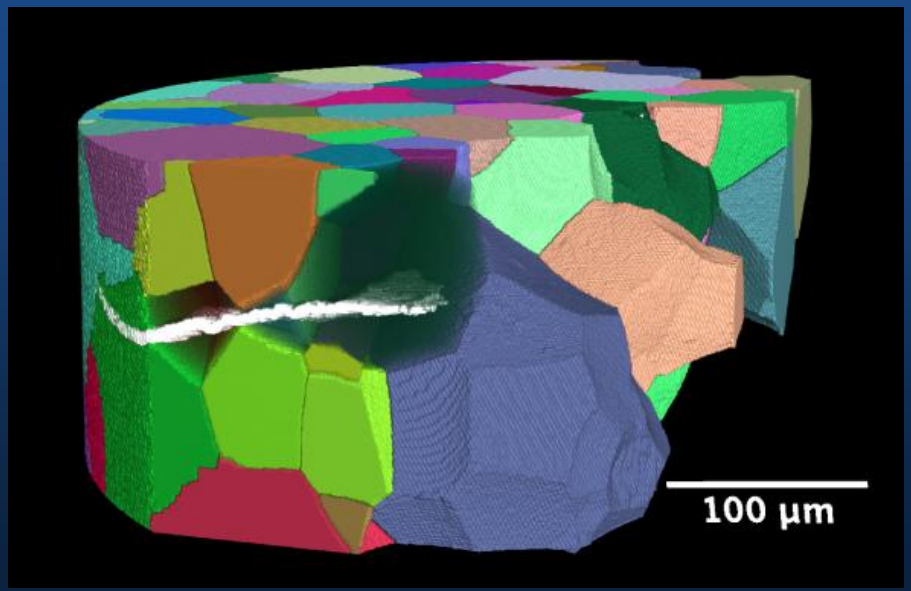
3D X-ray Diffraction Imaging Techniques

W. Ludwig, N. Vigano, A. King, P. Reischig, N. Gueninchault, H. Proudhon, M. Herbig, L. Nervo

(1) Mateis, INSA Lyon, (2) ESRF, Grenoble, (3) CWI, Amsterdam, (4) CdM Mines, Paris



Motivation

Study *evolution* of 3D grain microstructures during

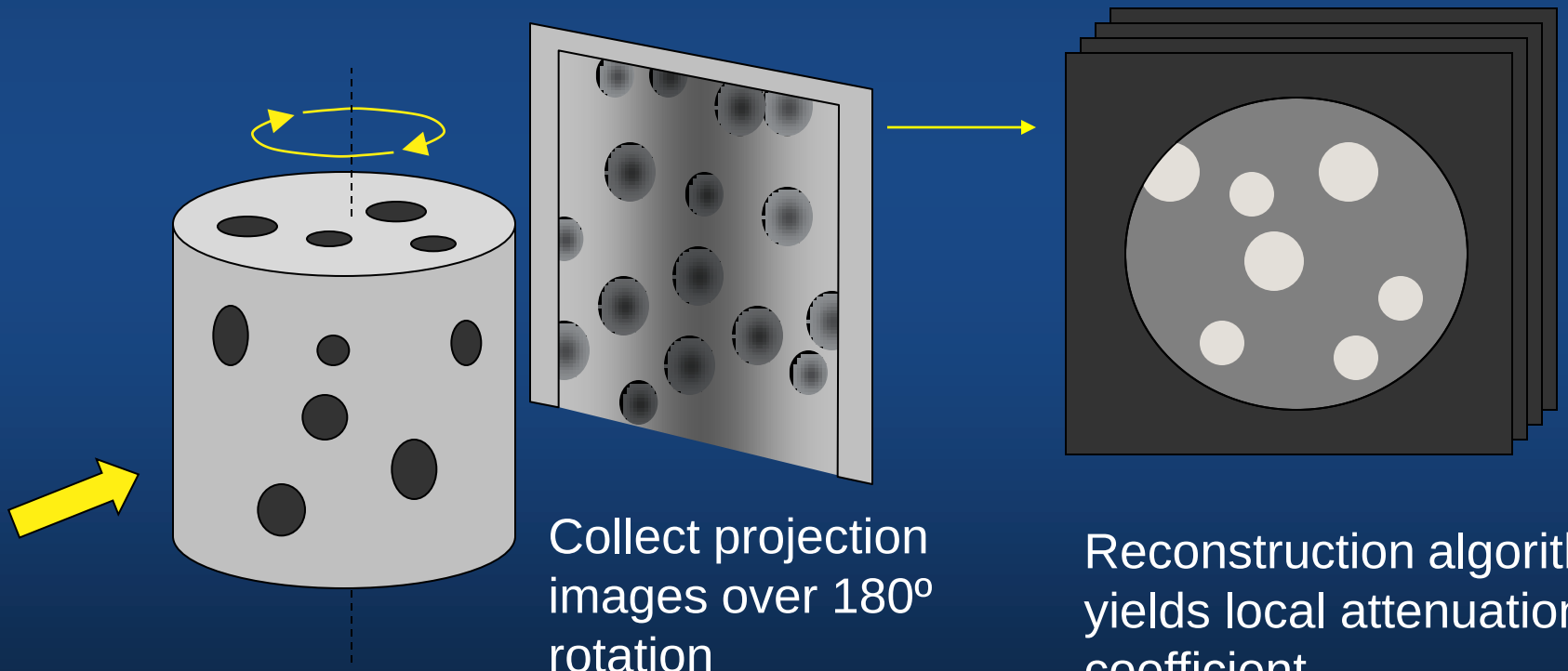
- deformation
- heat treatment
- corrosive environment

Comparison of experimental observations to numerical simulations:

→ test & improve models

Synchrotron X-ray microtomography

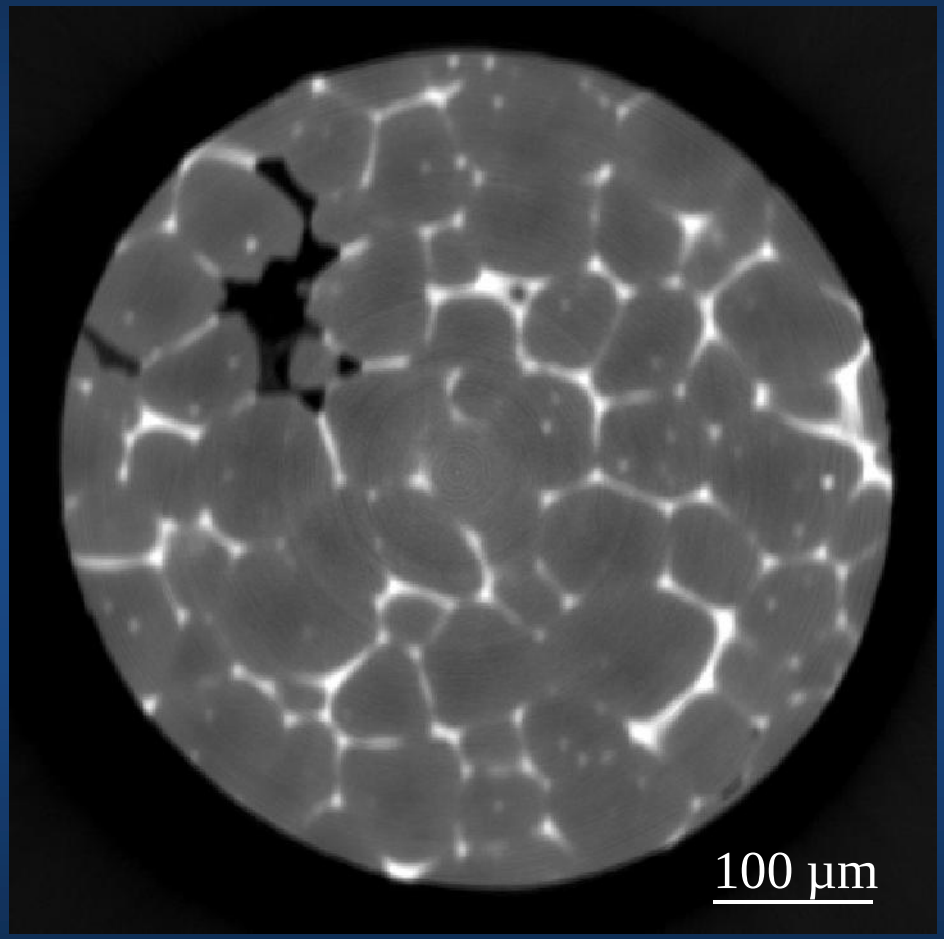
- based on absorption (phase) contrast
- multiphase materials, pores, cracks…



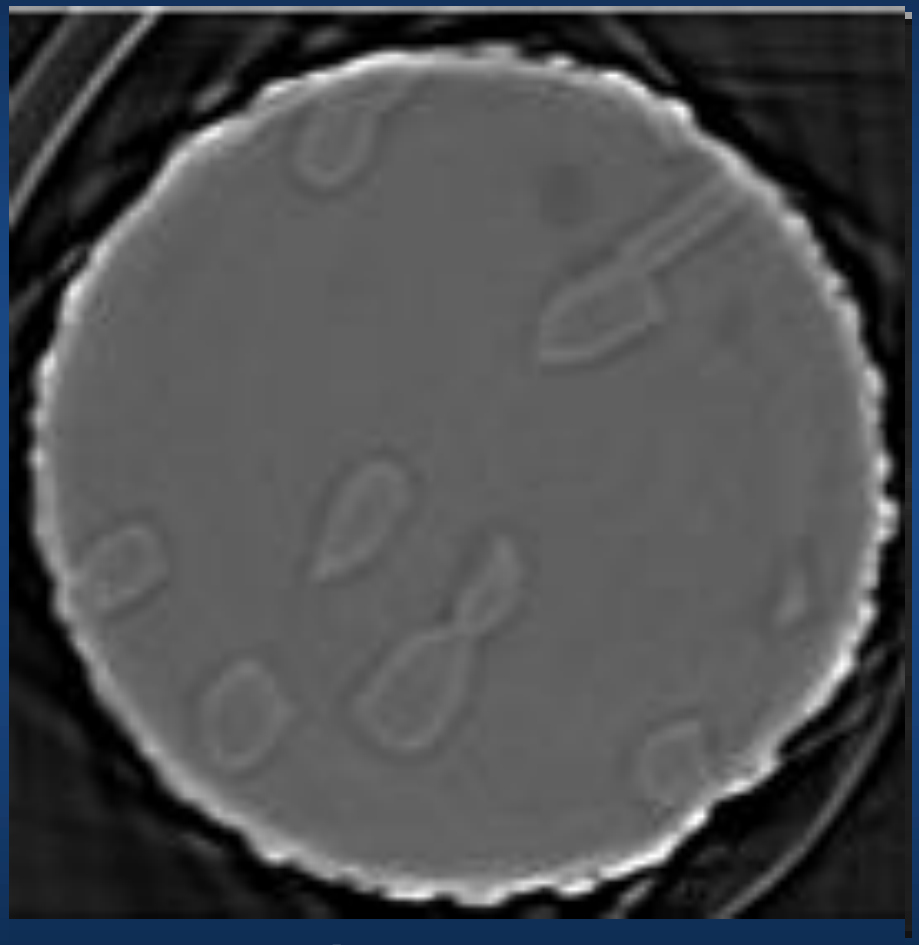
Collect projection images over 180º rotation

Reconstruction algorithm yields local attenuation coefficient

Characterization of grain microstructures by absorption or phase contrast tomography



Al-Cu alloy: after tensile test in the semi-solid state (L. Salvo, Simap)

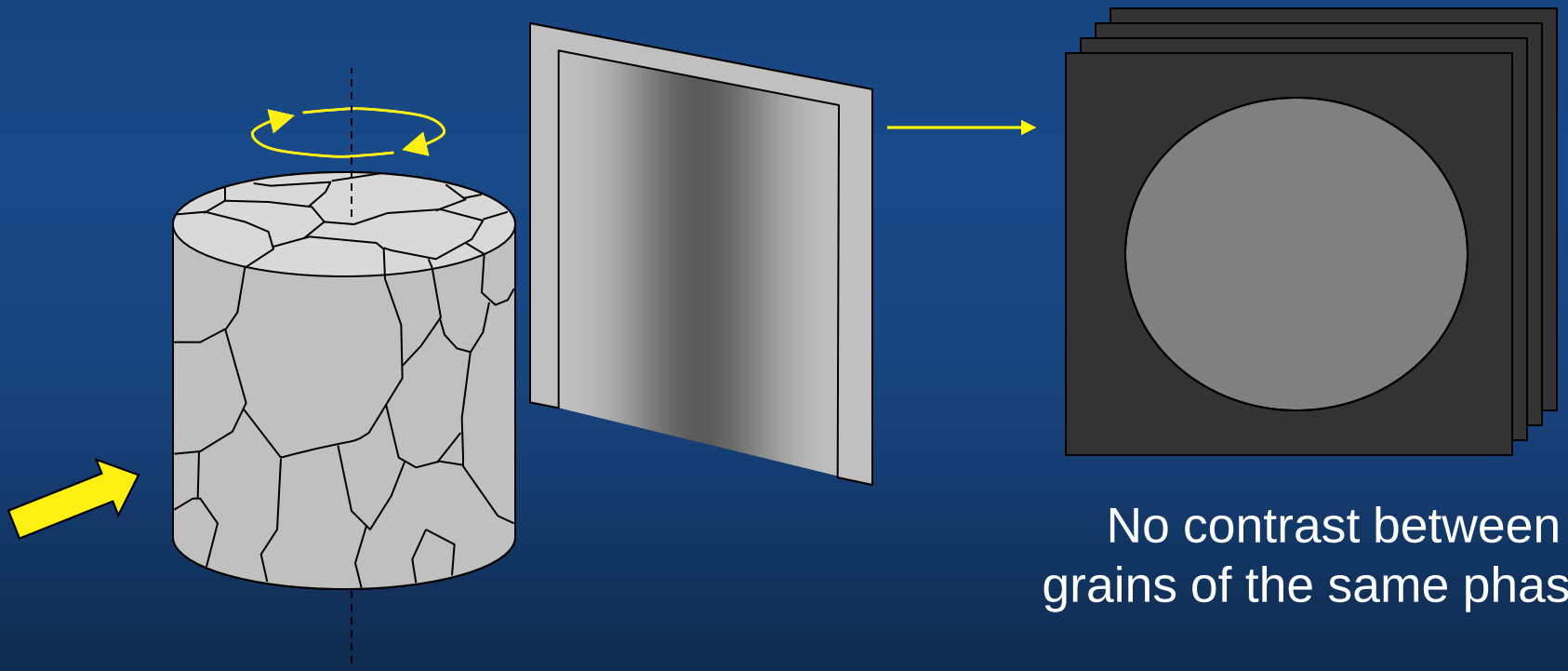


Phase contrast

$(\mu + \Delta\delta)$

Tomography of monophase materials

No crystallographic orientation or strain information !

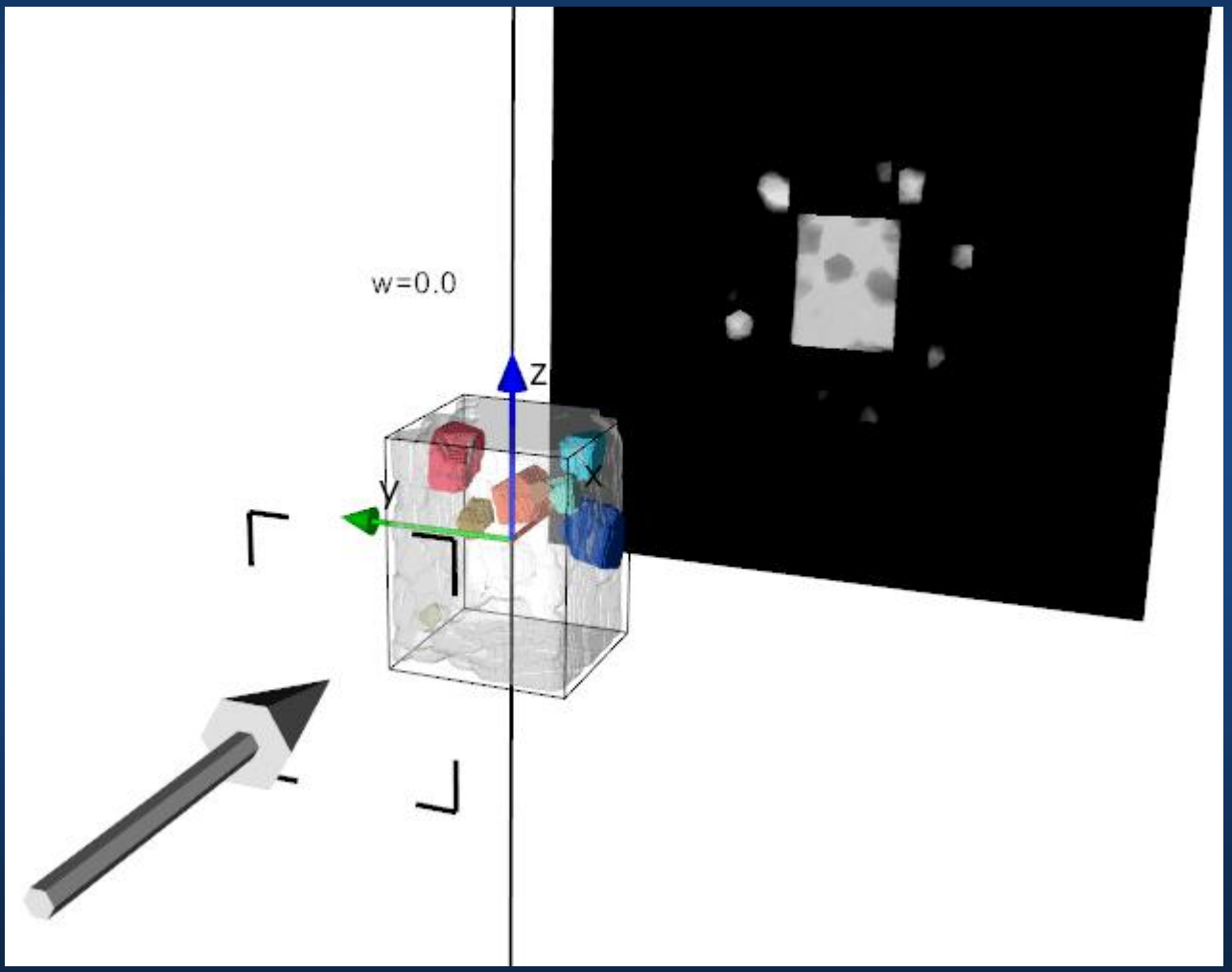


No contrast between grains of the same phase !

X-ray Diffraction Contrast Tomography

Non destructive characterization of 3D grain microstructures

(plastically undeformed, monophase materials)



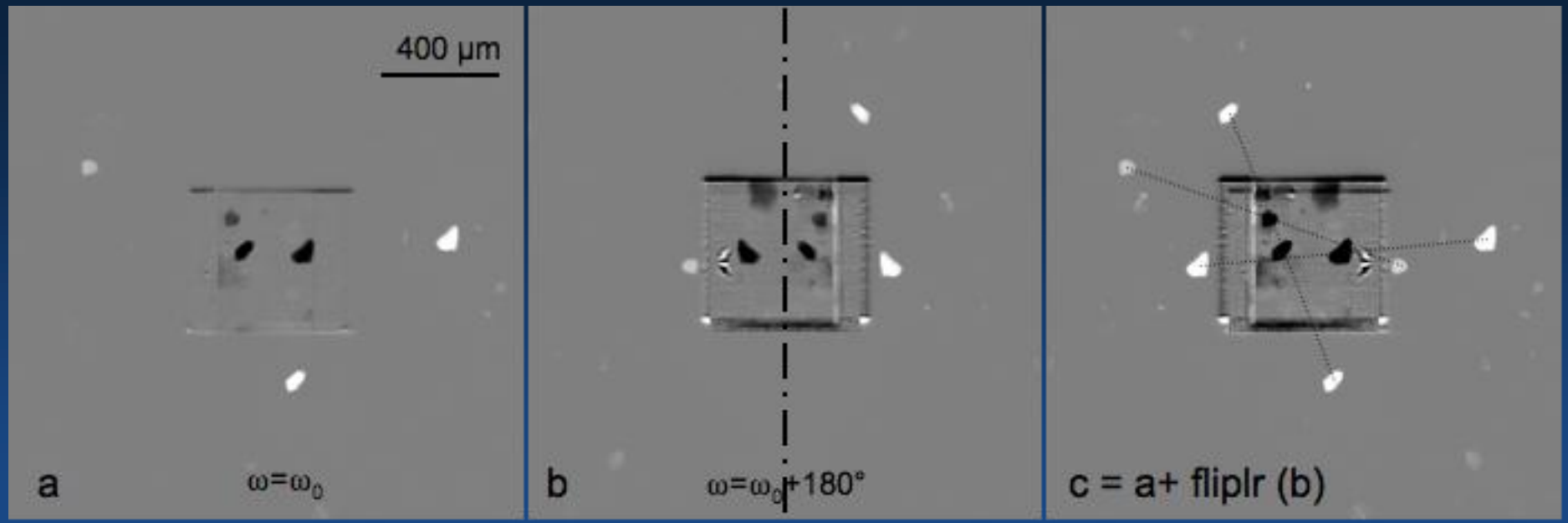
2D sample illumination with monochromatic beam

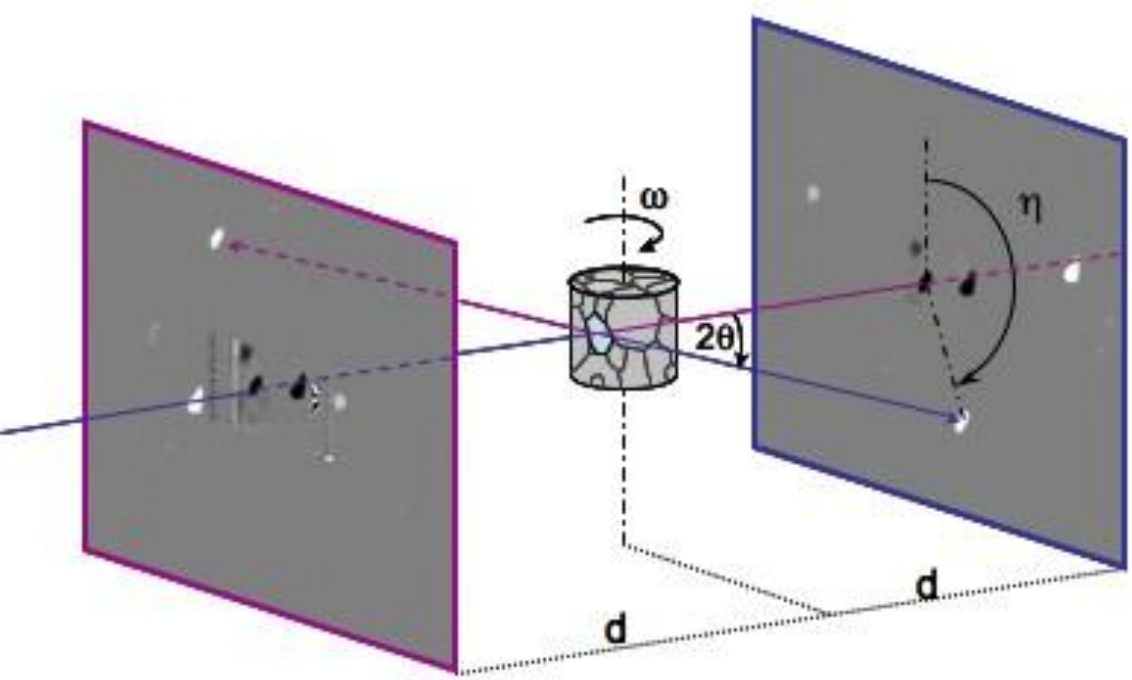
Continuous rotation over 360° typically in steps of 0.1°

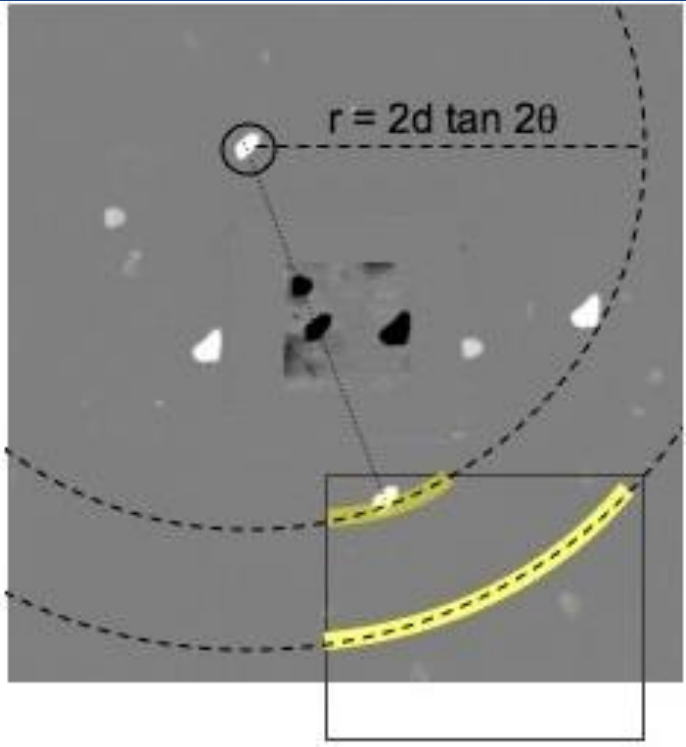
Simultaneous acquisition of transmitted and diffracted beam

Acquisition times : ~ 0.1 - 10 h

W. Ludwig et al., Rev. Sci. Instr. 2009, Reischig et al., J. Appl. Cryst. 2013

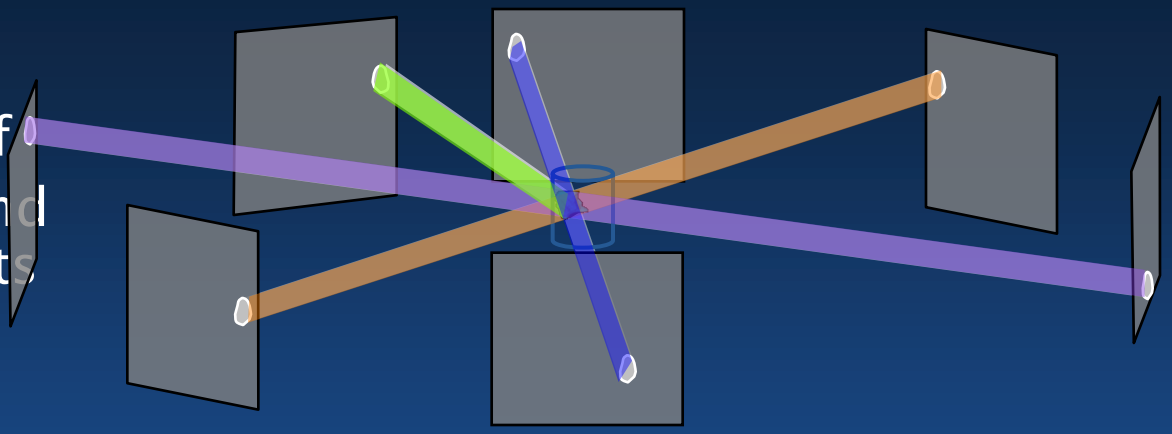

400 µm
a
ω=ω0
b
ω=ω0+180°
c = a+ fliplr (b)


ω
η
2θ
d
d

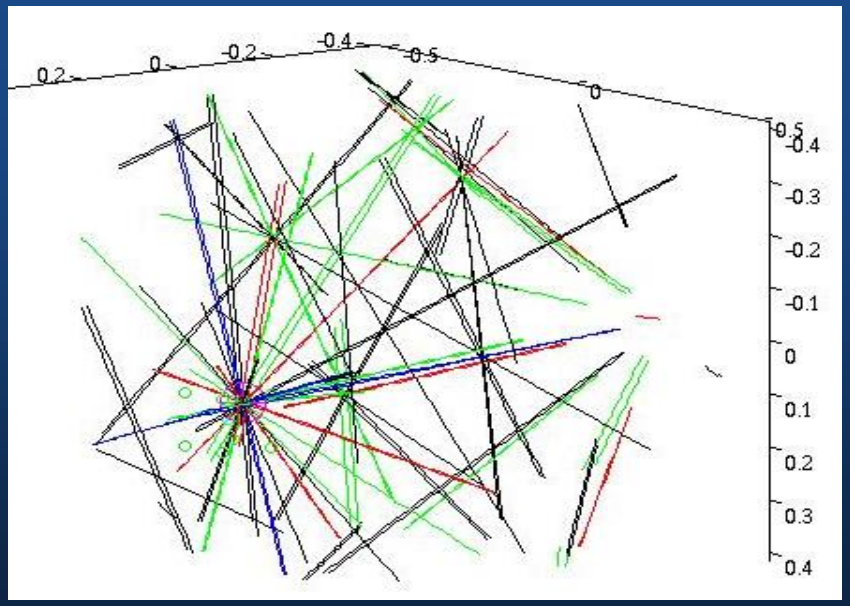

r = 2d tan 2θ

Indexing

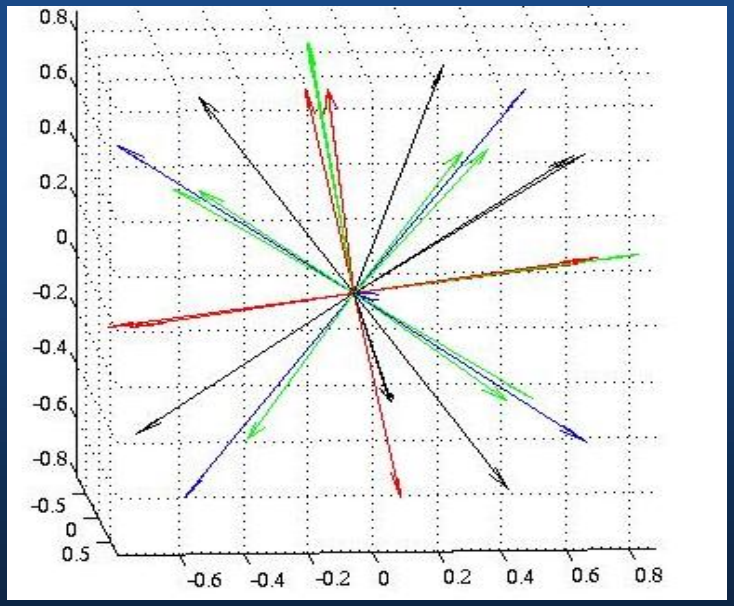
Based on a combination of shape, intensity, spatial and crystallographic constraints



Pairs from a given grain must have a common intersection point in real space and orientation space. Interplanar angles must correspond to those allowed by the spacegroup of the material.

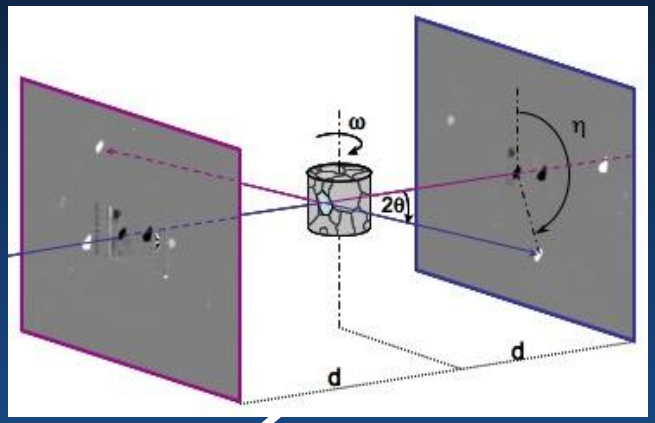


Rodrigues' space → orientation

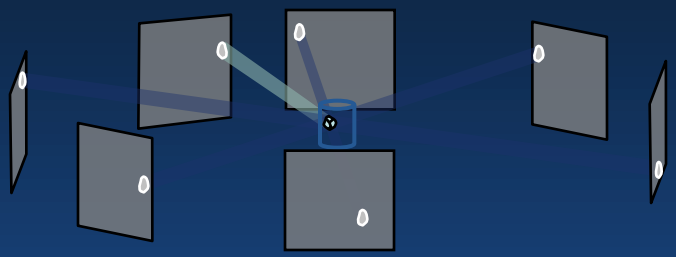


Scattering vectors show symmetry

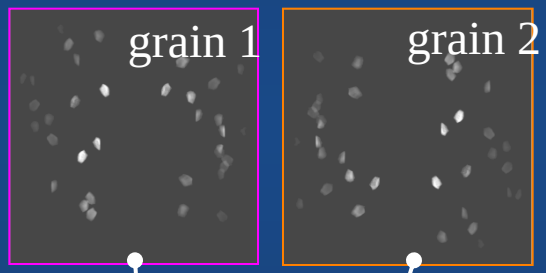
Pair Matching



Indexing

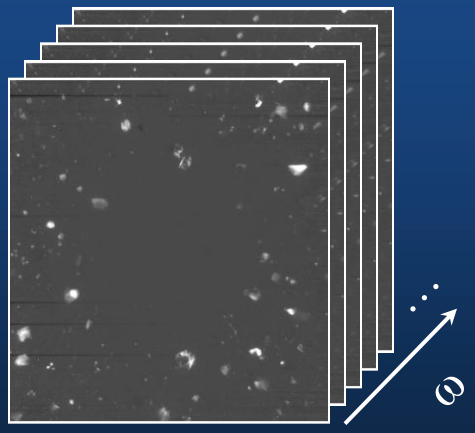


Reconstruction



Segmentation

Acquisition

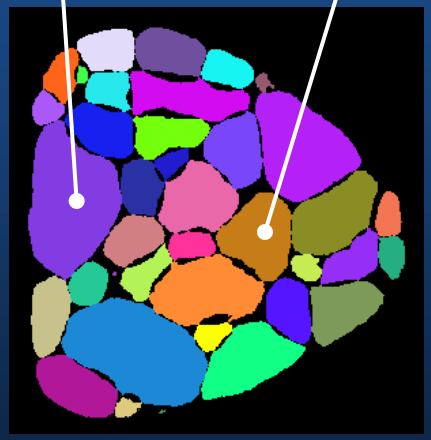


Raw data (7200 images)

~ 100.000 spots

principal steps of DCT data analysis

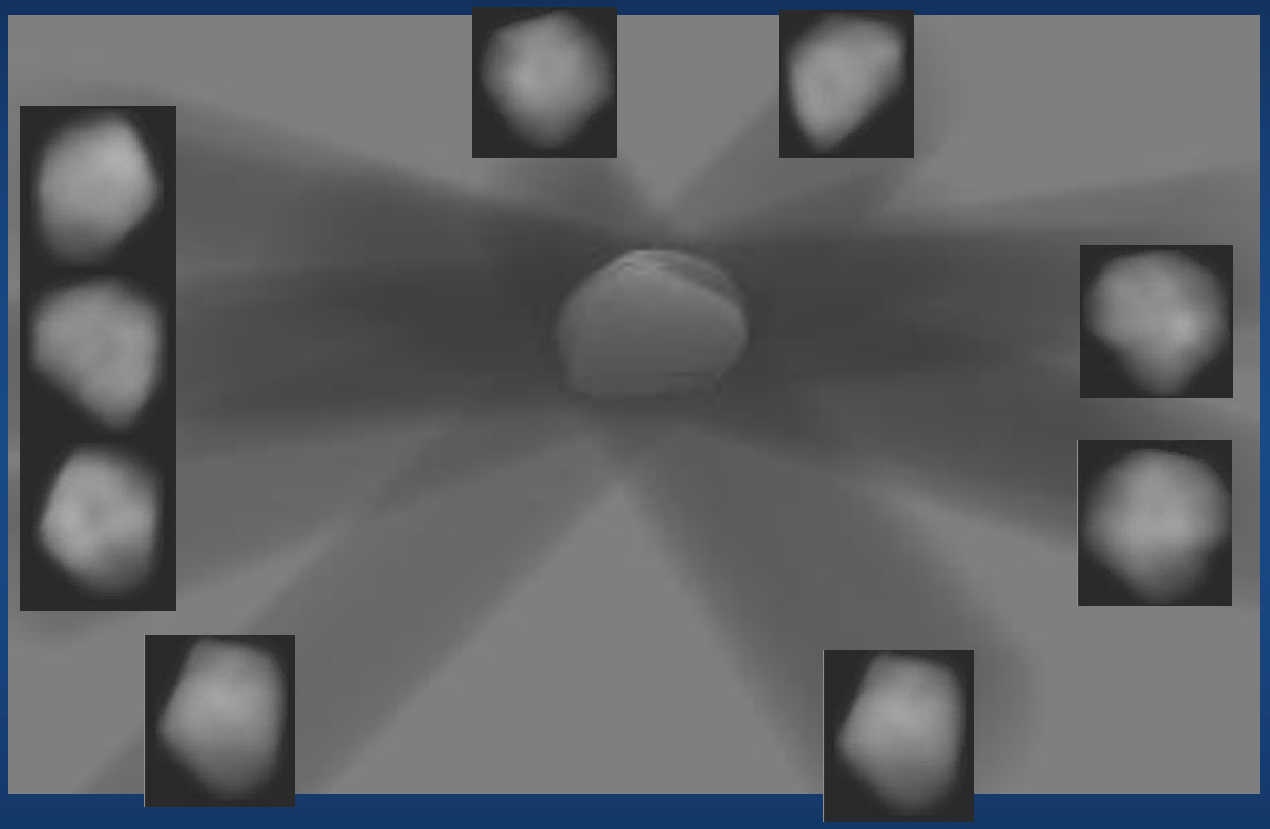
(~ 1 day / scan)



ART reconstruction

1000 grains

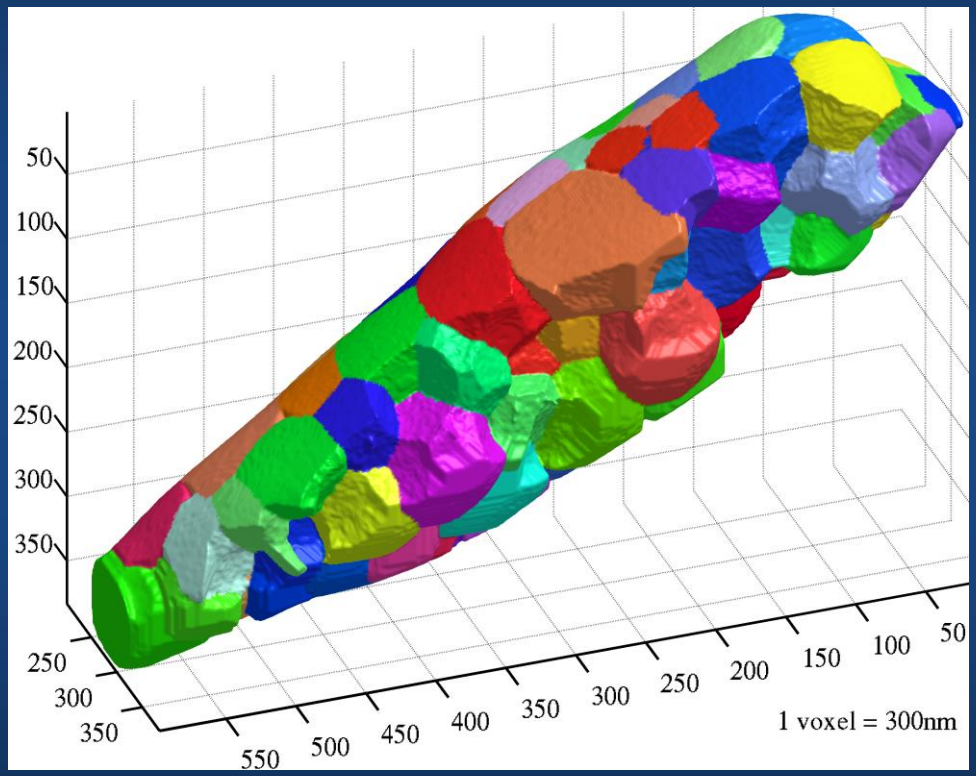
3D grain reconstruction from 2D projection images



3D SIRT algorithm Astra tomography library, Vision Lab, Belgium

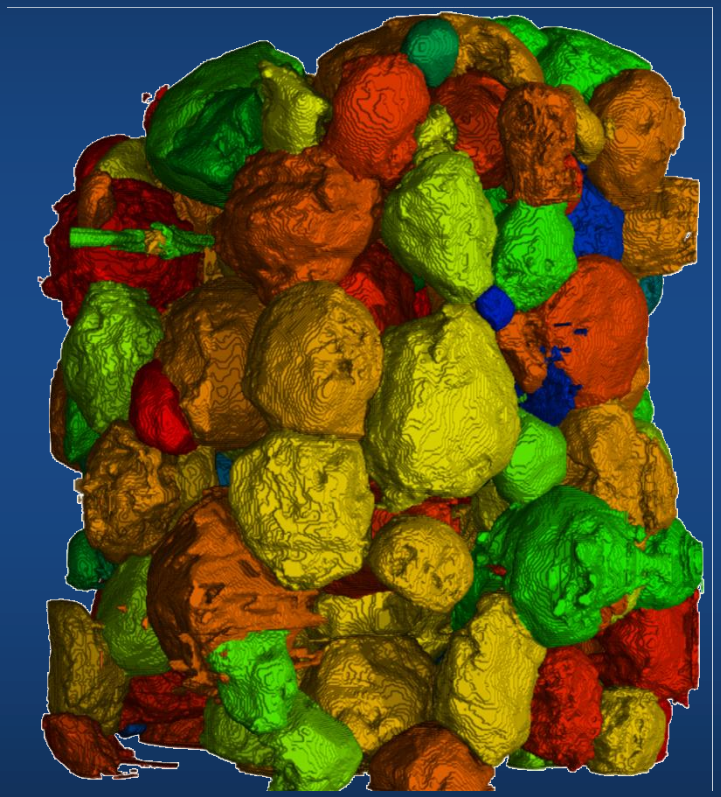
Spatial resolution & density : from UO_2 to H_2O

H. Palancher et al, CEA



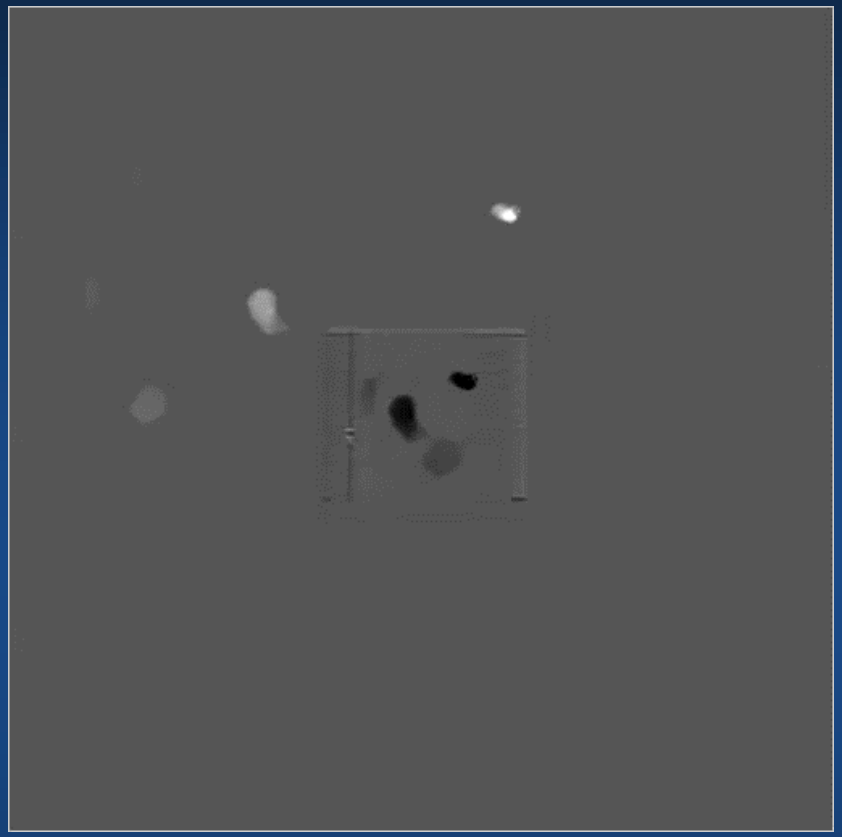
Voxel size: 0.3 µm
Average grain size: 14 µm
Acquisition time: 3 h (ID11)

J. Meyssonnier et al, LGGE

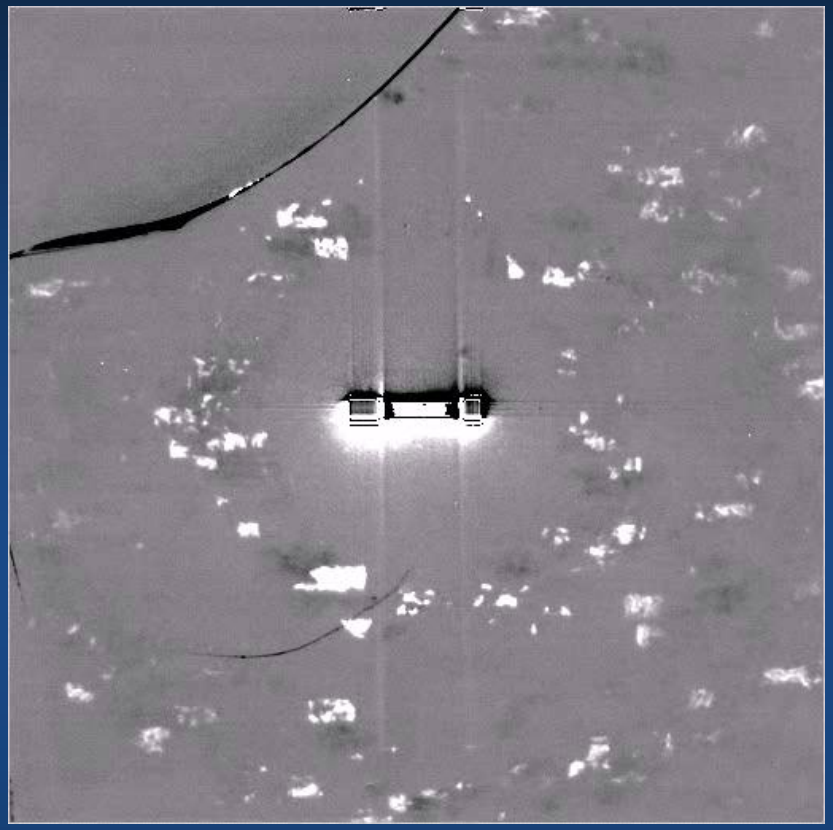


Voxel size: 30 µm
Average grain size: 1 mm
Acquisition time: 0.5 h (ID19)

What about less perfect / plastically deformed materials?



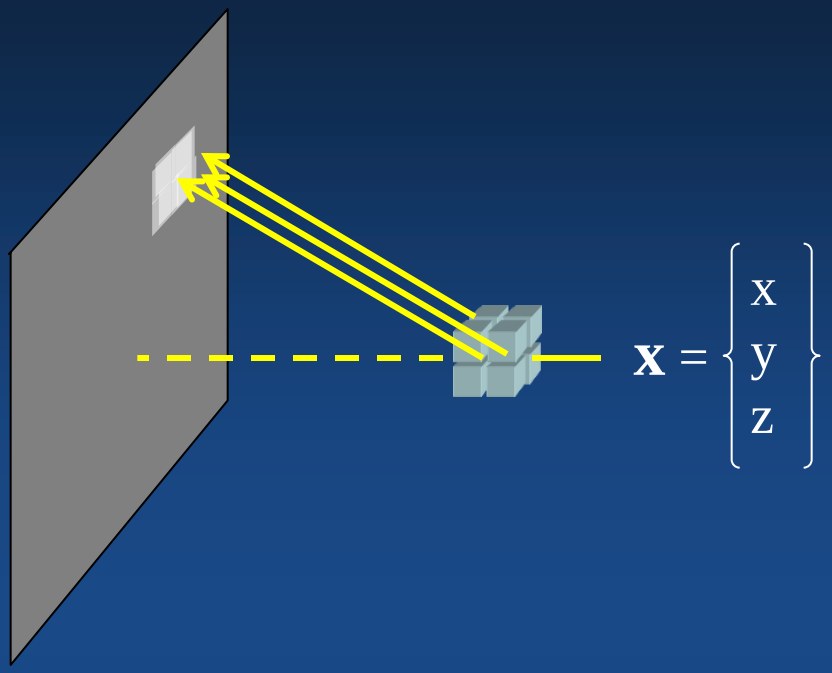
Ti alloy 55531
Rotation increment 0.1˚



Al alloy 2198 T8
Rotation Increment 1 ˚

Problem - spots no longer correspond to parallel projections
- increasing probability of spot overlap
- development of orientation ‘distributions’ and subgrain structures

3D reconstruction in undeformed material



$$\mathbf{x} = \begin{Bmatrix} x \\ y \\ z \end{Bmatrix}$$

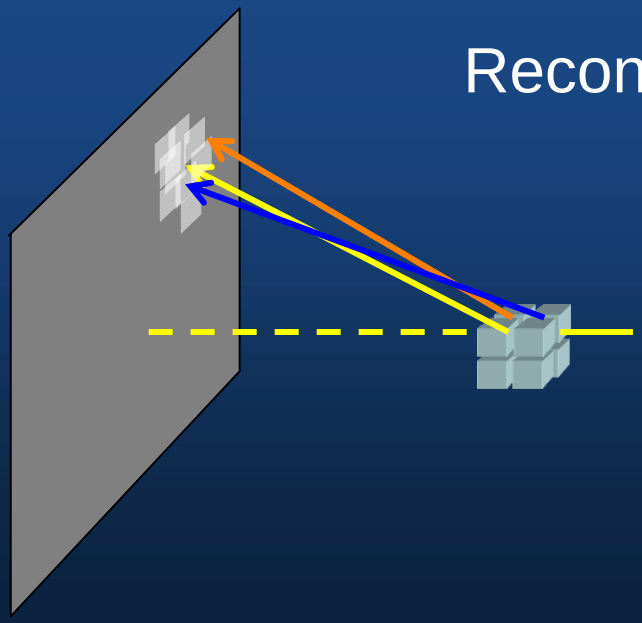
Same projection angle for all voxels

Simple (line) backprojection geometry

Can be handled as 3D (oblique angle)
ART problem: $\mathbf{A}\,x = b$

$(x \in R^3)$: scalar ~ "scattering power"

Reconstruction in deformed materials…



$$\mathbf{x} = \begin{Bmatrix} x \\ y \\ z \\ r_x \\ r_y \\ r_z \end{Bmatrix}$$

Describe local orientation (voxel)

- Vector field (r_x, r_y, r_z)

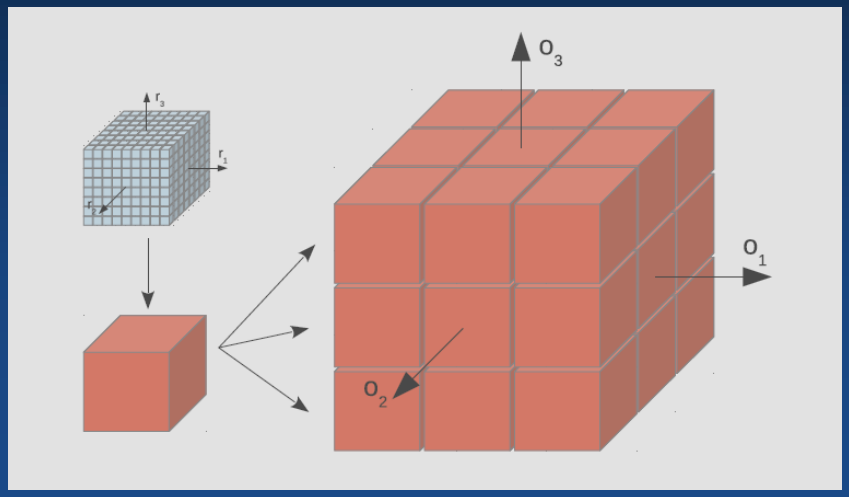
  Problem: $\mathbf{A}(x)\,x = b$: non-linear

- discretized, 3D orientation distribution

$(x \in R^6 = R^3 \otimes O^3)$

Reconstruction in a 6D position and orientation space

PhD thesis of Nicola Vigano (INSA Lyon); Vigano et al., J. Appl. Cryst, **47** (2014)



Discrete representation of the 6D reconstruction space as a collection of 3D real space volumes

$$A\underline{x} = A_1\underline{x}_1 + A_2\underline{x}_2 + \ldots + A_N\underline{x}_N = \sum_i^N A_i\underline{x}_i = \underline{b}$$

We have increased the number of unknowns by a factor of ~1000 (!)

The problem is heavily underdetermined

Need prior to select physical meaningful solution

Assumptions: kinematic scattering
neglect absorption, extinction,…

Prior (TV min): constant scattering power per real space voxel

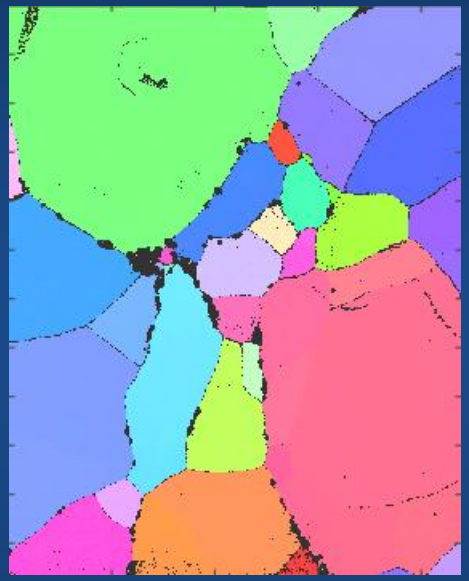
$$\underline{x}^* = \arg\min_{\underline{x}} \{\|\mathbf{A}\underline{x} - \underline{b}\|_2^2 + \lambda \mathrm{TV}(\mathbf{S}\underline{x})\}$$

$$\text{subject to} : \underline{x} \geq \underline{0}$$

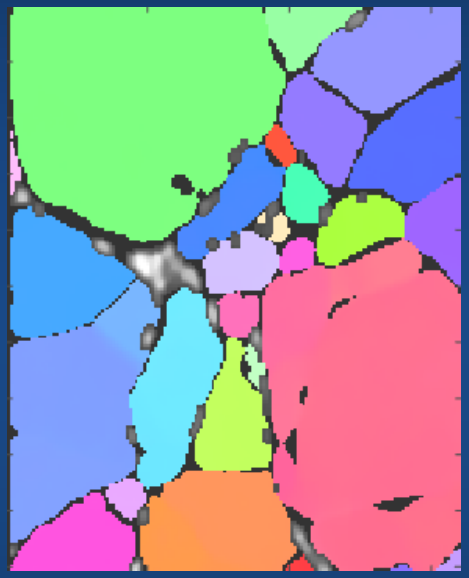
TV : total variation operator in real space
S : Sum over orientation space

Validation of the 6D reconstruction framework against EBSD

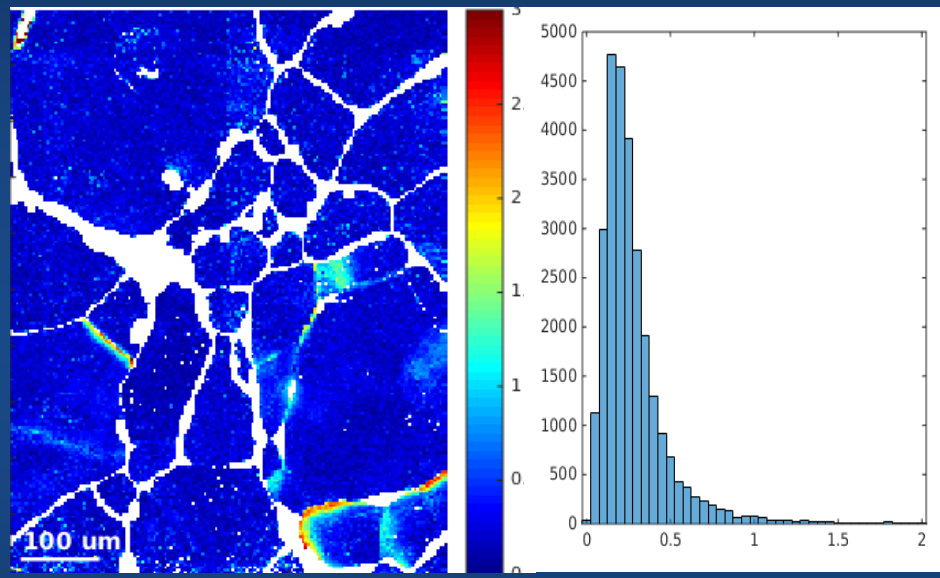
Material : NaCl sample containig fine disperion of Cu particles



EBSD



6D-DCT
(extended algorithm)

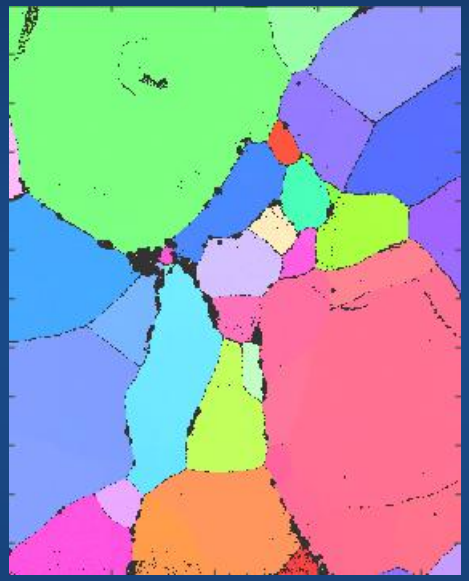


relative misorientation

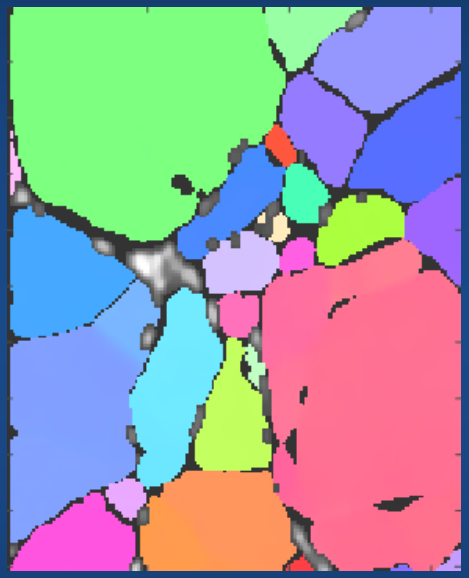
N. Vigano et al., Scientific Reports, 6 (2016)
N. Vigano et al., J. Appl. Cryst., 47 (2014)

Validation of the 6D reconstruction framework against EBSD

Material : NaCl sample containig fine disperion of Cu particles



EBSD



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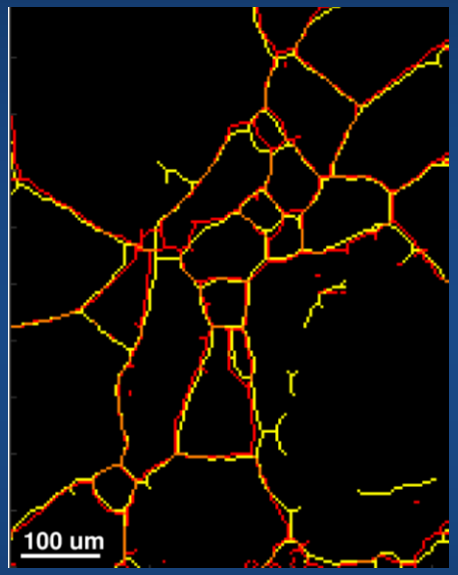
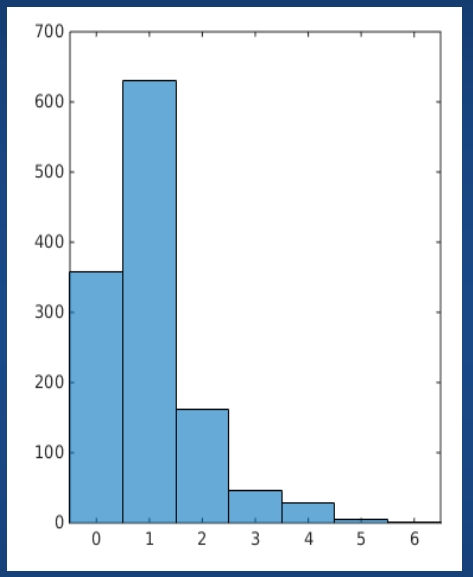
(extended algorithm)

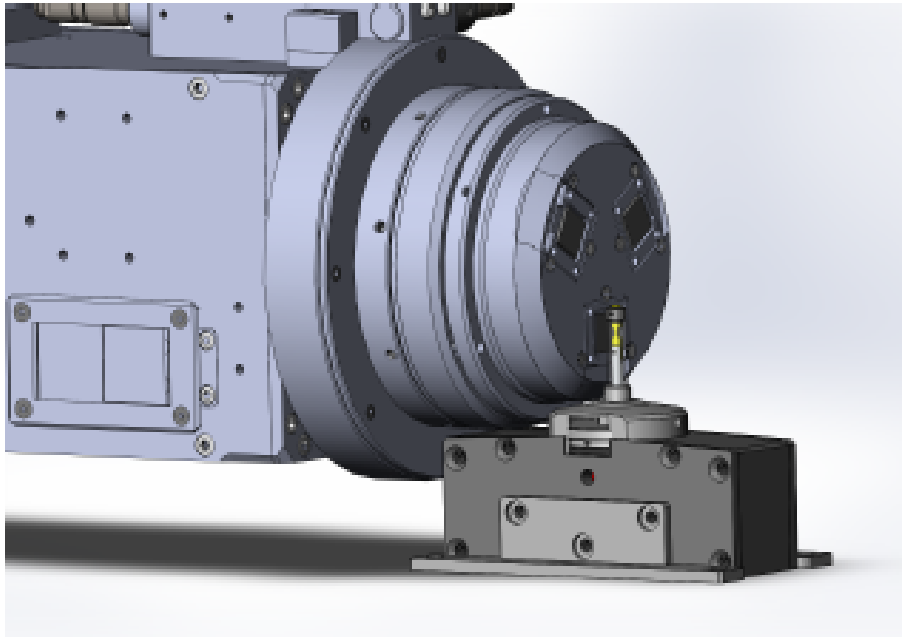




distance from grain boundary (pixel)

N. Vigano et al., Scientific Reports, 6 (2016)
N. Vigano et al., J. Appl. Cryst., 47 (2014)

Nanox : an in-situ stress rig dedicated to synchrotron radiation imaging



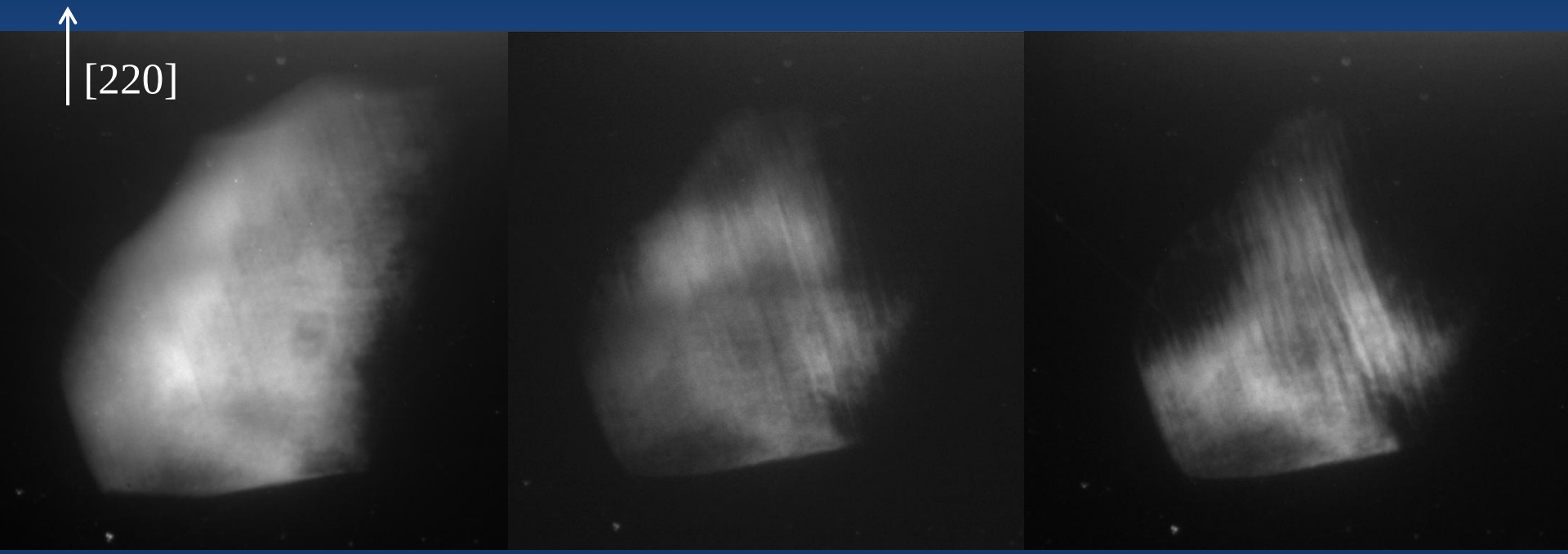
(Guéninchault et al., 2016)

Characteristics :

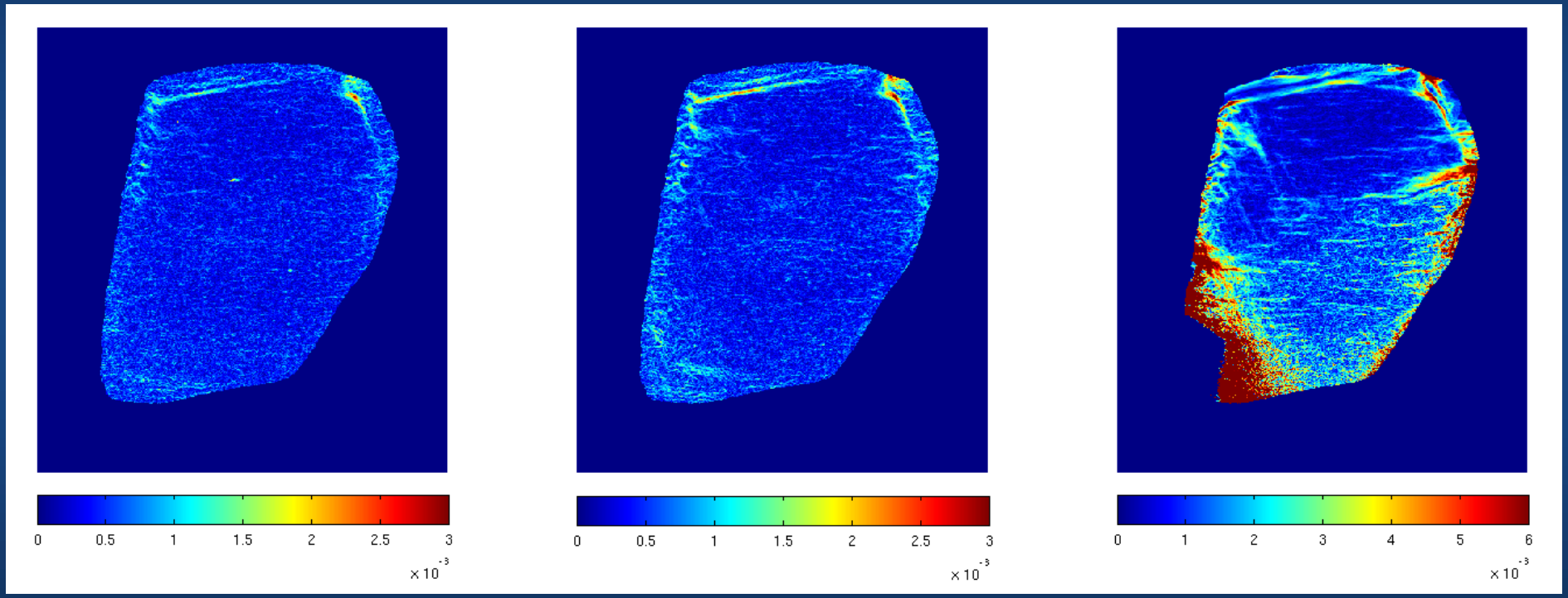
- up to 650 N and 150µ m displacement
- tensile and fatigue experiment up to 50 Hz
- allow 360°rotation for full visibility during tomographic imaging
- load precision and repeatibility $\sim$ 1 N
- high resolution imaging detector may approach the rotation center as close as 3 mm
- designed to fit into ID11 4 circles diffractometer

Evolution of topographic contrast during tensile loading

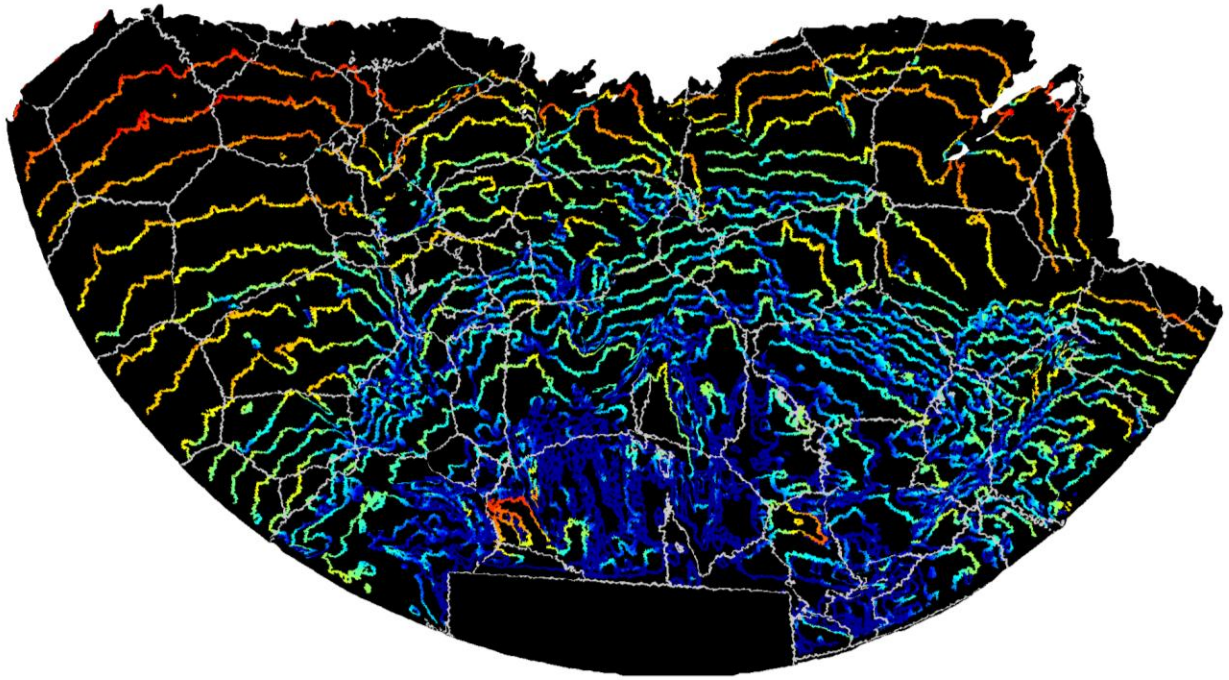
Projection topographs revealing the presence and multiplication of slip bands

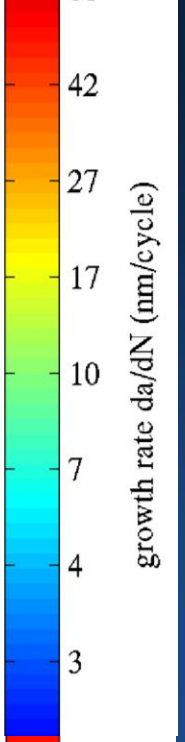


Topographic contrast from slip bands

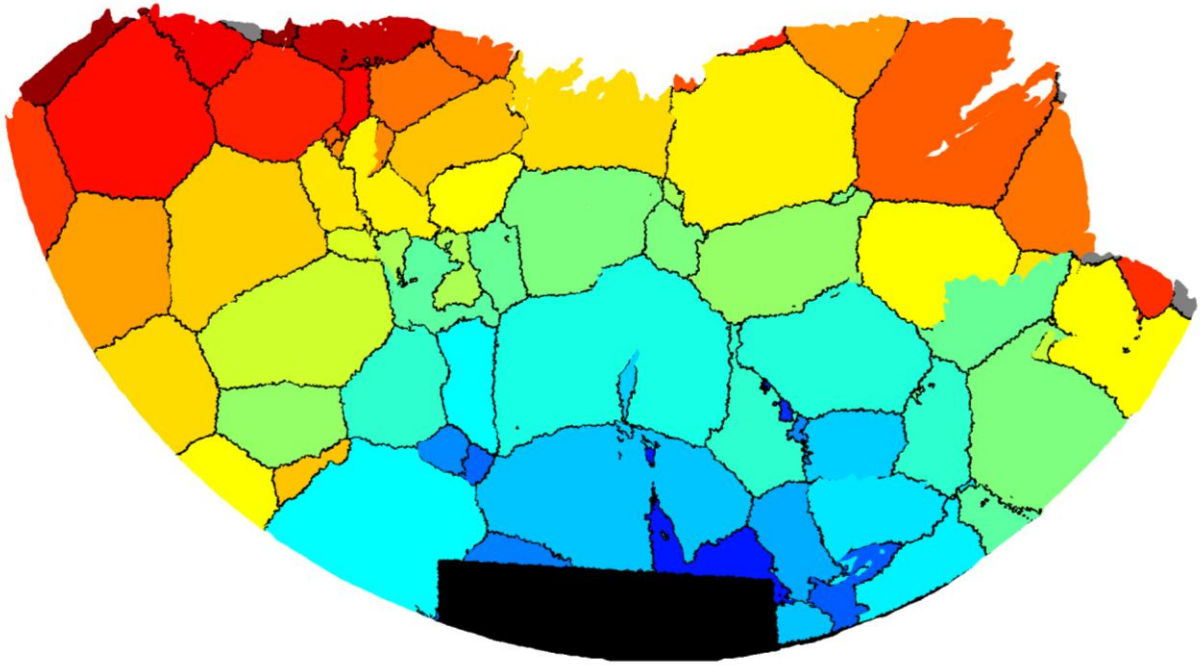


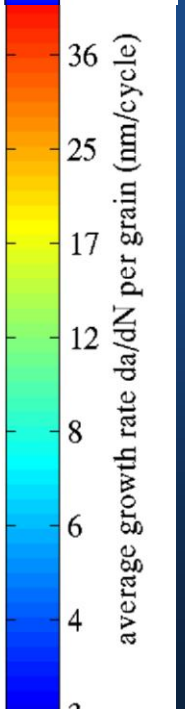
Tomographic reconstruction -





Local crack growth rate da/dN

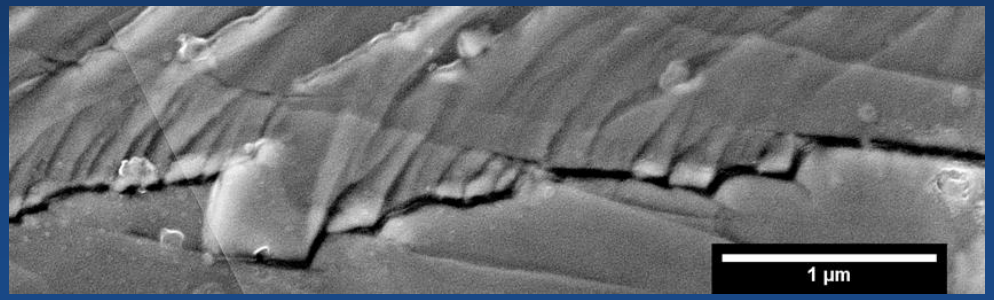




Average crack growth rate per grain

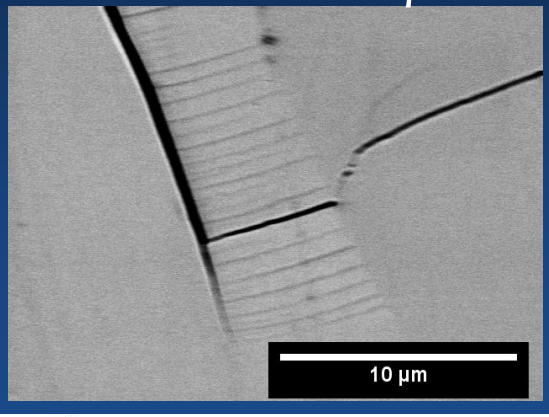
High spatial resolution is crucial for interpretation

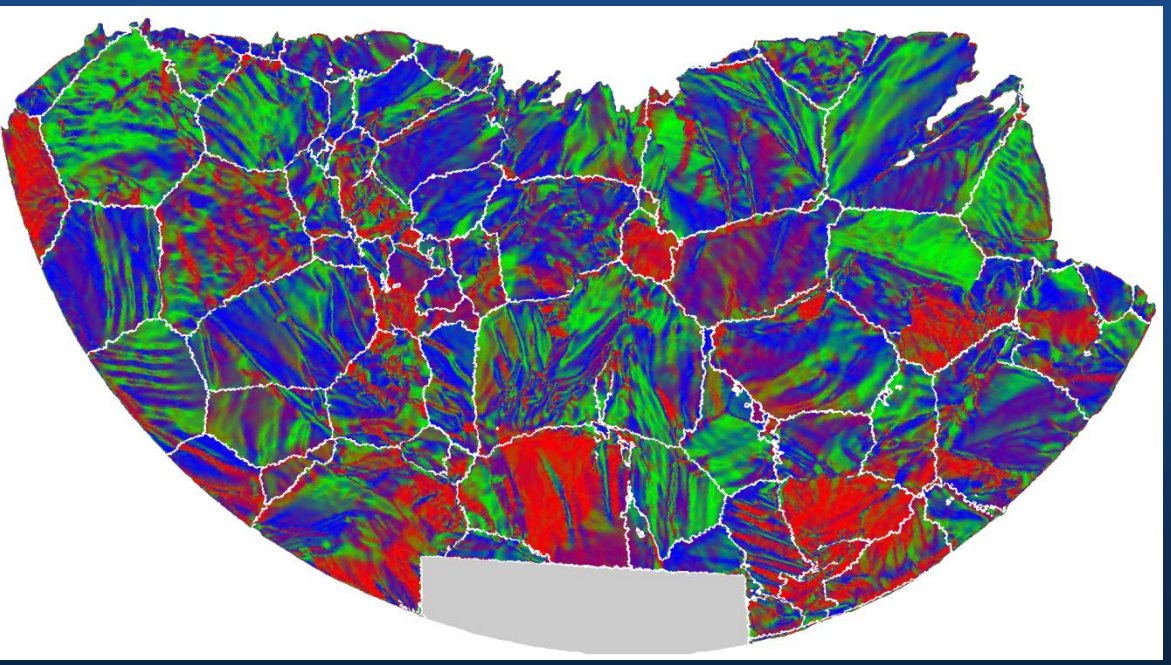
Ti alloy 21S: facets not resolved in µ-CT

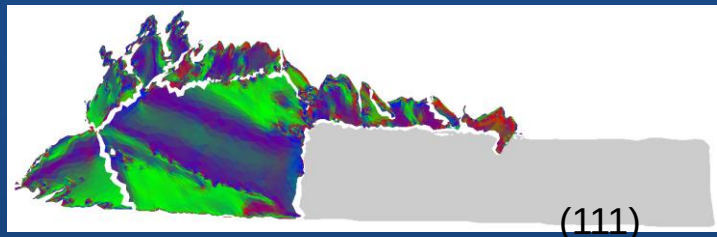


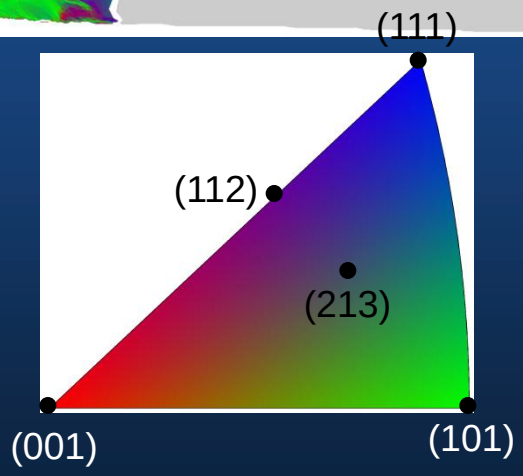
FEG - SEM

Ti alloy 55531: facets well resolved in µ-CT









Conclusions

- Motivation: development of characterization techniques which provide insight on deformation and damage mechanisms in polycrystalline materials

- Different approaches established so far (µ-Laue, 3DXRD, DCT, …)
  choice is problem dependent (resolution / speed / access to local orientations)

- X-ray diffraction contrast tomography:
  - *combination of imaging and diffraction mode*

- Analysis of fatigue crack propagation mechanisms in bulk of polycrystals
  - crystallographic propagation of stage I fatigue cracks

- Extensions of the methodology are under investigation with goal to
  - improve spatial resolution
  - access local orientation & strain inside grains

Valuable input for comparison with numerical simulations based on real microstructure (crystal plasticity, grain growth, crack propagation, …)

Acknowledgements

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Henning Friis Poulsen
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Université de Grenoble:
J. Meyssonnier, A. Philipe

TU Delft:
Sybrand van der Zwaag,
Erik Offerman

Mines Paris
Henry Proudhon, Samuel Forest,
Nicolas Gueninchault