

A Light for Science



European Synchrotron Radiation Facility

X-ray imaging



Alexander Rack

European Synchrotron Radiation Facility, France

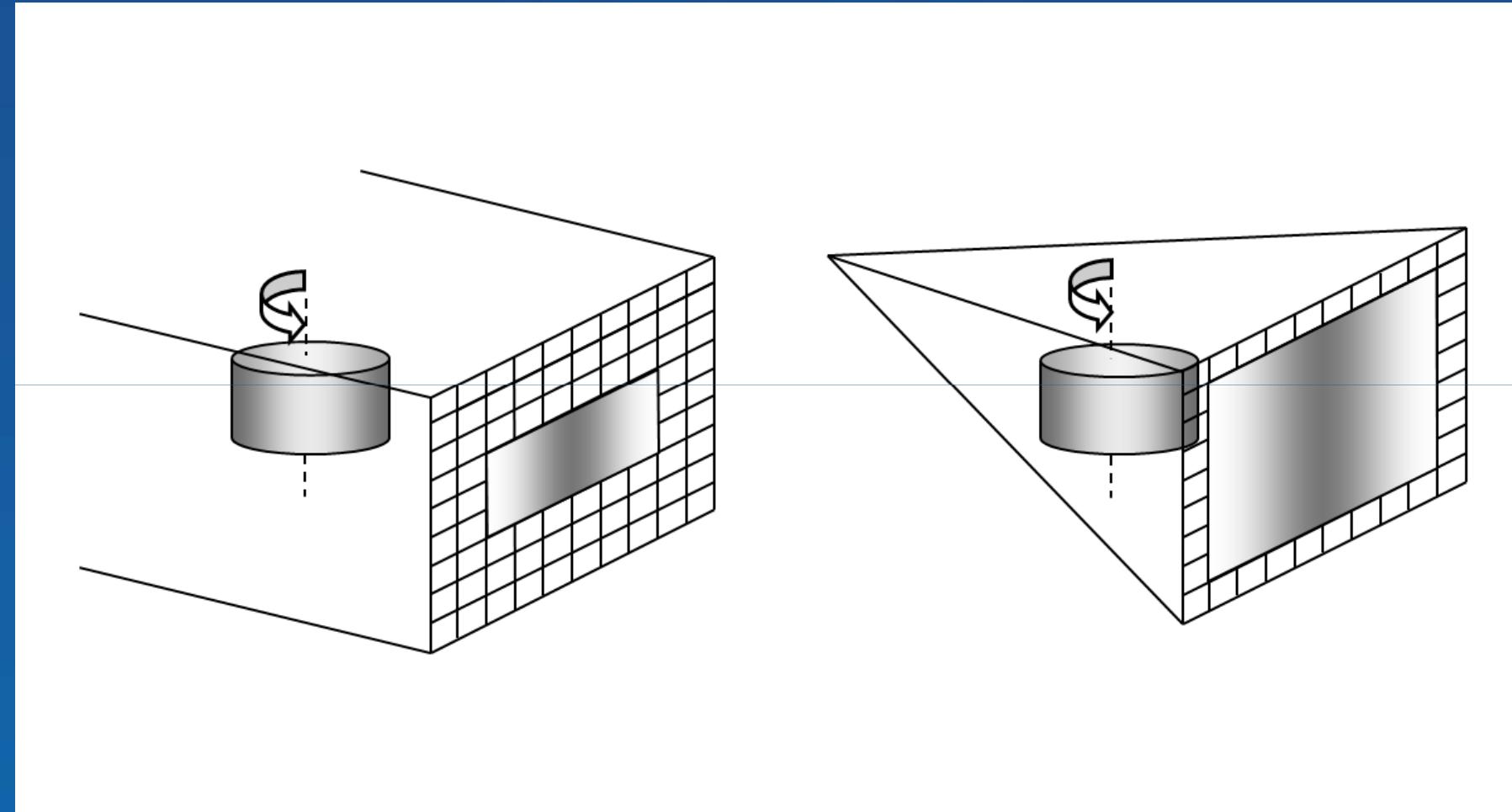
European Synchrotron Radiation Facility

Introduction

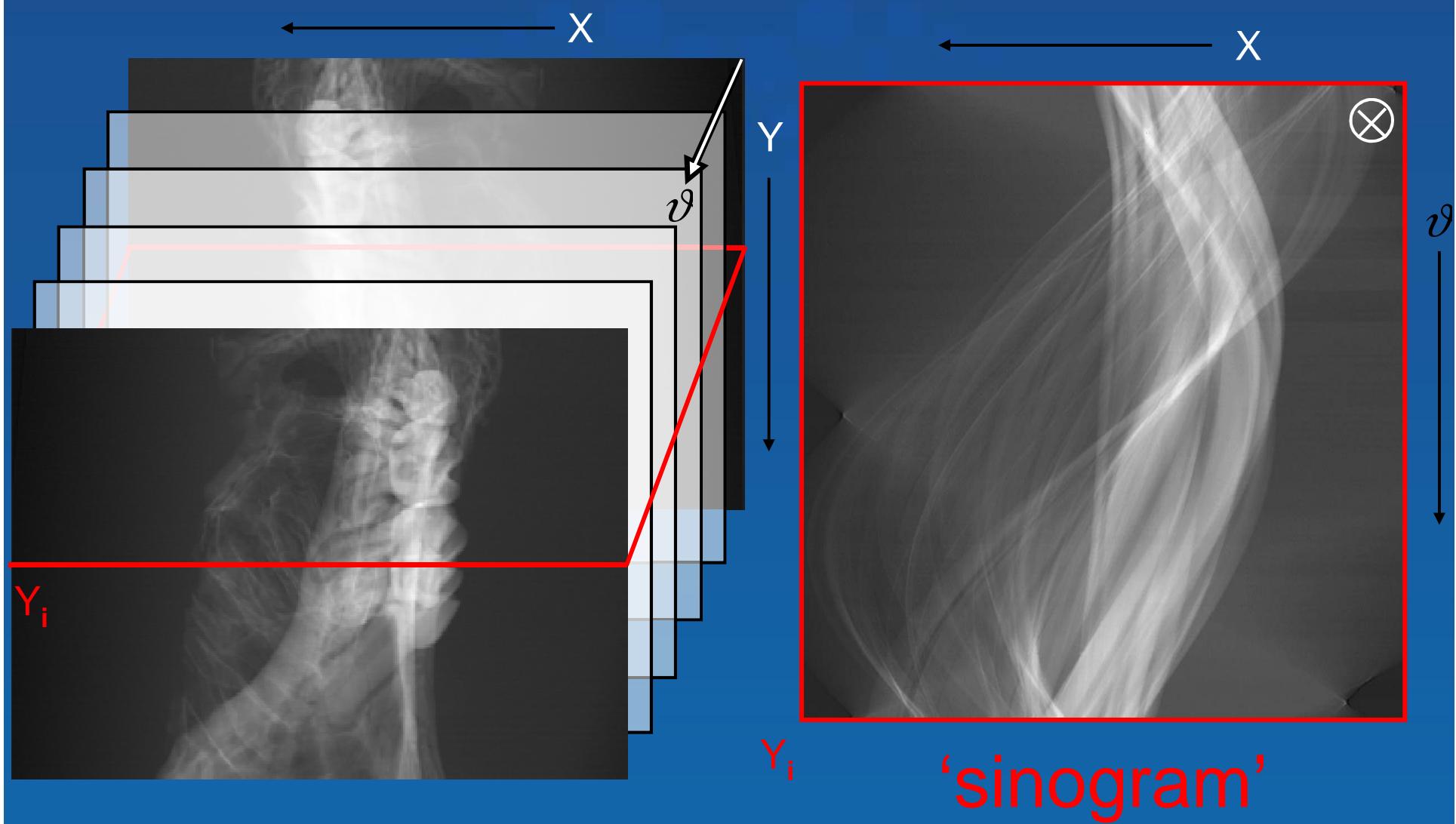
- X-ray Imaging, Contrast & Spatial Resolution -

Full-field X-ray Imaging

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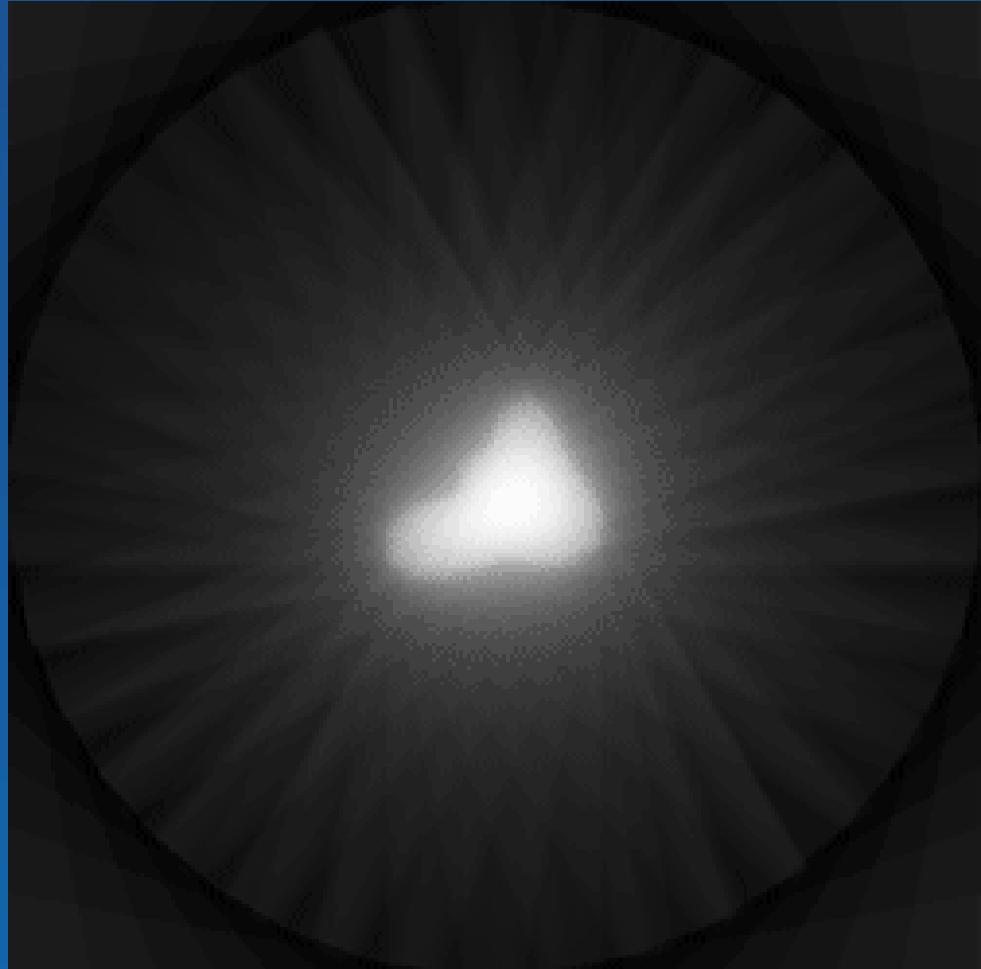


Tomographic Reconstruction



$$\frac{\text{number of projections}}{\text{pixels per line}} \geq \frac{\pi}{2}$$

(Shannon's theorem)



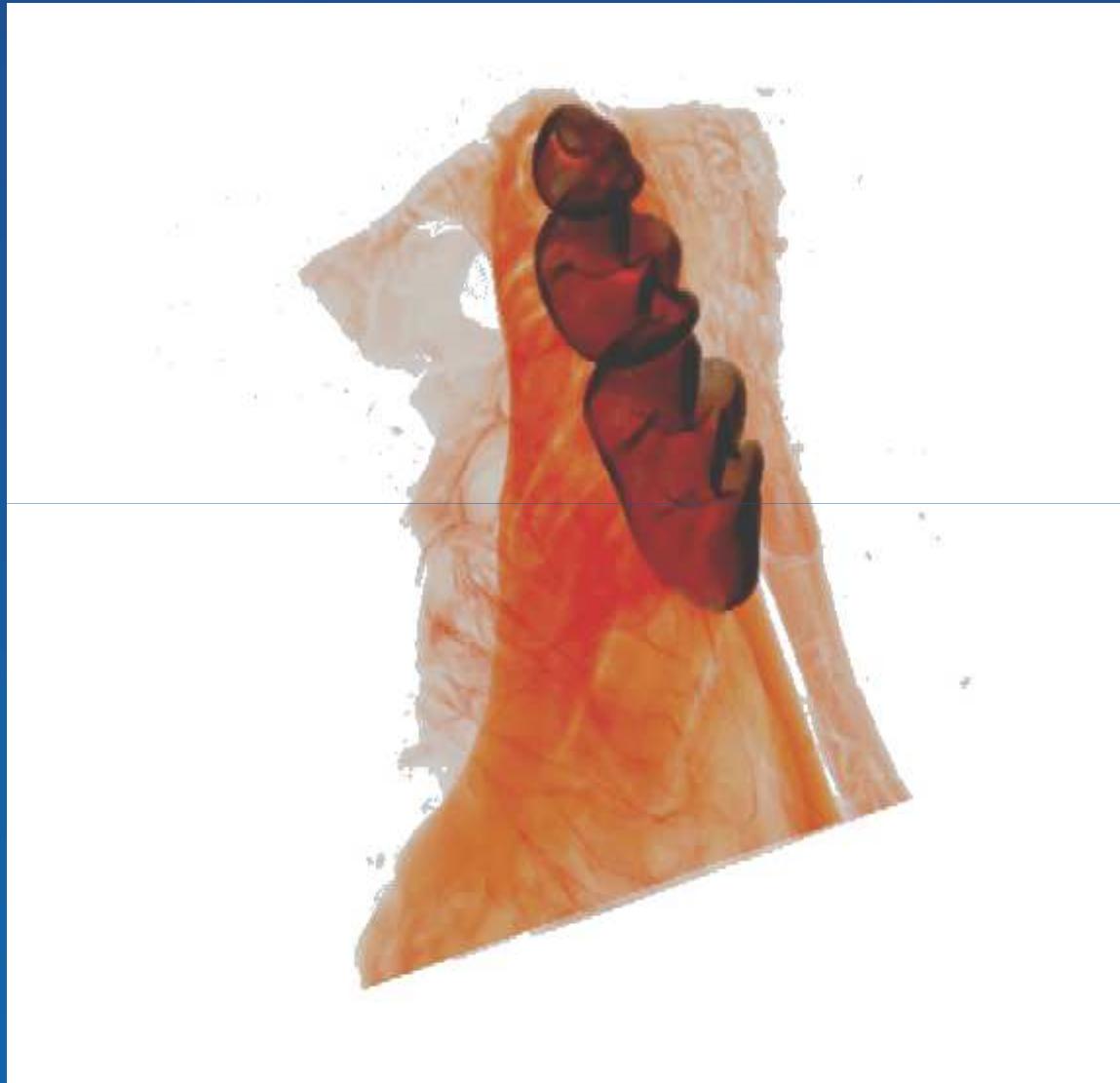


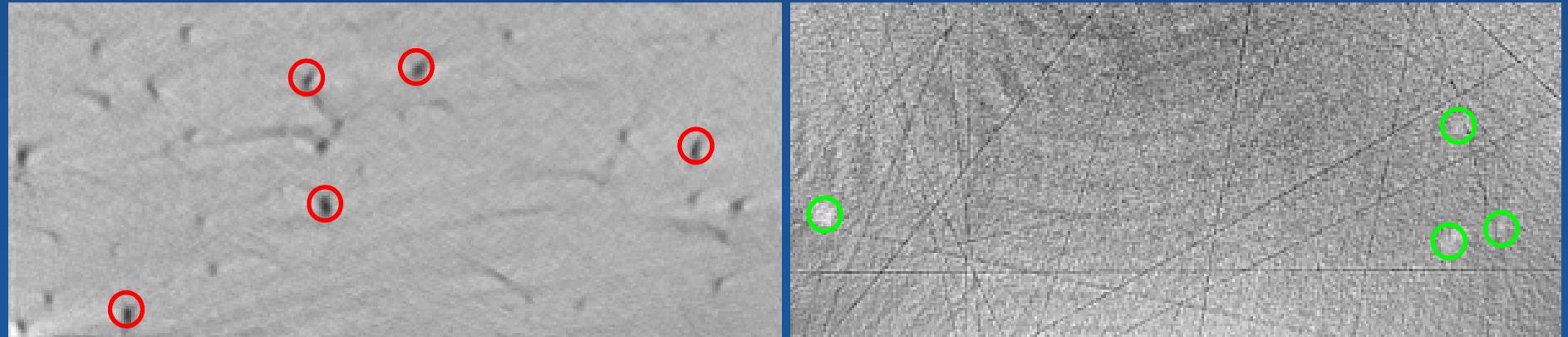
FBP



Volume Image = Stack of Slices

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Zn+ TiH_2
bulk

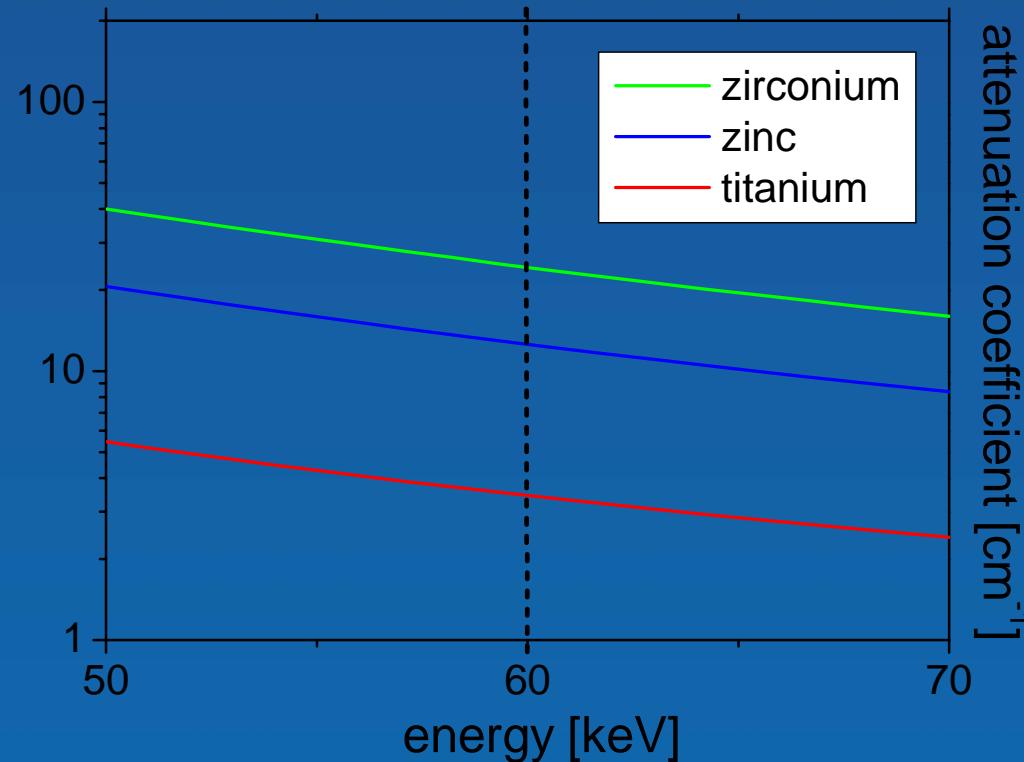
pre-cursor:

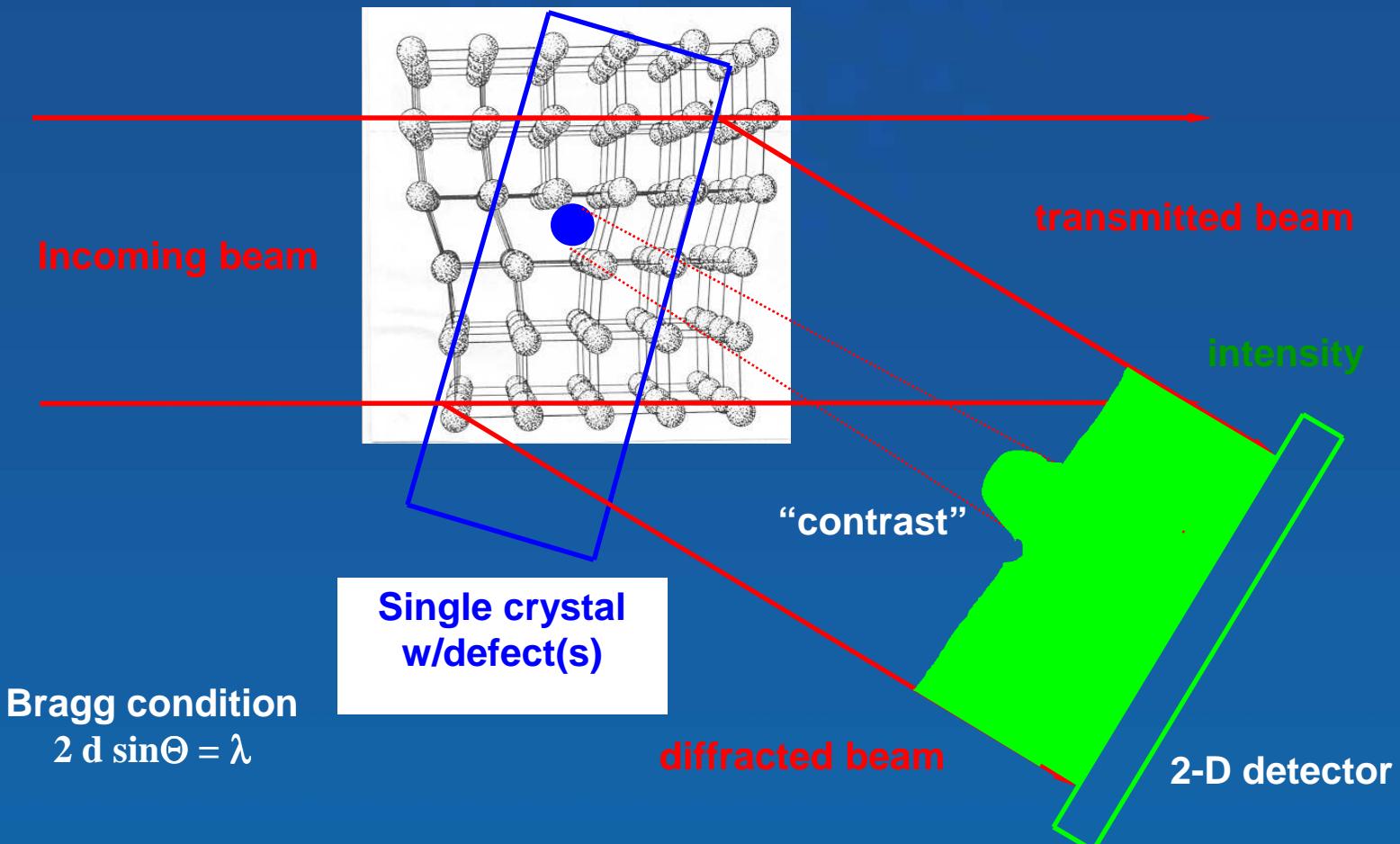
0.50 vol%

Zn+ ZrH_2
bulk

pre-cursor:

0.66 vol%



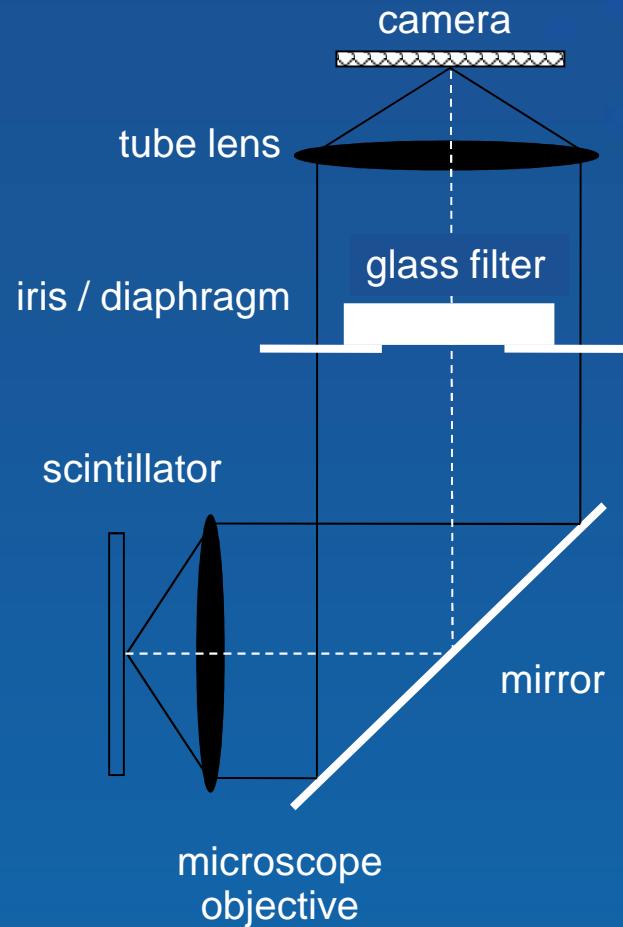


courtesy: J. Härtwig (ESRF)

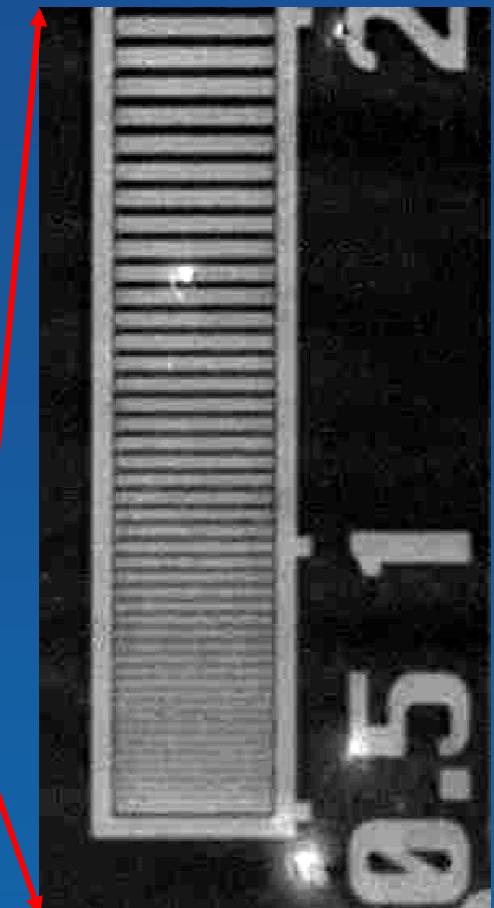
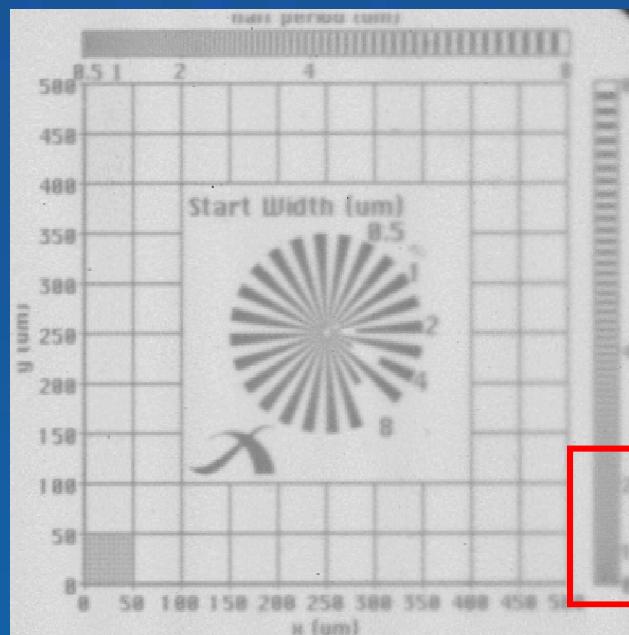
energy-dispersive	scanning	2D/3D XRF, XAS	<i>silicon drift diode</i>
high resolution full-field	full-field	magnified (holo-)μCT	<i>indirect detection</i>
single photon counting	scanning	ptychography	<i>pixel detector vs. integrating</i>
very large field of view	scanning / full-field	2D/3D XRD	<i>CCD w/taper</i>

dynamic range, efficiency, noise, read-out, radiation hardness ...

Bonse, Busch 1996



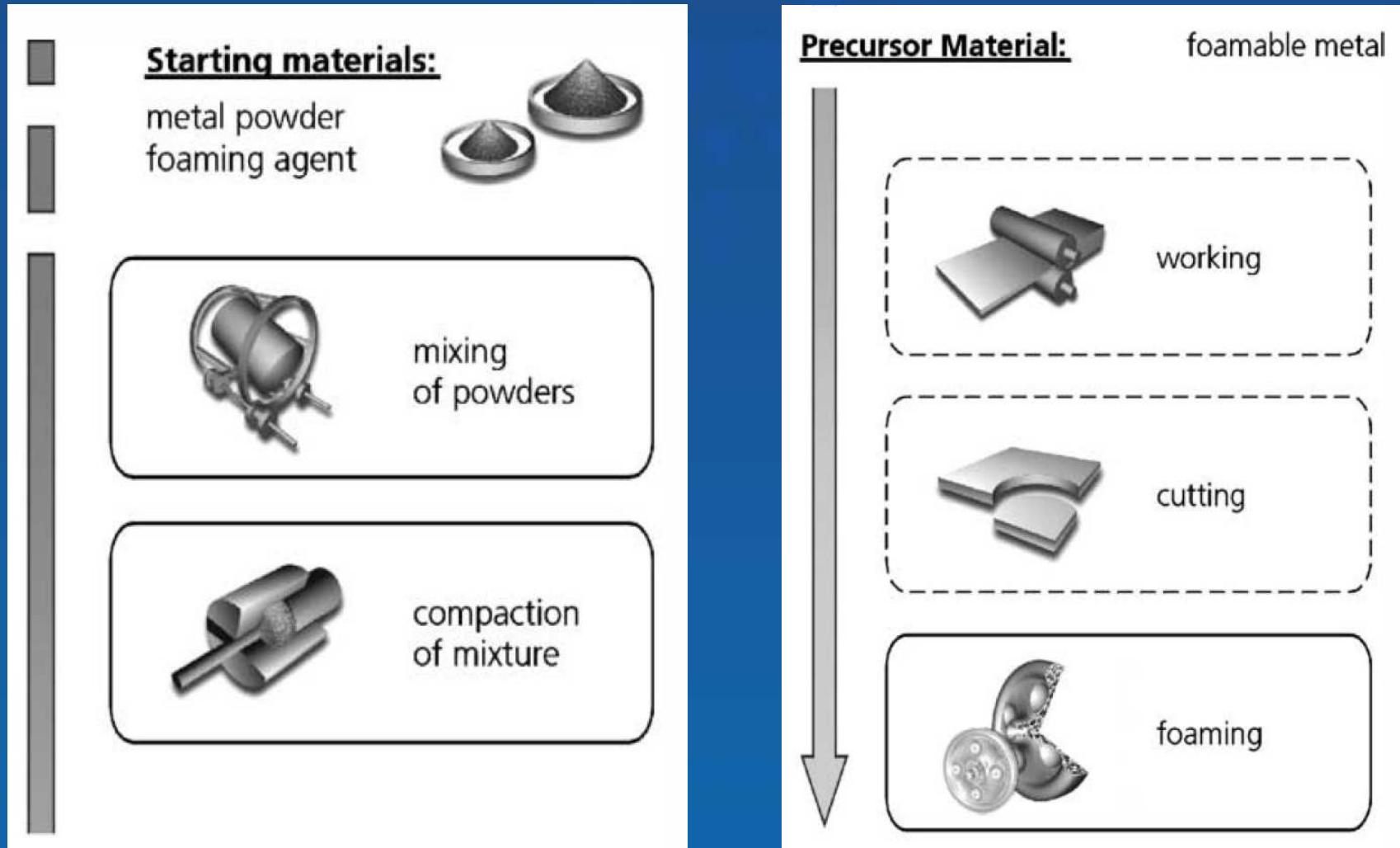
resolution, efficiency



Rack et al., Nucl Instr Meth B 267 (2009)

Metallic Foams

- Microtomography & Image Analysis -



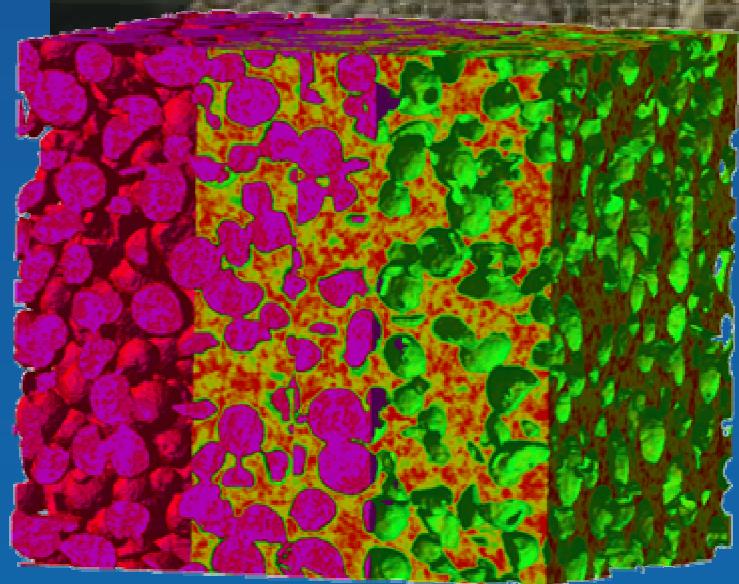
J. Banhart, Prog Mat Sci (2001)

Metallic Foams

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HZB Berlin

1 cm



1 mm

European Synchrotron Radiation Facility



Fraunhofer IFAM Bremen

A. Rack - X-ray Imaging, ESRF/ILL Summer School

15

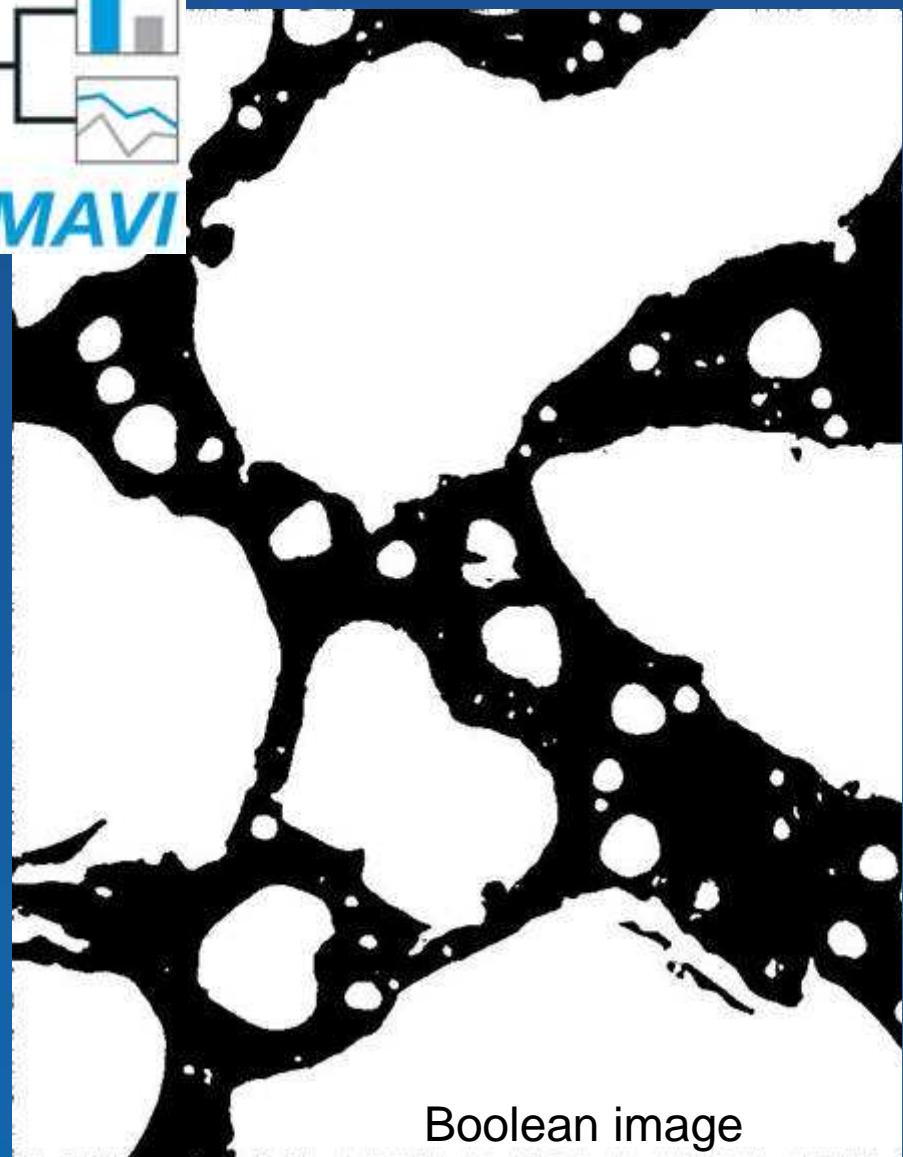
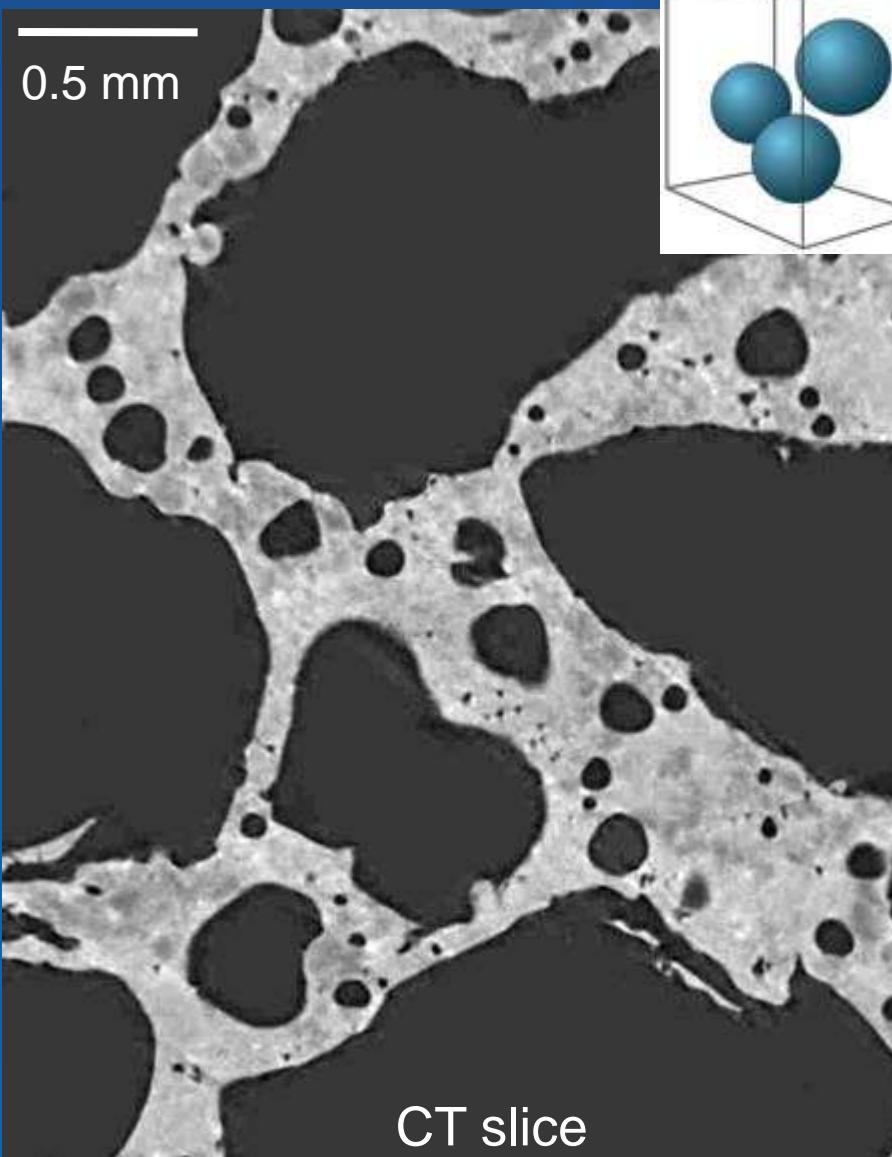
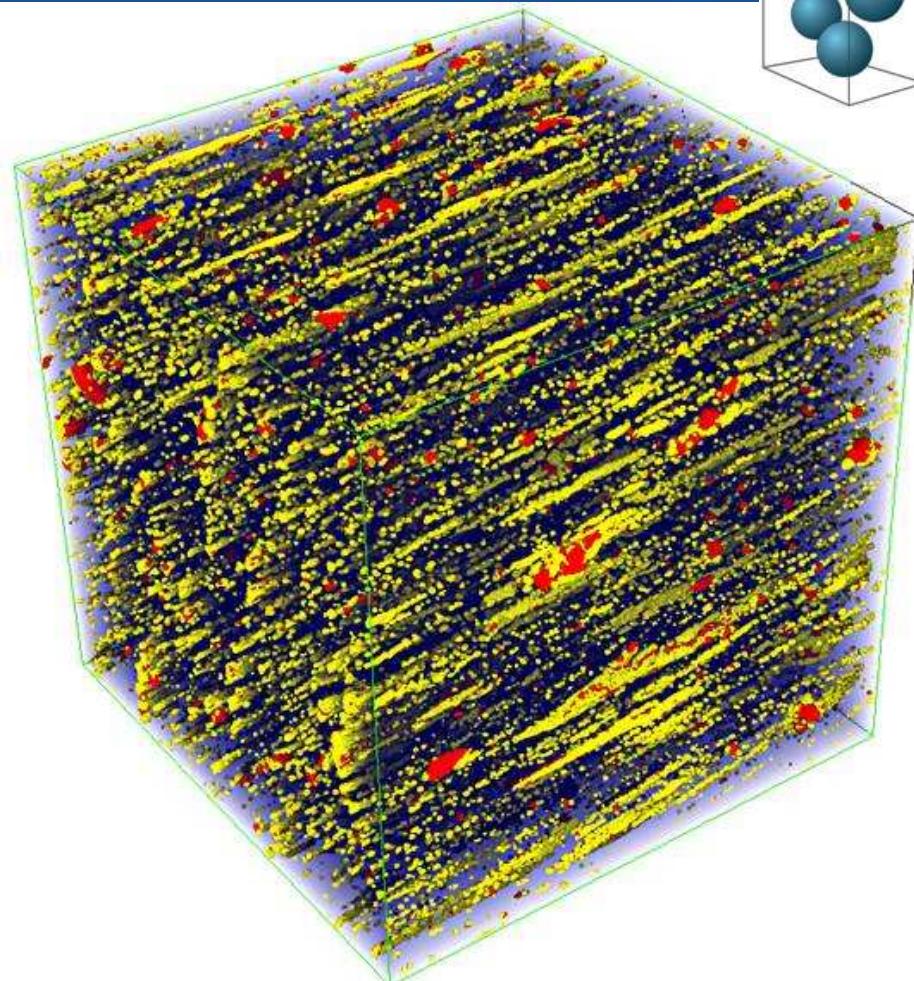
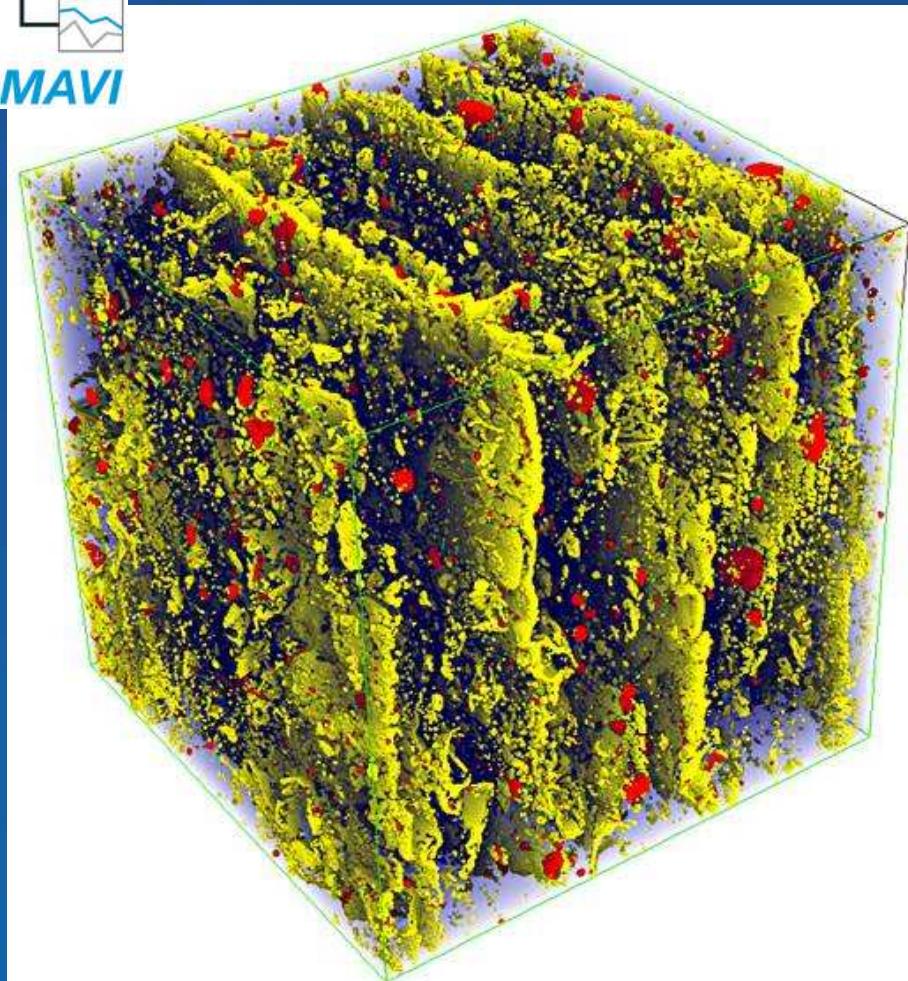


Image Analysis

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Al6061 (commercial AlSi alloy)

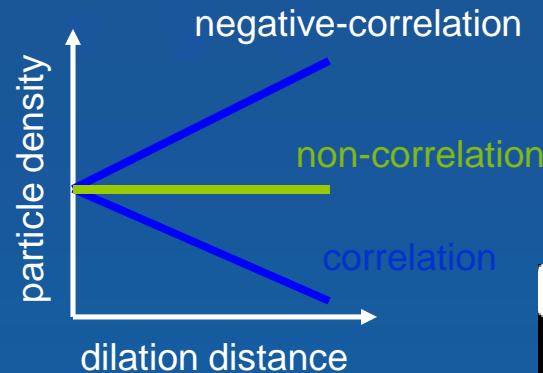
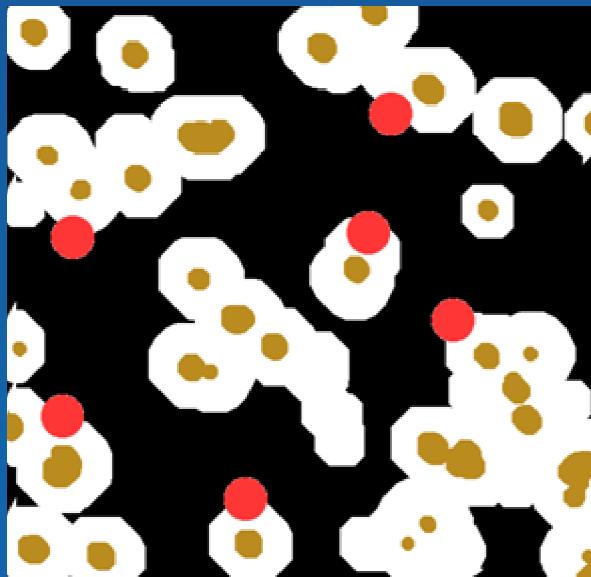


AlSi7 (elemental Al-Si mixture)

pore dilation \boxtimes calculation of the particle density found in each pore neighborhood

decreasing/increasing
particle density

correlation/negative-correlation

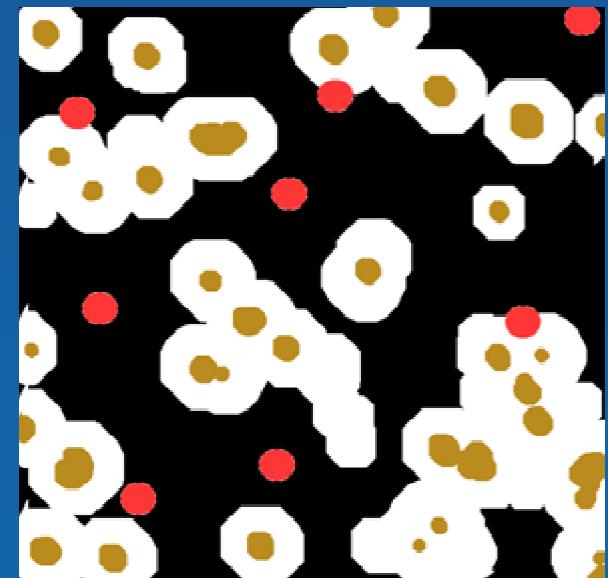


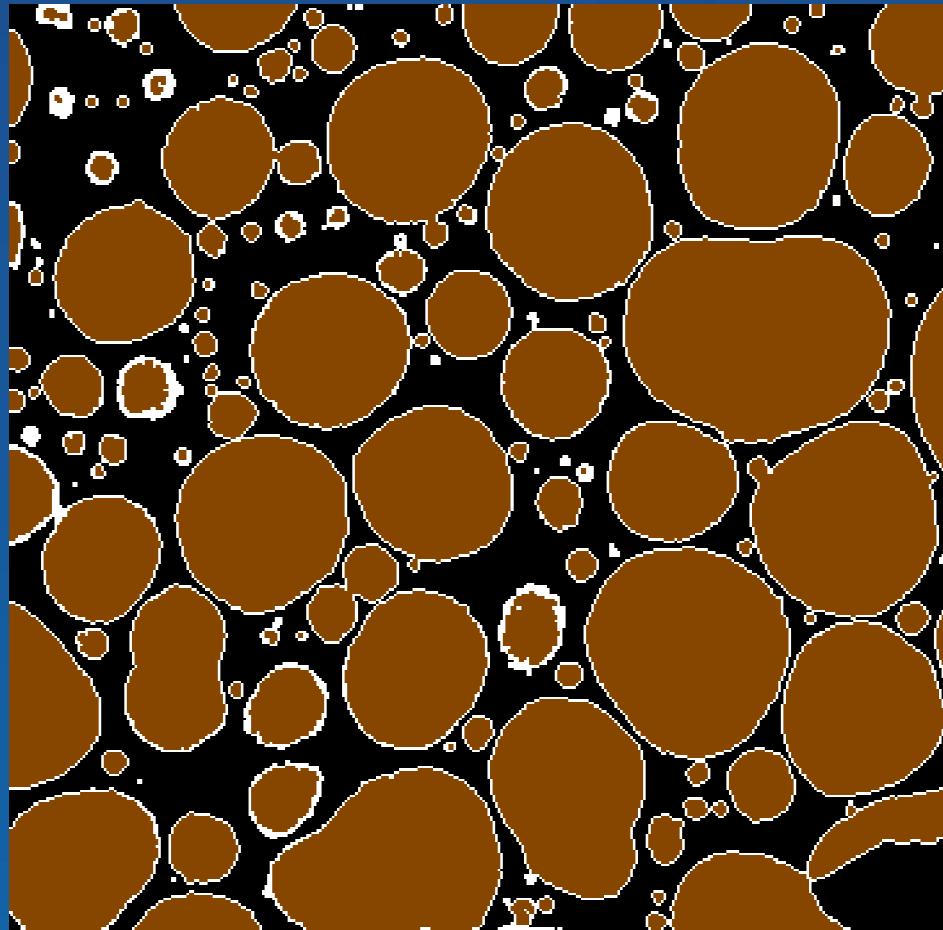
- particle
- pore neighborhood
- pores

Helfen, Ohser, Schladitz et al., Proc. SPIE 2003

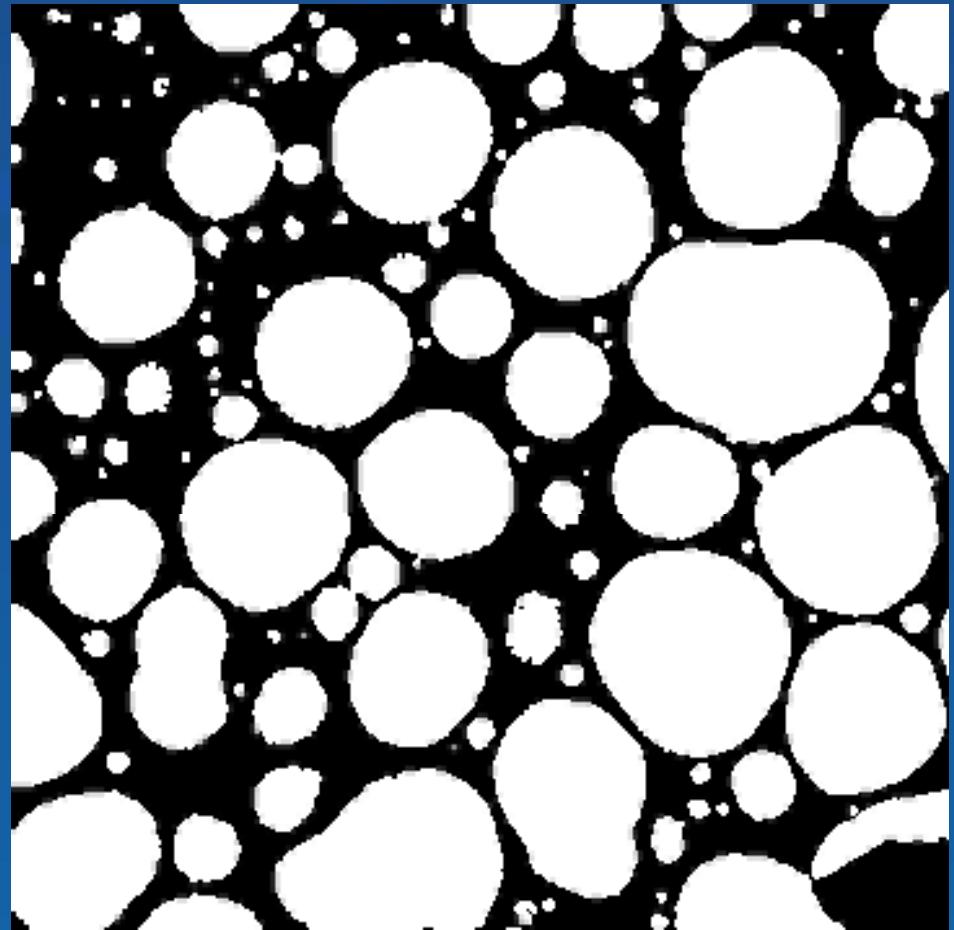
constant particle density

non-correlation



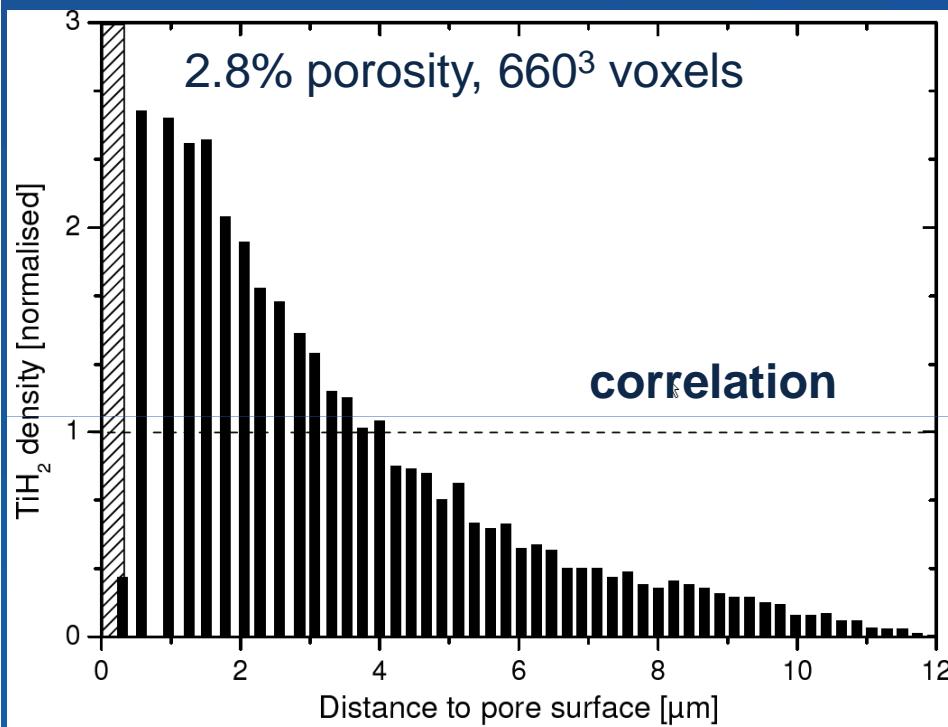


First dilation step

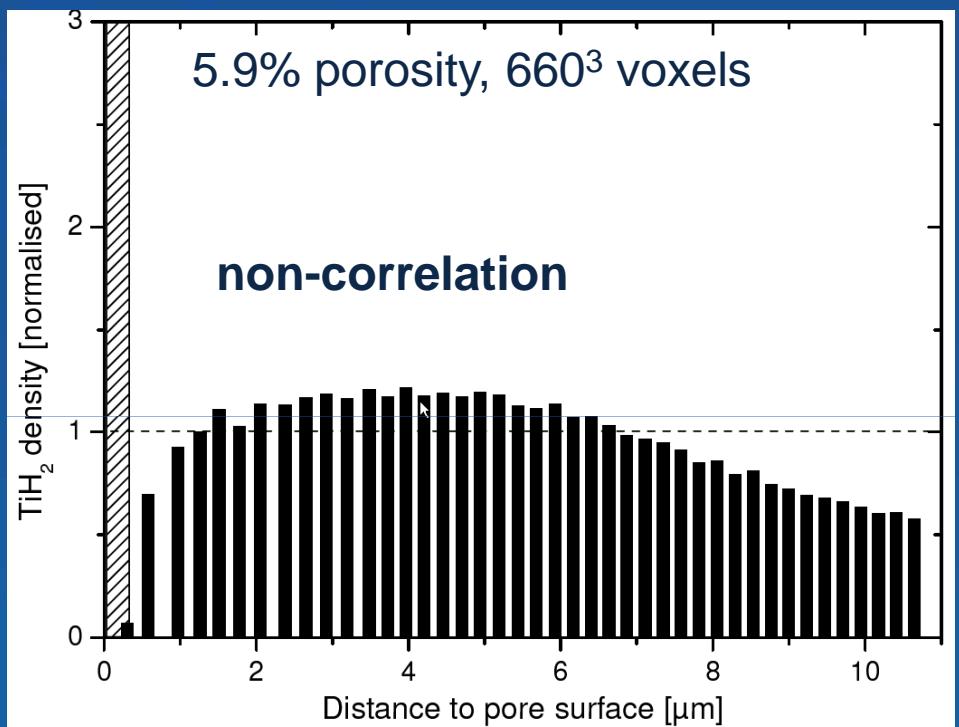


Dilation of pore volume in successive steps

Al6061 (commercial AlSi alloy)



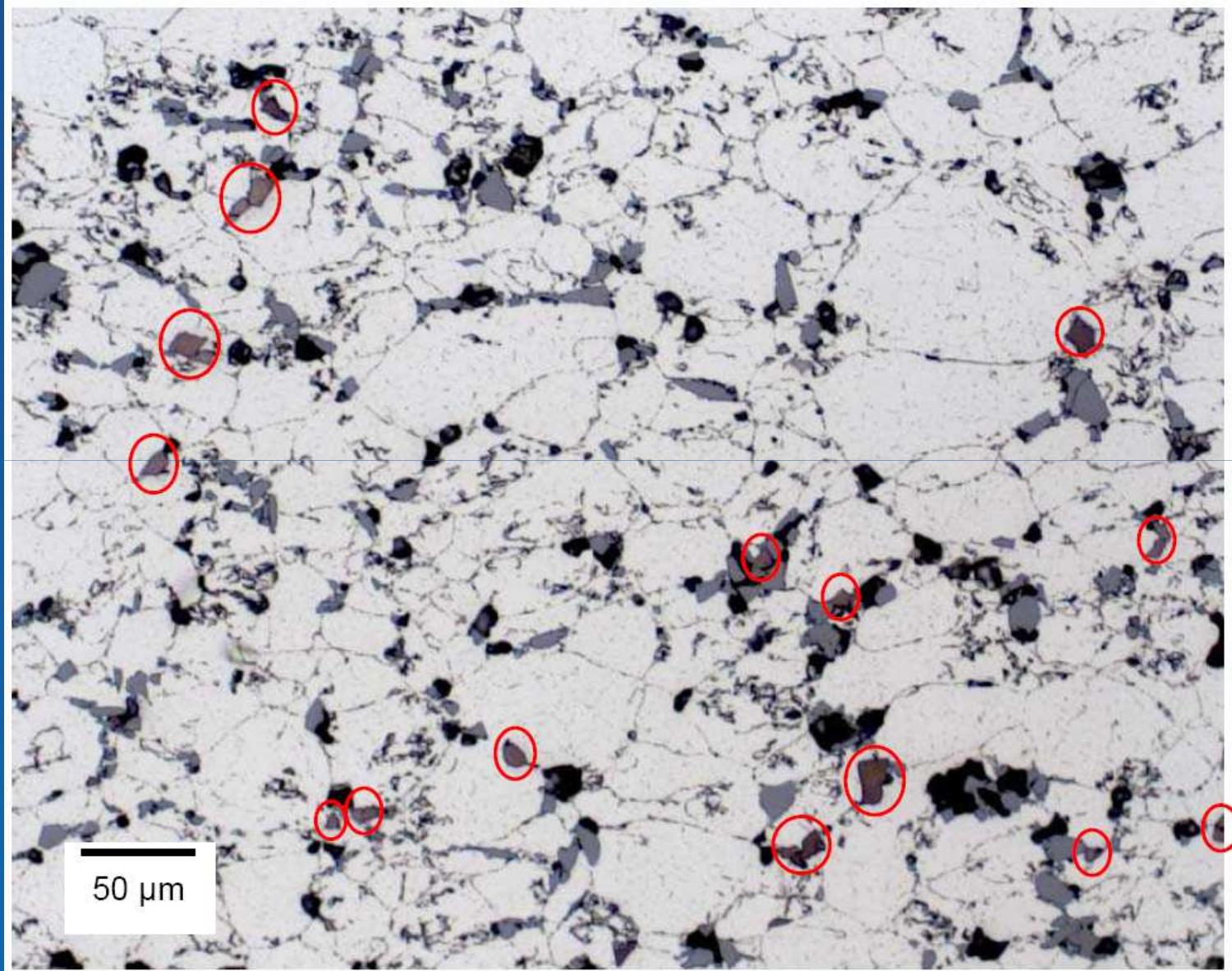
AISi7 (elemental Al-Si mixture)

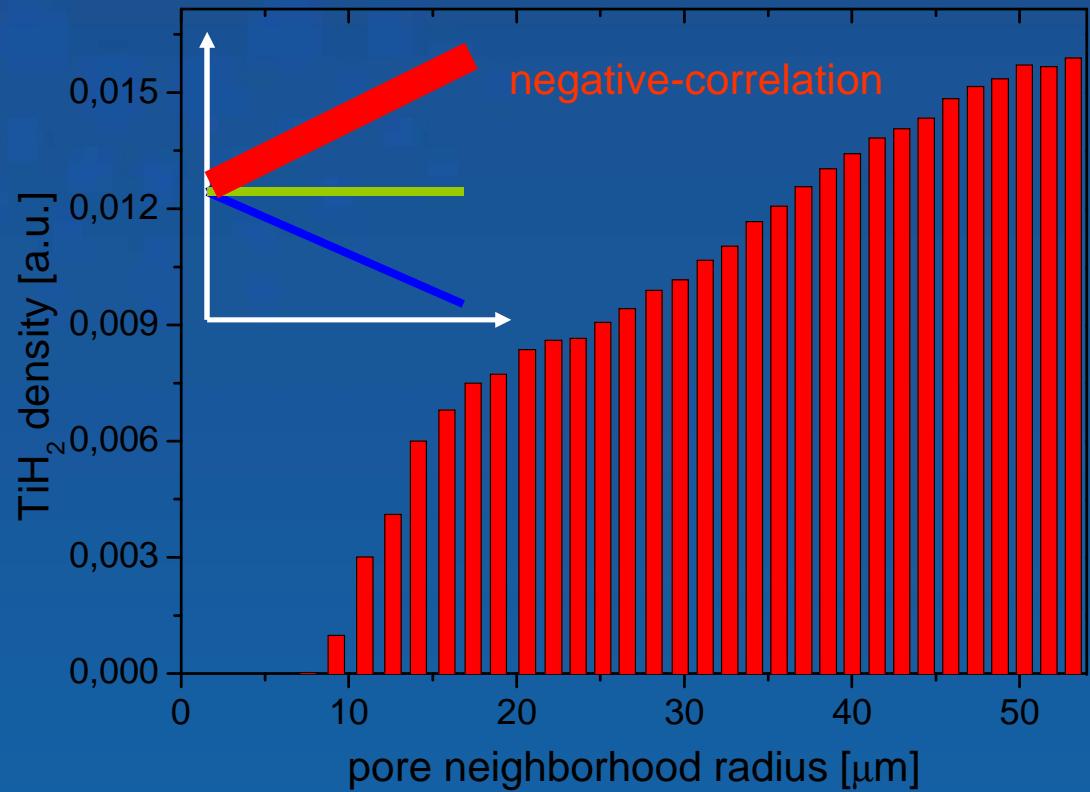
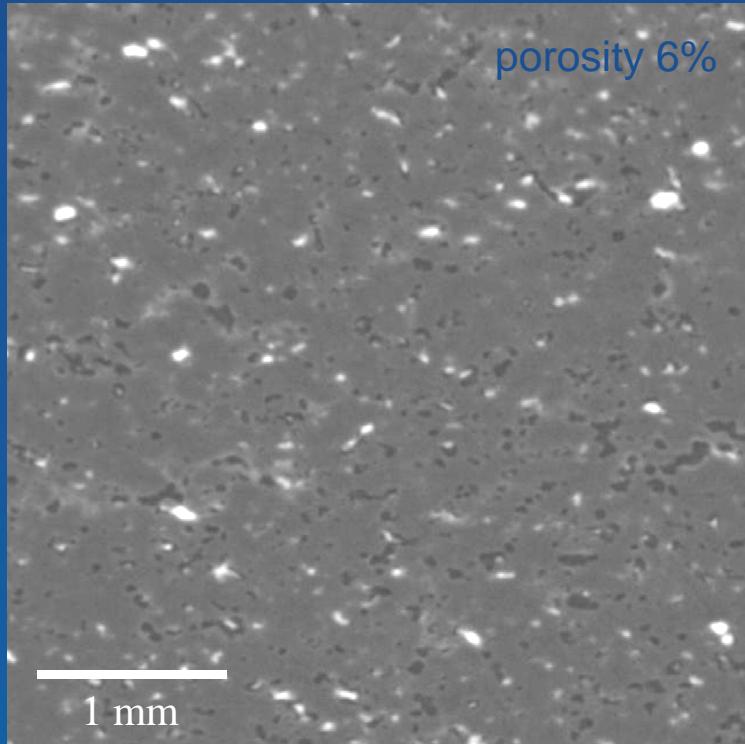


$$t_2(r) = \frac{F_{\Xi, \Psi'}(r)}{F_{\Xi, \Xi^c}(r)}, \quad r \geq 0$$

Rack, Ohser, Schladitz, Helfen et al., Journal Microscopy, 2008

Metallographic image of AlSi7

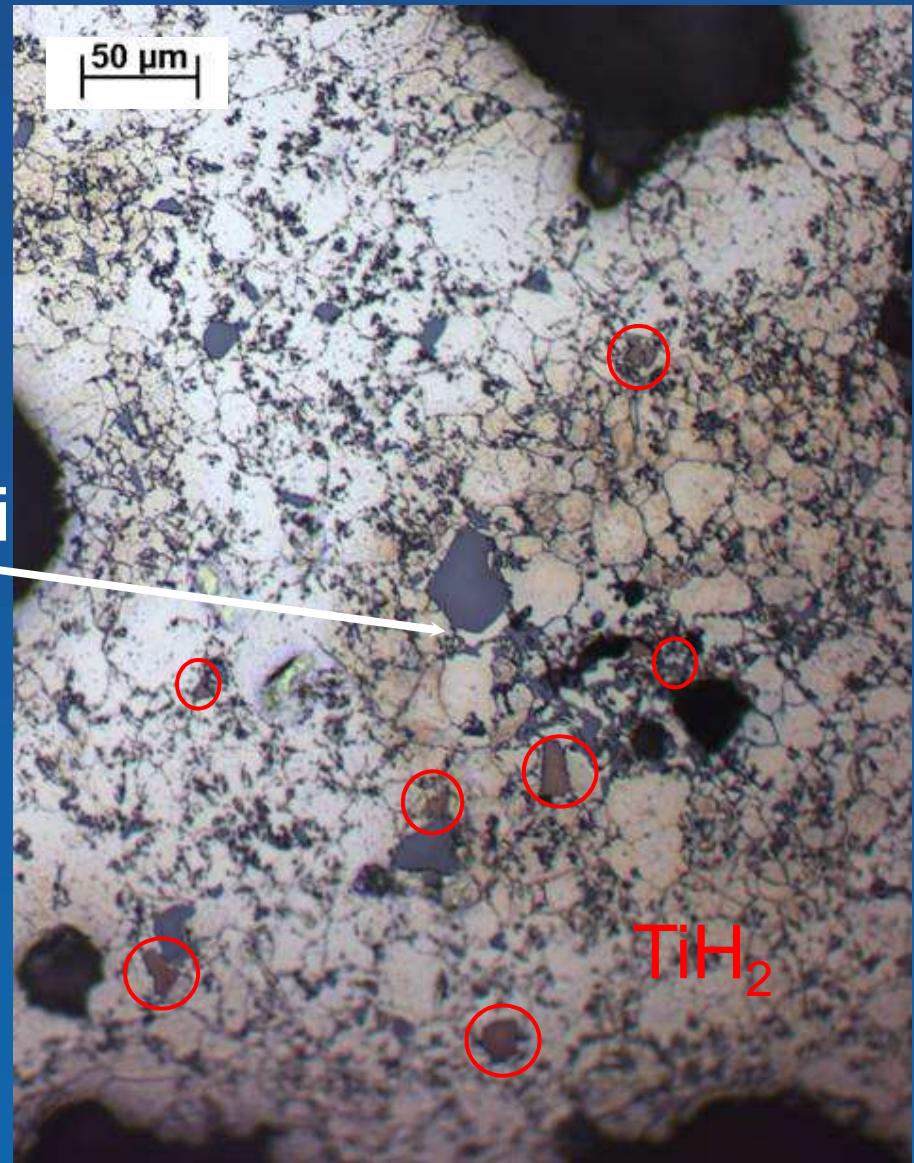




spatial negative-correlation between pores and blowing agent

Rack, Bülow, Banhart, et al., Acta Materialia 2009

Metallic Foams - AlSiCu



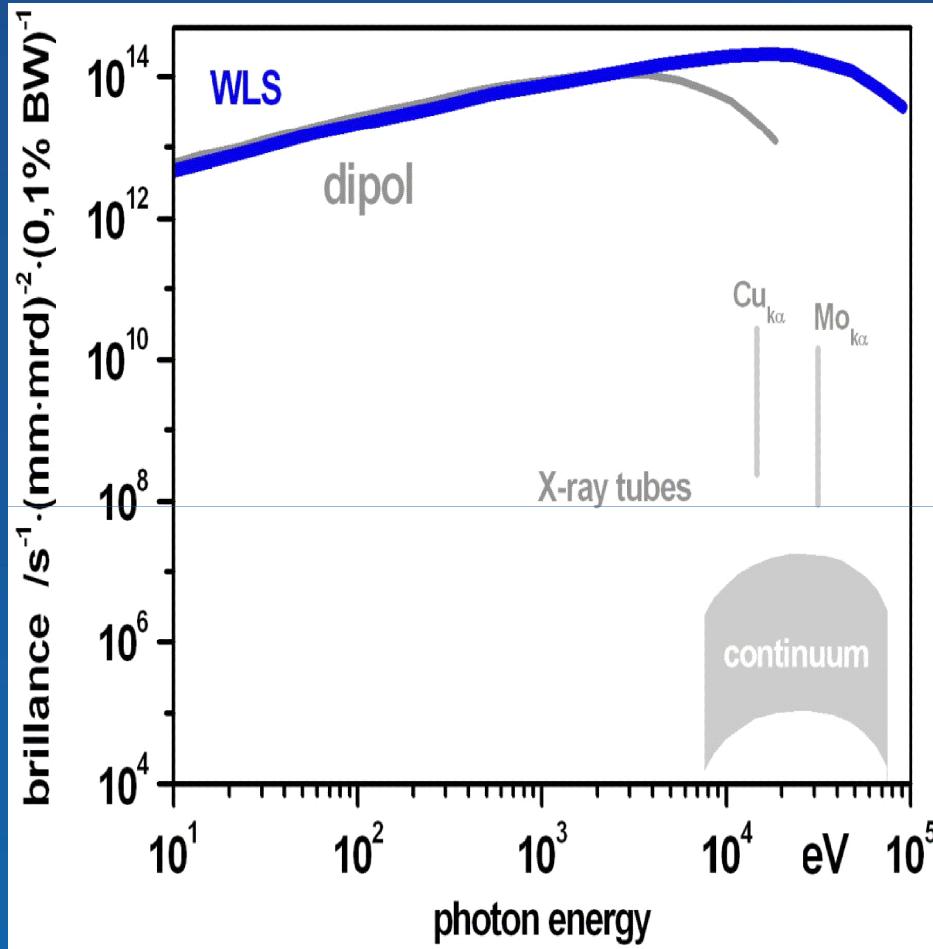
Exploiting Contrast with Tomography

- Synchrotron Light Sources, Scanning Techniques -

Synchrotron Light Sources

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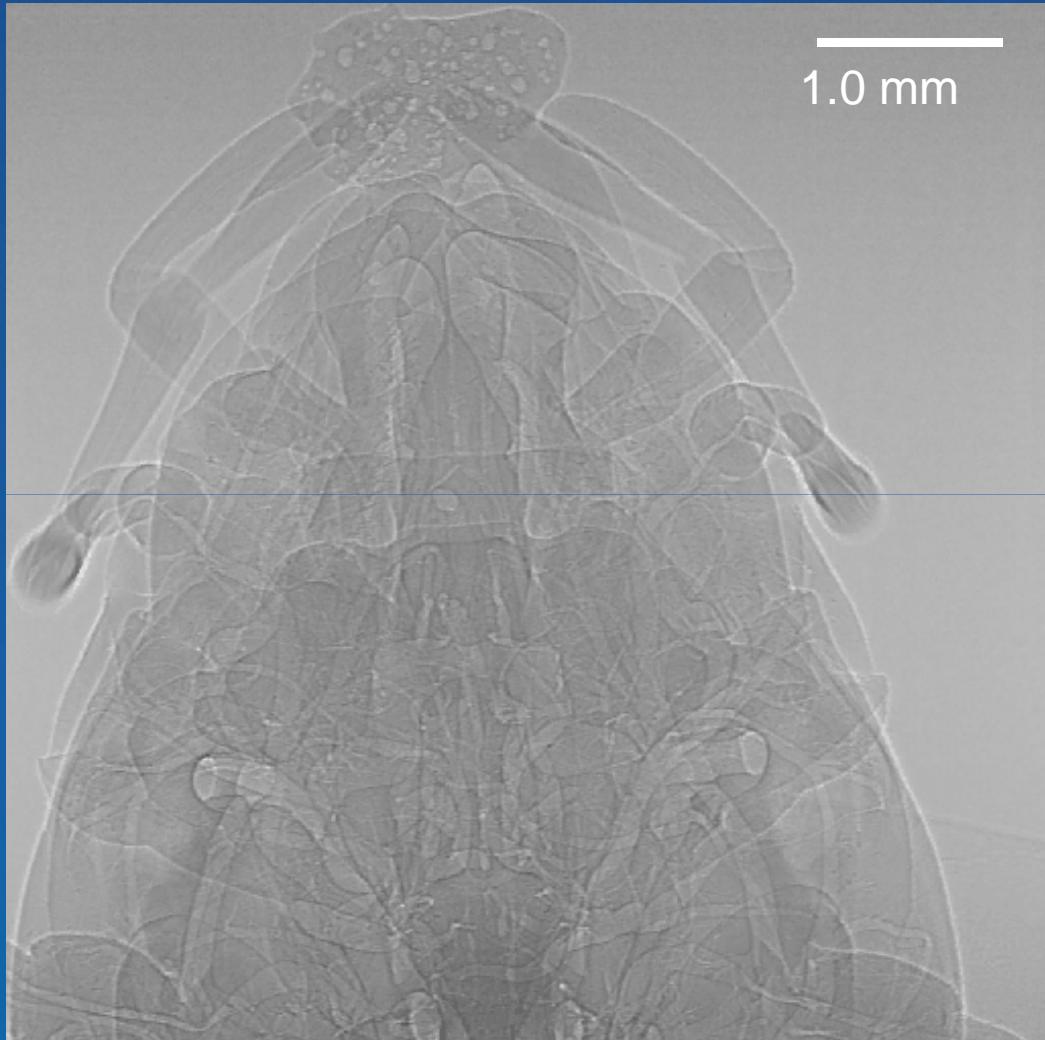


B.R. Müller et al., DGZfP 2005

- higher flux → allows the use monochromators:
 - higher contrast
 - no beamhardening artifacts
- quasi parallel beam
- partial spatial coherence - use of different contrast modes for higher sensitivity, e.g. the local electron density (holo-CT)

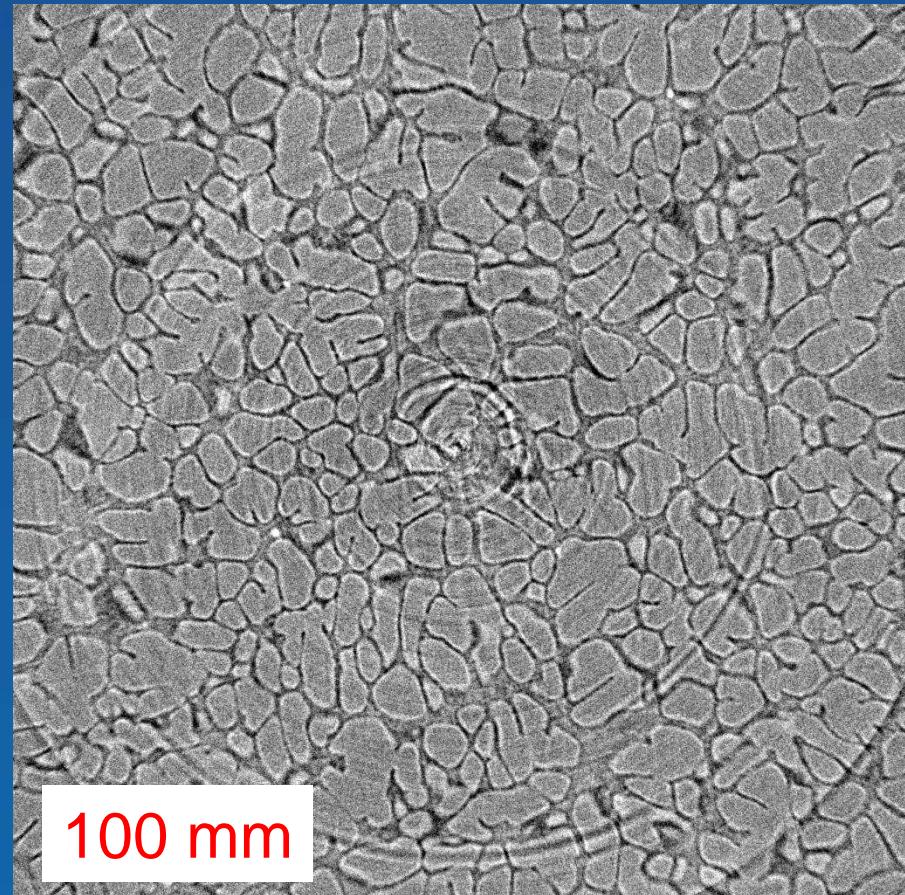
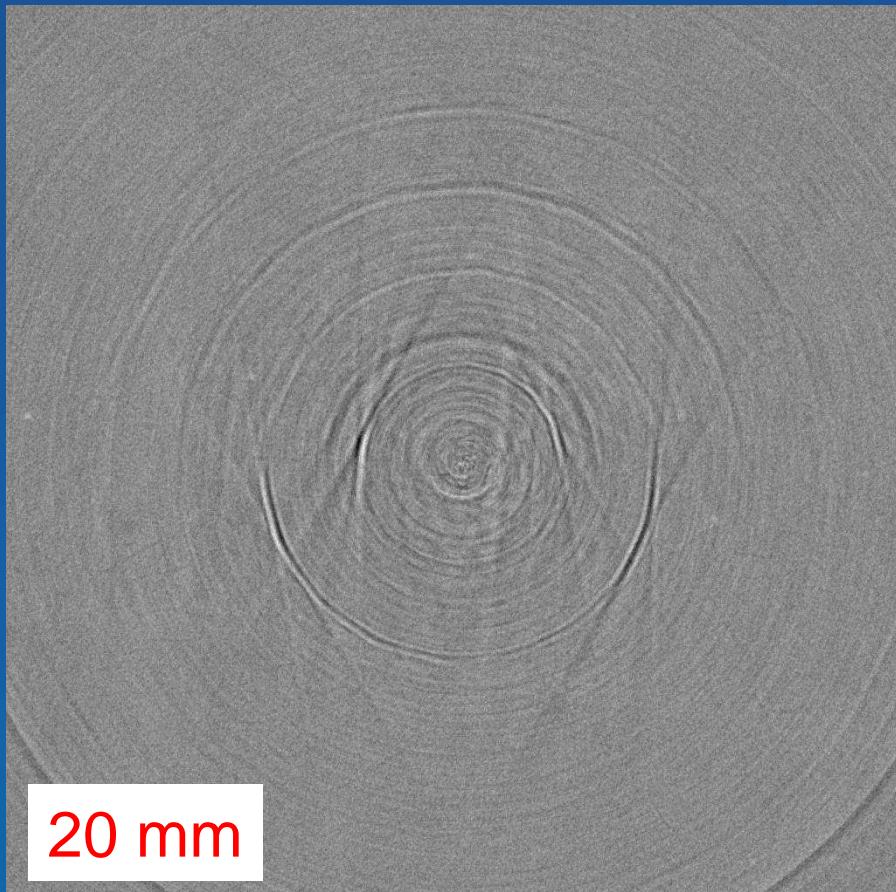
but:

- relative expensive
- only limited amount of beamtime available

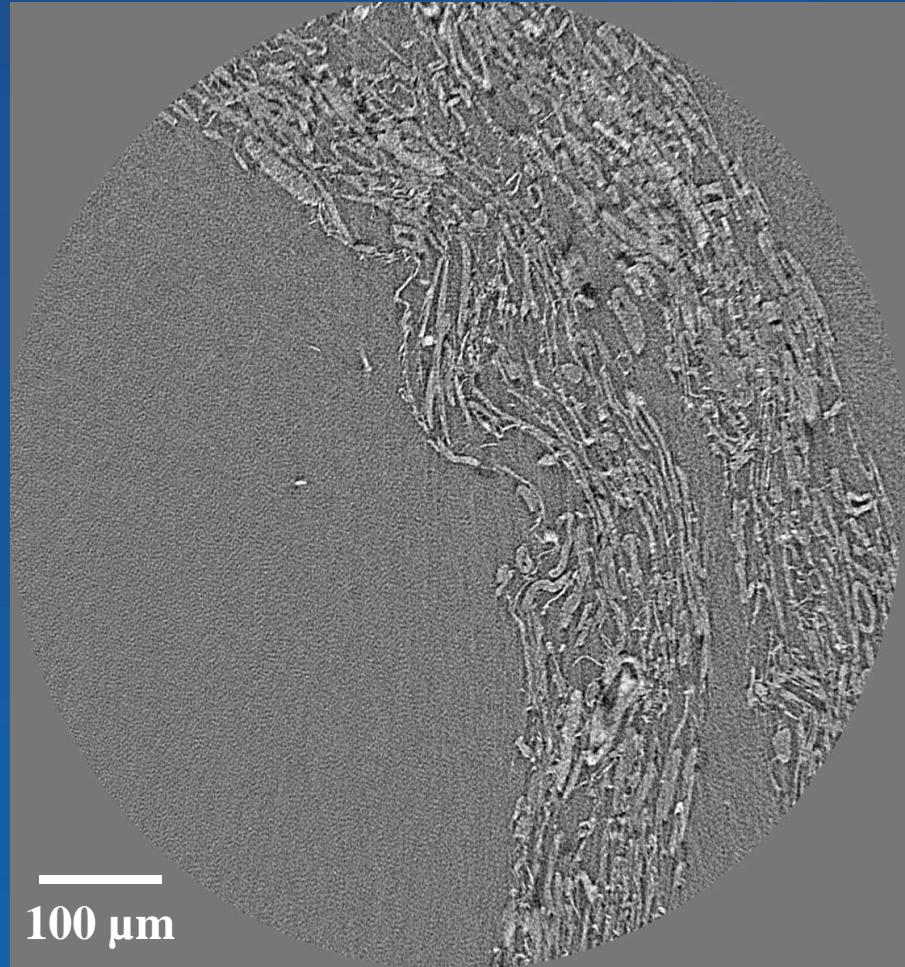


- mouthpart kinematics during feeding
- 125 FPS
- 15 μm spatial detector resolution
- Betz, Rack et al., Synch Rad News '08 & J. Exp. Biol. '14
- Westneat, Betz et al., Science '03
- TopoTomo @ ANKA

commercial Aluminum alloy A357 (Al, Si, Mg)
(18 keV, 0.8 μm pixel size / <2 μm resolution)



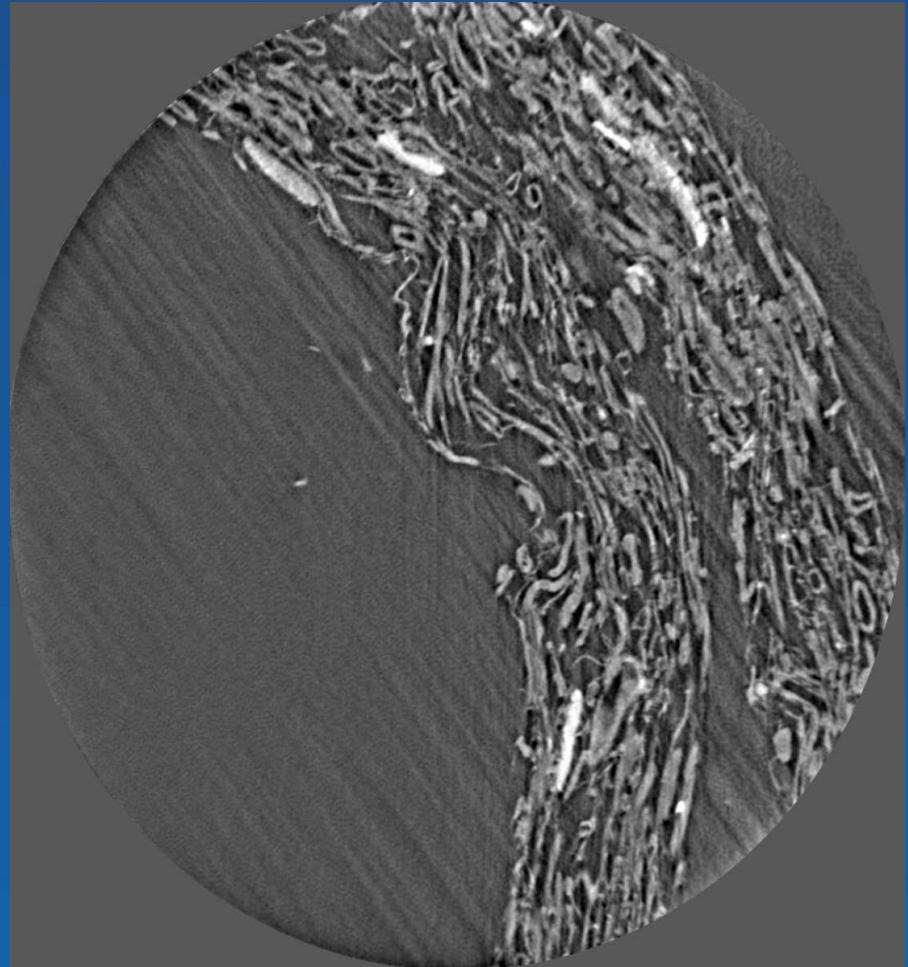
Simon Zabler, PhD thesis



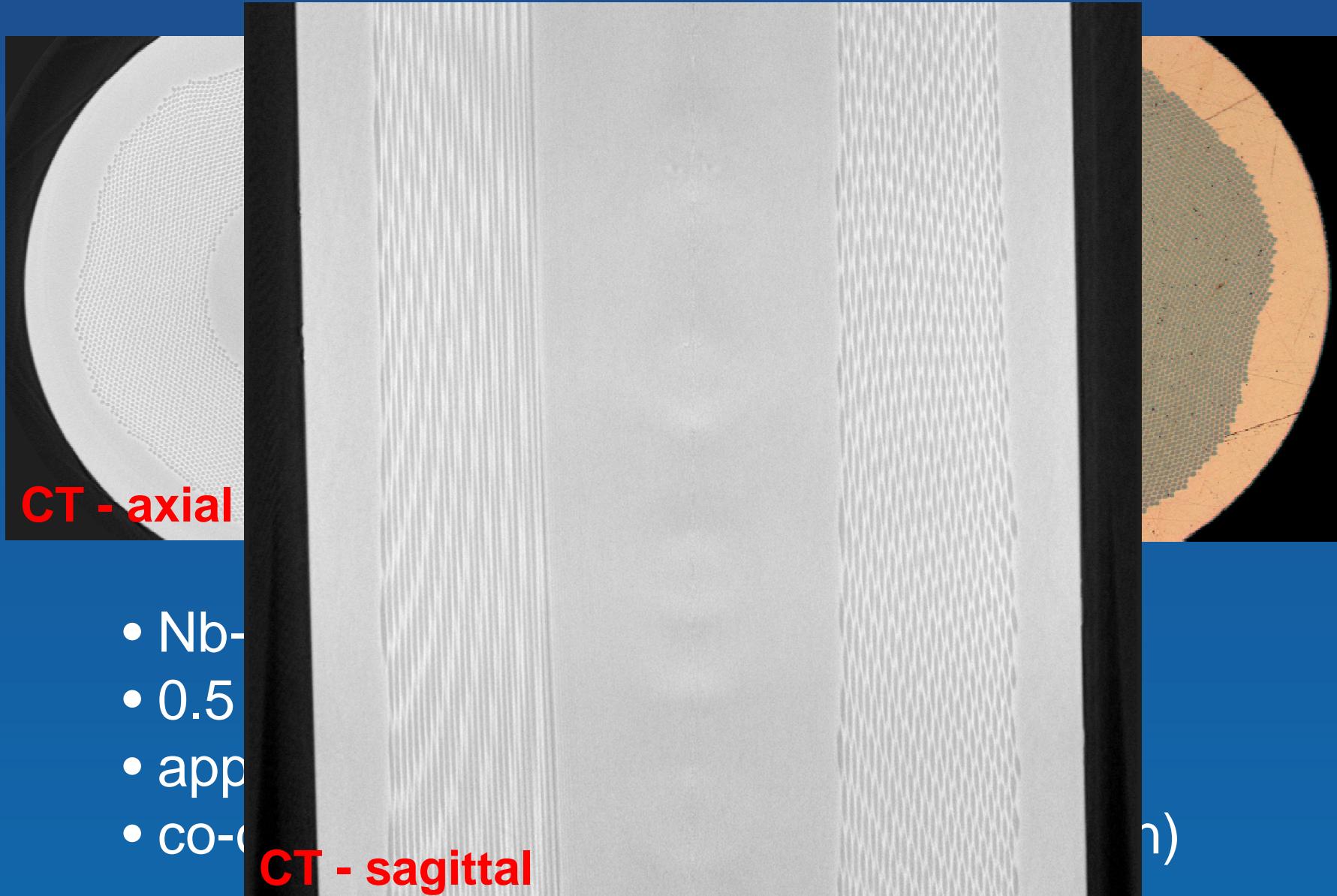
standard reconstruction

8 keV, 0.7 μm pixel size, Si (111) mono (ESRF-ID22)

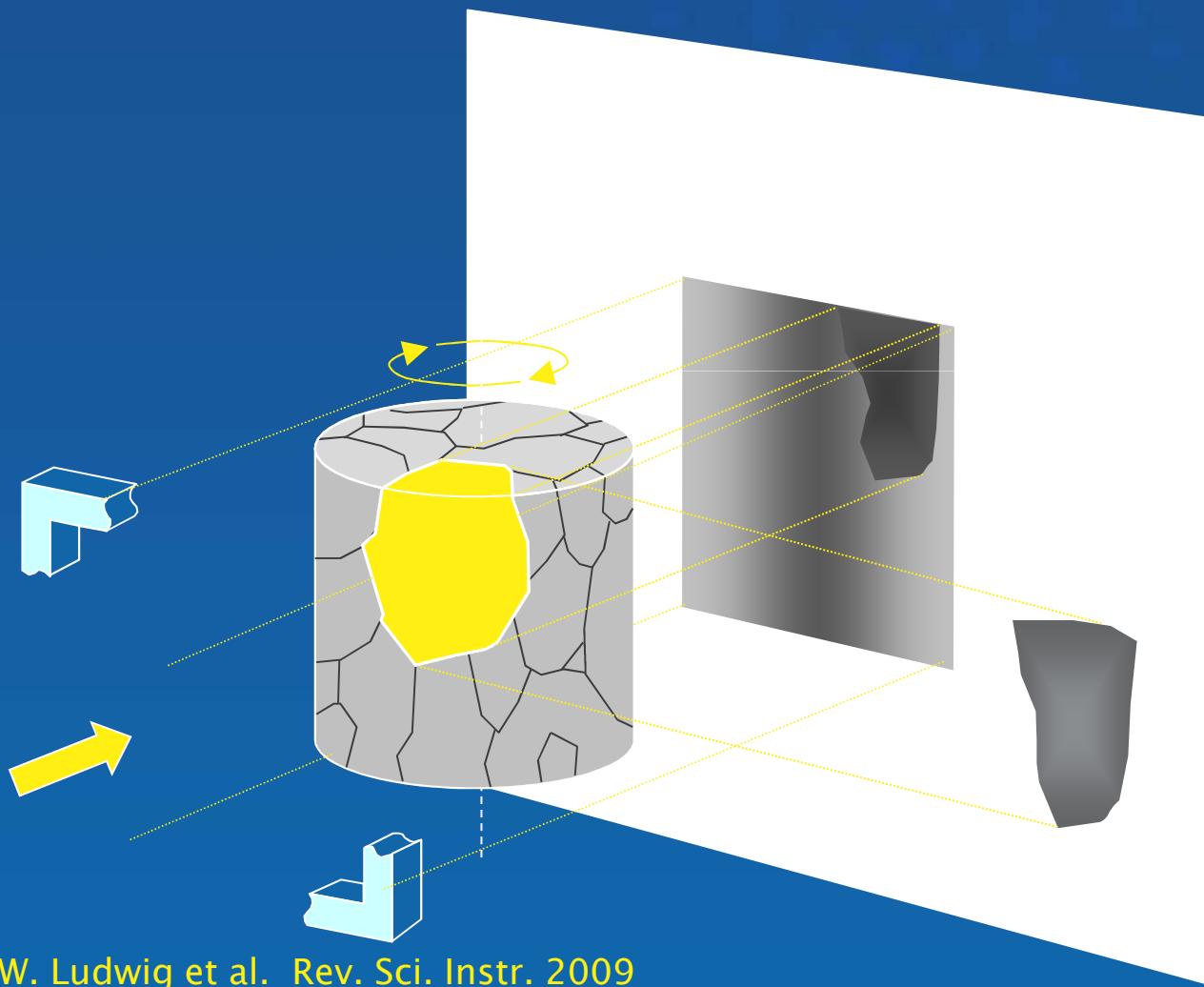
Ohser et al. Image Analysis & Stereol. 28 (2009); Weitkamp, Rack et al., J Synch Rad 18 (2011)



ANKAphase retrieval



Conventional tomography setup, large detector with high dynamic range, use slits to confine the beam to sample, monochromatic beam ($\Delta\lambda/\lambda \sim 10^{-4}$), continuous rotation, large number of images (7200 /360°).

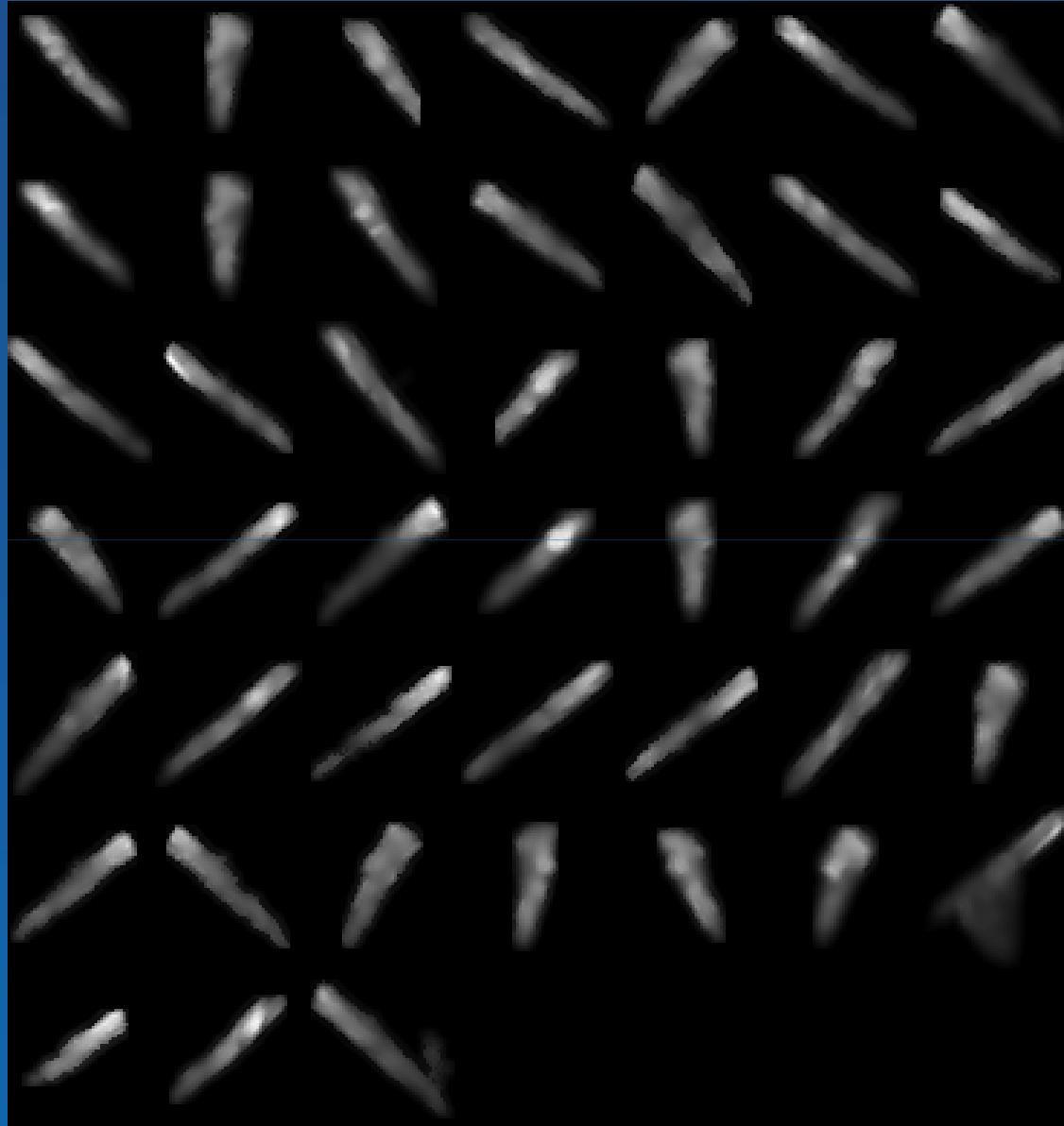


W. Ludwig et al. Rev. Sci. Instr. 2009

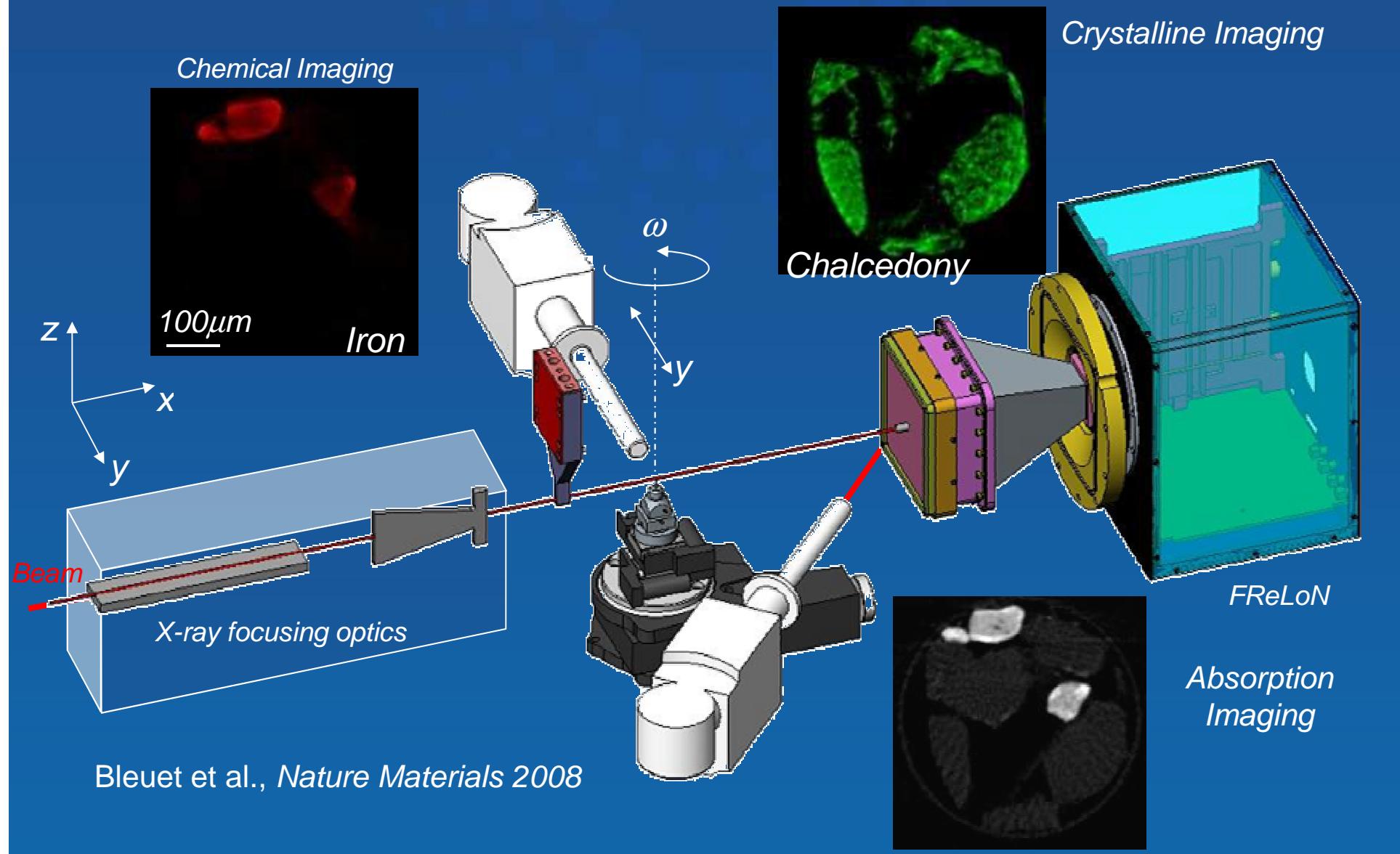
- During rotation, grains pass through Diffracting alignments
- grains with small spreads in orientation:
 - “extinction” spot visible in direct beam
 - both spots can be approximated as grain projections

Chironex fleckeri

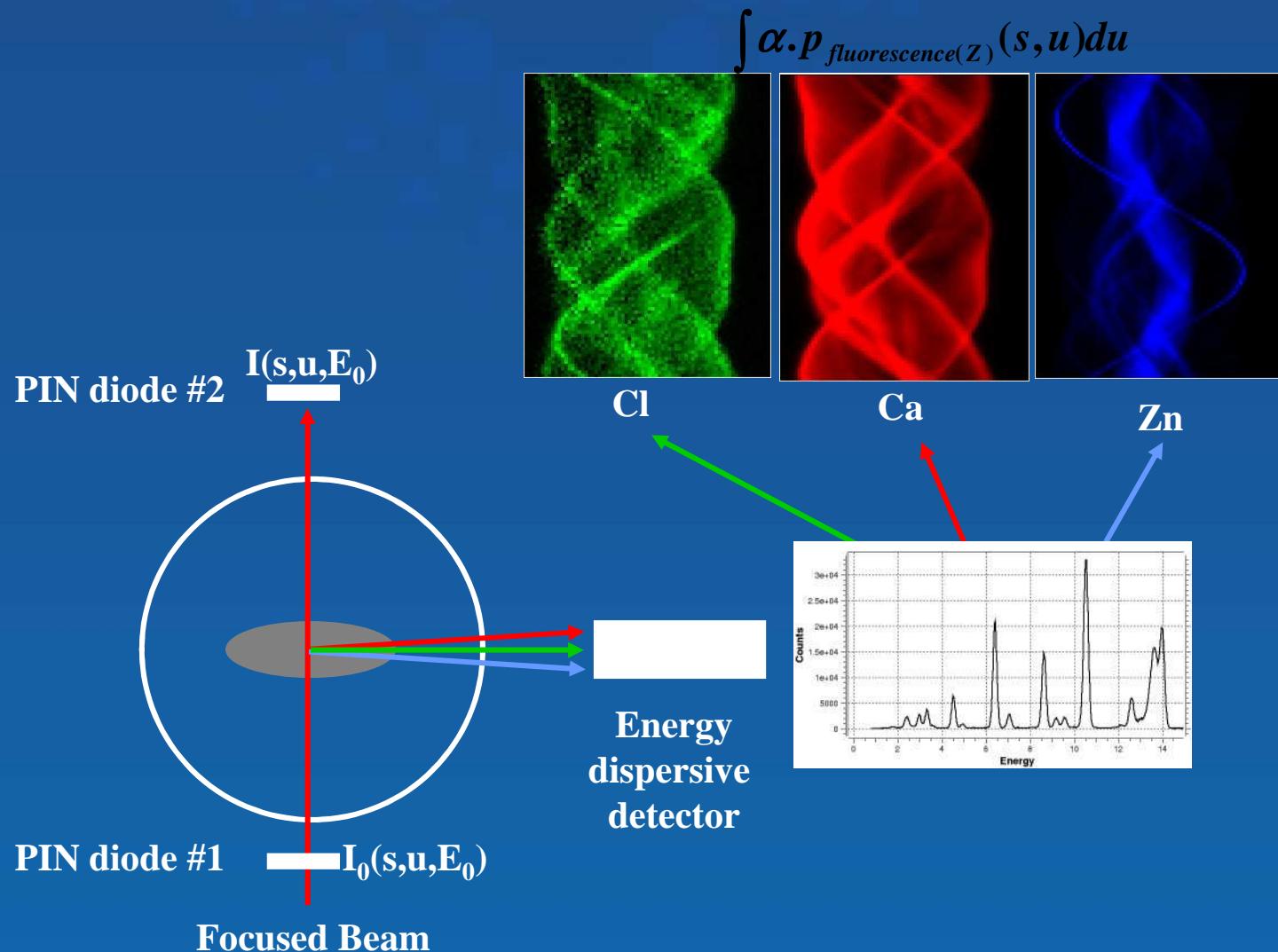
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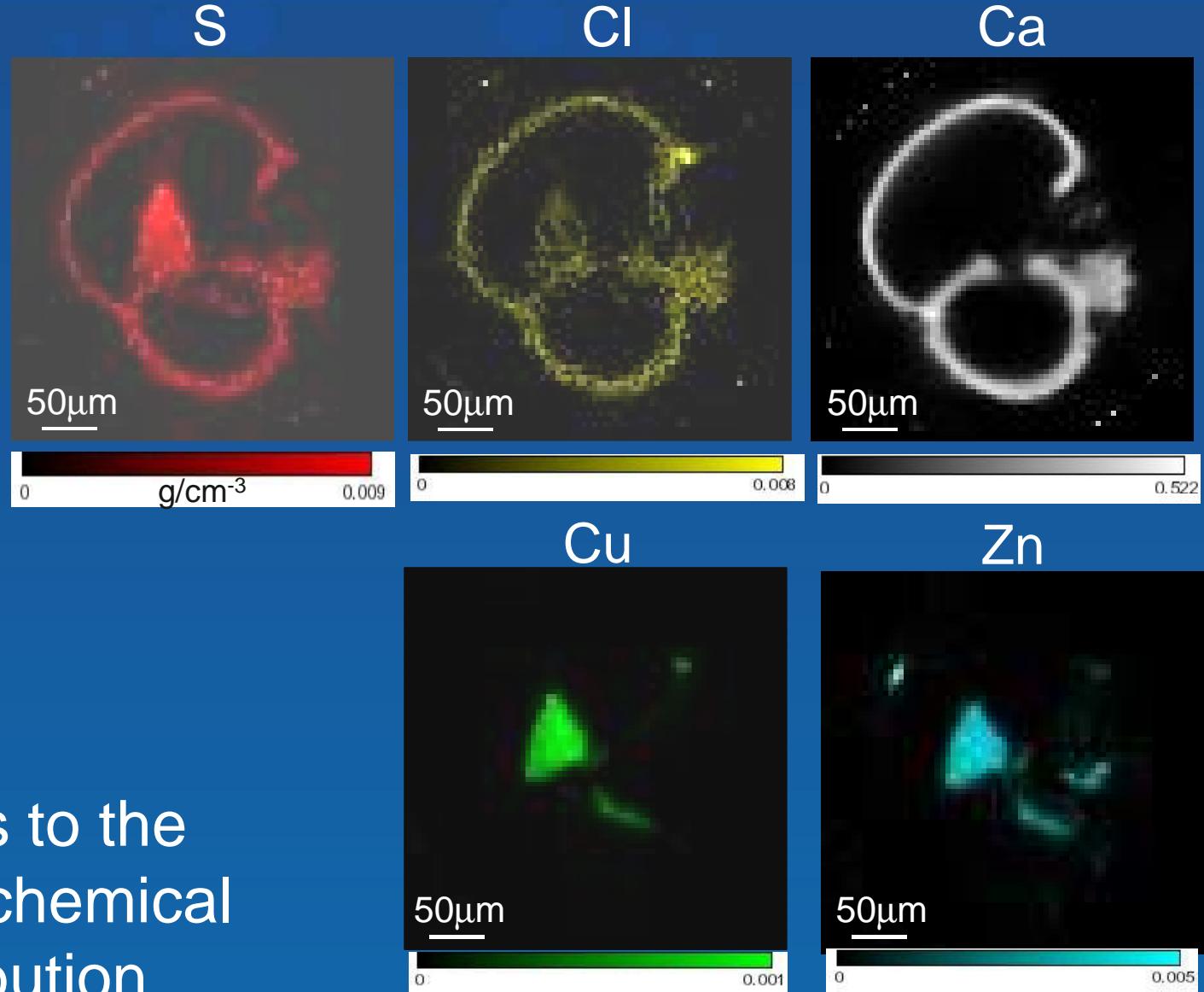
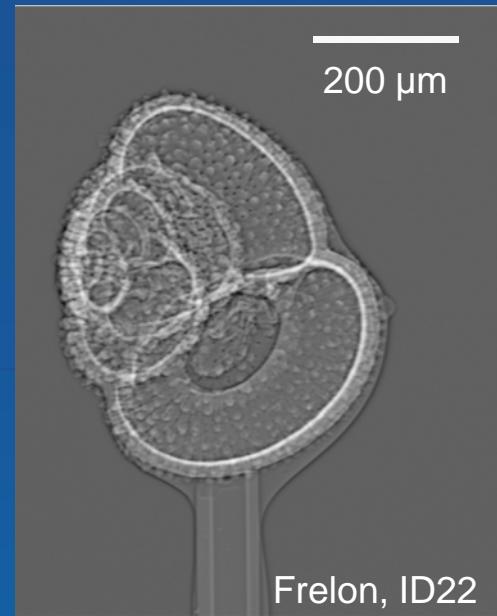
Sötje et al., Marine
Biology (2009)



Bleuet et al., *Nature Materials* 2008

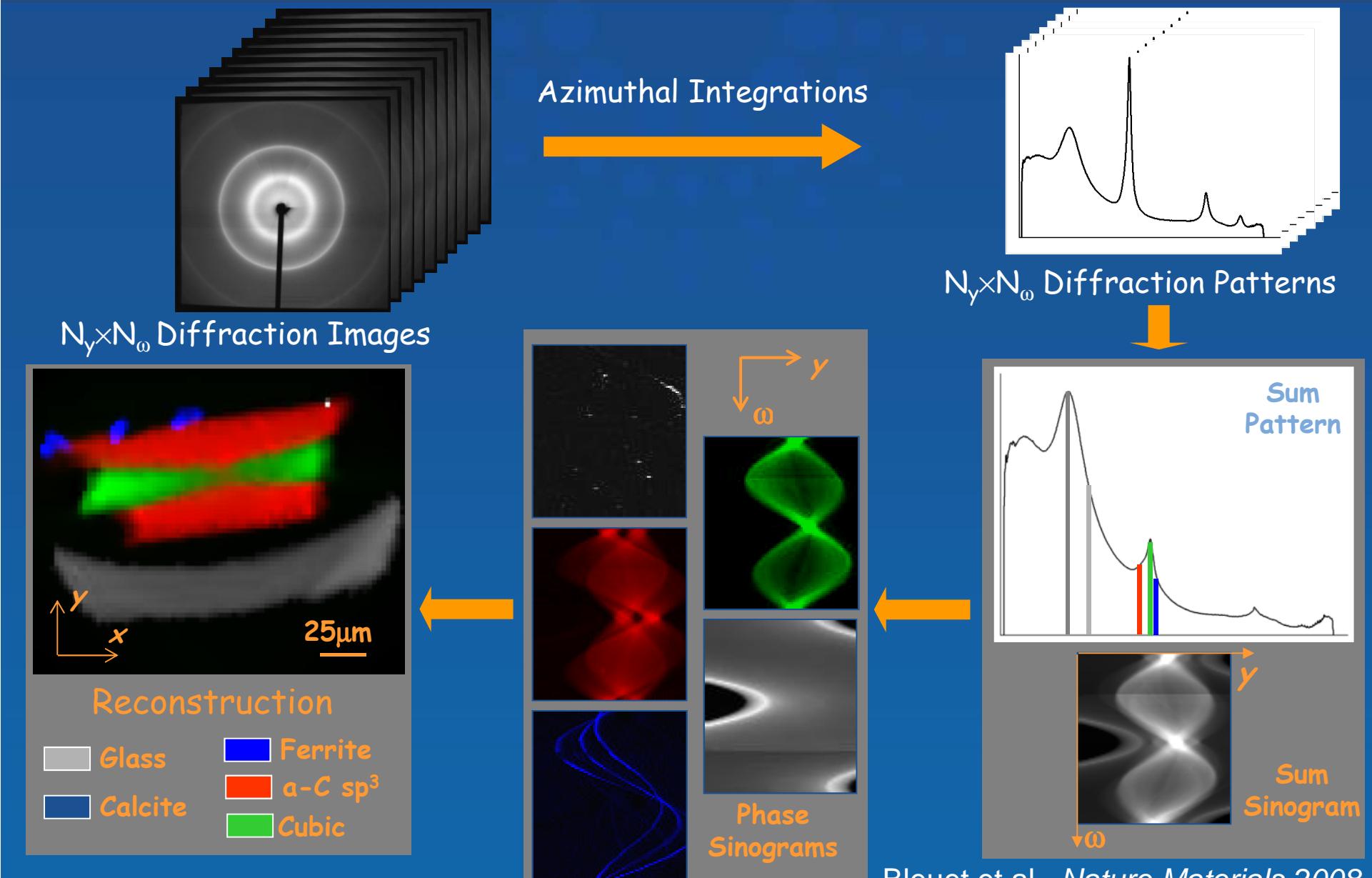


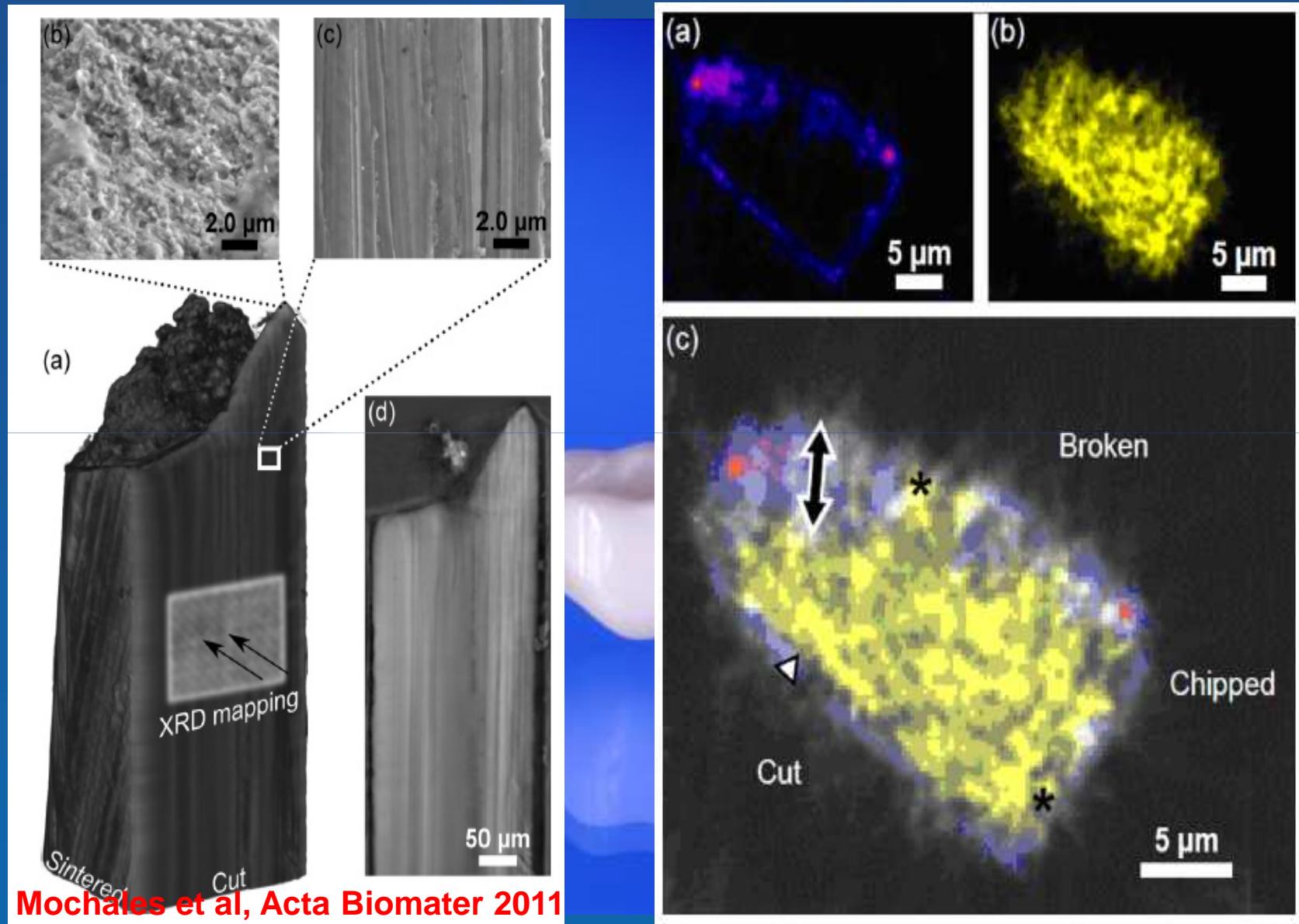
Bleuet et al., SPIE, 2006



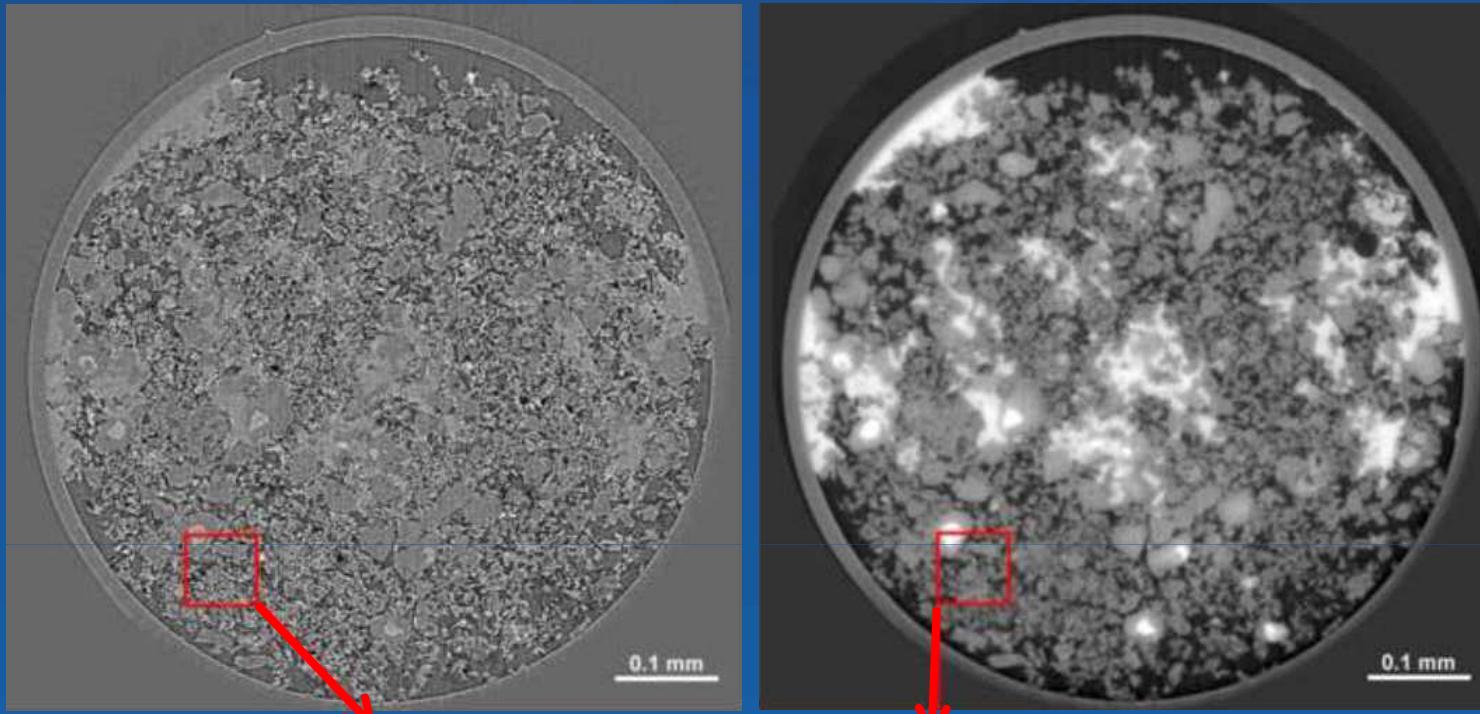
Access to the
internal chemical
distribution

Bleuet et al., SPIE, 2006

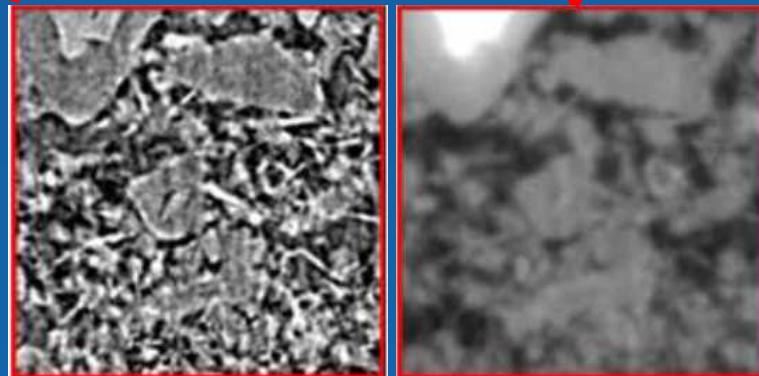




Mochales et al, Acta Biomater 2011



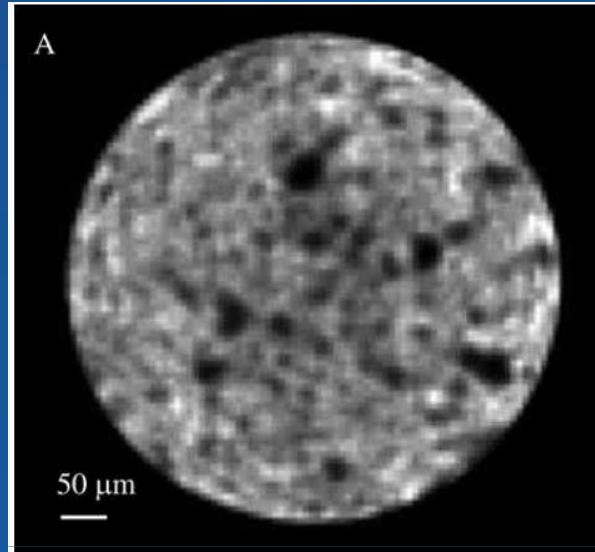
- microCT, ID22
- 14 keV, 0.7 μ m
- inline X-ray phase contrast
- Si(111) mono



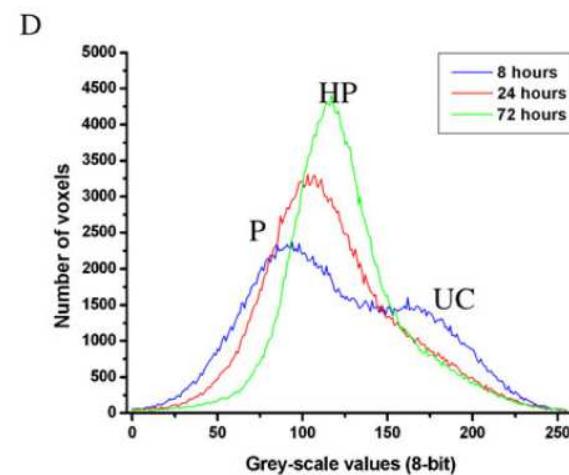
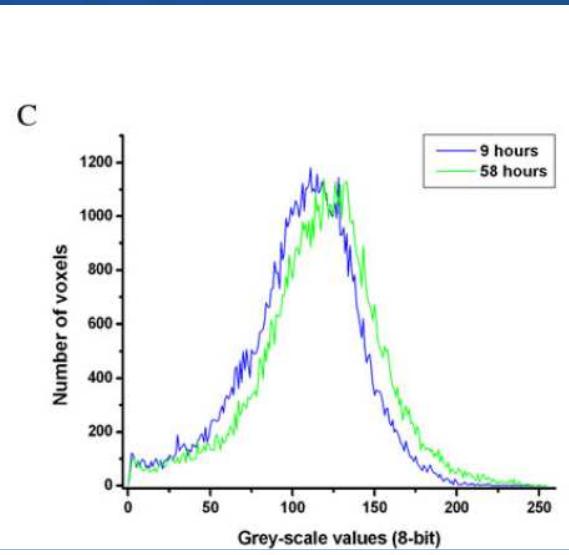
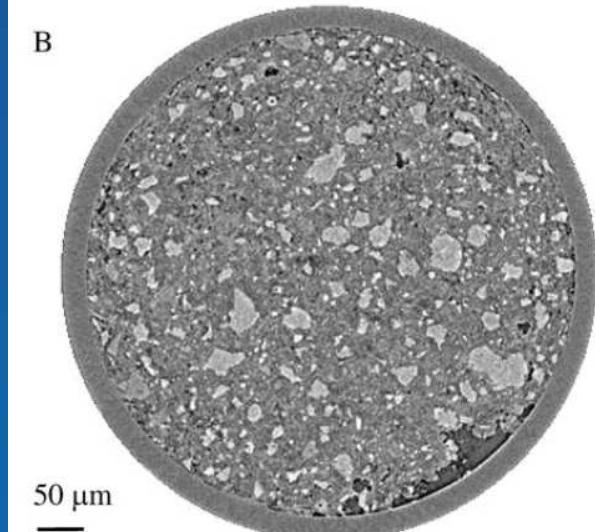
- single-distance phase retrieval
- ANKAphase

Artioli et al., Int J Mater Res, 103 (2), 145 (2012)

- XRD- μ CT, ID22
- 2 μ m x 4 μ m spot, 18 keV
- 001 peak intensity
- 58 h hydration



- microCT, ID22
- 14 keV, 0.7 μ m
- inline X-ray phase contrast
- Si(111) mono

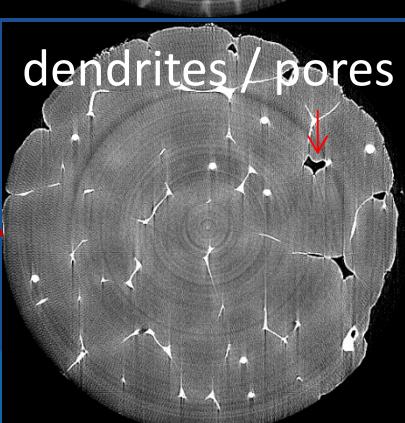
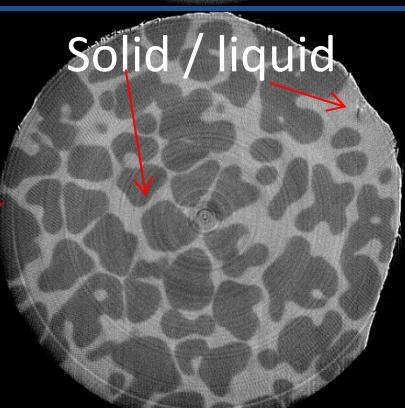
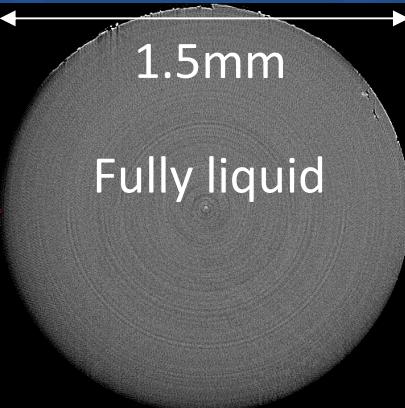
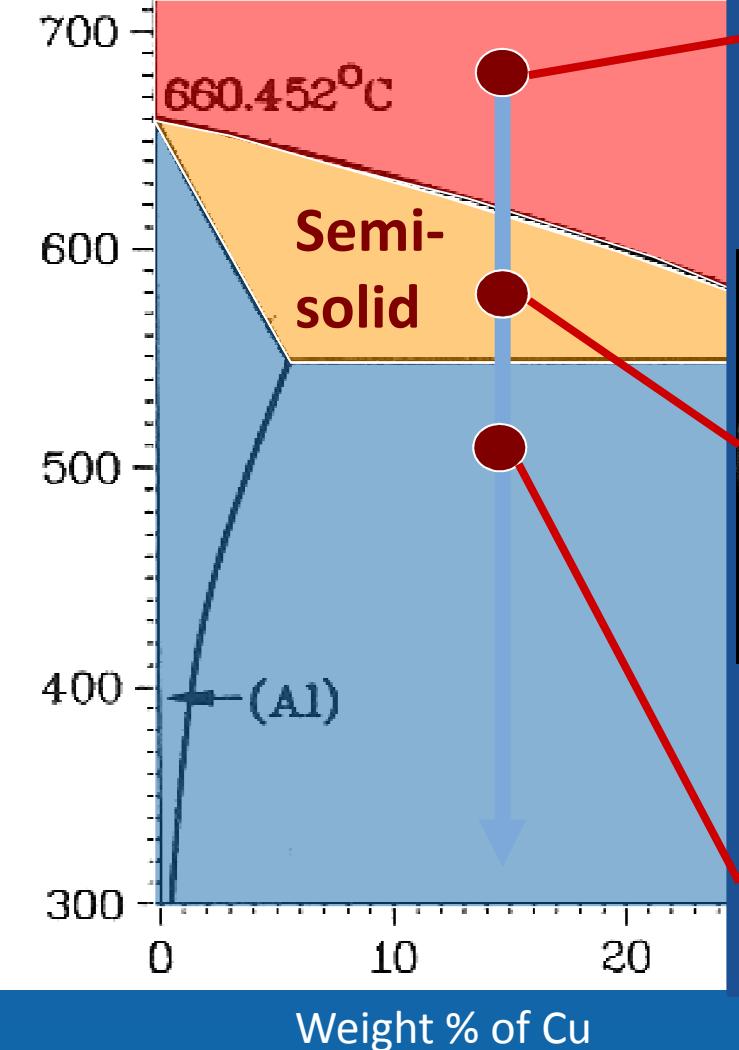


Artioli et al., Analyt. & Bioanalyt. Chem. (2010)

Time-resolved Microtomography

Solidification

Temperature °C



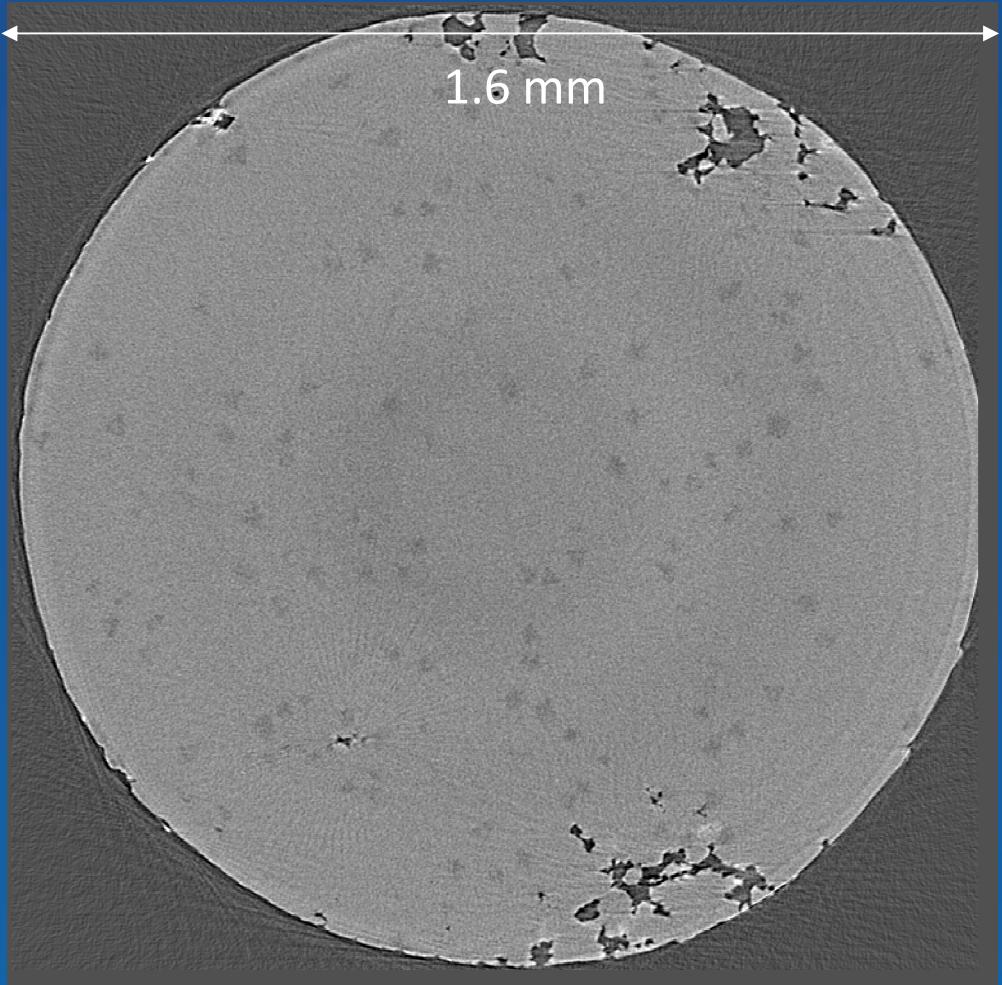
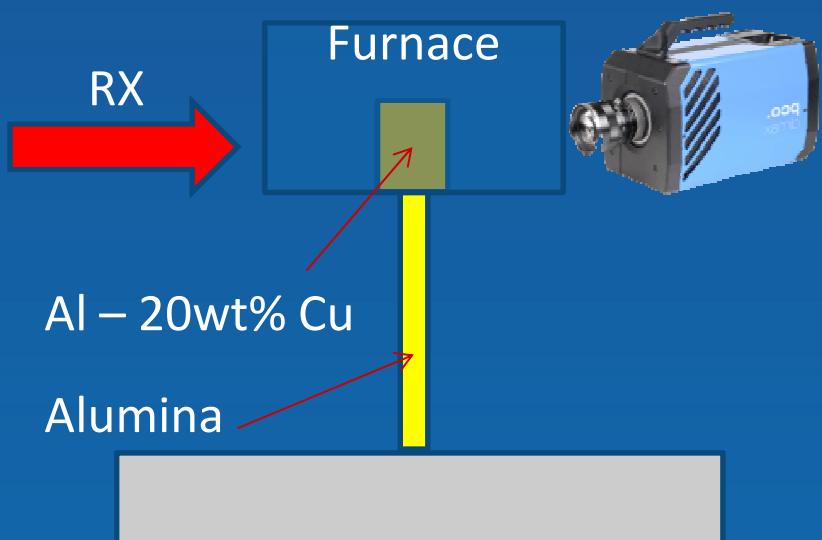
Solid fraction evolution
Solid coherency
Dendrite growth
Pore formation

Relevant scale
 $1\mu\text{m} - \text{few }\mu\text{m}$

Typical cooling rates
 $0.1 - 10\text{ }^{\circ}\text{C/s}$

Courtesy Luc Salvo, CNRS

Solidification Al 20% Cu
ID15A
PCO DIMAX, Size 780 x 600
600 projections, Optics 2.2 μ m
White beam (gap open)
Scan time 0.15 seconds
70 scans,



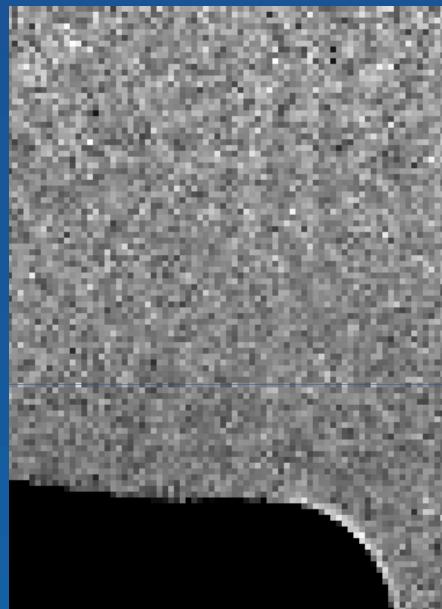
Solidification rate $\sim 5^{\circ}\text{C/s}$
Solidification time 10s !!

Courtesy Luc Salvo, CNRS

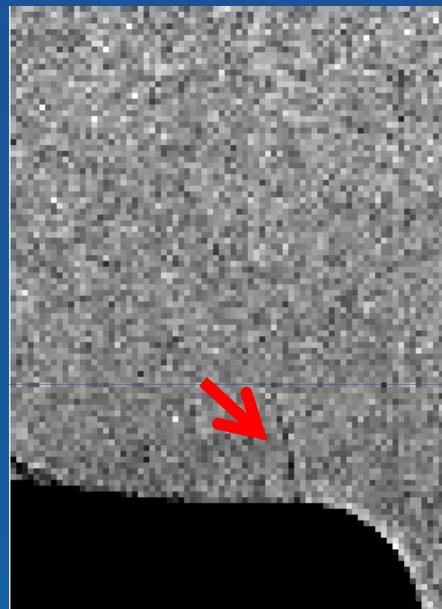
Ultimate Resolution in Time

1 mm

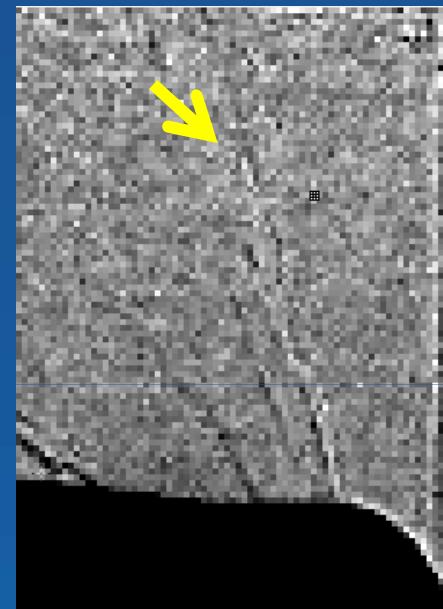
35 μm pixel size, 500 LuAG:Ce



0 μs



28 μs



56 μs

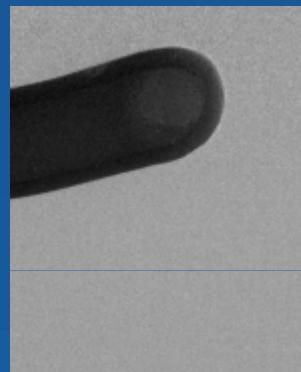
2.4 μs exposure = 1 bunch / 35504 FPS (28 μs)

ID19: 1x 32u @ 11.5 mm (ca. 10 mA = single-bunch mode)

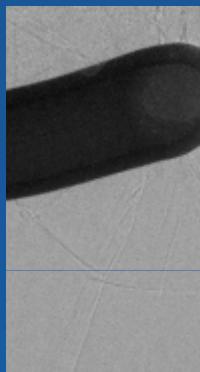
Rack, Scheel, Reichert et al., J Synch Rad 21, 815-818 (2014)

1 mm
—

500 LuAG:Ce



0 μ s



28 μ s

2.26 μ s

ID19: 2

Rack, Sche

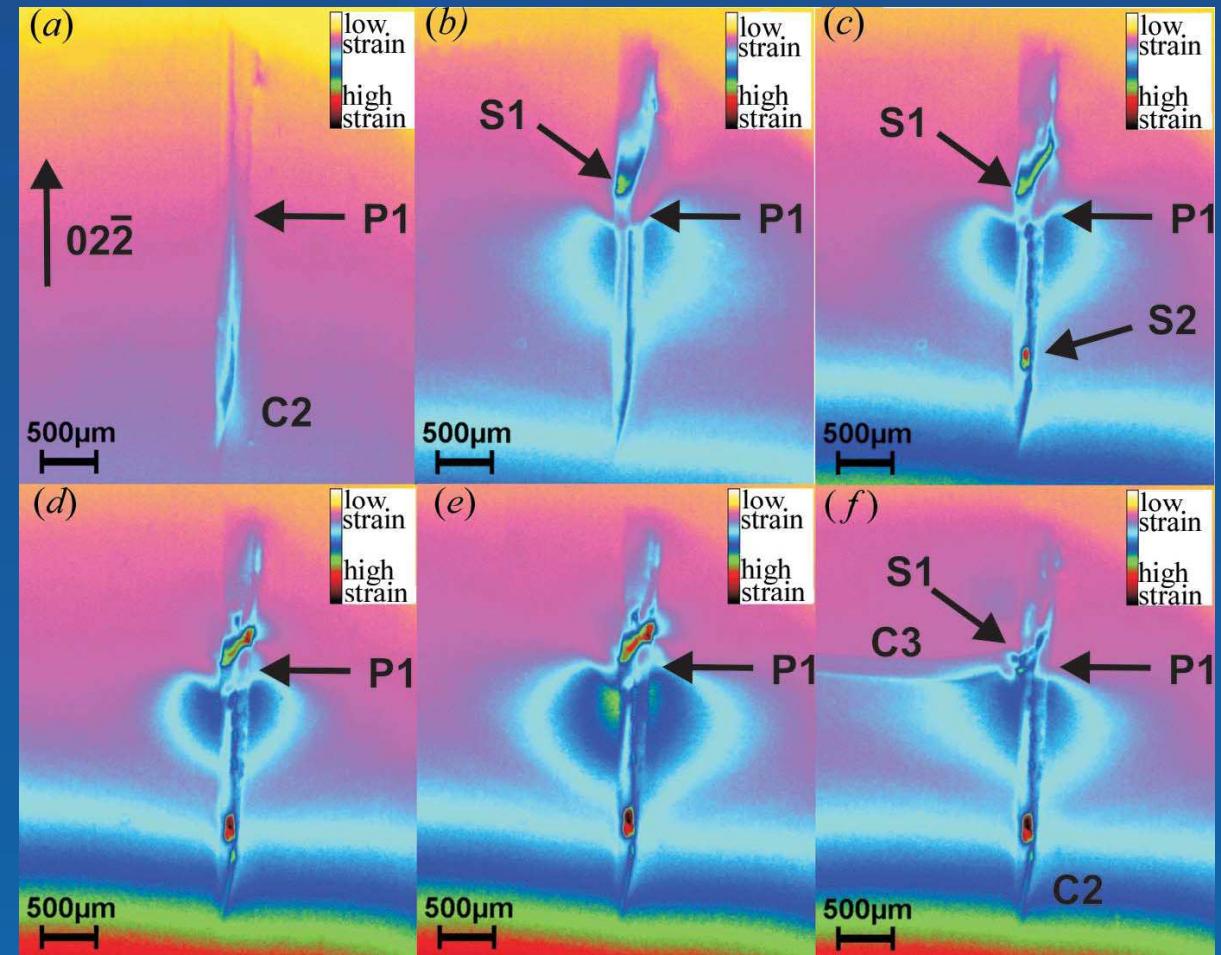
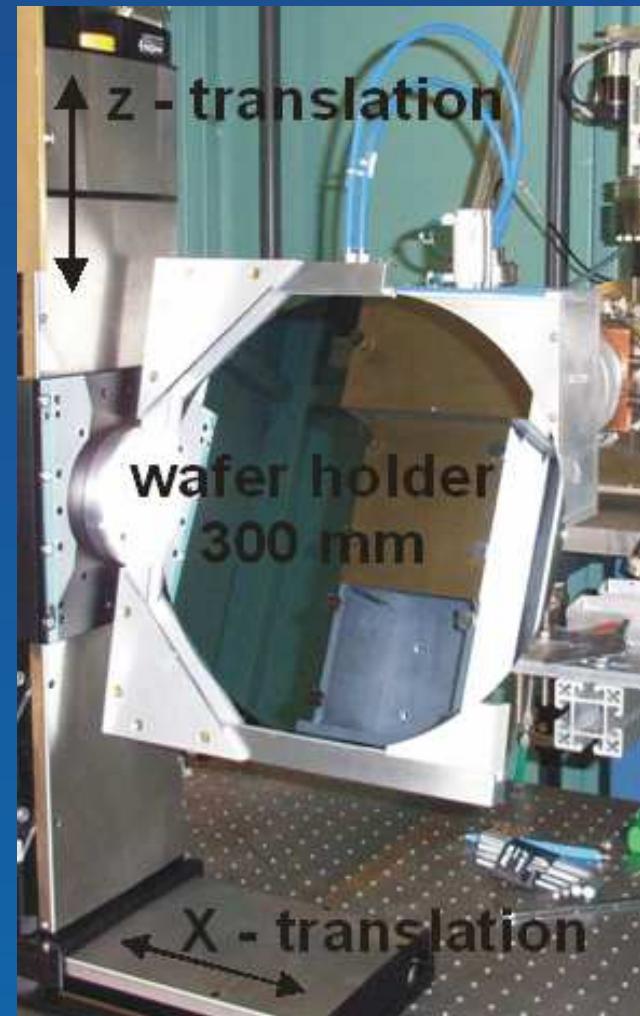


140 μ s

PS (28 μ s)

mm (6 mA)

14)



Danilewsky et al., “Crack propagation and fracture in Si wafers under thermal stress”, J Appl Cryst 46 (2013)



Acknowledgments: Simon Zabler, Stefan Kirste, Jürgen Goebbels, Gerd Weidemann, Heinrich Riesemeier, ID19 @ ESRF, Alexander Bütow, Katja Schladitz, Joachim Ohser, Holger Kropf, Biljana Matijasevic-Lux, Olaf Dalügge, Christine Knabe, Michael Stiller, Christian Koch, Bernd Müller, Timm Weitkamp, Andreas Danilewsky, Thierry Martin, Angelica Cecilia, Tatjana Rack, Lukas Helfen, Peter Cloetens, Sondes Bauer-Trabelsi