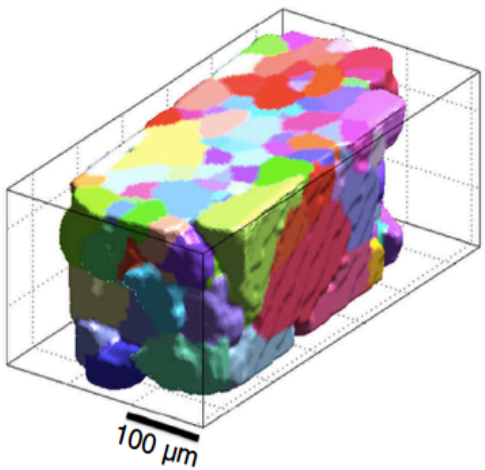
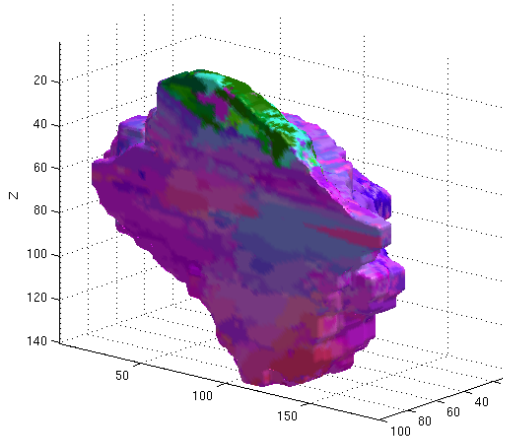
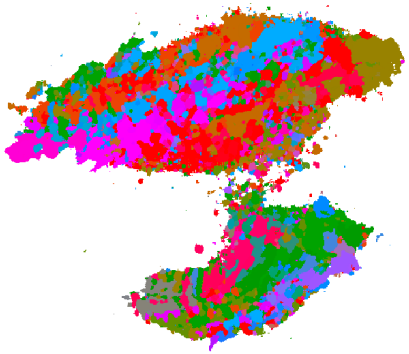
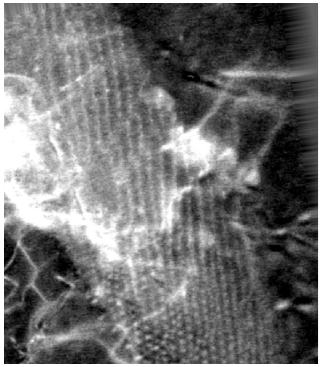
# Hard X-ray microscopy for multiscale materials science

## Zoom into bulk materials:





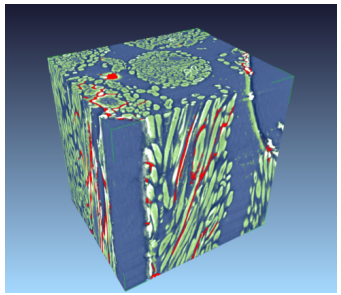
100 µm | 10 µm | 1 µm | 100 nm

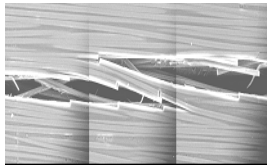
Compare 3D movies directly to 3D modelling on ”all scales”

”materials genome …”, Energy Frontier Research Centers, ICME

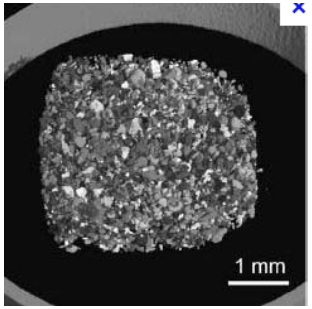
# Multiscale studies

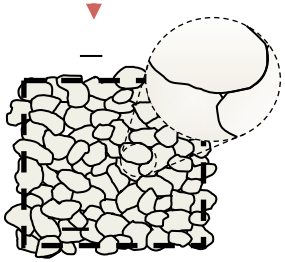
## Composites for wind



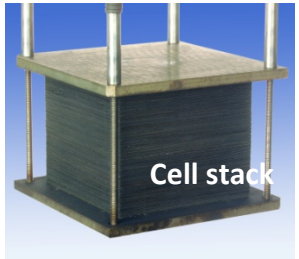


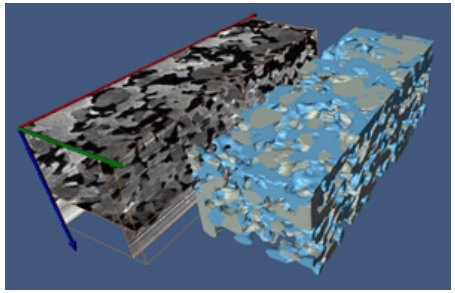
## Batteries

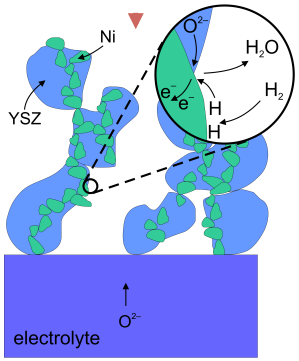


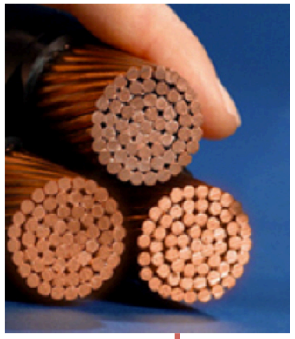
## Fuel cells

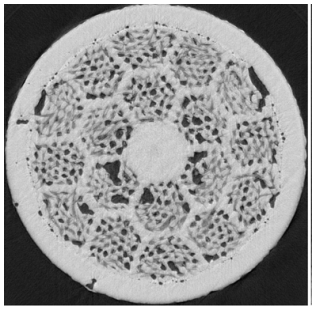


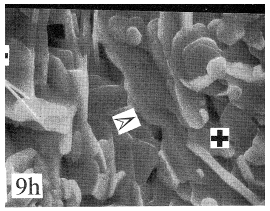






## Superconducting cables

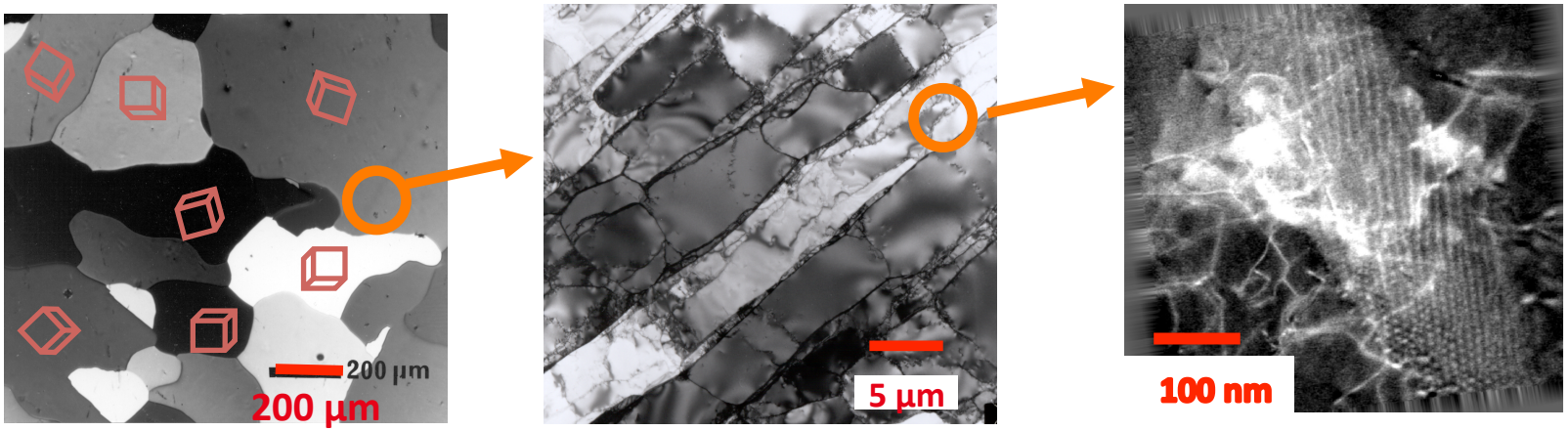






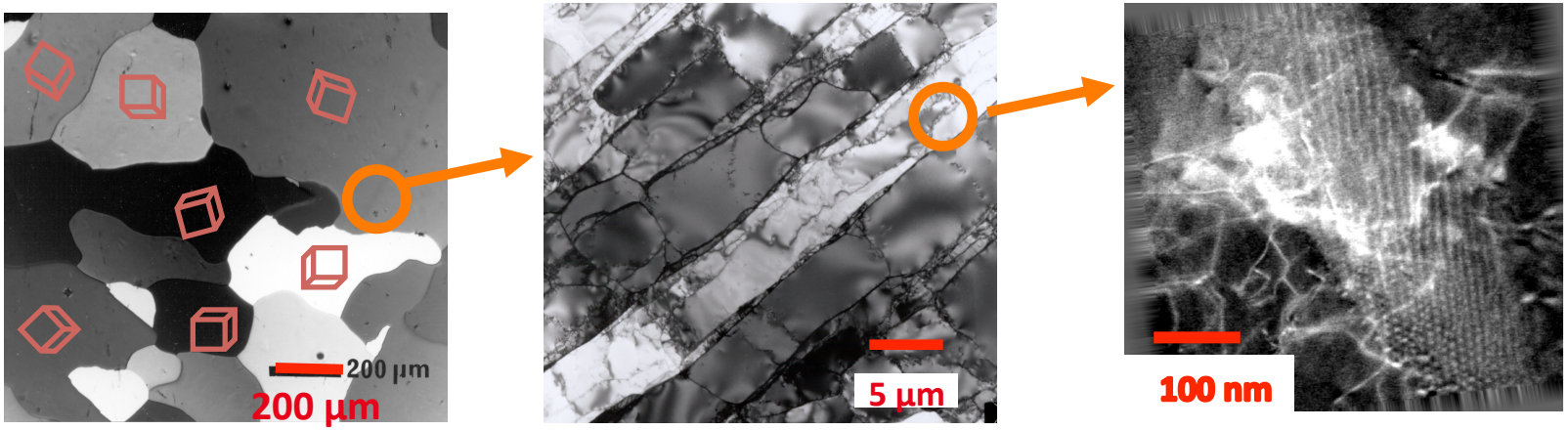
# Hard crystalline materials

Example: metals




Characterize multi-scale structural evolution in 3D

# Multi-scale multi-modal imaging




Tomography
- Phase contrast
- 3DXRD

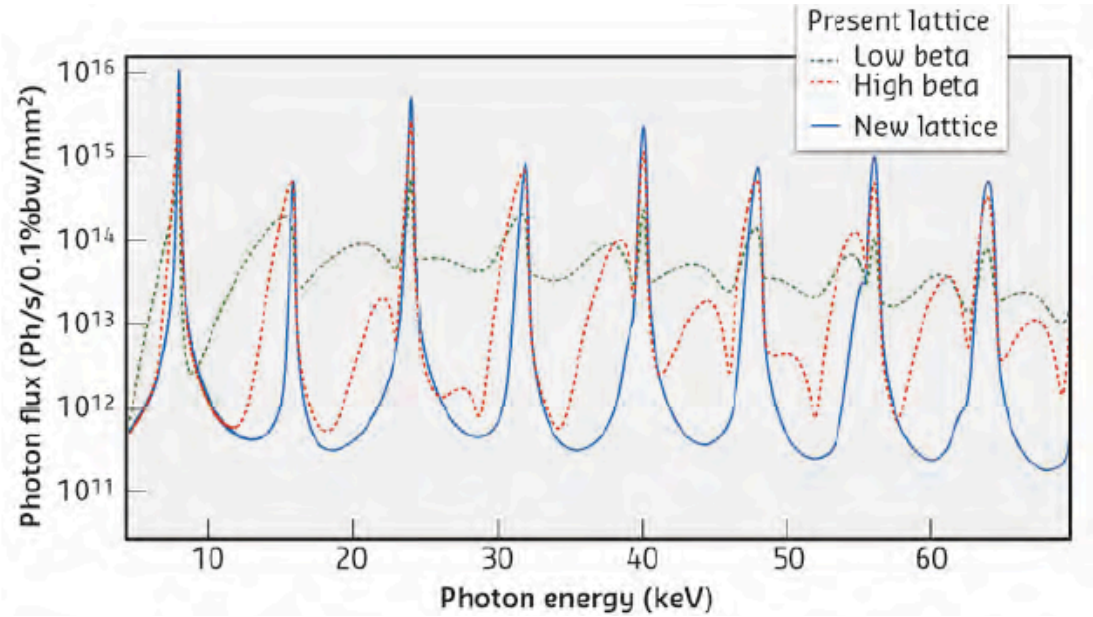
Microscopy
- Bright field
- Dark field

Coherent diffraction

# A tomography beamline ”without optics”

Divergence → 1 mm illumination at sample

Spectrum*




→ Multi energy pink beam operation

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- No optics hutch
- Full wavefront preservation
- Phase + energy contrast

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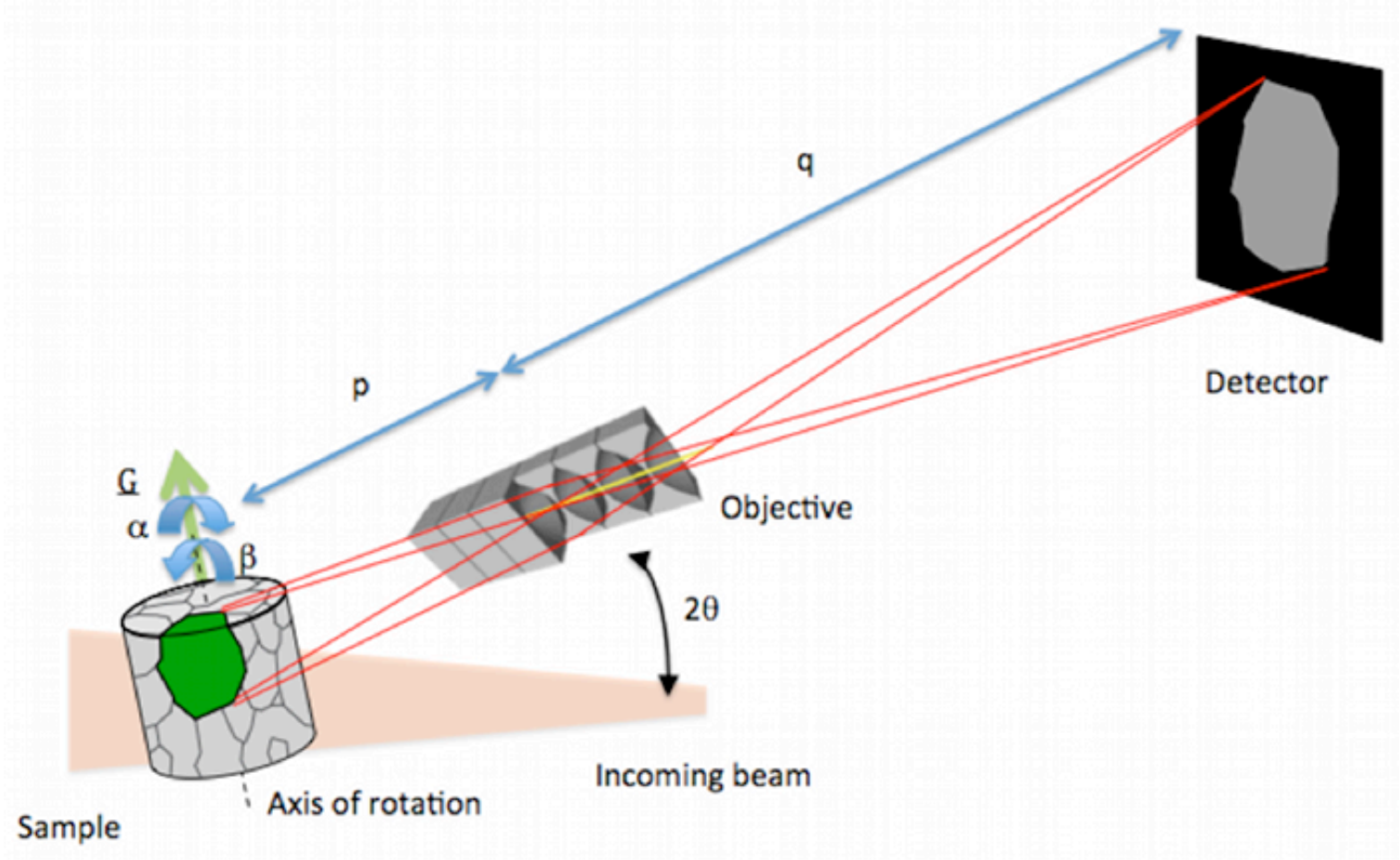
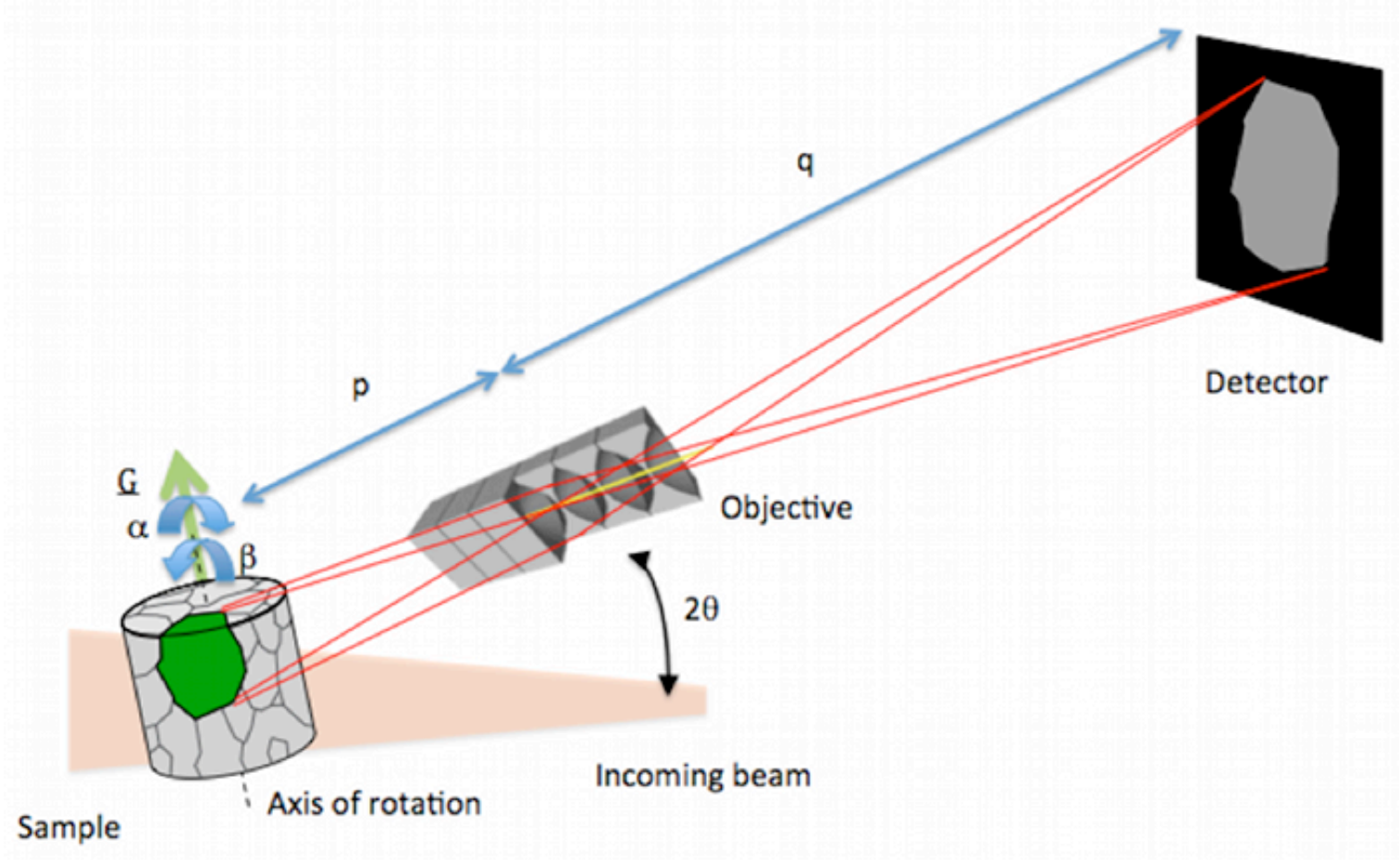
*From ESRF Orange book

# Basic principle of dark field microscopy




Zoom in direct space: today 100 nm (theory: 10 nm)

# Basic principle of dark field microscopy




Zoom in reciprocal space: angular resolution down to 0.003 deg

can avoid spot overlap despite illuminating $10^{10}$ cells

# Hard x-ray microscopy at ID06, ESRF

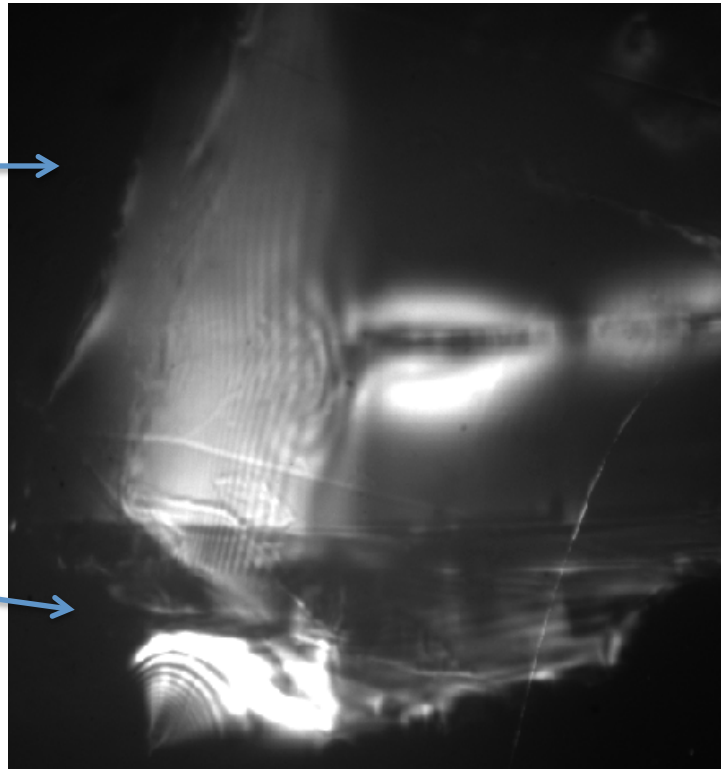
Resolution: 50-200 nm, energy: 15-35 keV

Team ESRF: **C. Detlefs**, A. King, A. Snigirev, I. Snigireva. INSA-Lyon: **W. Ludwig**, NTNU: R.H. Mathiessen, Utrecht: A.V. Petukhov, DTU: S. Ahl, F. Stöhr, J.X. Trujillo, N. F. Gade-Nielsen, A.C. Jakobsen, **H. Simons**, J. Bowen, W. Pantleon, H. F. Poulsen, S. Schmidt, Y. Zhang

Demonstration on embedded 100 μm thick Si grain:

Pendullösing fringes

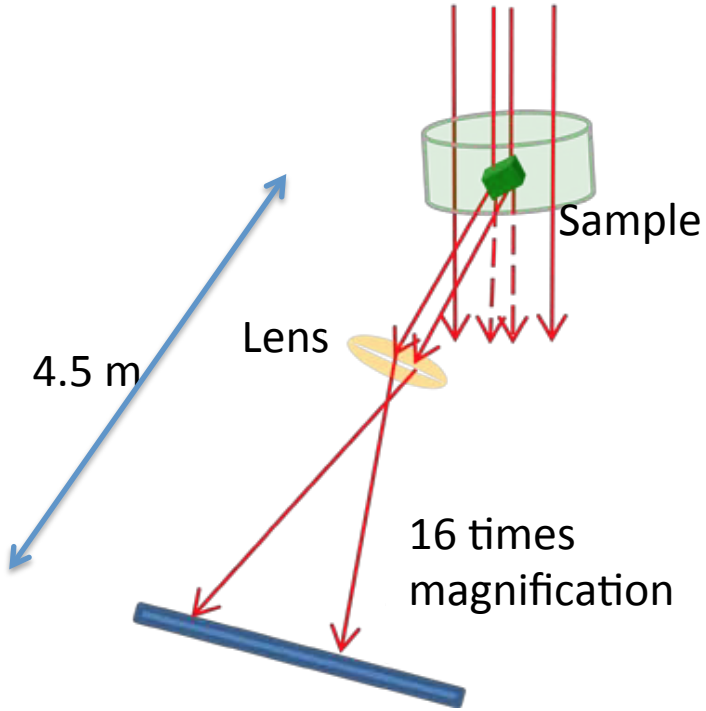
Stress concentration



# Demonstration on deformed Al

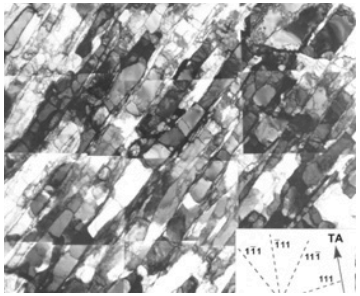
**Experimental**

ID06, 17 keV x-rays




**Sample**

5% tensile deformed Al

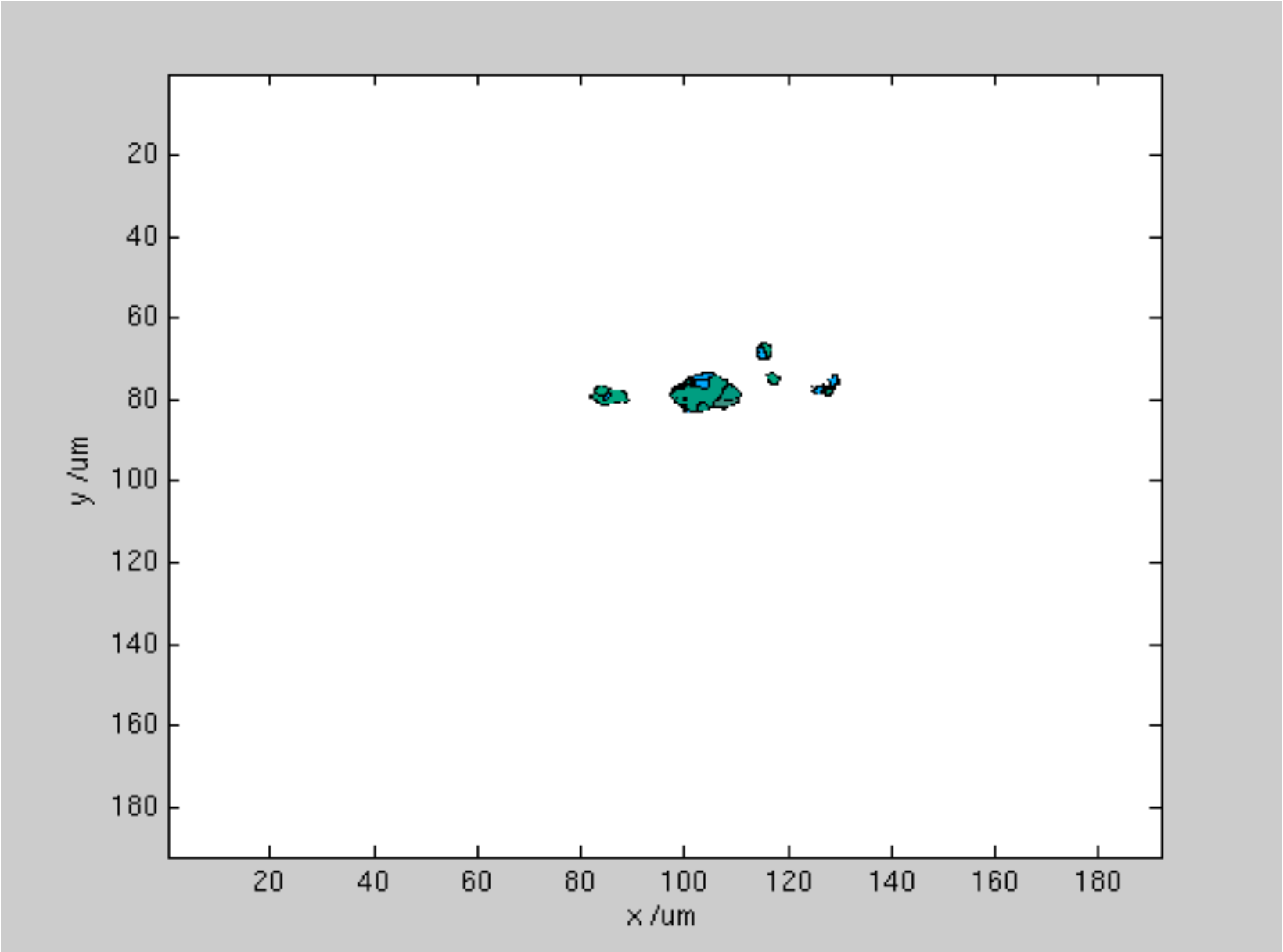


Sample: 700x300 μm

Grain size: 100 μm

Cell size: 2 μm

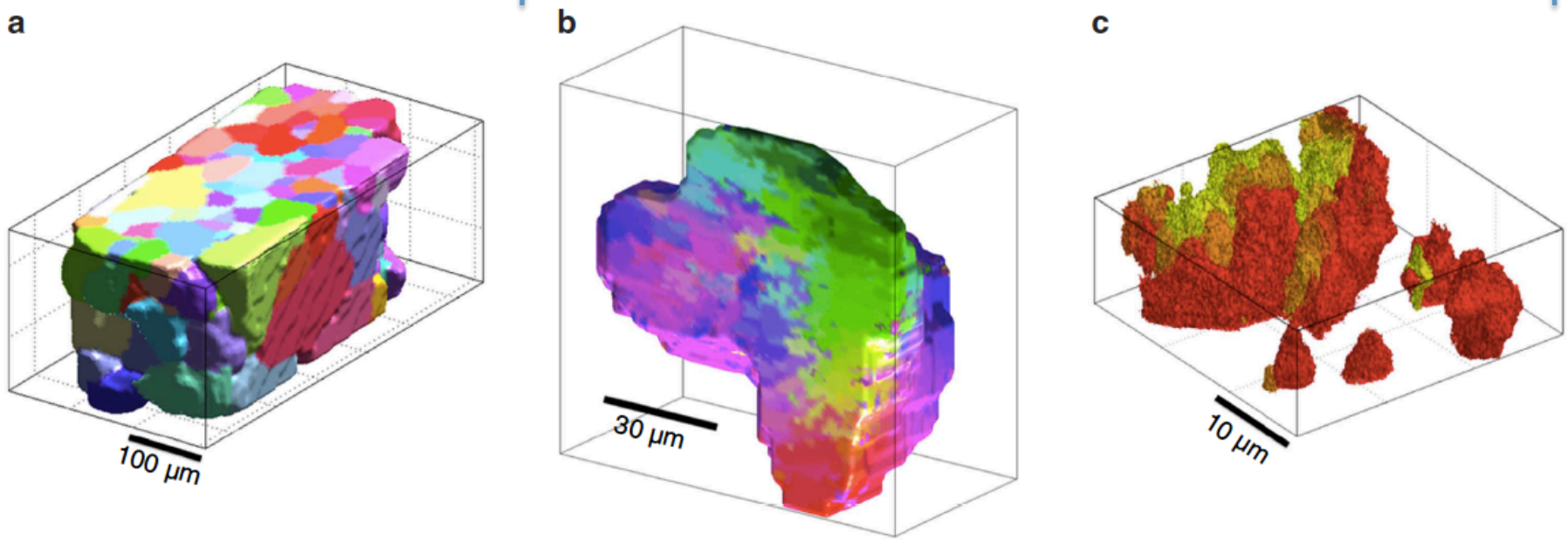
# “Grain reconstruction”



# Al study

Diffraction contrast tomography

Dark field microscopy




2 µm
0.5 deg

200 nm
0.15 deg

200 nm
0.01 deg

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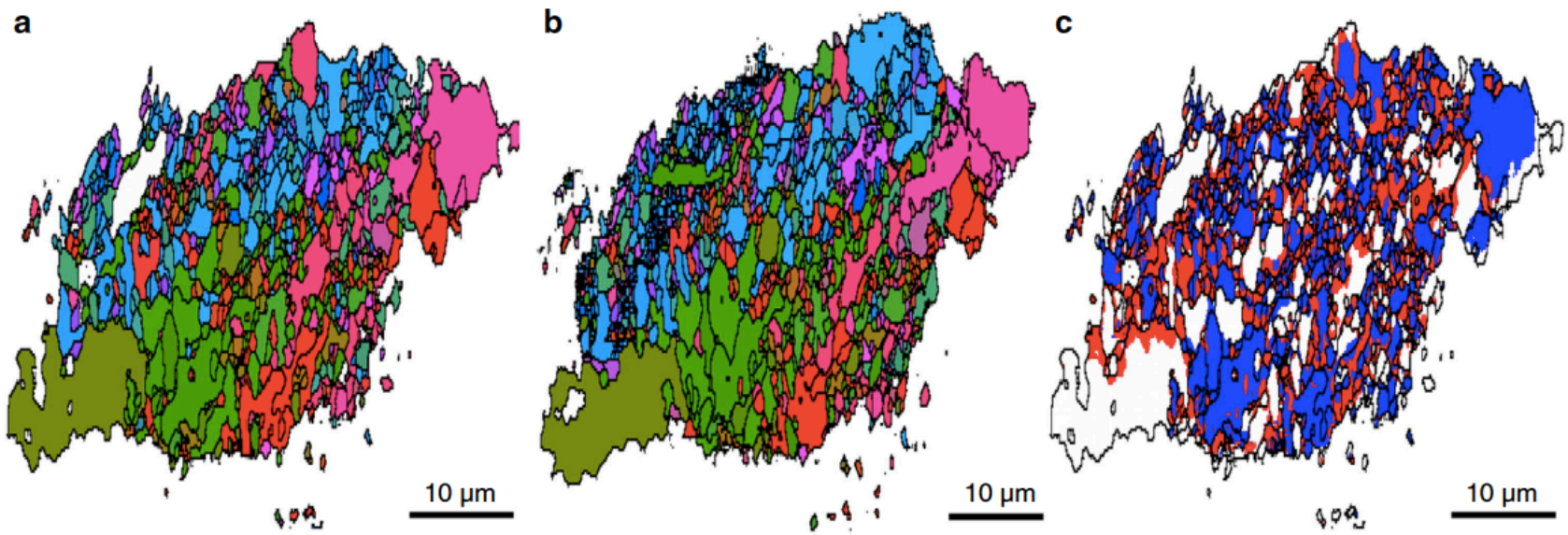
H. Simons, A. King, W. Ludwig, C. Detlefs, W. Pantleon, S. Schmidt, F. Stöhr, I. Snigireva, A. Snigirev, H.F. Poulsen. *Nature Communications* **6**, 6098

# Time evolution of one layer

T = 246 C

T = 257 C

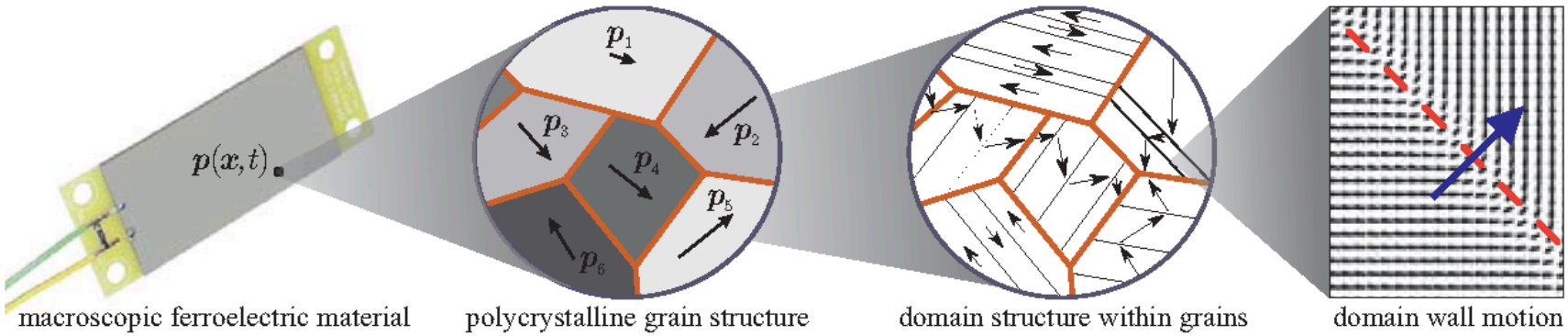
Difference




< 0.01° < 0.2° > 0.2°

Evidence for subgrain boundary motion

# Domain evolution in ferroelectric materials




Micromechanical modeling

Phase field modeling

DFT

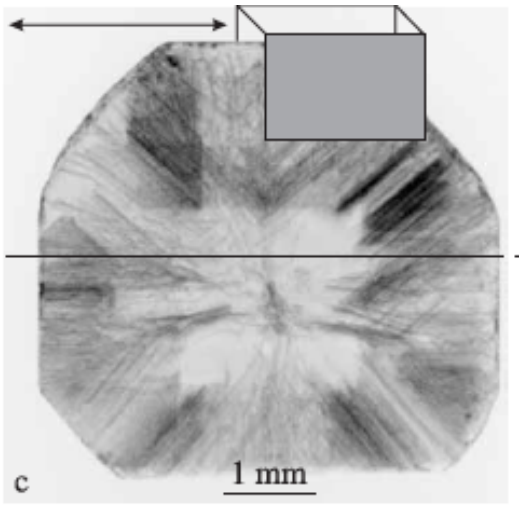
3DXRD

Dark field microscopy

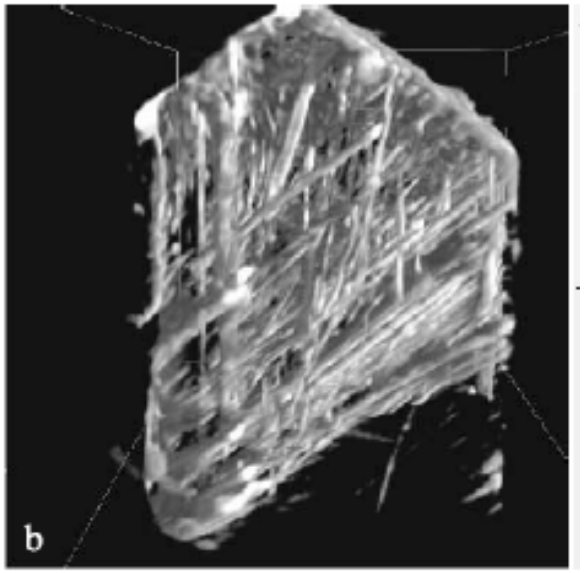
Coherent diffraction

# Dislocation structures in 3D

Topography

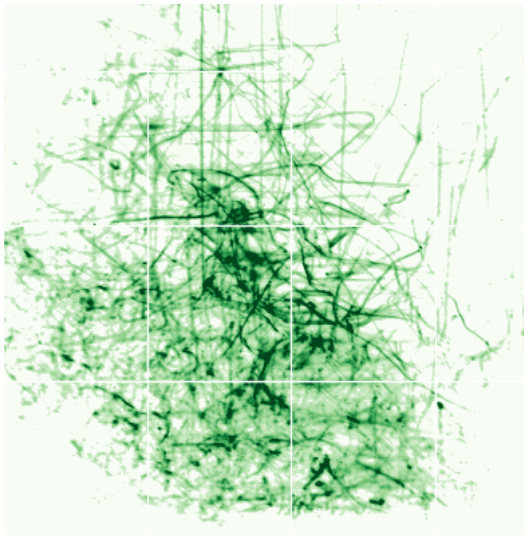



”Direct beam Topo-Tomography”*




intrinsically limited to ~10 μm

Off-peak microscopy**



Resolution: 200 nm

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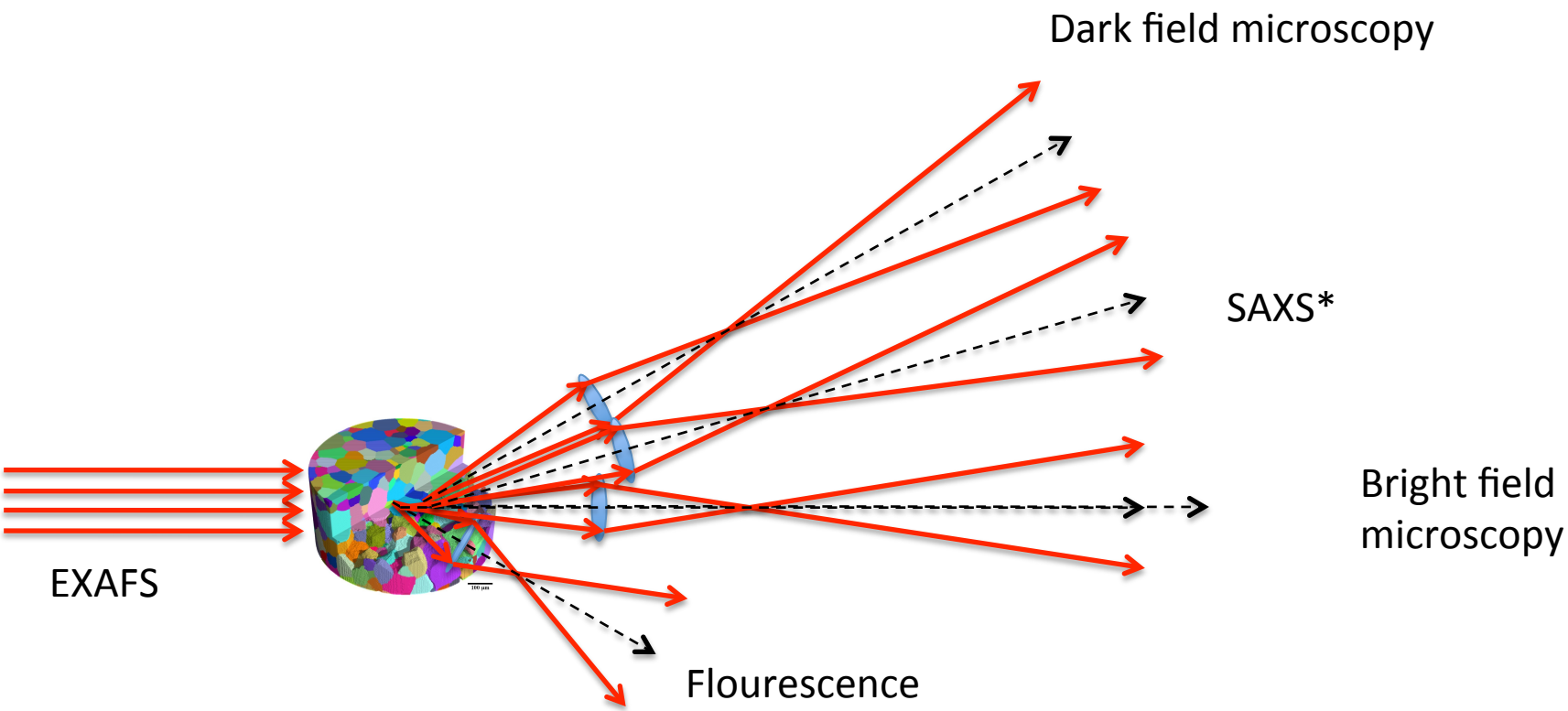
*Ludwig *et al. J. Appl. Cryst*. (2001). 34, 602-607

** A.C. Jakobsen, H. Simons, W. Ludwig, Y. Zhang, C. Detlefs, H.F. Poulsen. Work in progress

# Multimodal and 4π coverage

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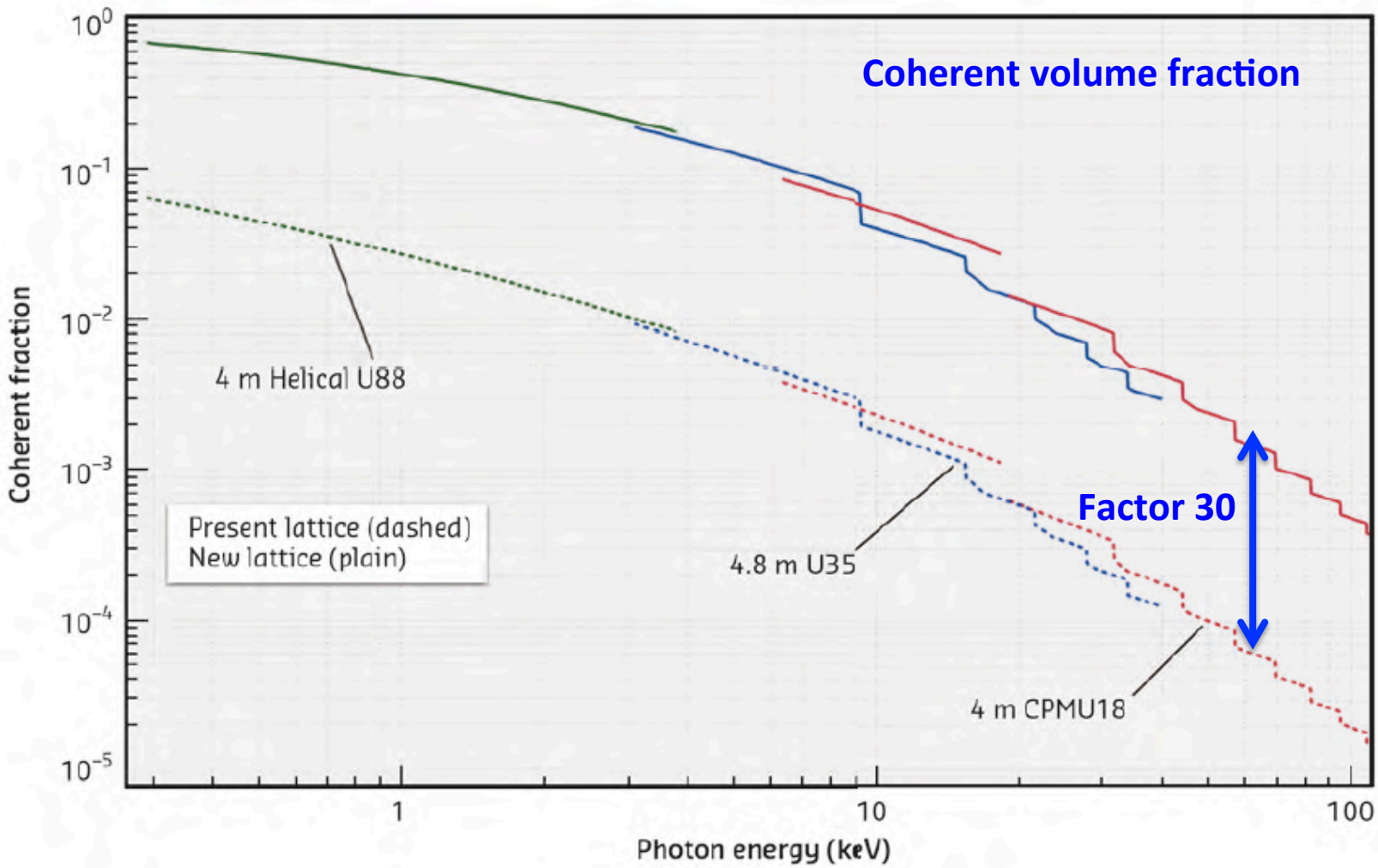
*A. Bosak, I. Snigireva, K.S.Napolskii, A. Snigirev *Adv. Mat*. **22**, 3256 (2010).

# Ultimate resolution: use of coherence

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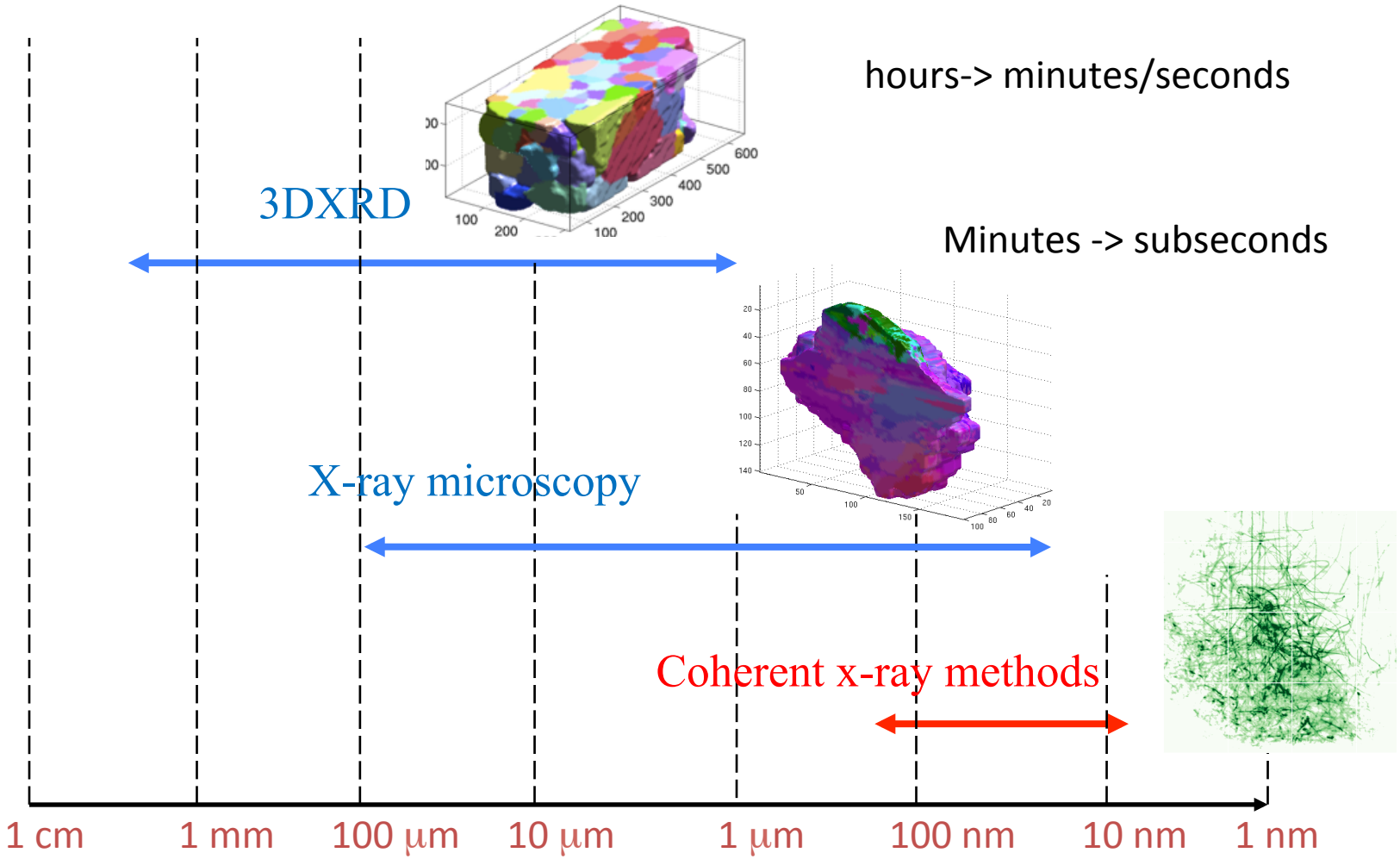
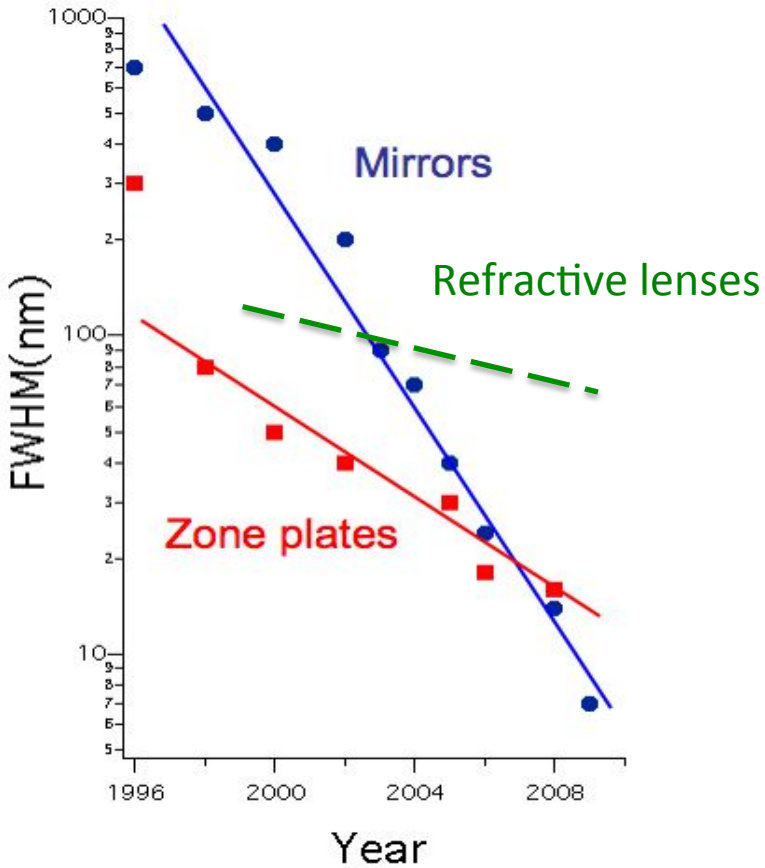
*From ESRF "Orange book"

# Multiscale studies at “ultimate storage rings”



# Challenge: refractive optics




New manufactuing technologies needed

Minituarisation superior route*

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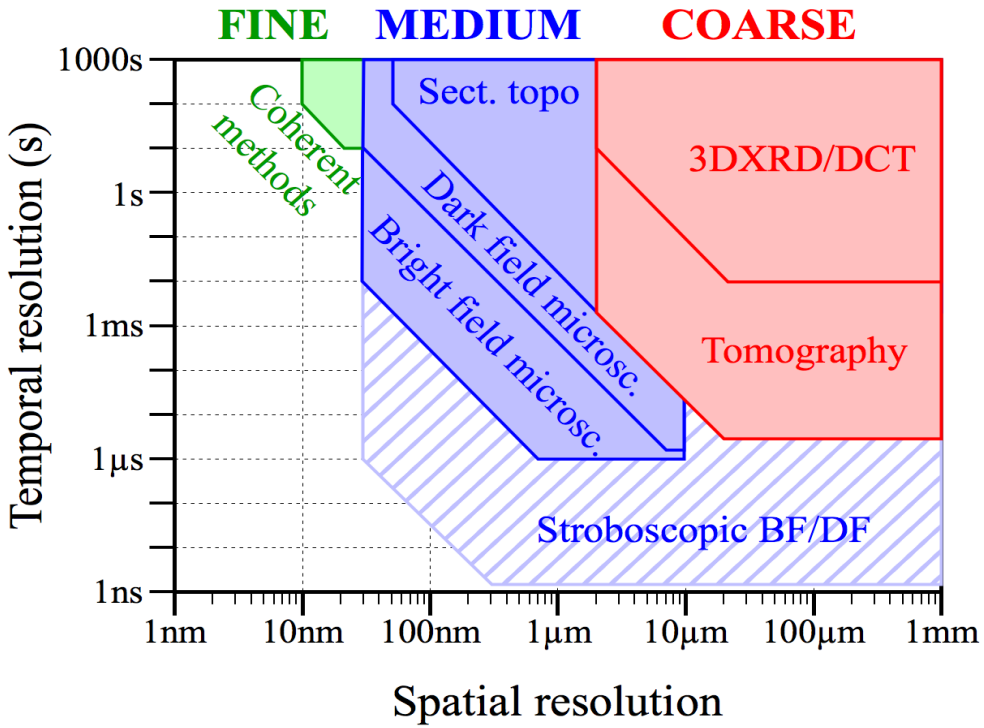
*H. Simons, F. Stöhr, J.M. Lindhard, F. Jensen, O. Hansen, C. Detlefs, H.F. Poulsen. arXiv 1506.06677v1 (2015)

# A microscope on an ultimate storage ring

20-80 keV x-rays




Science cases: onset of plasticity, workhardening, local electrochemistry, liquid flow in reservoirs, crack nucleation in composites, flexoelasticity, biomimicry, 3D printing, fluctuations in phase transformations, pinning in superconductors, molecular electronics, bone structure/healing, colloidal particle selforganisation, …

Support from ERC Advanced grant ”d-TXM”, ESRF, and DTU