FEMaS tutorial, Rosenheim, May 9, 2011

Application of Synchrotron-based Techniques for Fusion Materials

Henning Friis Poulsen
Center for metal structures in four dimensions
Risø DTU

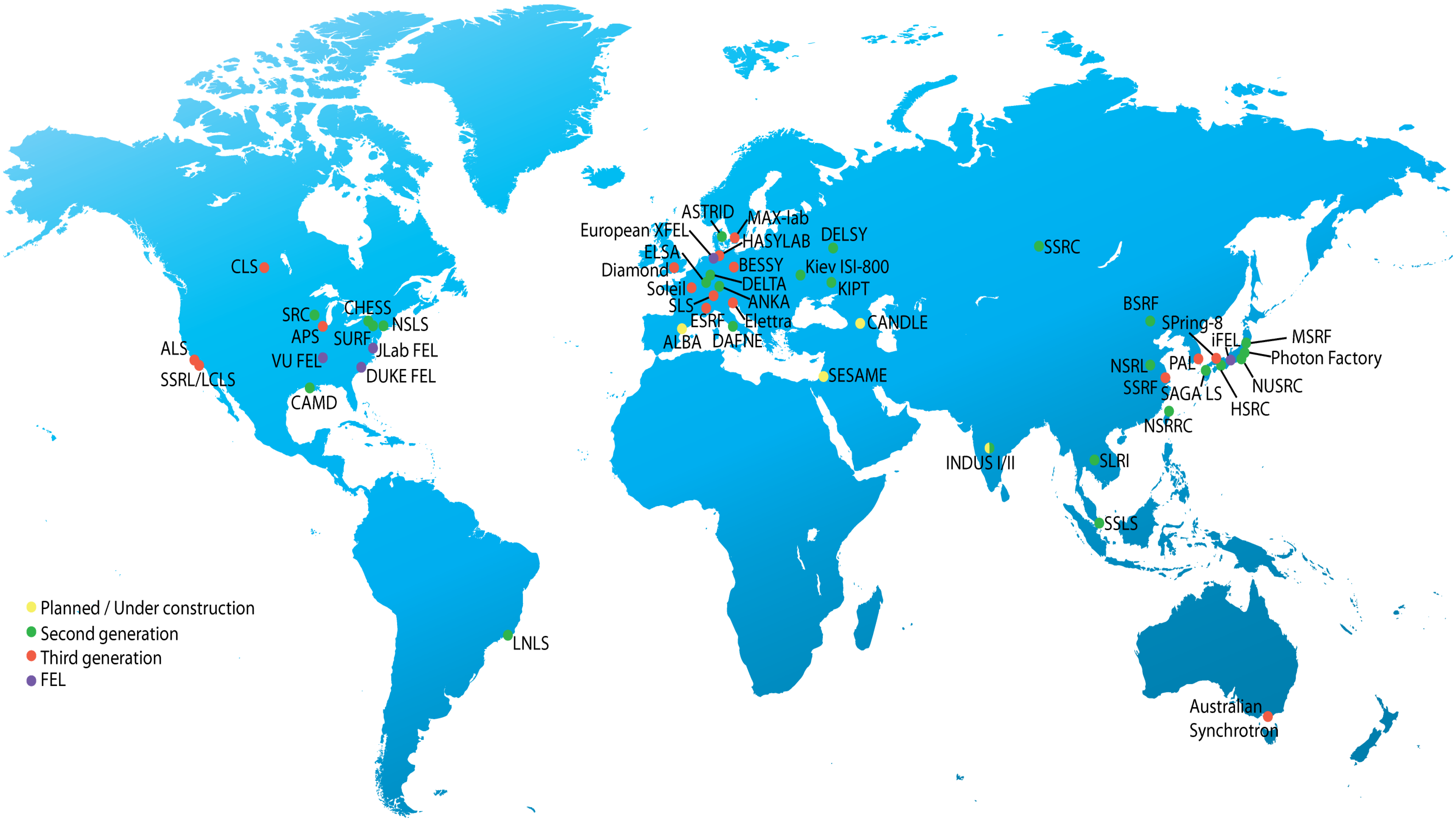
General:
Risø: D. Juul Jensen, E.M. Lauridsen, A. Lyckegaard, L. Margulies, J. Oddershede, U.L. Olsen, W. Pantleon, S. Poulsen, S. Schmidt, H.O. Sørensen, G. Winther

ESRF, Grenoble: A. King, W. Ludwig, P. Reischig, G. Vaughan, J. Wright

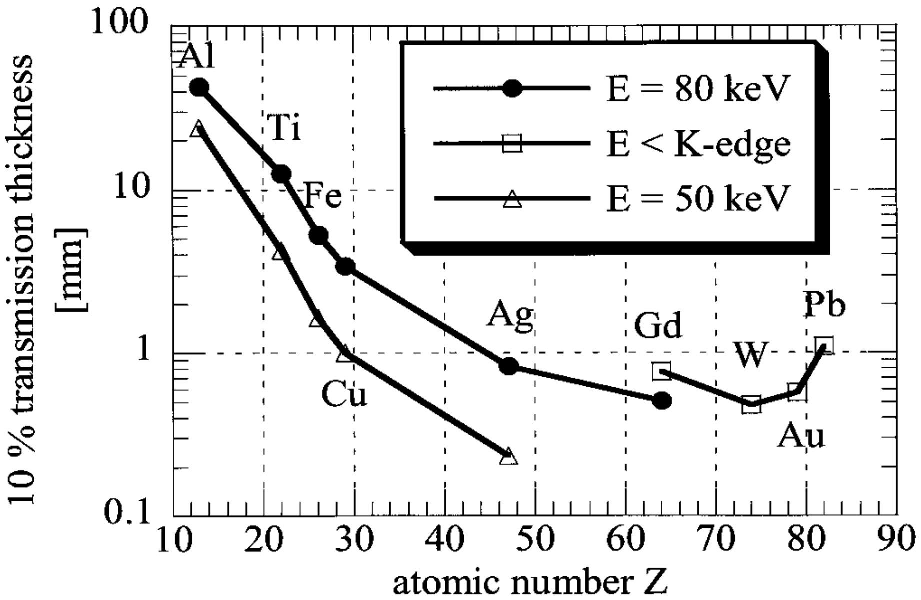
Fusion materials:

Slides by **Christian Linsmeier, IPP**

Map of synchrotrons



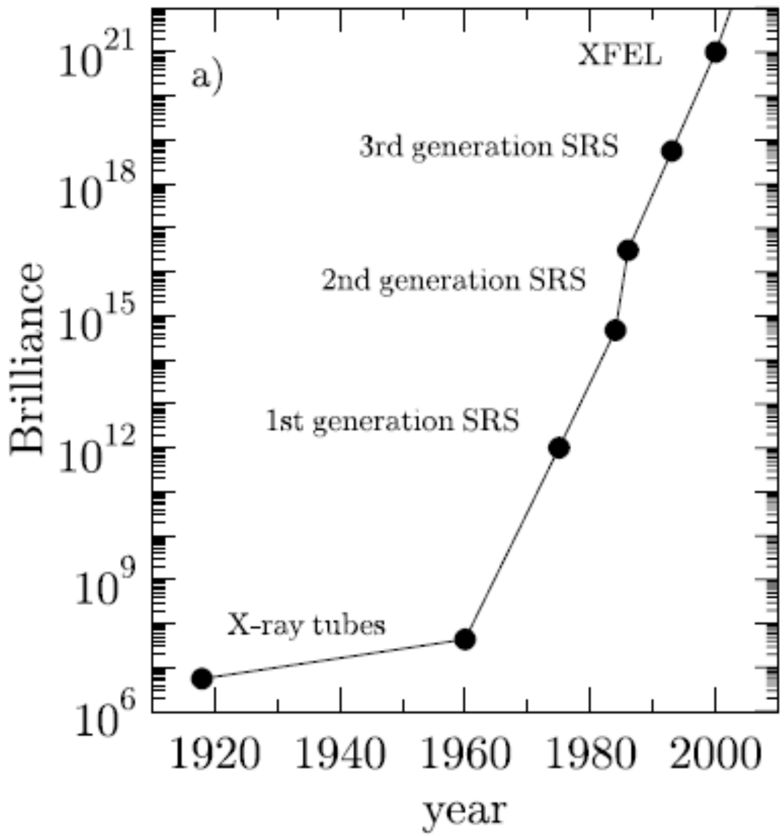
Hard x-rays



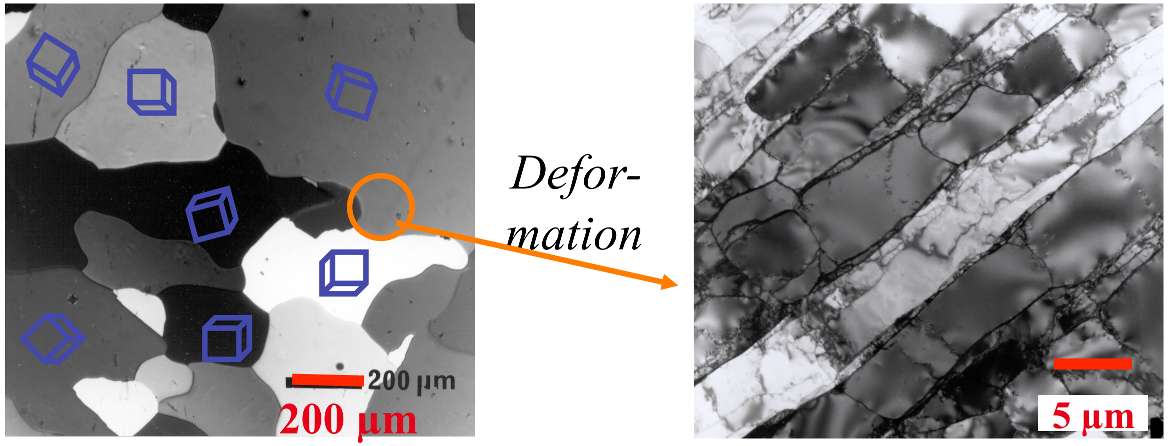
50 - 100 keV x-rays:

- \+ *Penetration*
- \+ *Sample surroundings*
- \+ *No absorption and extinction*

Brilliance



Materials science in 2D



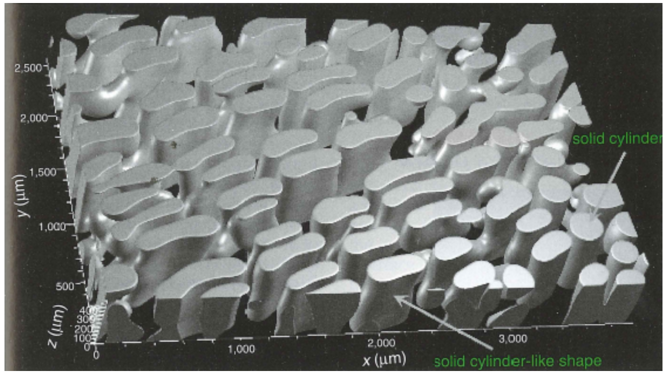
Problems:

- *Objects are 3D*
- Limited statistics, heterogeniety
- Cannot predict the dynamics

Materials science in 3D

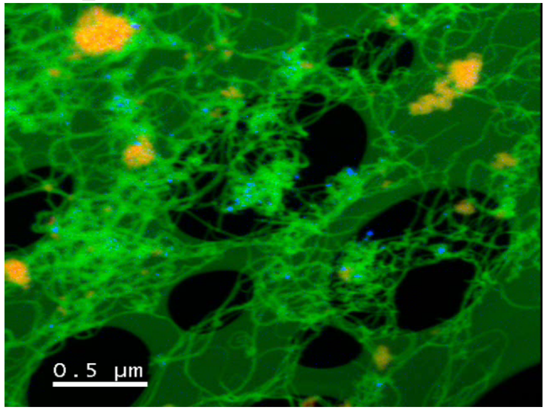
Sectioning + optical microscopy

Sample 1cm, Res: 2 µm



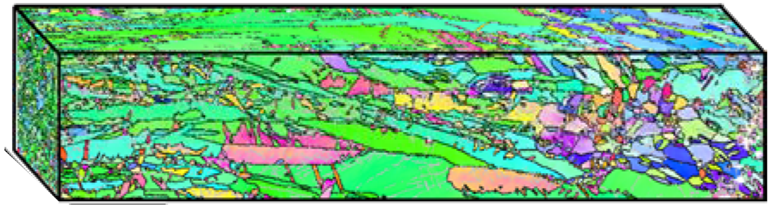
TEM tomography

Sample 500 nm. Res 5 nm



FIB+EBSD:

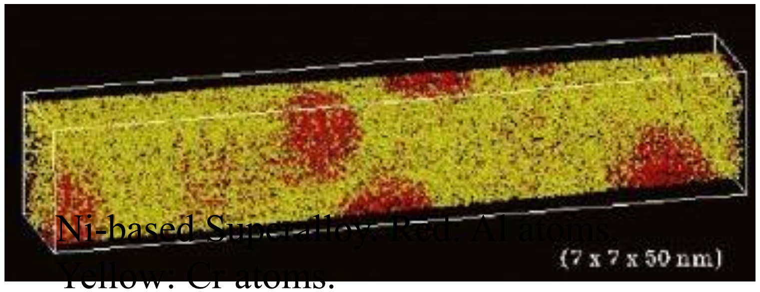
Sample 20 µm, Res: 30 nm



10 µm

3D Atom Probe

Sample 30 nm. Res 1 Å



Ni-based Superalloy. Red: Al atoms. Yellow: Cr atoms.

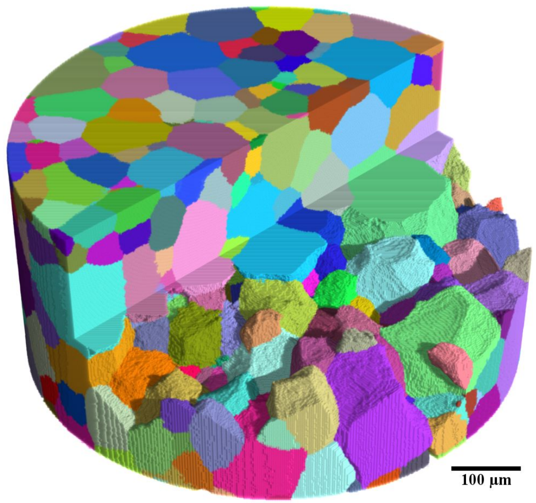
Metal Structures in 4D

Aim: 4D Movies

Crystalline materials
for each grain:

- Phase
- Center-of-mass & morphology
- Orientation of lattice
- Elastic strain (stress)
- Plastic strain

- Defect populations
- Dislocation densities



Metal Structures in 4D

Aim: 4D Movies

Crystalline materials
for each grain:
- Phase
- Center-of-mass & morphology
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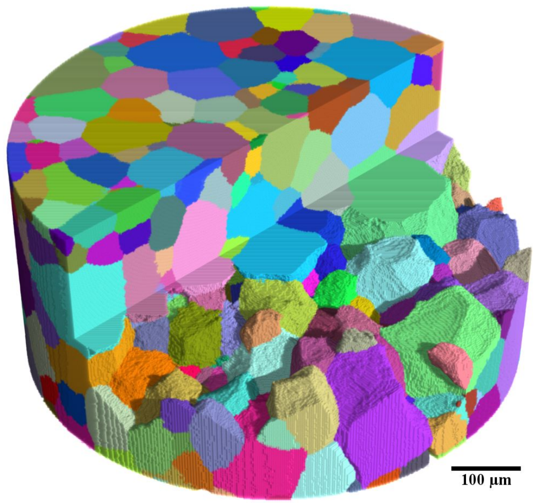
- Defect populations
- Dislocation densities

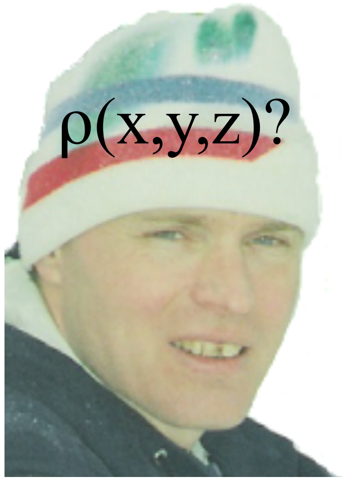
Tomography: density



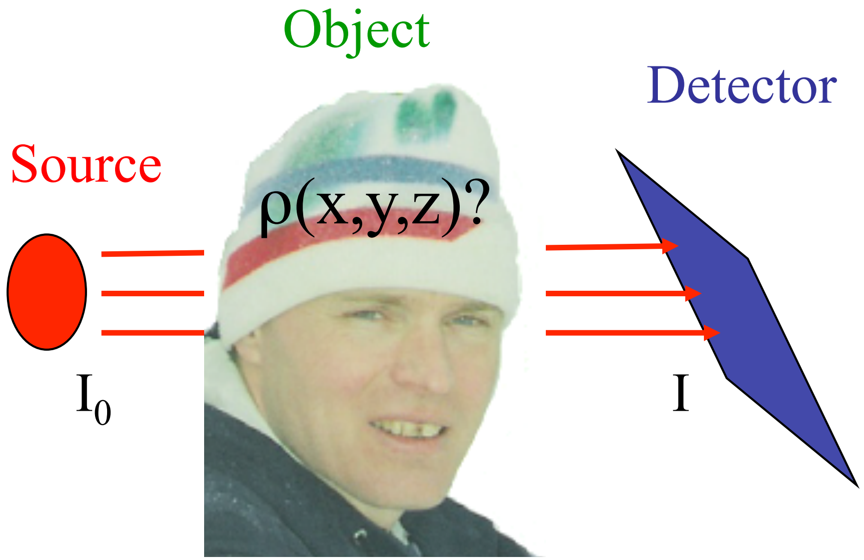
Tomography

Absorption contrast tomography

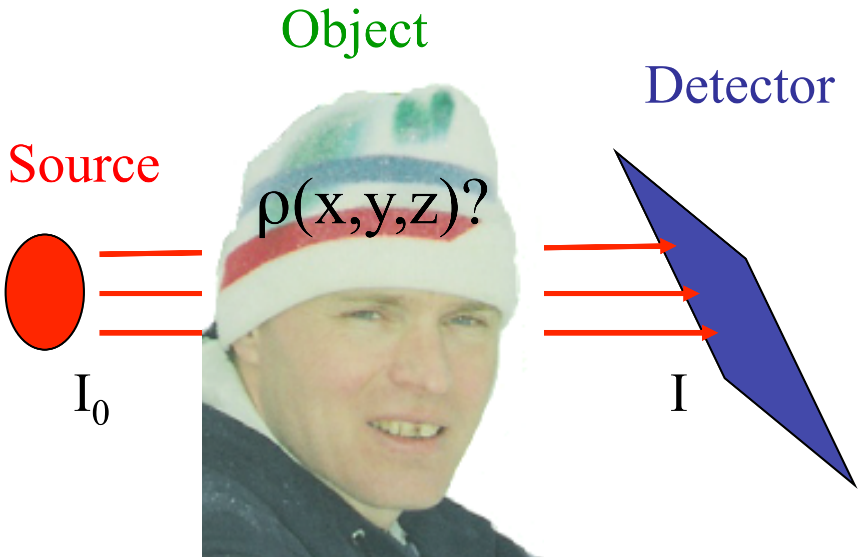
Object



Absorption contrast tomography



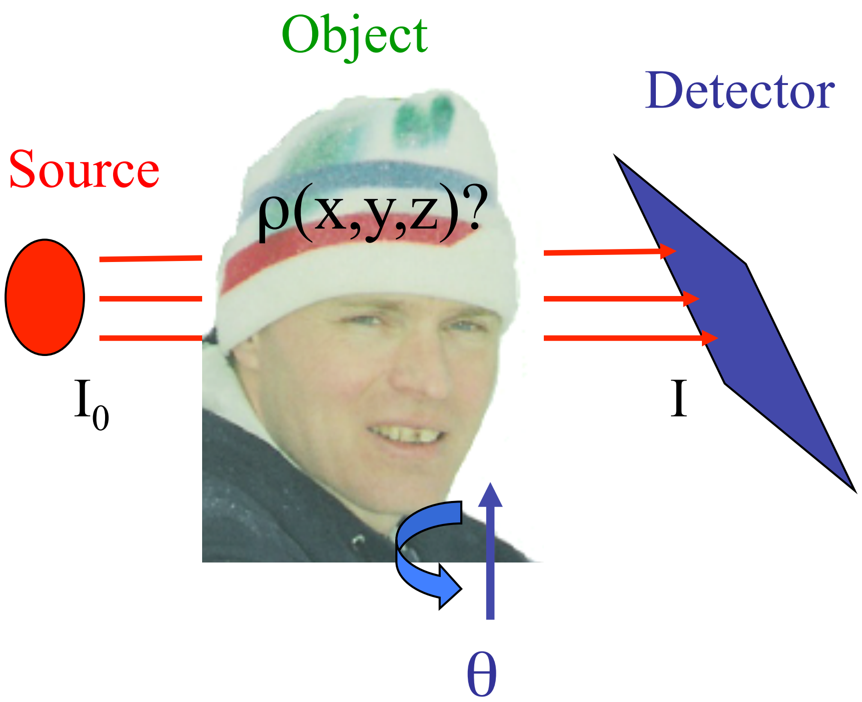
$$\frac{dI}{ds} = -\mu(s)I$$



$$\frac{dI}{ds} = -\mu(s) I$$

$$-\ln\left(\frac{I}{I_0}\right) = \int \mu \, ds$$

Absorption contrast tomography

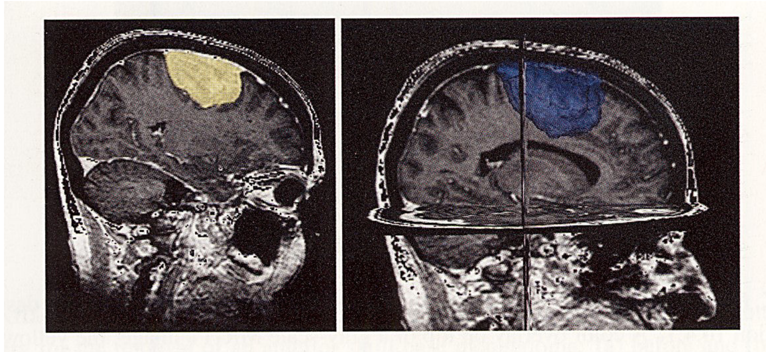


$$\frac{dI}{ds} = -\mu(s)I$$

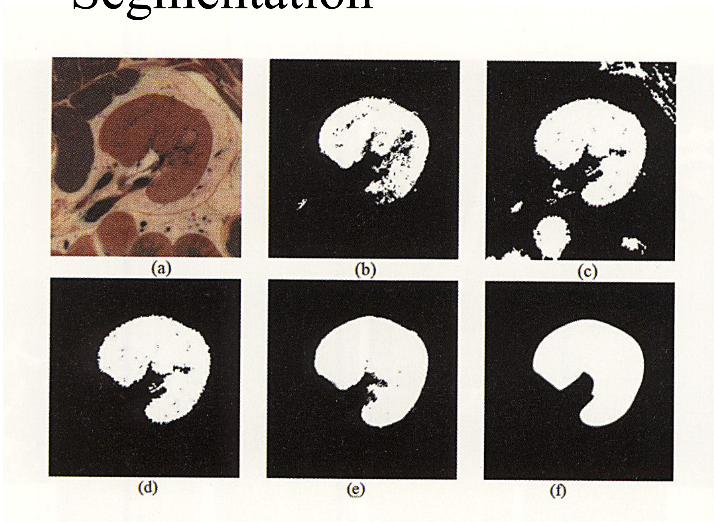
$$-\ln\left(\frac{I}{I_0}\right) = \int \mu \, ds$$

Inverse problem.
Solution: reconstruction

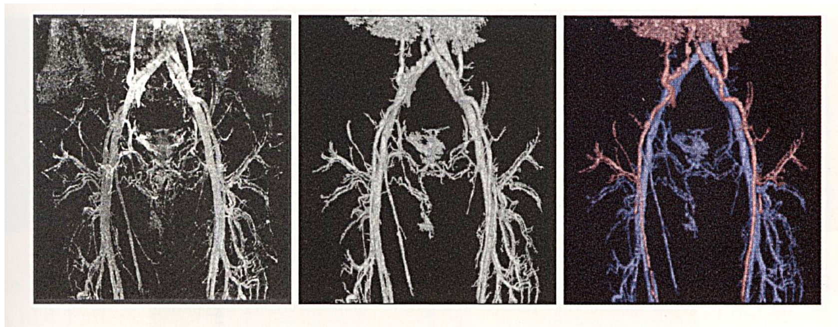
Reconstruction → Segmentation → *Quantification*



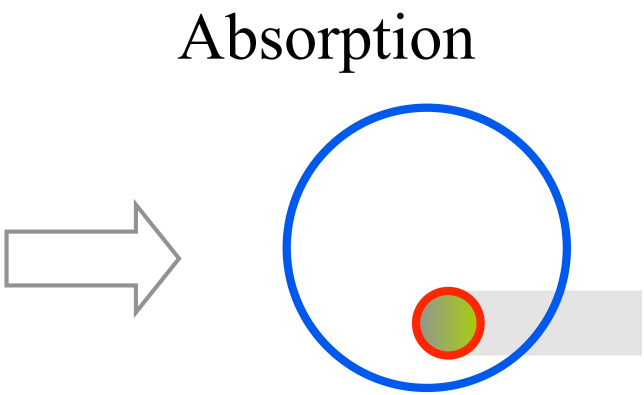
Segmentation

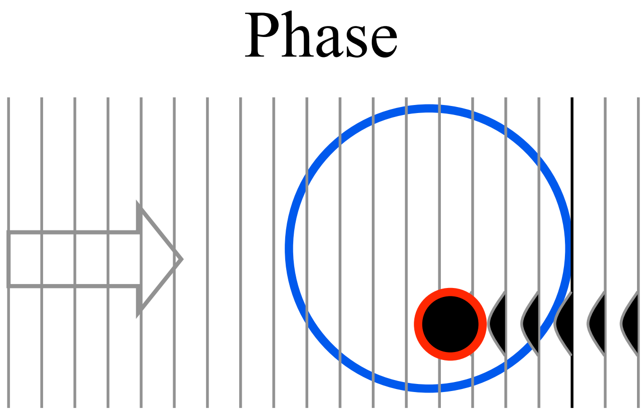


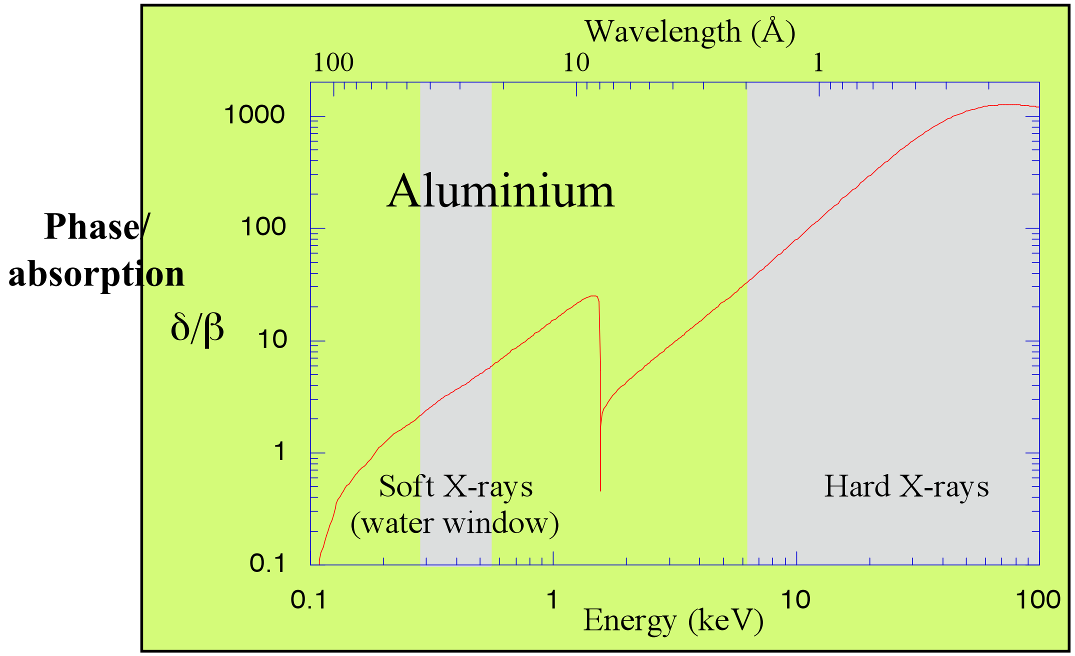
Fuzzy connectivity



Phase Contrast Tomography







Smallest detectable hole at 25 keV in a 4 mm thick sample:

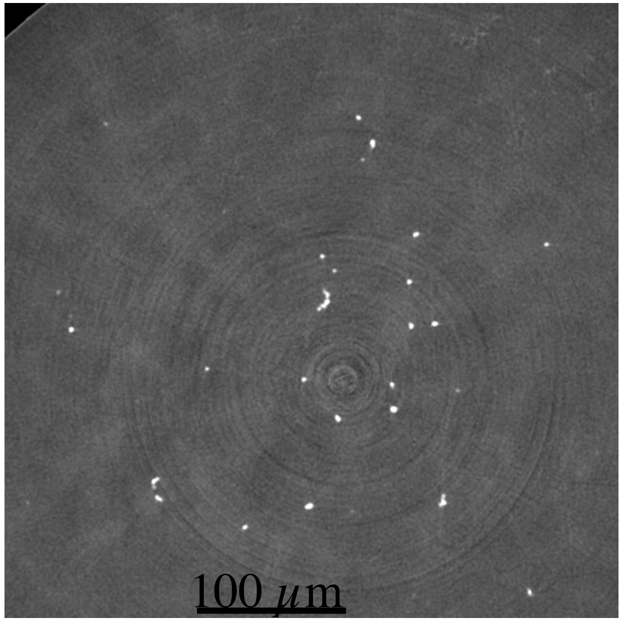
Absorption:	20 µm
Phase:	0.05 µm

Ex.: Holotomography of semisolid Al/Si alloy

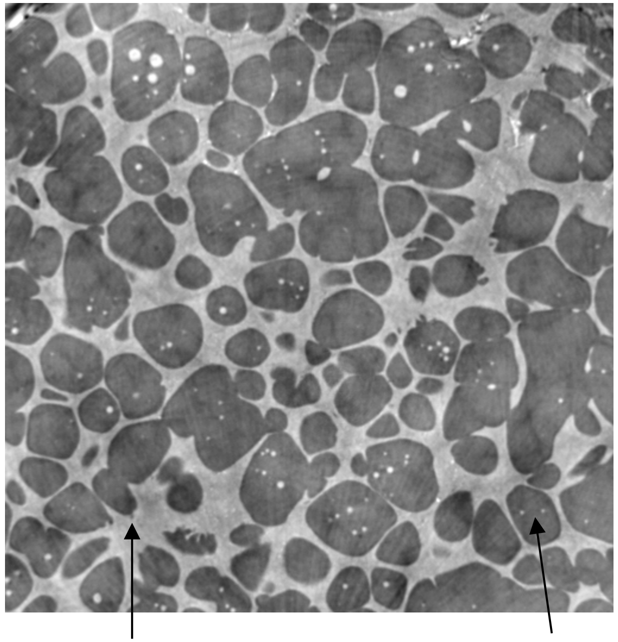
800 angular positions
Total time ≈ 40 minutes

E = 18 keV

Absorption



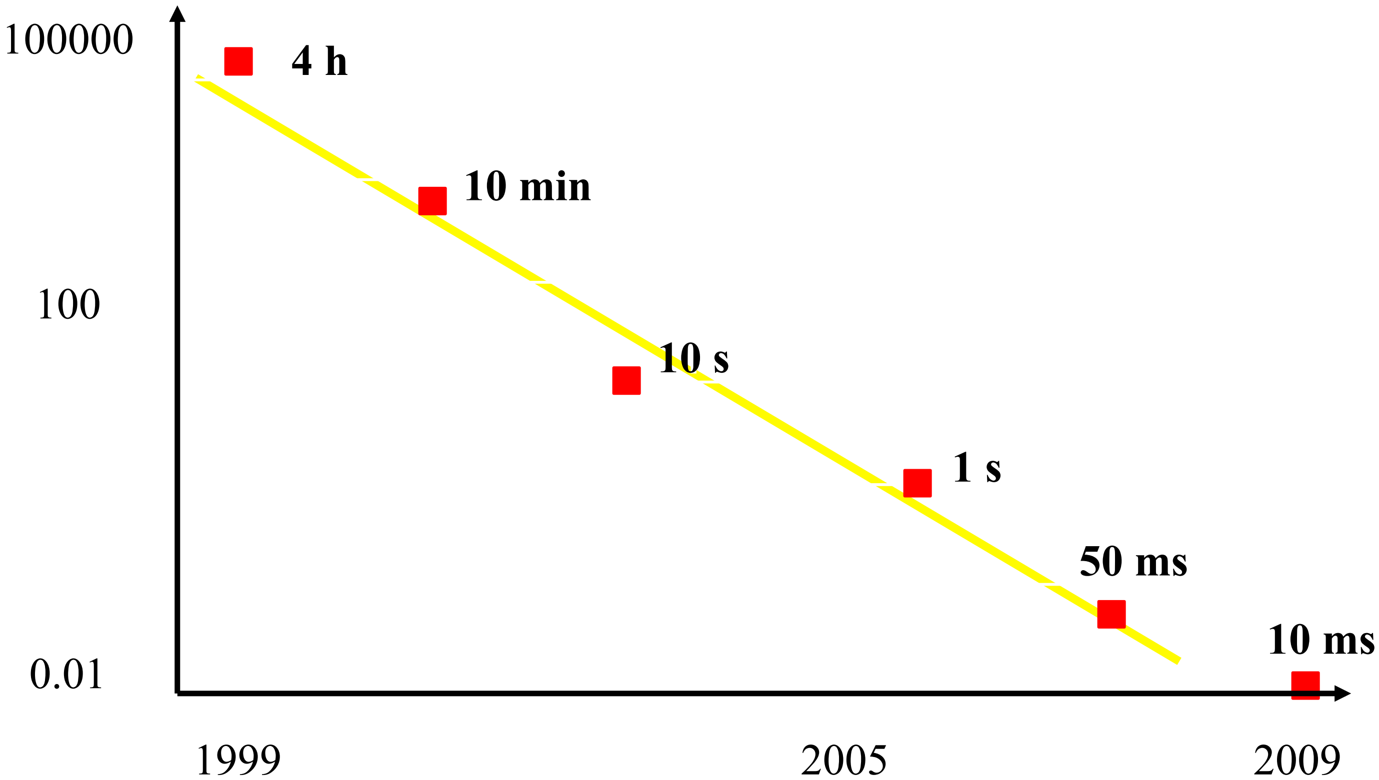
Holotomography



Work by P. Cloetens, ID19, ESRF

Fast micro-tomography

Time for 500 frames at ID15A, ESRF (s)



Courtesy of Honkimäki

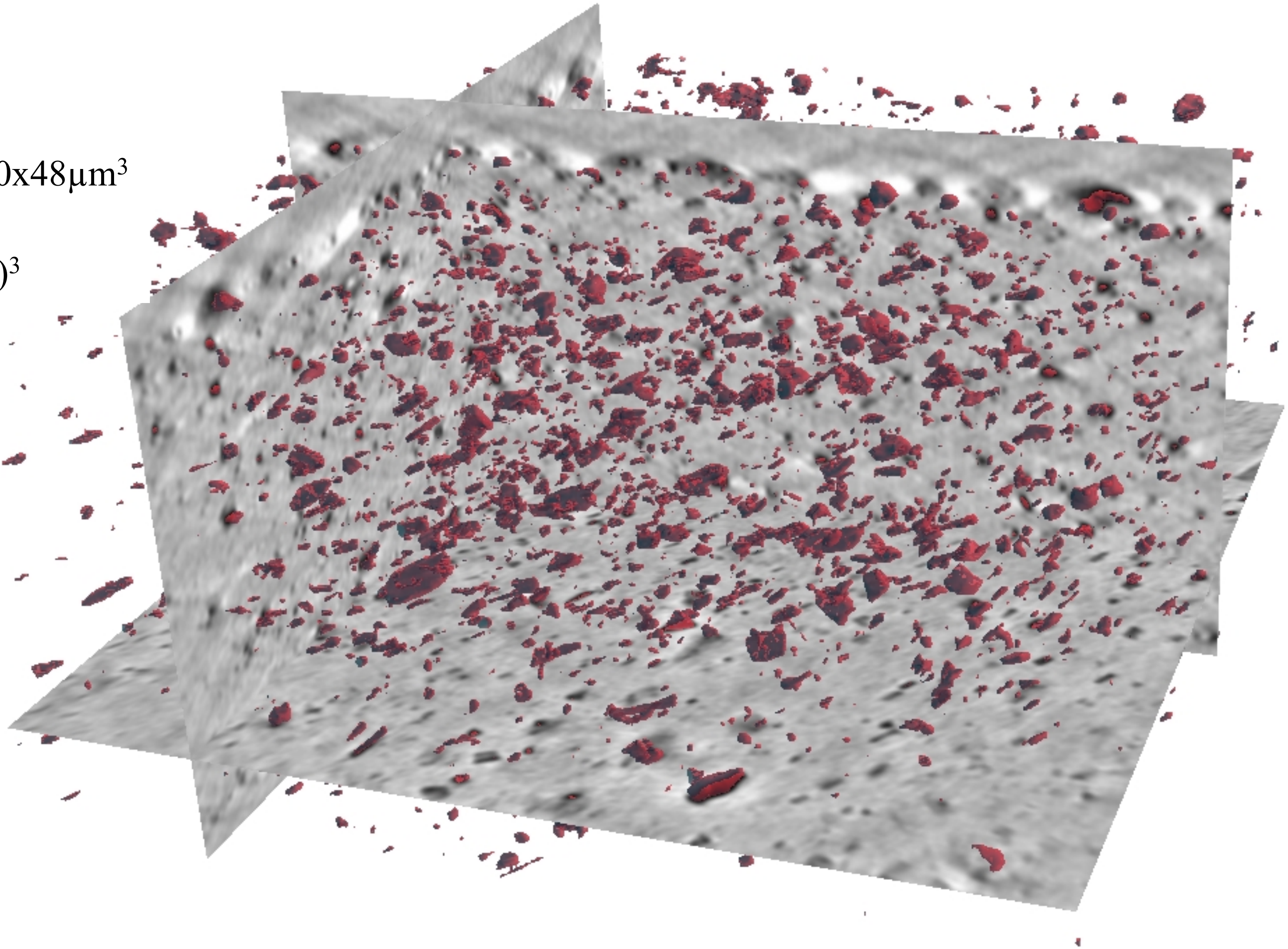
Nano-tomography of Al foil

Material: Alcan Al foil

Energy: 17.5 keV

Volume size: 90x90x48μm^3

Voxel size: (60 nm)^3



Courtesy of P. Cloetens, ESRF

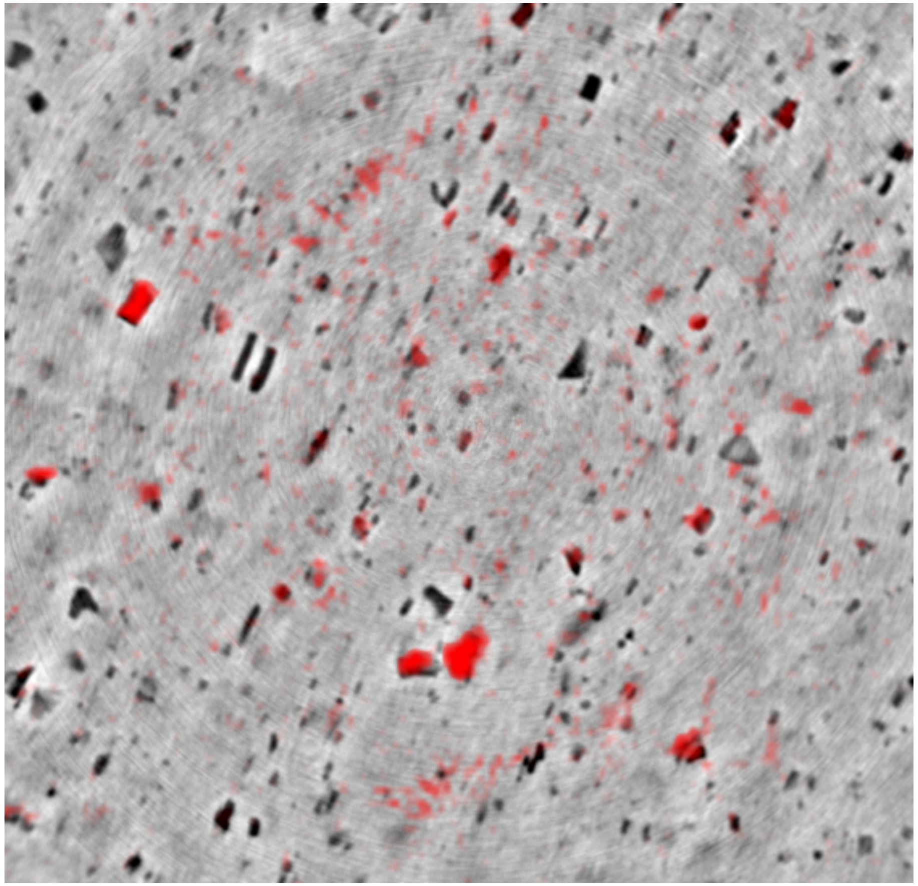
Fluorescence Nano-tomography

Reconstructed slice 5 μm

below surface

Colorcode:

- RED = Ni



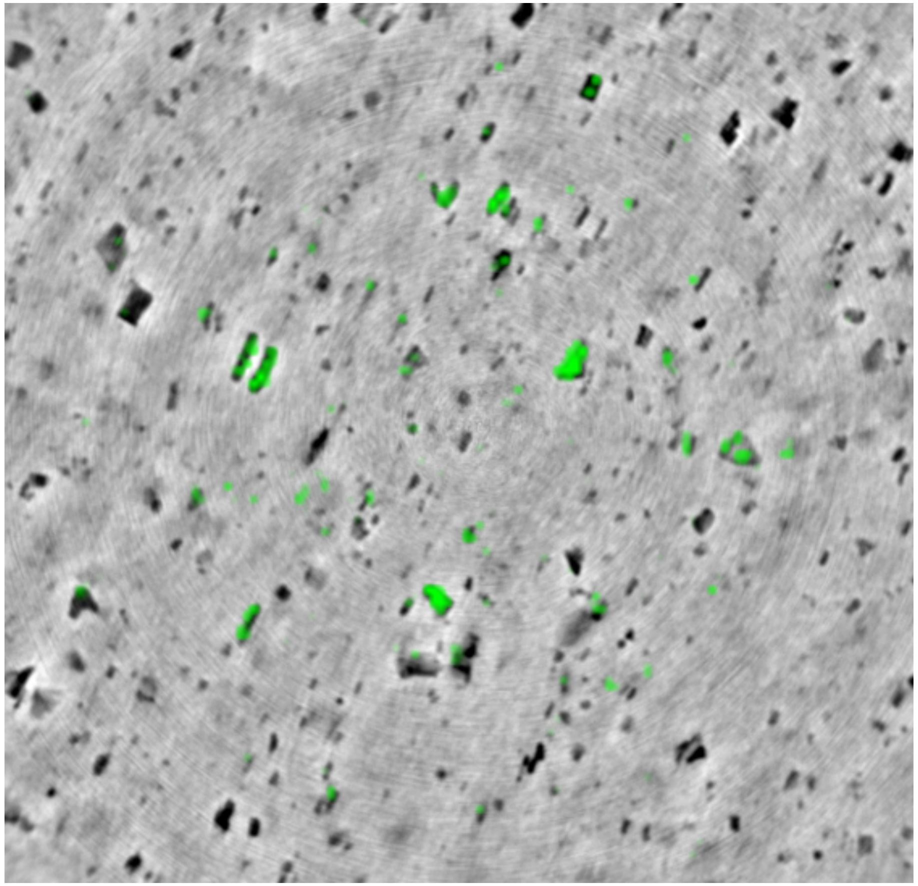
Courtesy of P. Cloetens, ESRF

Fluorescence Nano-tomography

Reconstructed slice 5 μm below surface

Colorcode:

- RED = Ni
- GREEN = Cu



Courtesy of P. Cloetens, ESRF

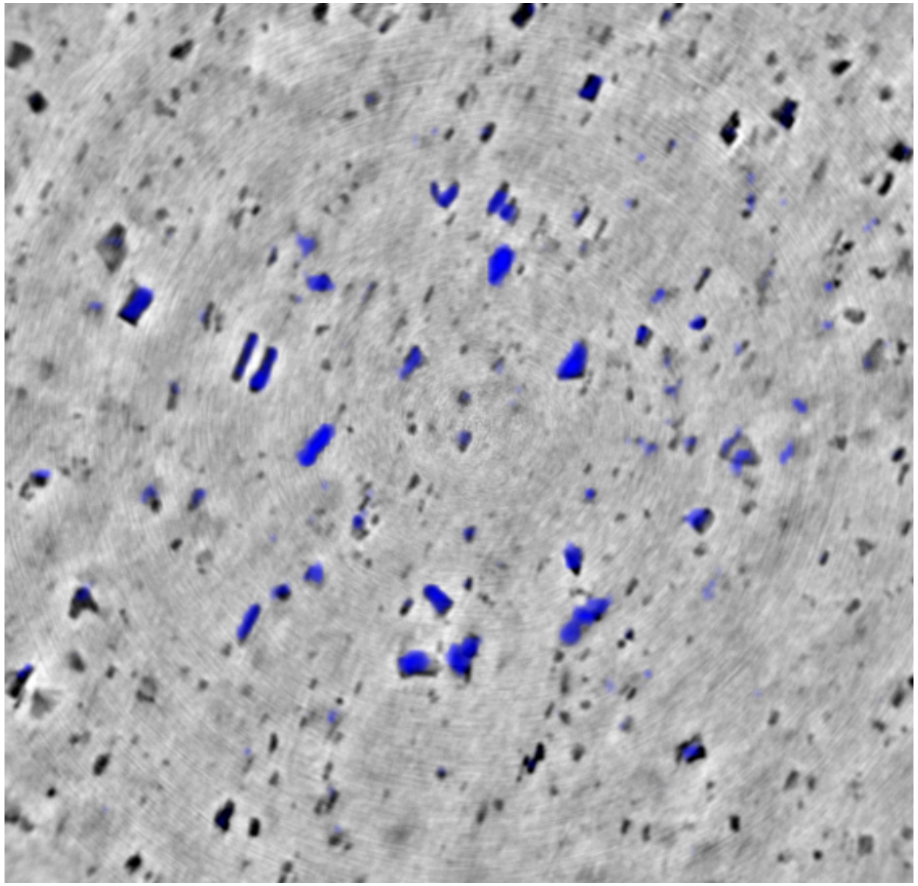
Fluorescence Nano-tomography

Reconstructed slice 5 μm

below surface

Colorcode:

- RED = Ni
- GREEN = Cu
- BLUE = Fe



Courtesy of P. Cloetens, ESRF

Fluorescence Nano-tomography

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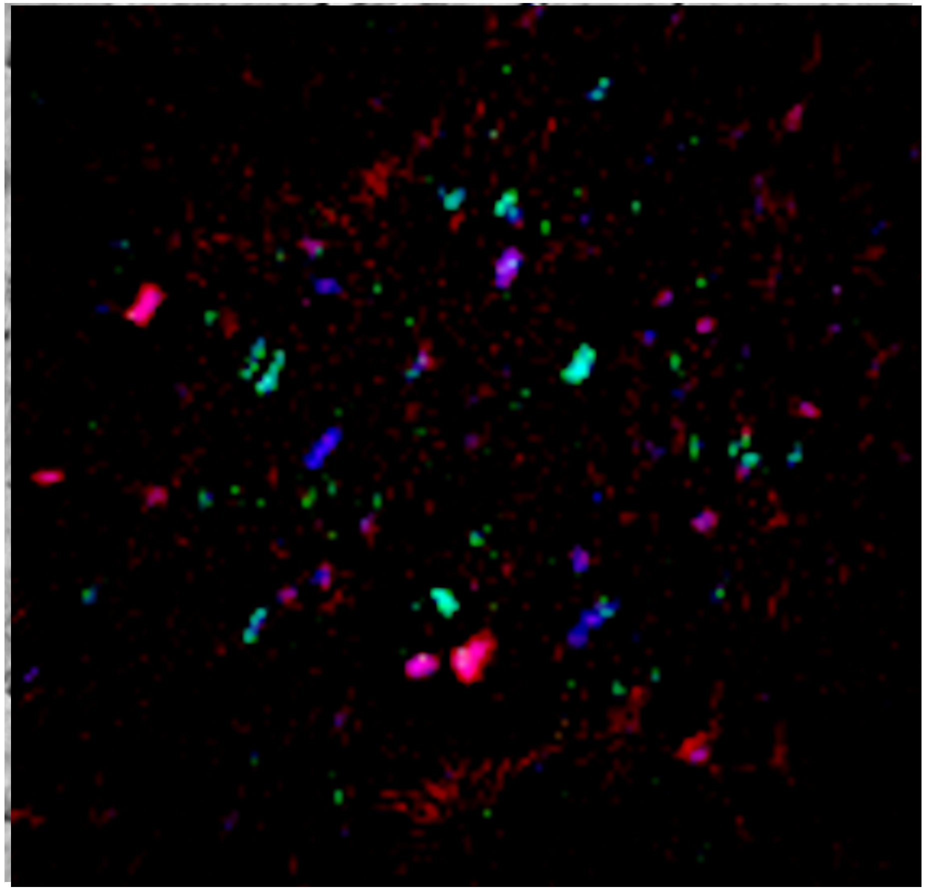

Courtesy of P. Cloetens, ESRF

Fluorescence Nano-tomography

Reconstructed slice 5 µm

below surface

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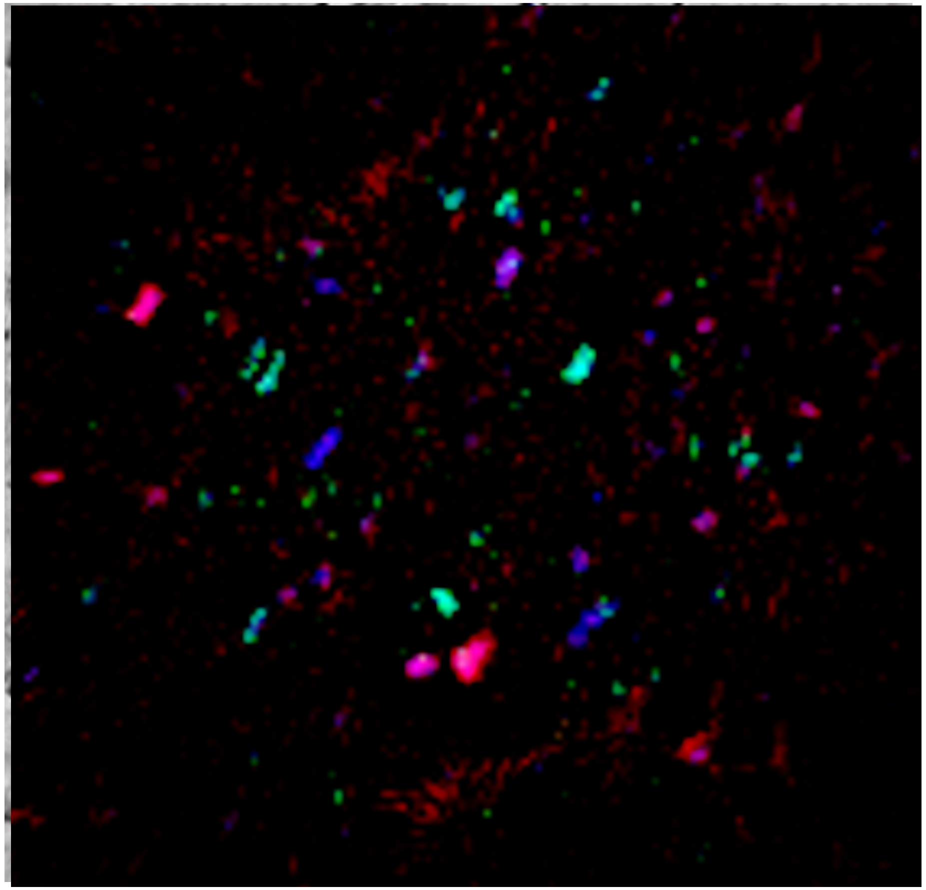


Courtesy of P. Cloetens, ESRF

Micro-tomography at VPS-W on steel

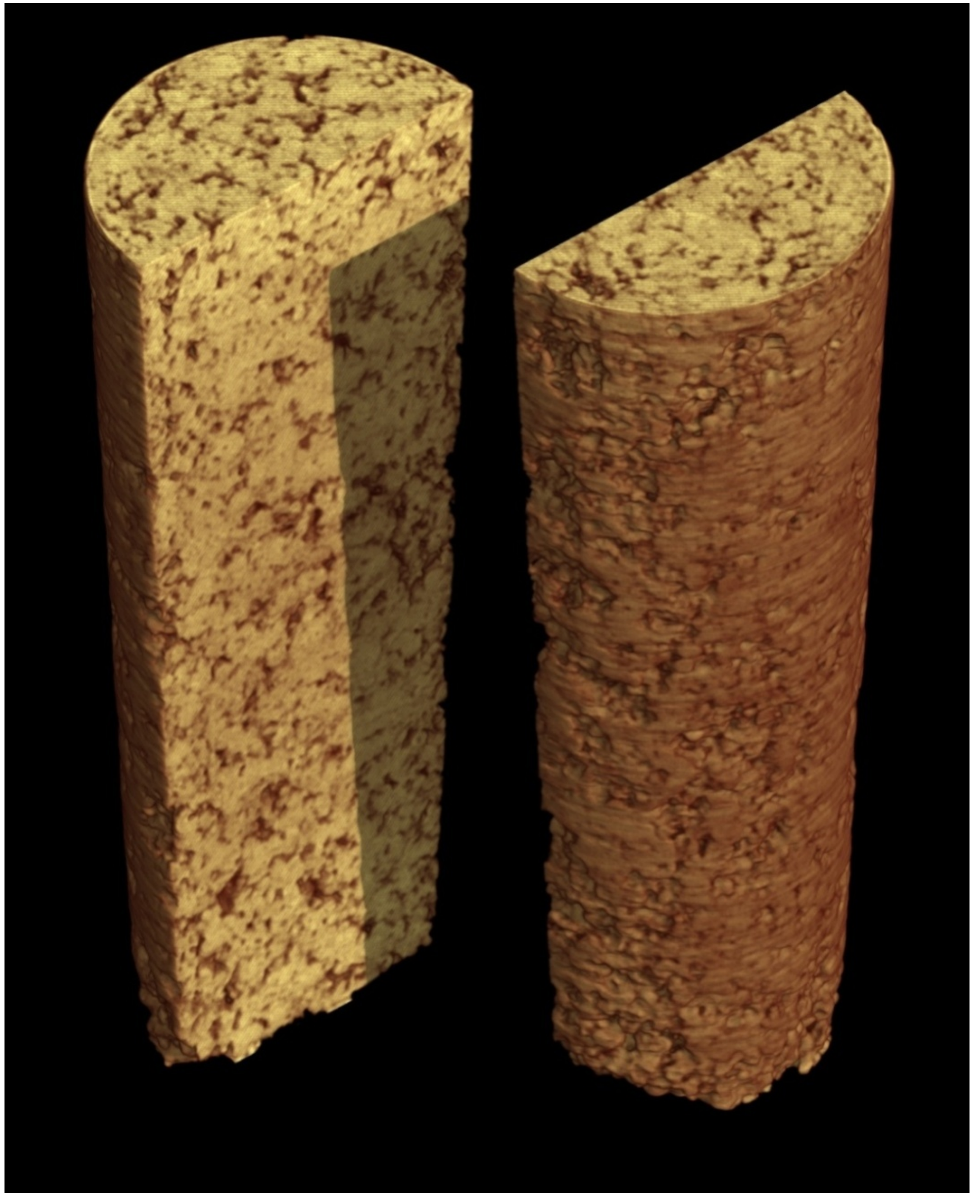
Objective:

- Pore distribution in W -> FEM
- Interface W/steel
- Crack propagation

Challenges:

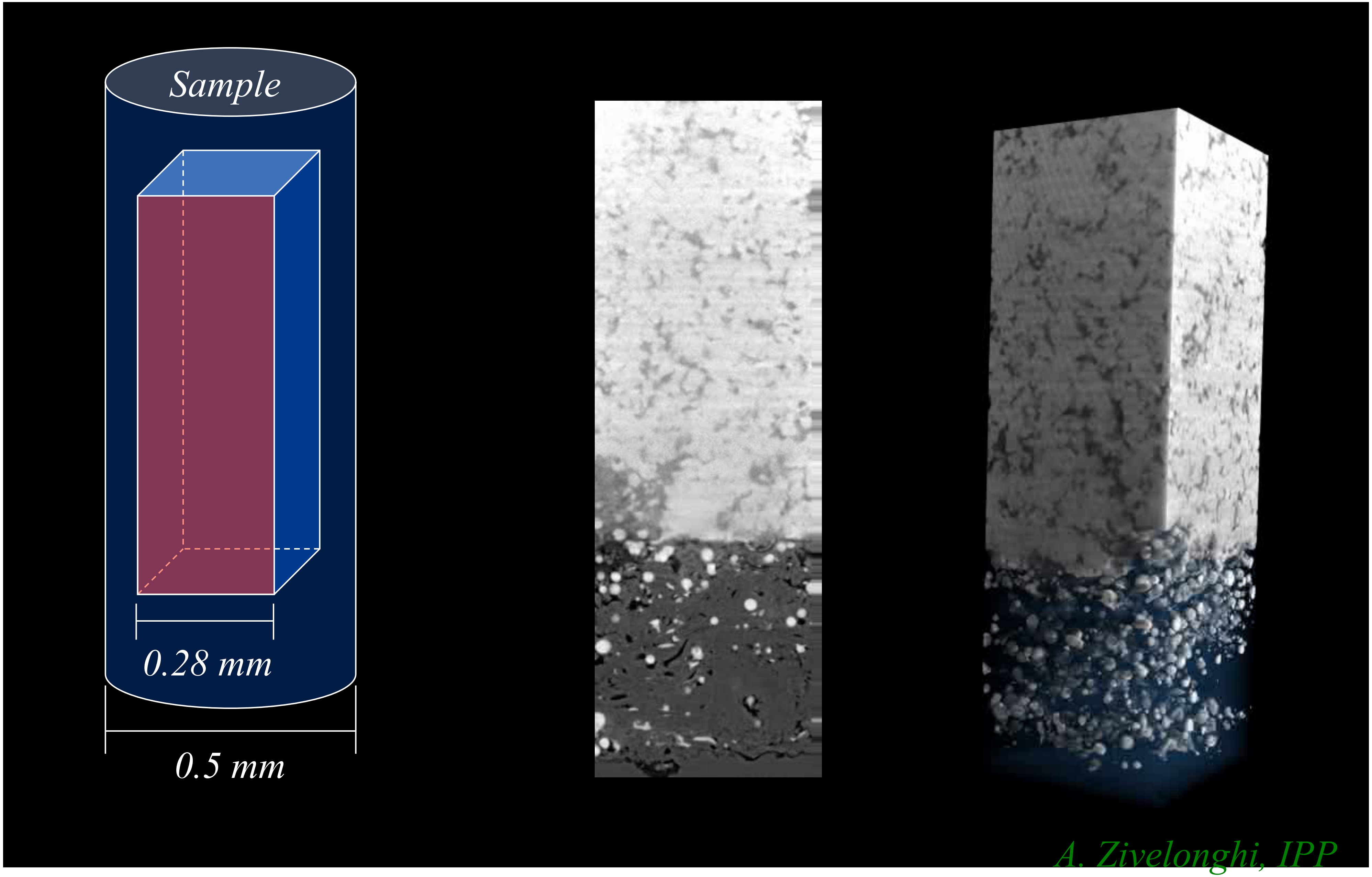
- strong absorption in W

➔ ESRF ID-19:
52 keV, 1.4 µm/pixel



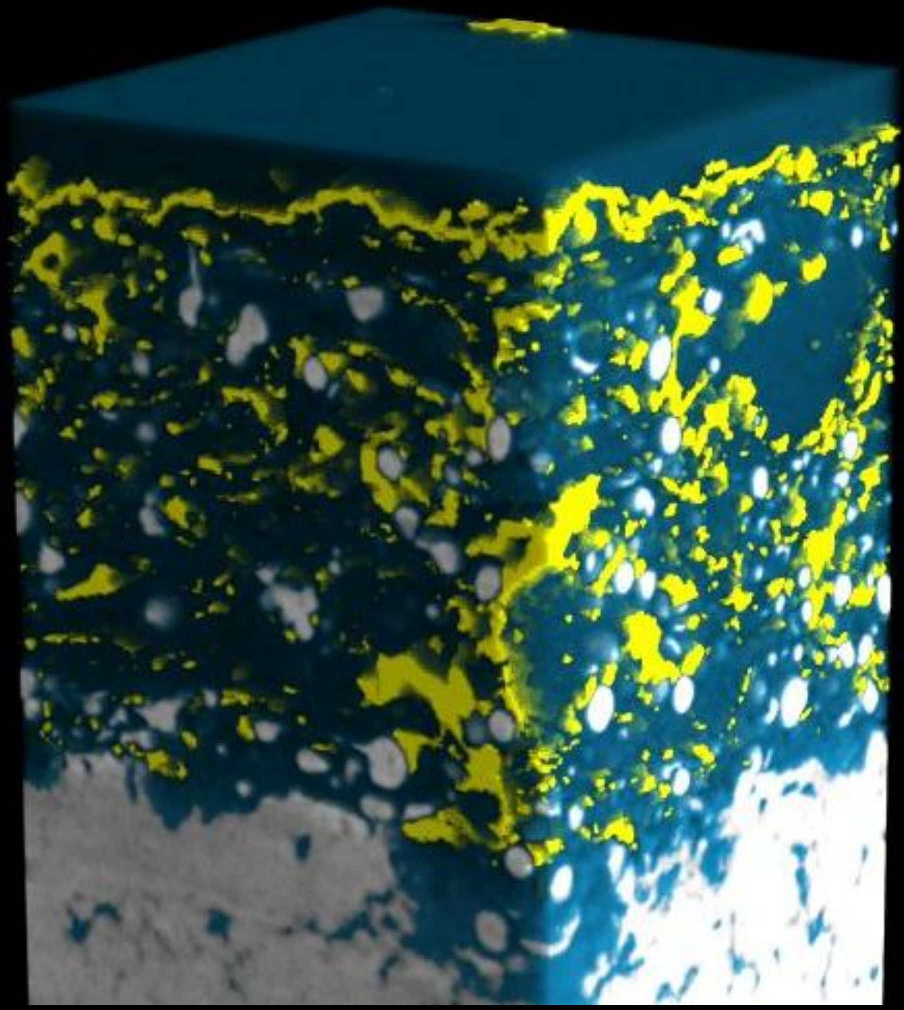
A. Zivelonghi, IPP
T. Weitkamp, ESRF

VPS-W coating, interface to steel



A. Zivelonghi, IPP
T. Weitkamp, ESRF

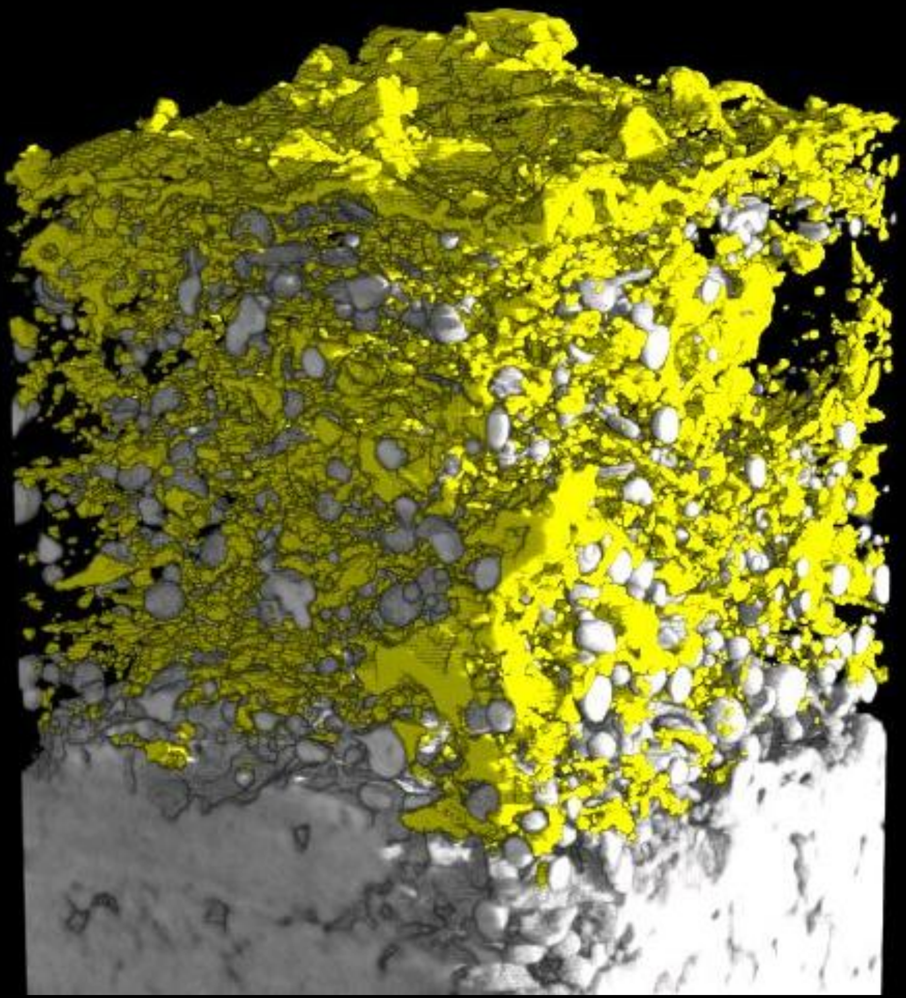
VPS-W coating, steel matrix, W particles, pores



Blue: *steel*
White: *W*
Yellow: *pores in steel*

A. Zivelonghi, IPP
T. Weitkamp, ESRF

VPS-W interlayer: W particles, pores



White: *W*
Yellow: *pores in steel*

Micro-tomography:
quantitative analysis of
real 3D microstructure

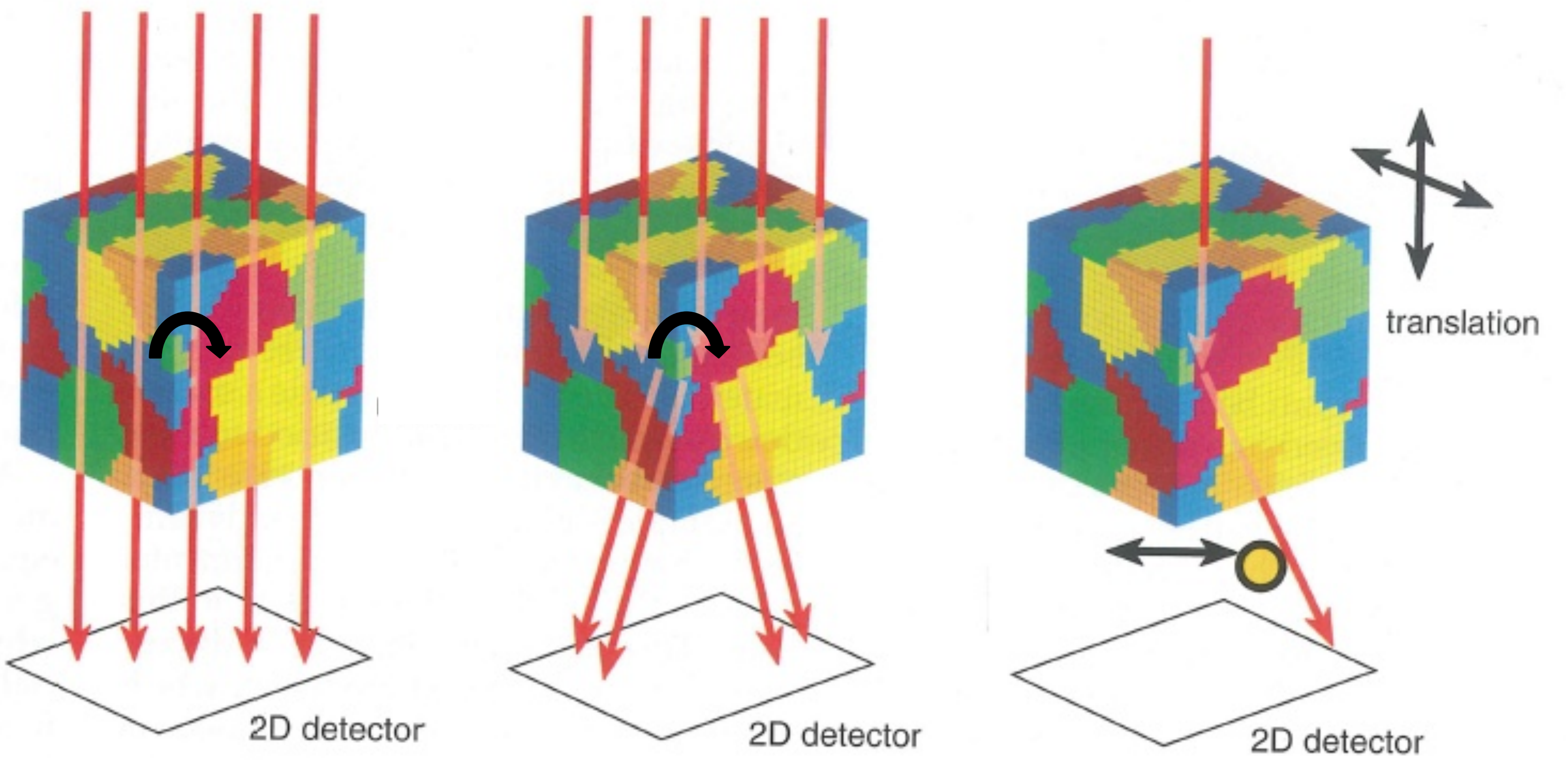
A. Zivelonghi, IPP
T. Weitkamp, ESRF

Three-Dimensional X-ray Diffraction: 3DXRD

Density contrast tomography

3DXRD microscopy

DAXM



X-ray scanning probes

Overview

Scanning a wire.

3D X-ray Structural Microscopy. Polychromatic µ-beam, 25 keV

B.C. Larson *et al*. (2002). *Nature* **415**, 887-890.

Scanning a 2D Capillary tube (collimator)

MAXIM. Monochromatic, DORIS: 8 keV, HARWI: hard x-rays

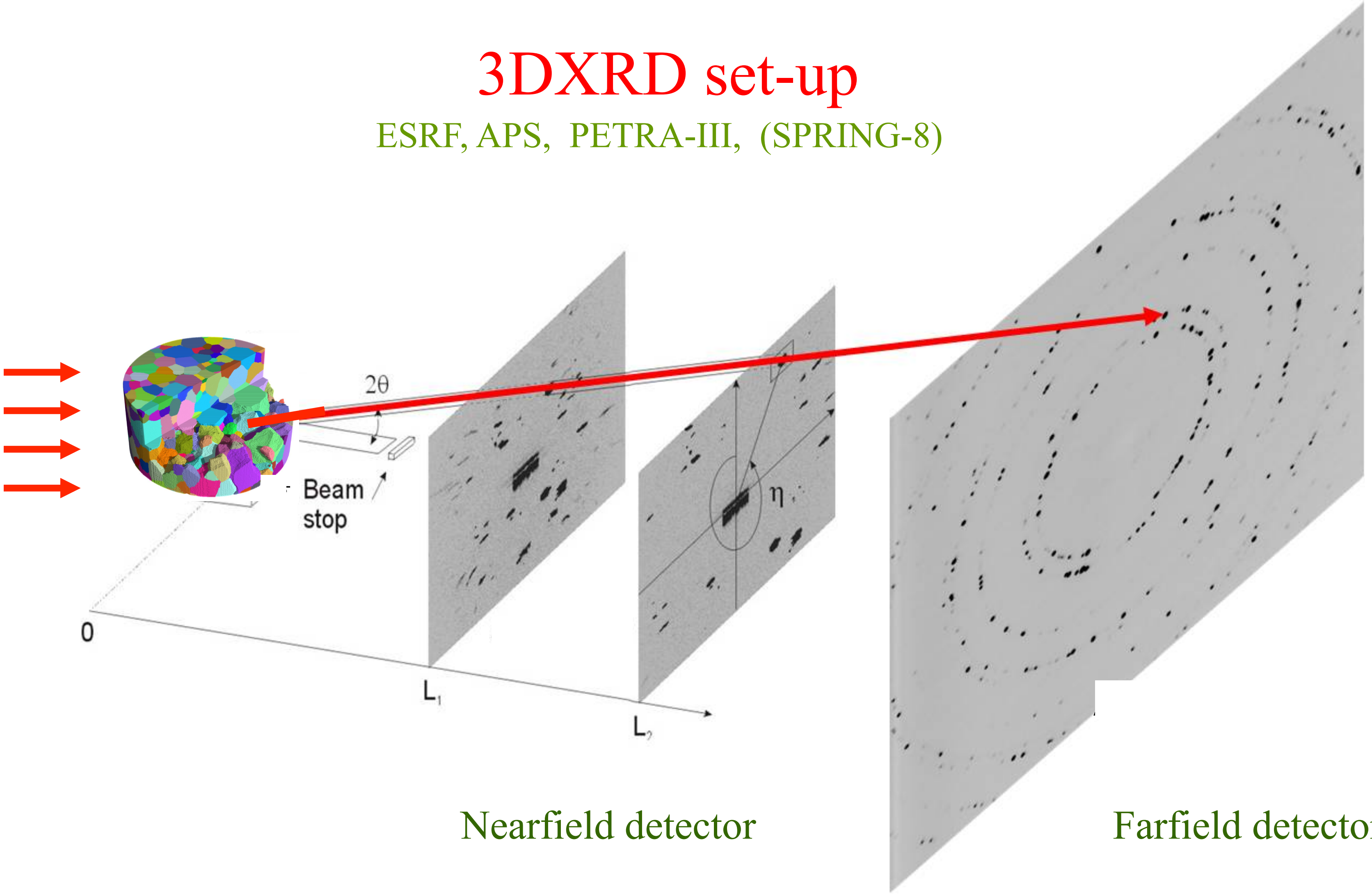
T. Wroblewski *et al.* (1999). *Nucl. Instr. Meth. A* **428,** 570-582.

Scanning tomography

P. Bleuet, E. Welcomme, E. Dooryhee, J. Susini, J.-L. Hodeau, P. Walter. *Nature Mater.* 7, 468-472 (2008)

3DXRD set-up

ESRF, APS, PETRA-III, (SPRING-8)



Nearfield detector

Position

Farfield detector

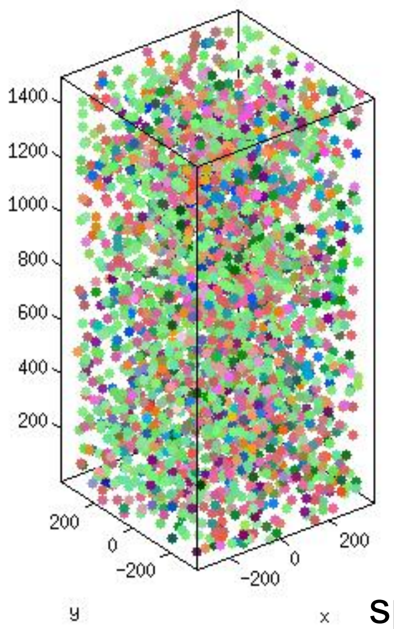
Orientation and strain

H.F. Poulsen: *Three-Dimensional X-ray Diffraction Microscopy* (Springer, 2004).

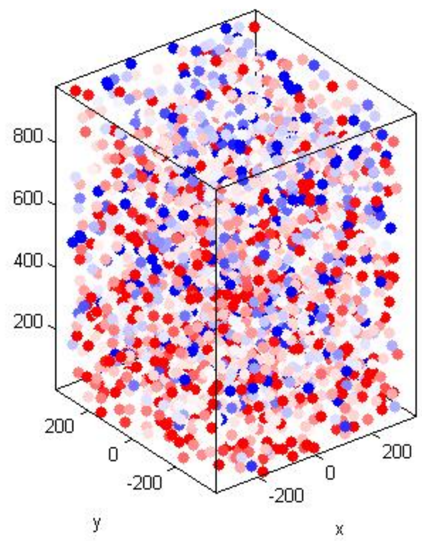
Grain center mapping: status

Ex: CMS position of 2842 grains
in an IF steel sample

Orientation map



Elastic strain map

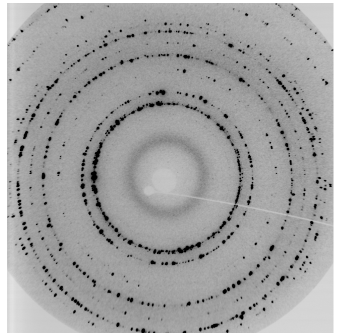


spatial resolution: 2 μm
orientation resolution: 0.1 deg
strain resolution: 10^-4

J. Oddershede, G. Winther, H.F. Poulsen, L. Margulies, M. Kobyashi,, S. Schmidt, J. Wright, W. Reimers

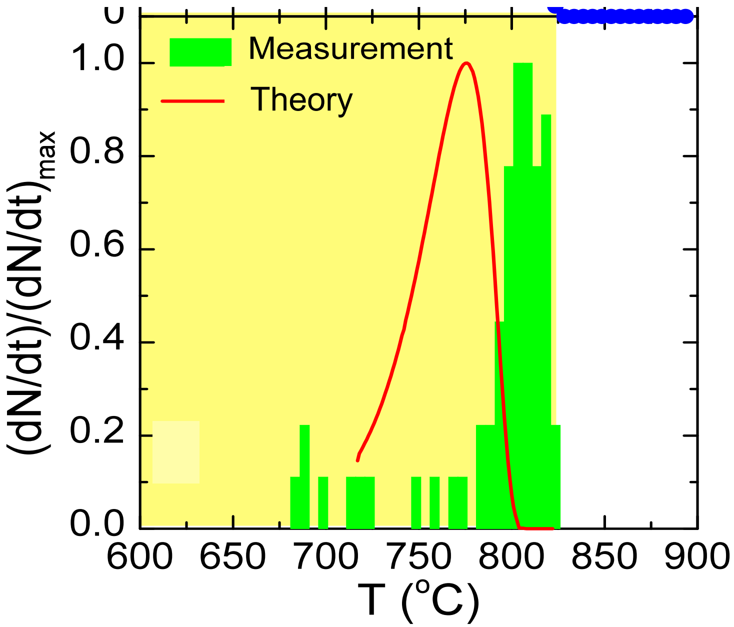
Nucelation

Study on Carbon steel with TU Delft



Cooling exp.:

Ferrite – Austenite



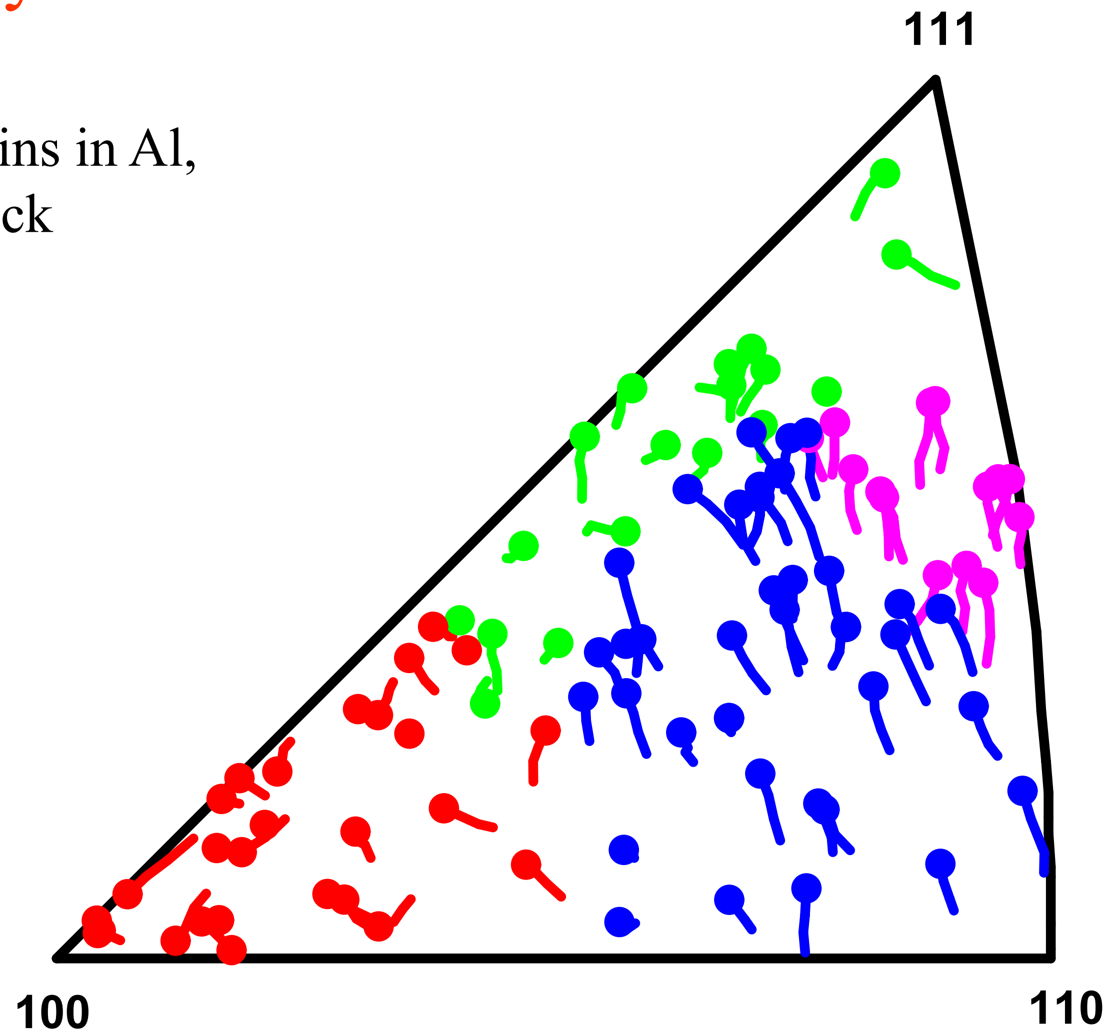
Activation energy off by 100!

S.E. Offerman *et al.* (2002). *Science* **298**, 1003.

Polycrystal Deformation

Grain rotation for 95 grains in Al,
100 μm grains, 5 mm thick

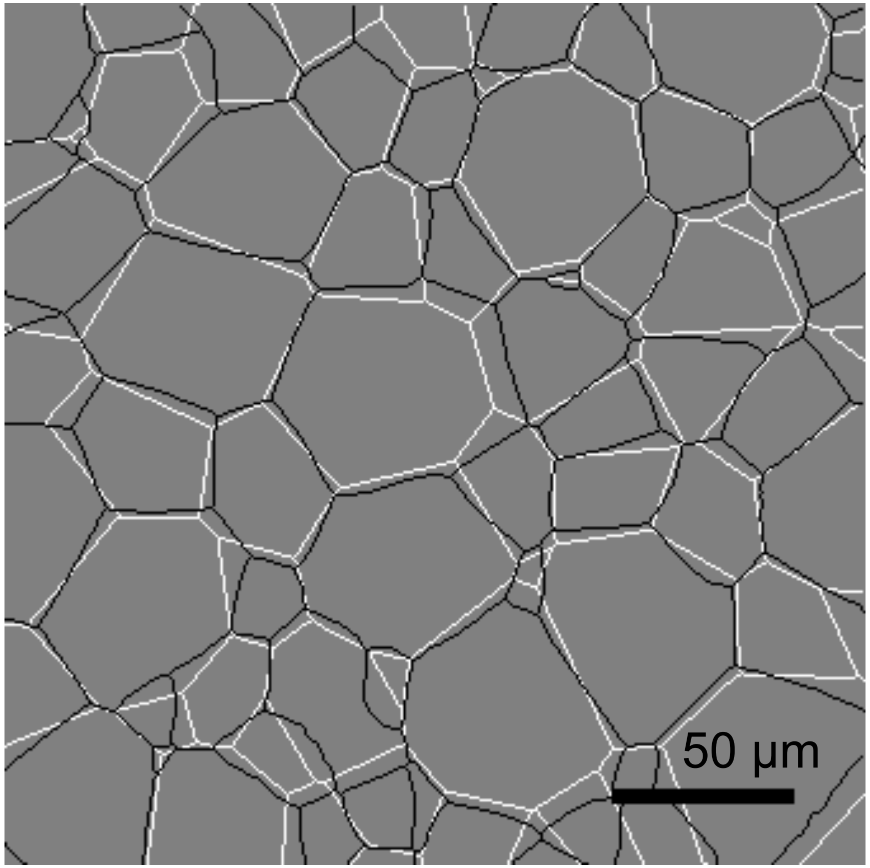
Tensile strain: 6 %



L. Margulies *et al*. (2001). *Science* **291**, 2392.
H.F. Poulsen *et al*. (2003). *Acta Mater*. **51**, 3821.
G. Winther *et al*. (2004). *Acta Mater*. **52**, 3821

Include neighbors: Laguerre tesselation

3D:



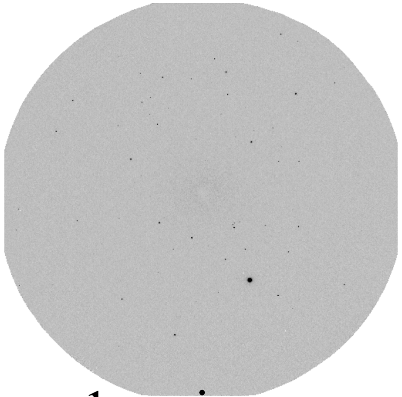
Voxels in wrong grain: 14%
Wrong neighbors per grain: 1.2

Future:
- reconstruction of shape

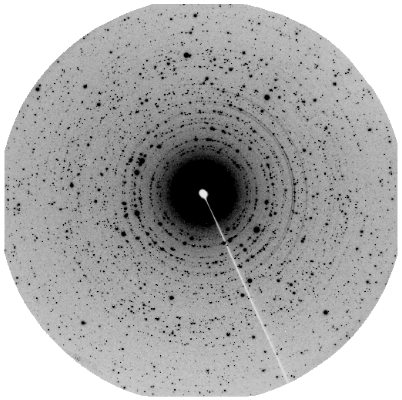
A. Lyckegaard, E.M. Lauridsen, W. Ludwig, R. Fonda, H.F. Poulsen, *Image Analysis Stereology*, in print.

Limitations

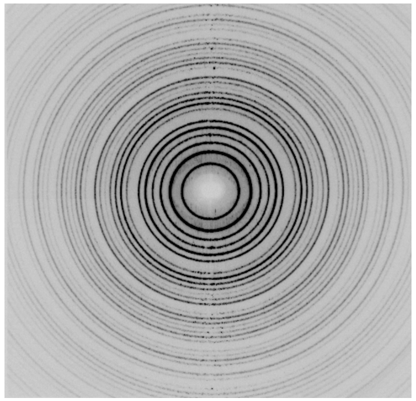
Number of grains



1 grain



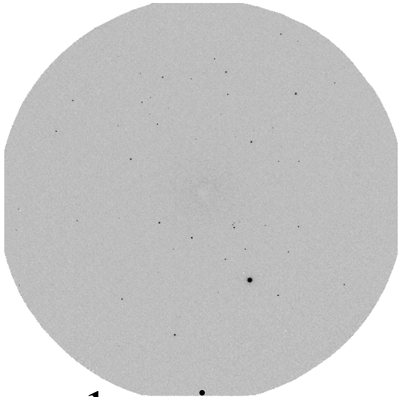
100 grains



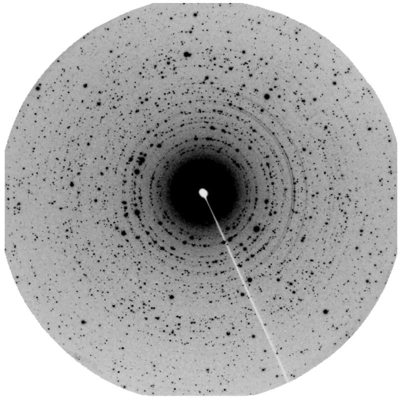
10,000 grains

Limitations

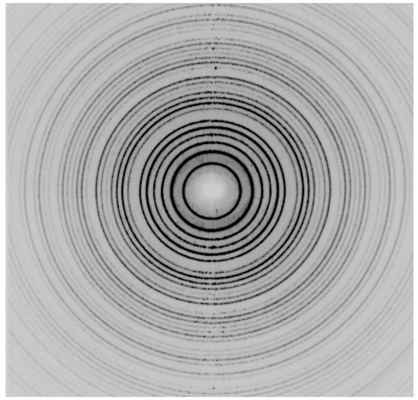
Number of grains



1 grain

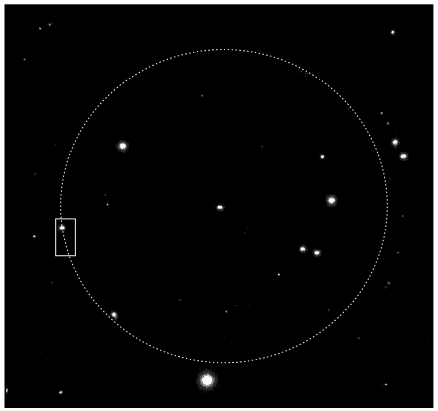


100 grains

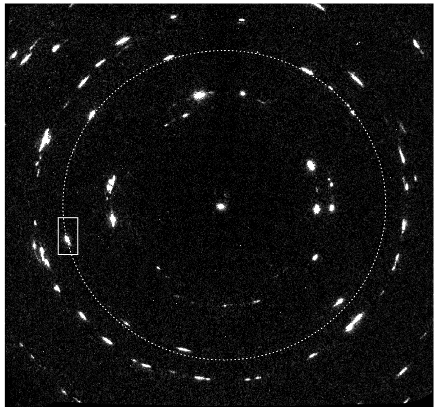


10,000 grains

Degree of deformation:

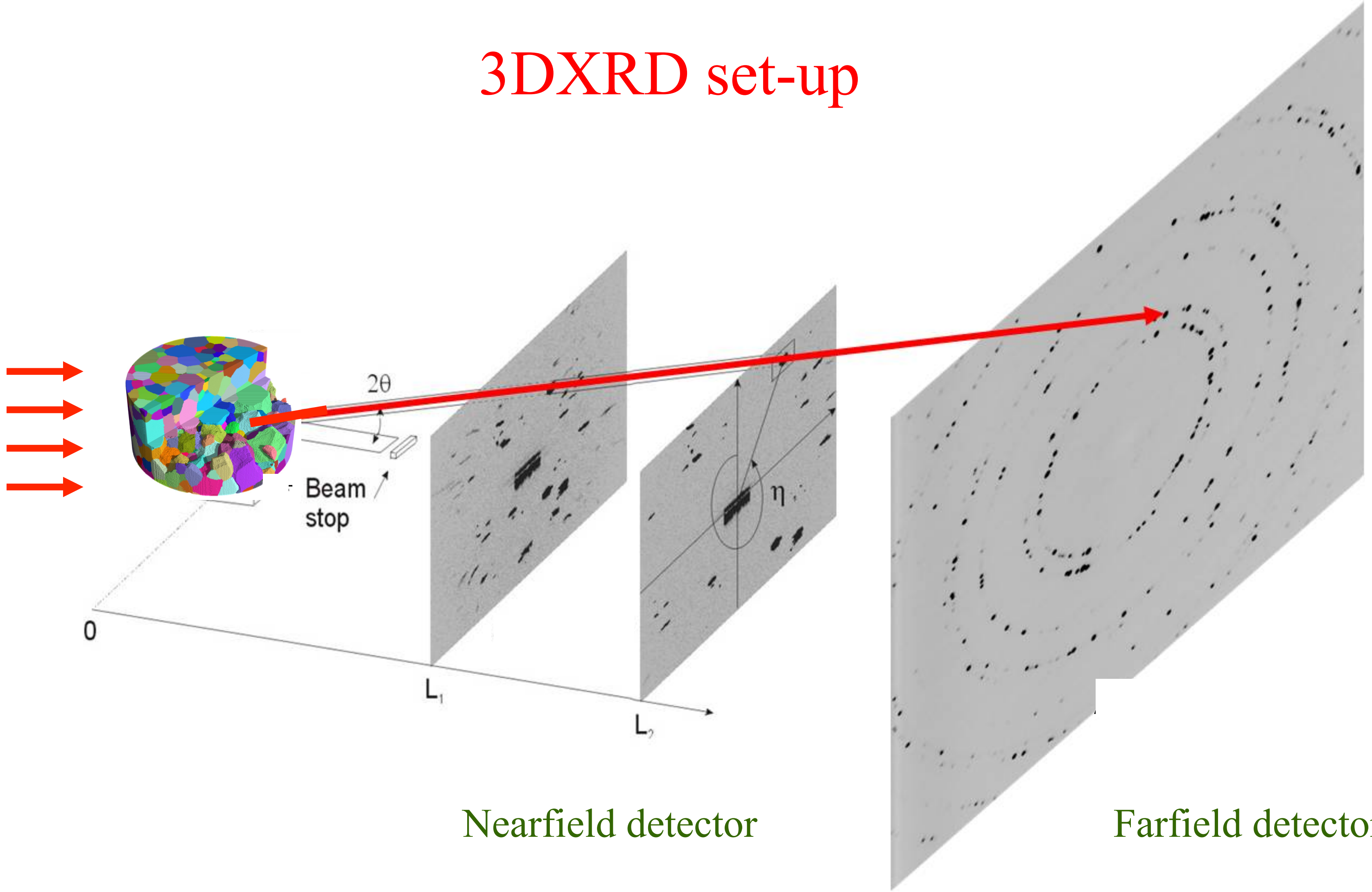


0%



11%

3DXRD set-up



Nearfield detector

Position

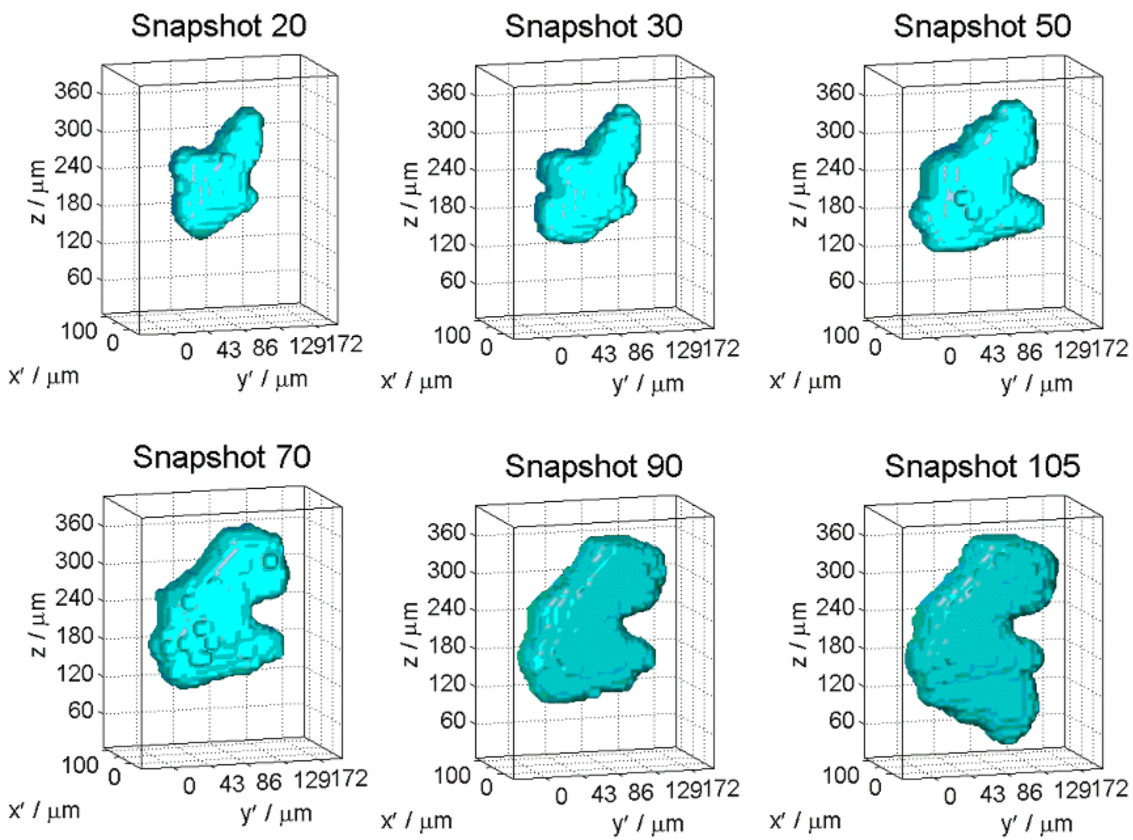
Farfield detector

Orientation and strain

H.F. Poulsen: *Three-Dimensional X-ray Diffraction Microscopy* (Springer, 2004).

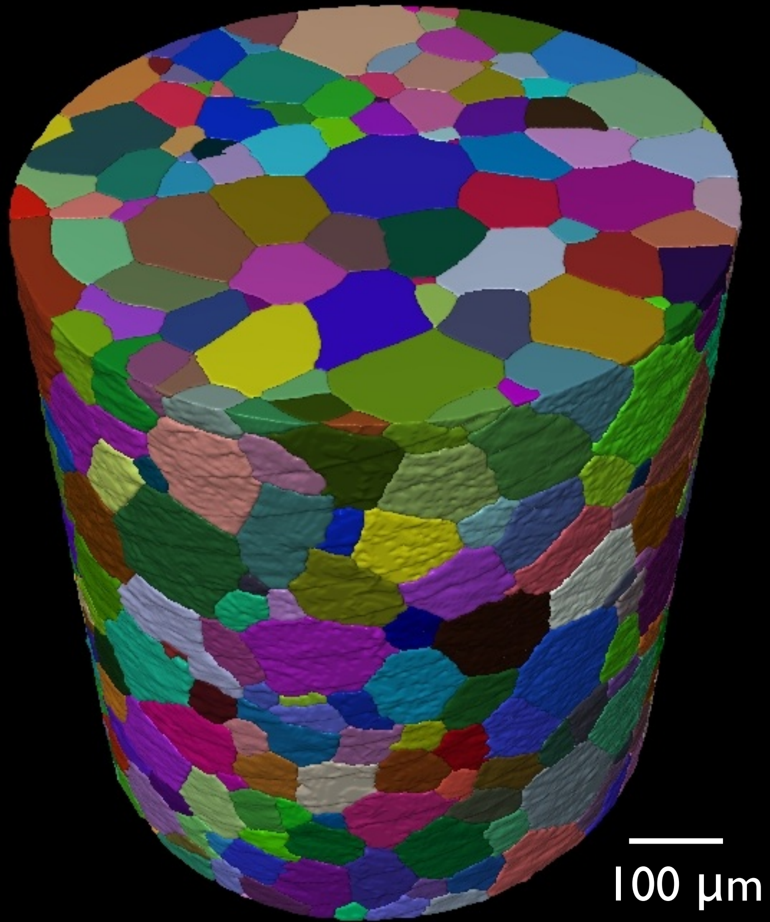
Video of growth of an internal grain

Recrystallization of 42% deformed pure Al during annealing at ~200 C.

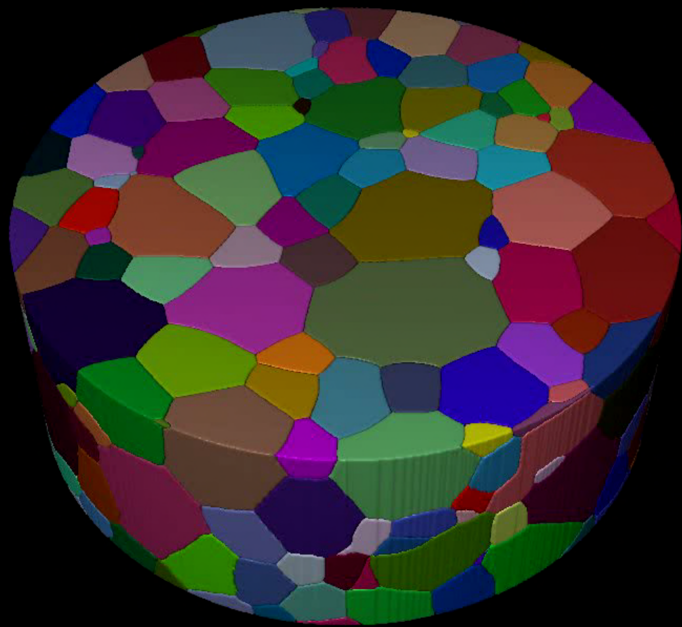


S. Schmidt, S. F. Nielsen, C. Gundlach, L. Margulies, X. Huang, D. Juul Jensen. *Science* **305**, 229 (2004)

Experimental results



Phase field simulations



Risø: E.M. Lauridsen, S. Poulsen, A. Lyckegaard.

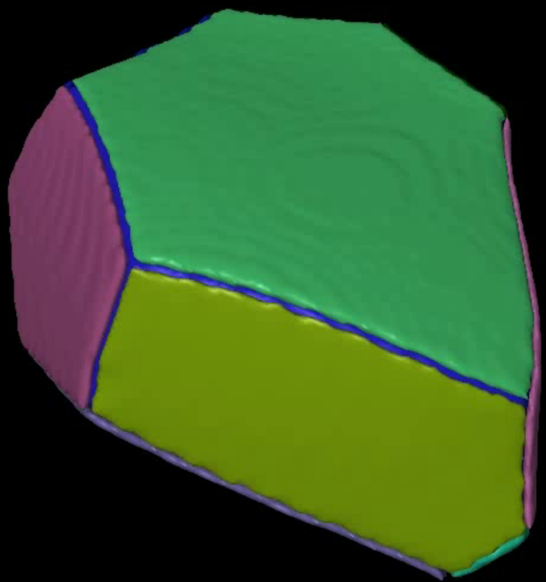
Northwestern: P. Voorhees, I. McKenna

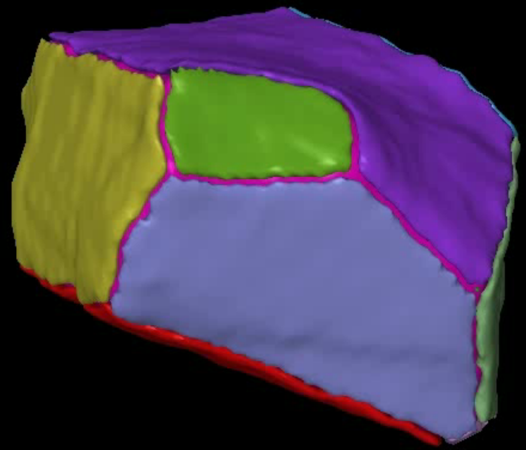
Navy Resarch Lab: R. Fonda

ESRF: W. Ludwig, A. King, S. Rolland

Simulations

Experimental



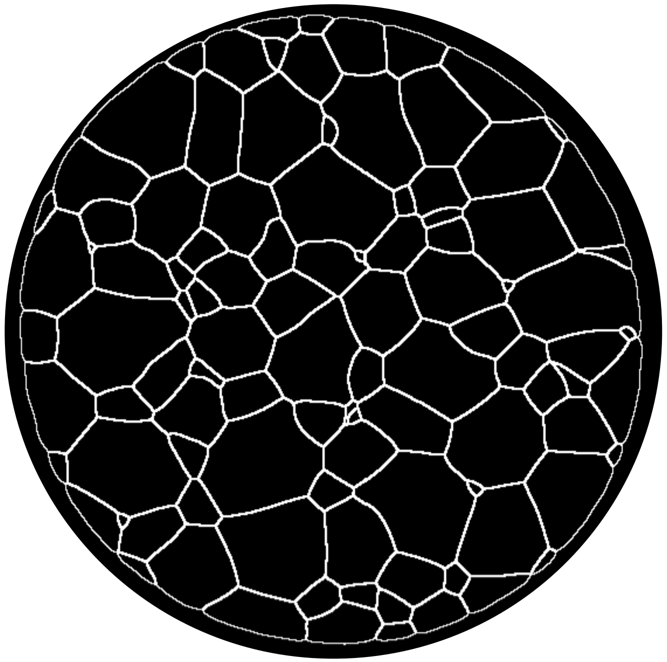


Faces = 12
Edges = 30
Volume = 314000 voxels

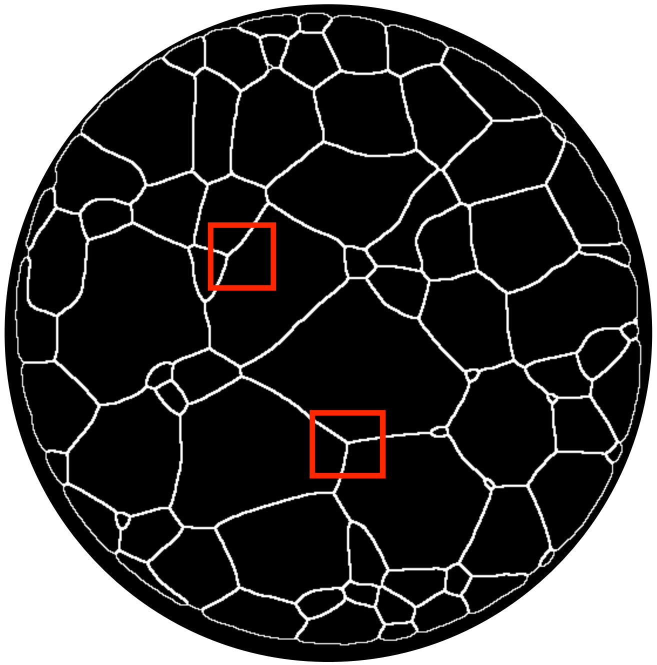
Faces = 13
Edges = 33
Volume = 262000 voxels

(Faces at time 1 = 22)

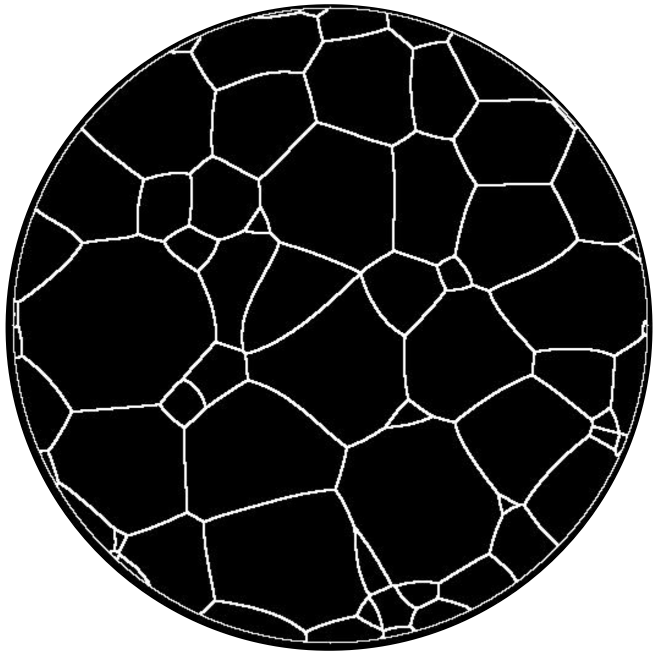
Grain Growth Kinetics in Titanium β21S



Initial microstructure



Final experimental microstructure

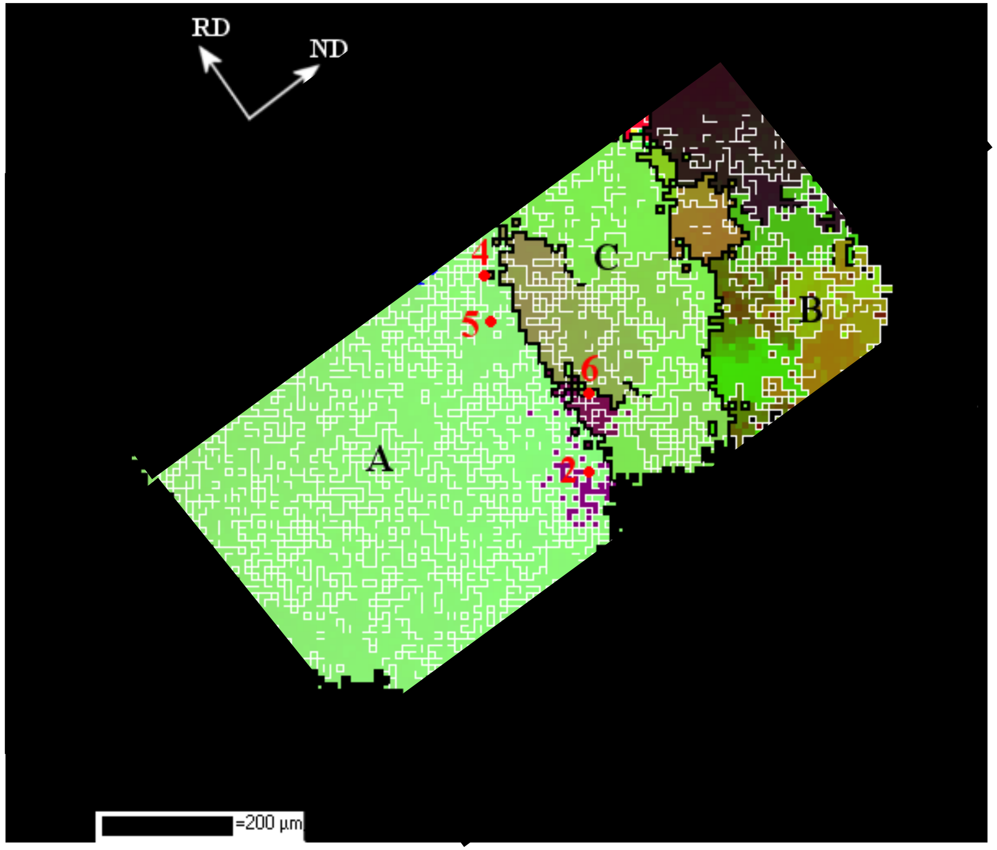


Final Phase field simulated microstructure

3D orientation imaging :
a nucleation study

30% cold-rolled Al

1 layer with farfield detector:

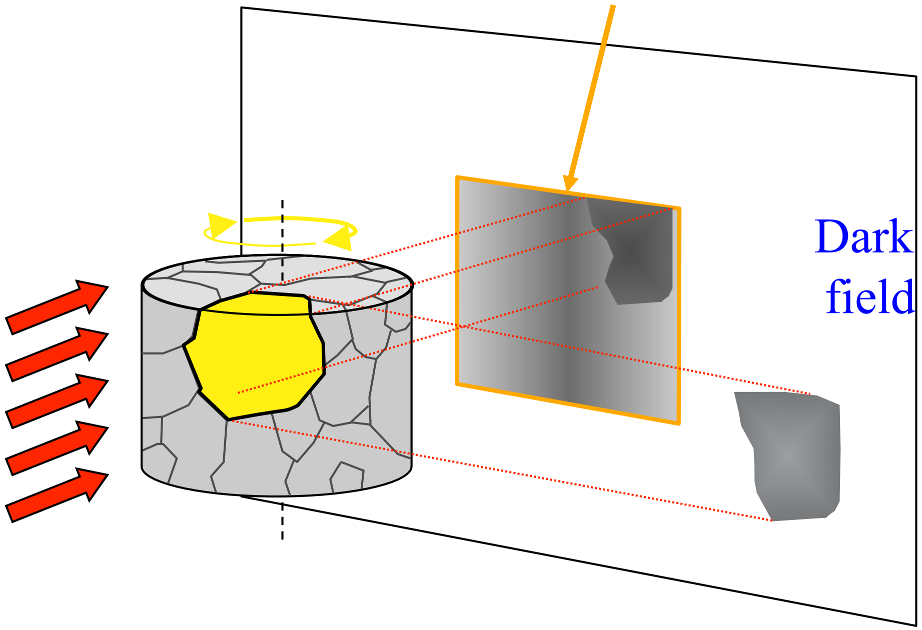


red dots are nuclei

Work by <u>S. Schmidt</u>, S. West, G. Winther, H.F. Poulsen, D. Juul Jensen

Tomography +
3DXRD

Diffraction Contrast Tomography



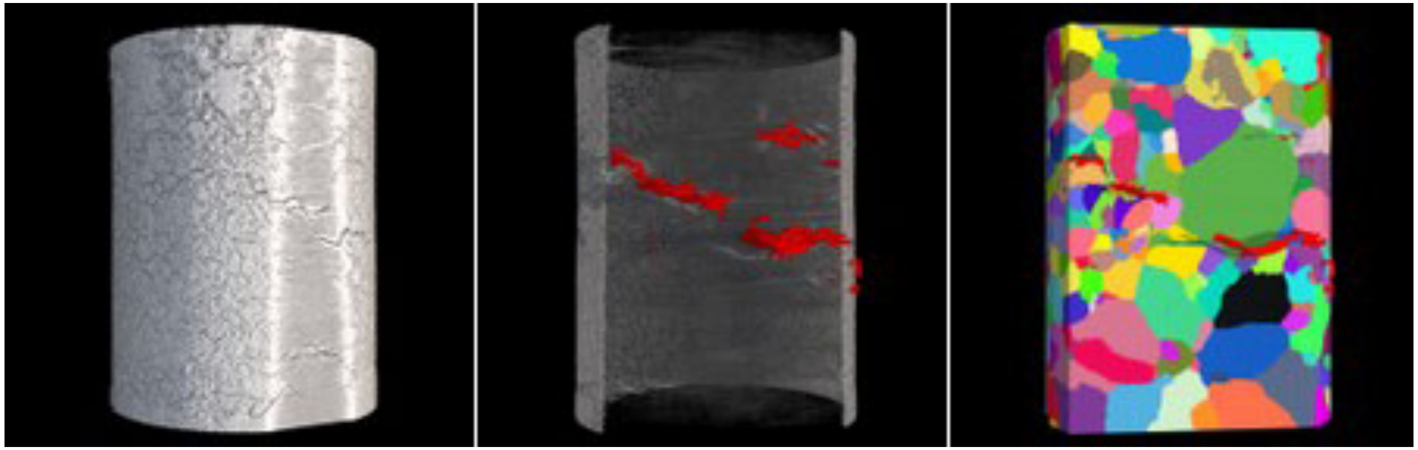
r

Used at ID19, ID11, ID06, …, at ESRF

J. Appl. Cryst. (2008). **41,** 302-309, W. Ludwig, S. Schmidt, E. M. Lauridsen, H. F. Poulsen
J. Appl. Cryst. (2008). **41**, 310-318, G. Johnson, A. King, M. G. Honnicke, J. Marrow and W. Ludwig
R. Sci. Instr. (2009), **80**, 033905, Ludwig, Reischig, King, Herbig, Lauridsen, Johnson, Marrow, Buffiere

Tomography + 3DXRD

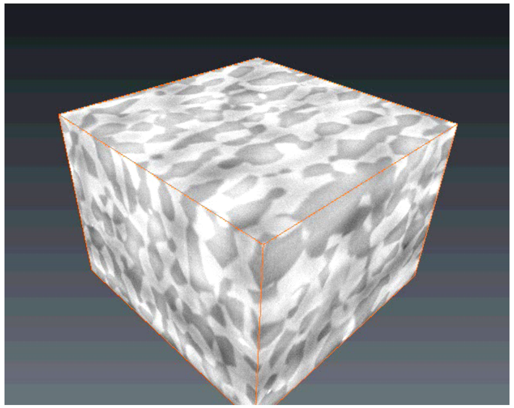
Intergranular Stress Corrosion Cracking in steel:



A. King, G. Johnson, D. Engelberg, W. Ludwig, and J. Marrow, *Science* (2008) **321**, 382 - 385

Multi-phase materials

Tomo:
resolution: 1 μm

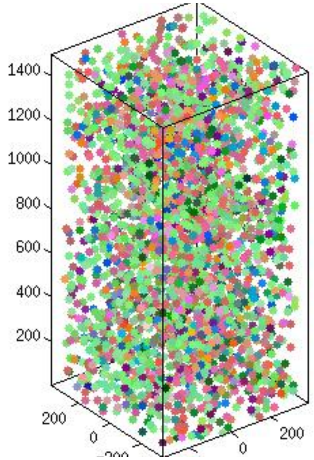


+ 3DXRD

R: Fonda, W. Ludwig, E.M. Lauridsen, S. Poulsen, in work

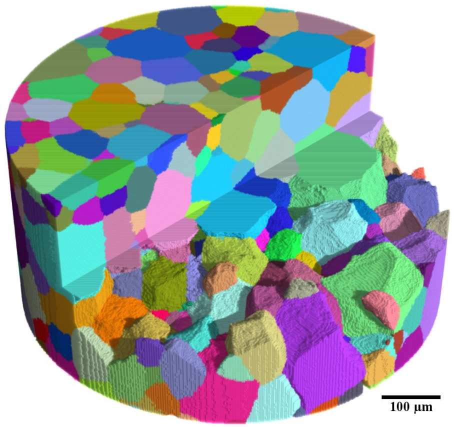
4D methods on micron-scale

Grain center +volume
+ orientation+stress



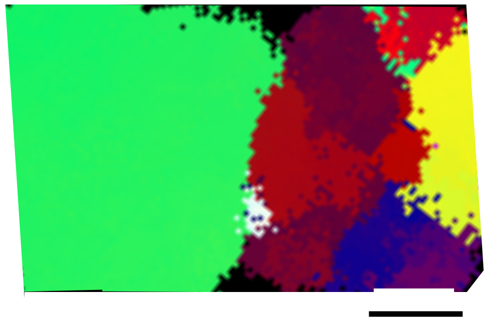
Position: 2 µm

Grain maps
(undeformed)



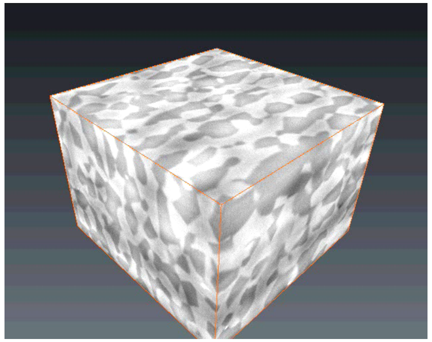
Spatial res: 0.5-5 µm

Orientation imaging
(deformed)



Spatial res: 3 µm

Phases + cracks



Spatial res: 1 µm

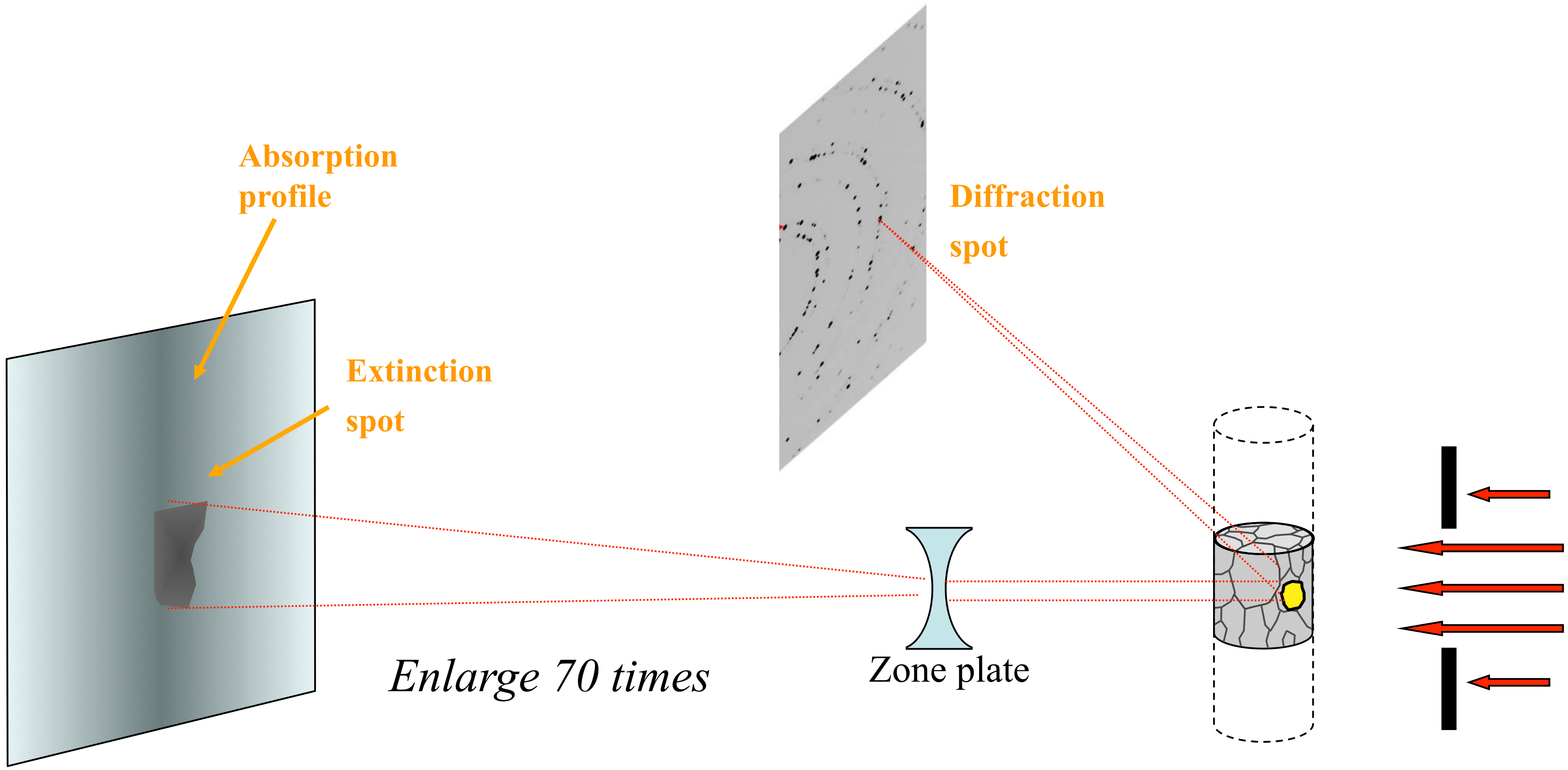
More ….

Plastic strain mapping
Structural refinement
Maps of defect populations

Outlook

Nano-3DXRD using full field microscope

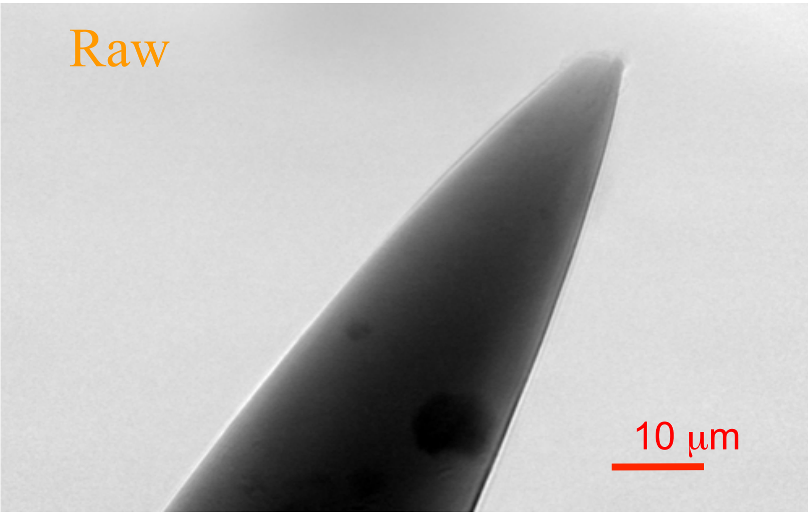
Demonstration at XU47 at SPring-8

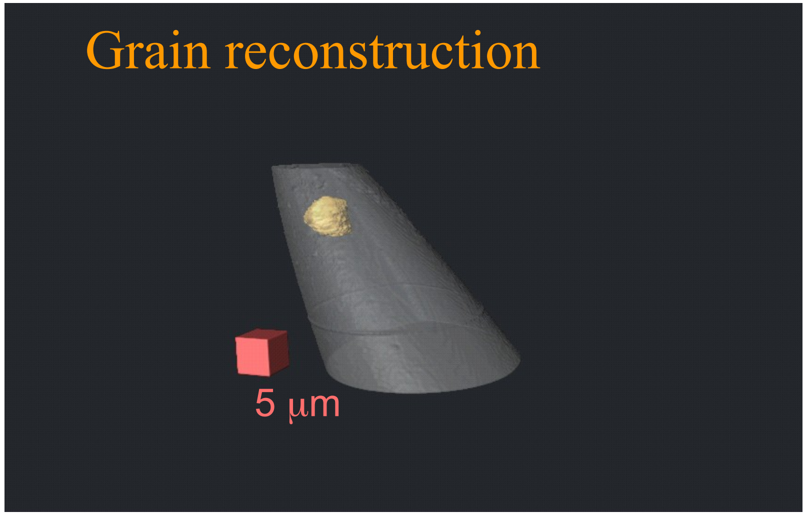


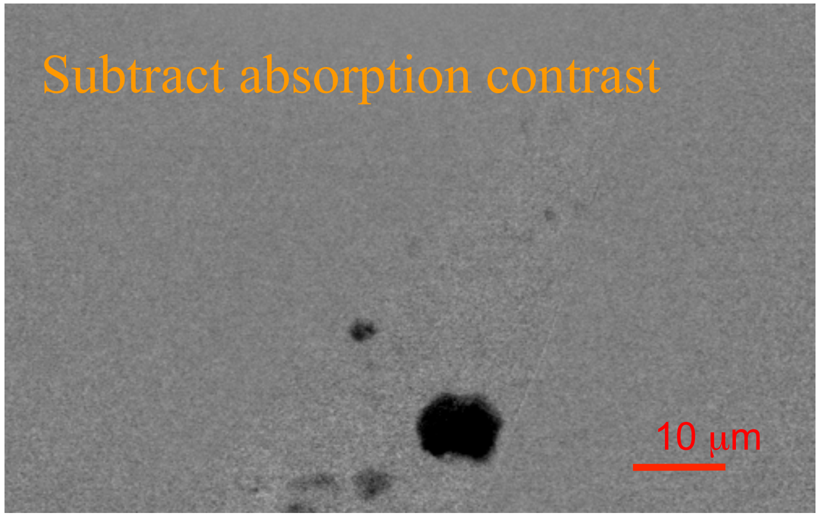
Risø DTU: E.M. Lauridsen, X. Huang, H.F. Poulsen
Toyohashi: M. Kobyashi. SPring-8: K. Uesugi

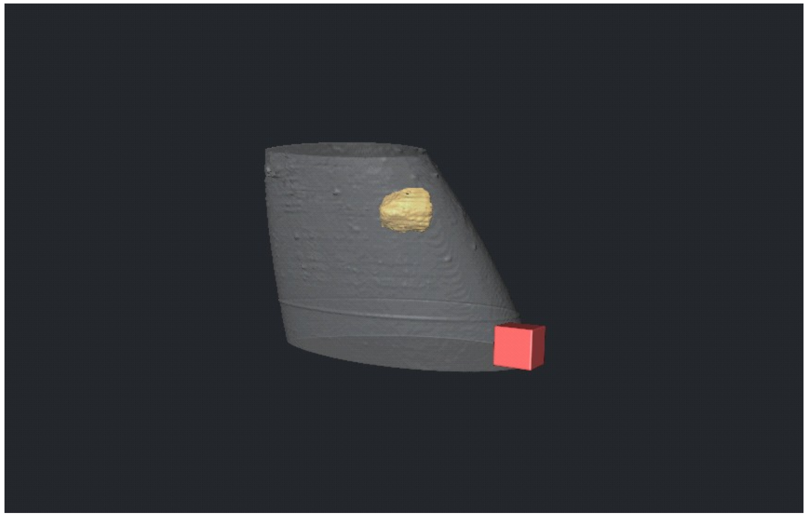
Nano-3DXRD

Sample: 99.99% pure Al. 6 cycles ARB => strain of 4.8









Beamlines for irradiated samples

Beamline MARS at Soleil, Paris: X ray powder-diffraction (XRD), X ray fluorescence (XRF), X ray absorption spectroscopy (XAS).

APS, Chicago: Workshop on Synchrotron Radiation for Nuclear Energy Systems, APS 27 Jan 2010.

More on 4D:

Søren Schmidt: I-12: *Latest Achievements Utilizing the 3DXRD Technique for Non-destructive Characterization of Microstructures*