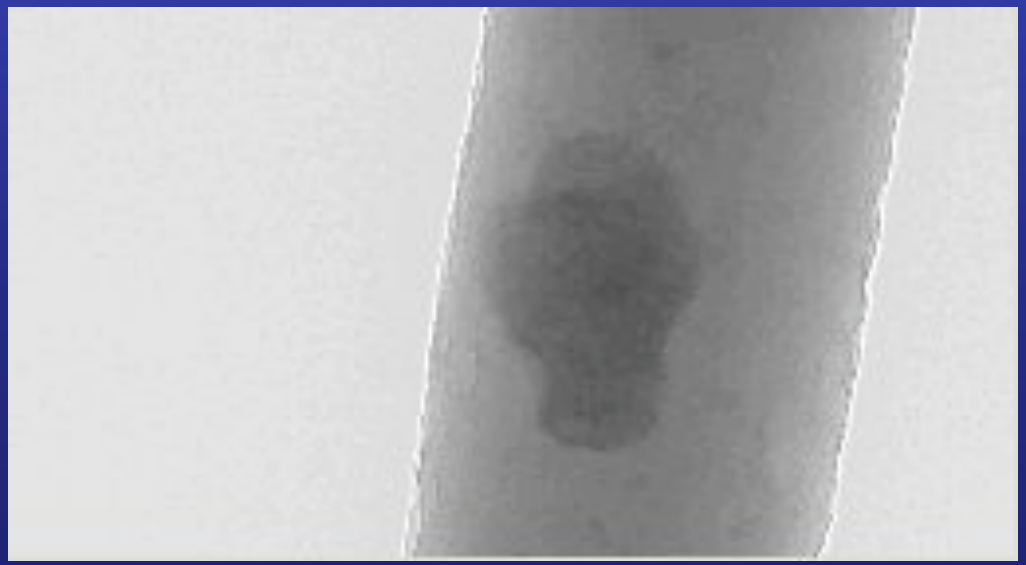


Topo-Tomography: data acquisition



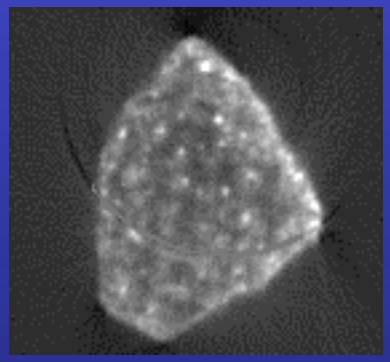
diffracted beam

100 µm

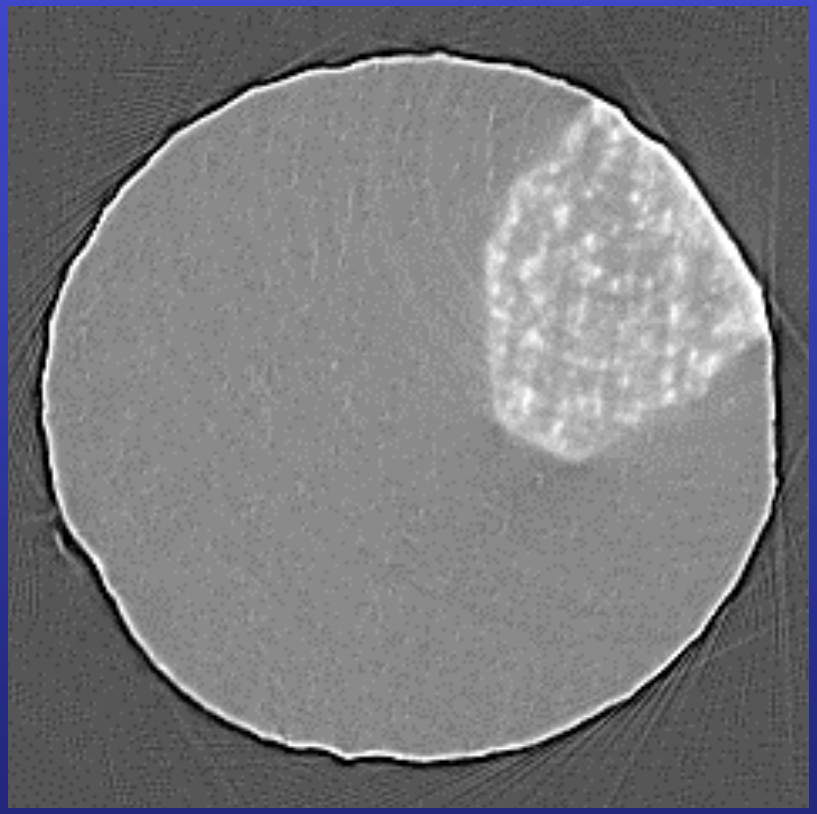


direct beam

Topotomography

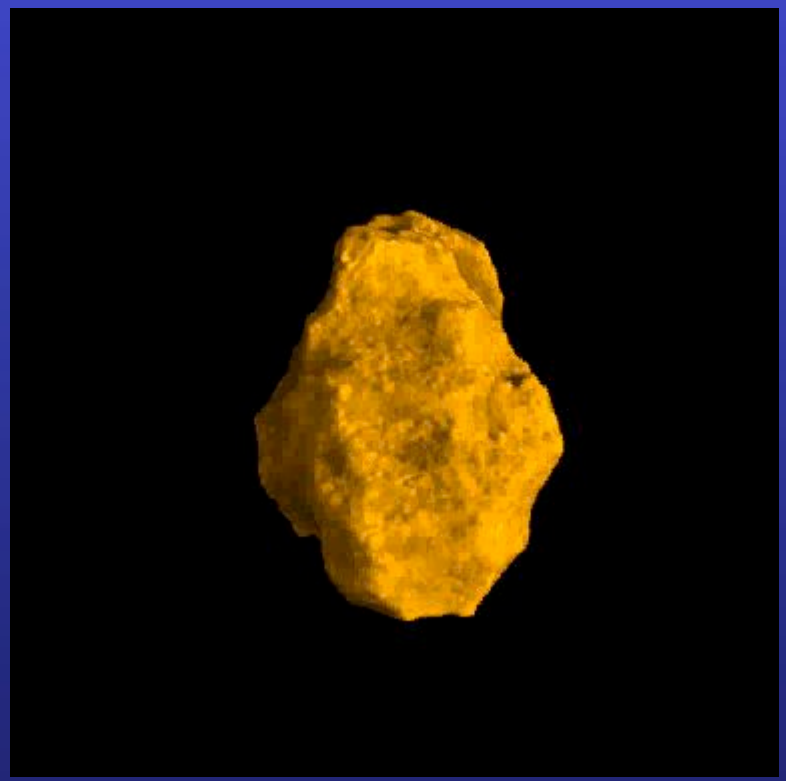


Reconstruction from diffracted beam (180 projections)

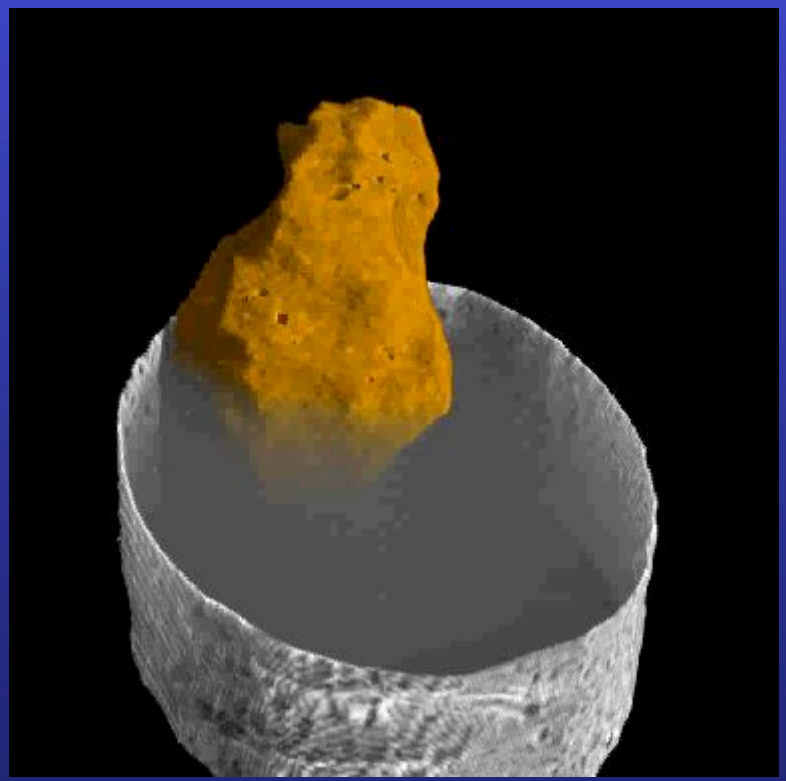


Reconstruction from direct beam (720 projections)

FBP reconstruction (cone beam algorithm)



Diffracted beam



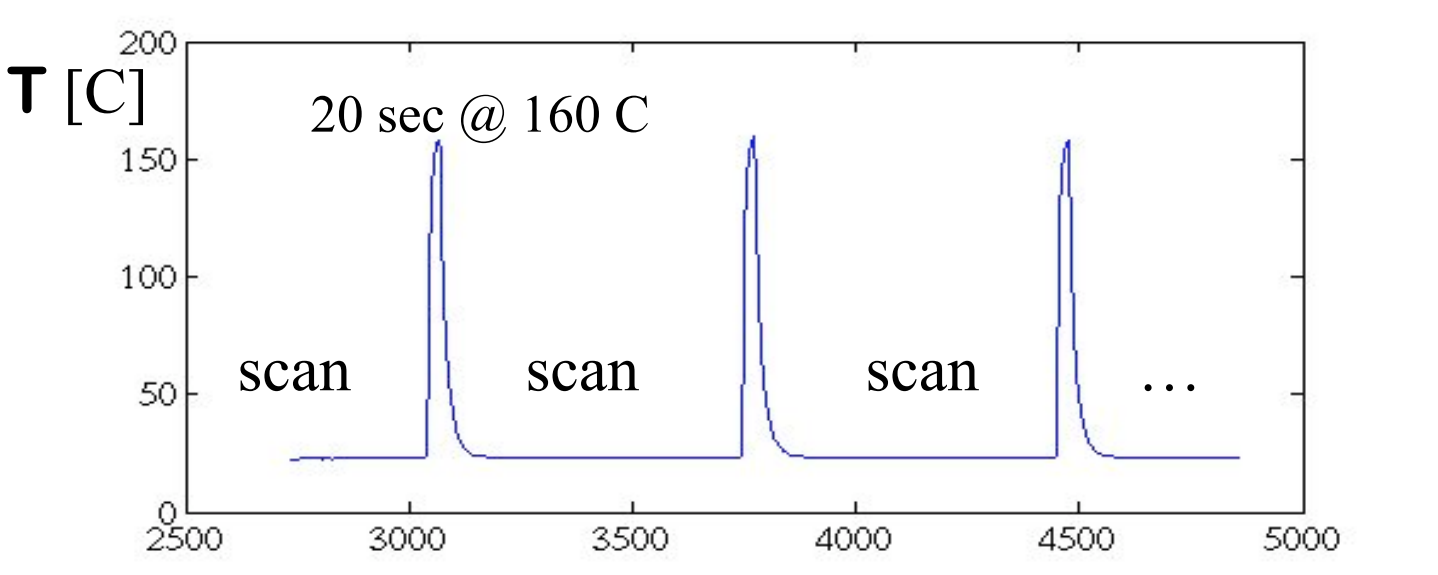
Direct beam

Topo-Tomography applied to recrystallisation (…first results)

sample: deformed Al single crystal

46 Topotomo scans (45 angles, 20 sec)

20 s @ 160 C annealing steps



Topo-Tomography applied to recrystallisation

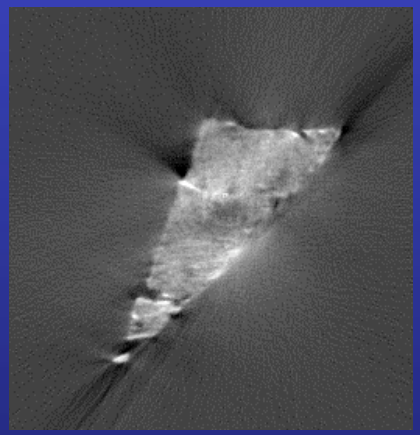
2D projection topographs

3D grain shape evolution during growth

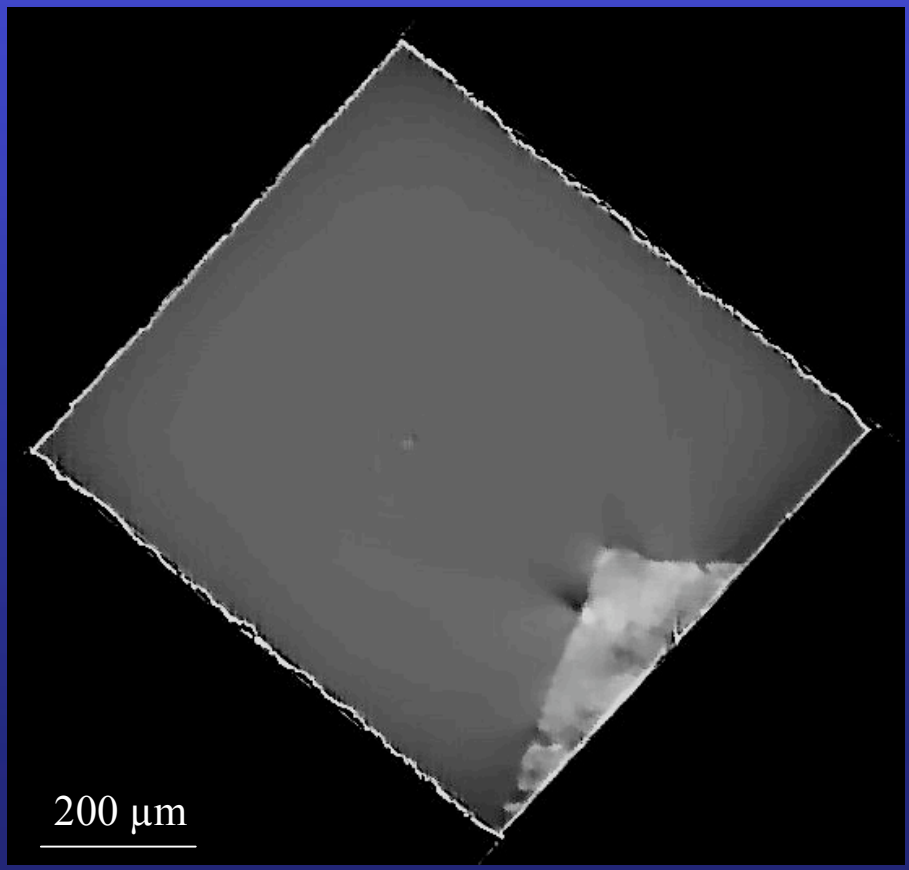
3D reconstruction
(diffracted beam)

ART reconstruction
45 projections

Tomographic reconstruction of final grain shape



diffracted beam



direct beam

Tomographic reconstruction of final grain shape

3D reconstruction
(diffracted beam)

FBP, 360 projections