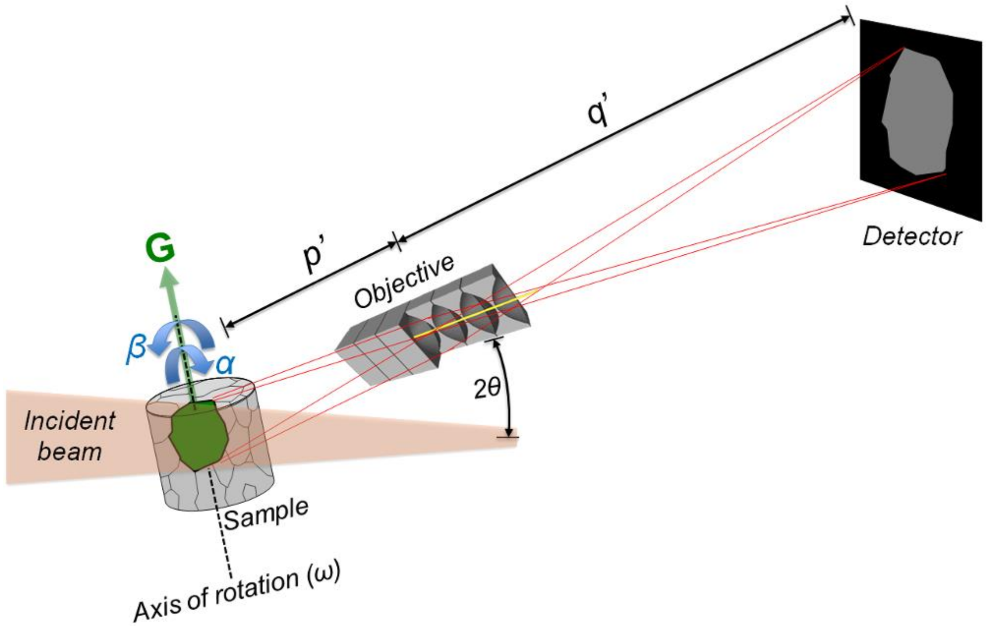
ID03 is the new ID06-HXM,
the ESRF Dark-Field X-ray microscopy beamline

STREAMLINE

C. Detlefs (ESRF), on behalf of the ID03 team:
Helena Isern
Raquel Rodriguez Lamas
Thierry Brochard
Thomas Dufrane
Can Yildirim
and many collaborators



DARK FIELD X-RAY MICROSCOPY

What is dark field x-ray microscopy?

- How does it work?
- What can it be used for?
  Overview
  Examples

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*H. Simons et al. (2014). Dark-field X-ray microscopy for multiscale structural characterization. Nature Communications 6, 6098.
DOI: 10.1038/ncomms7098*
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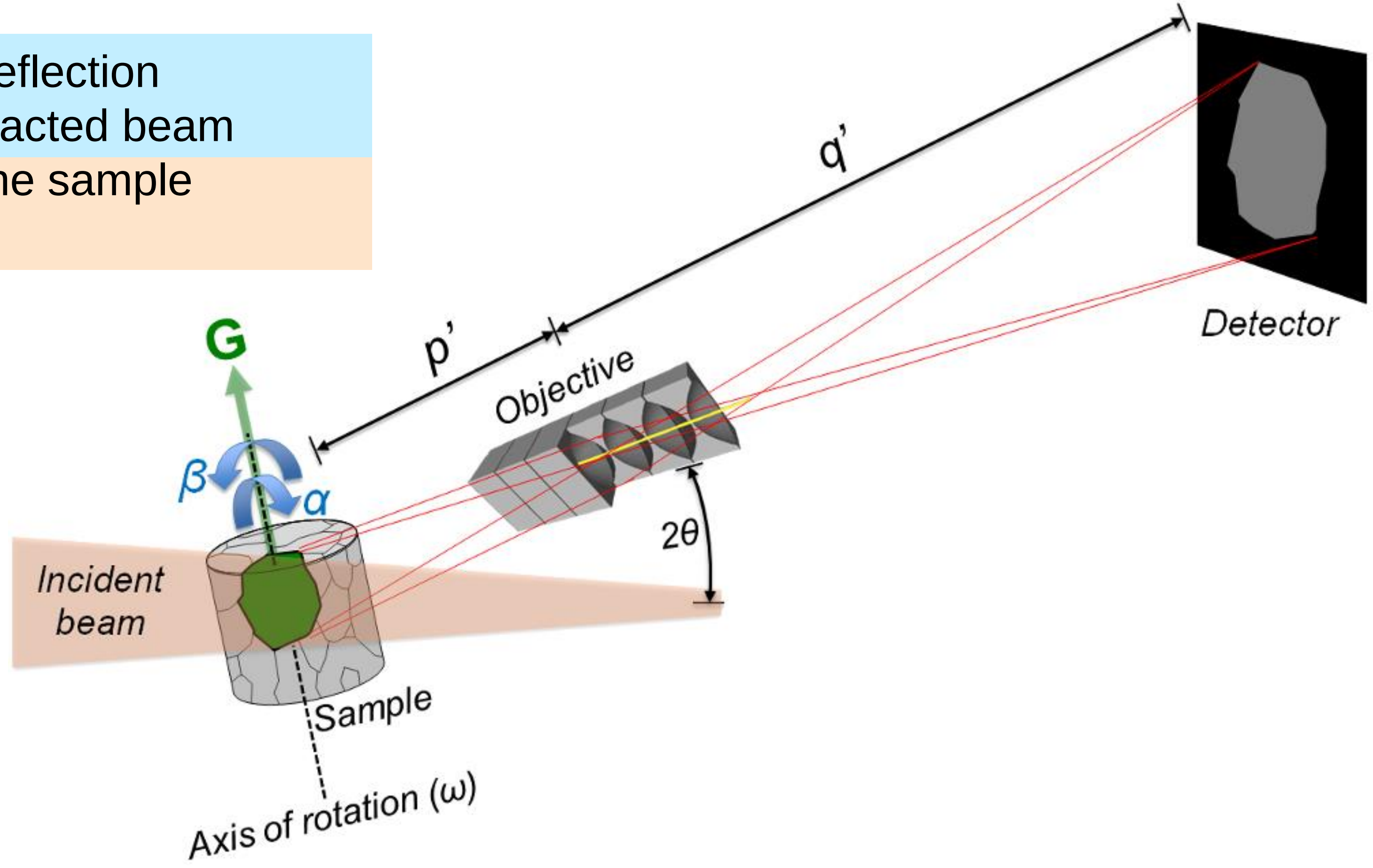
*C. Yildirim et al. (2020). Probing nanoscale structure and strain by dark-field x-ray microscopy. MRS Bulletin, 45, 277
DOI:10.1557/mrs.2020.89*

Magnified diffraction topography
using a lens between sample and detector

- You take a sample and align a Bragg reflection
- You put an imaging detector in the diffracted beam
- You insert an objective lens between the sample and the detector



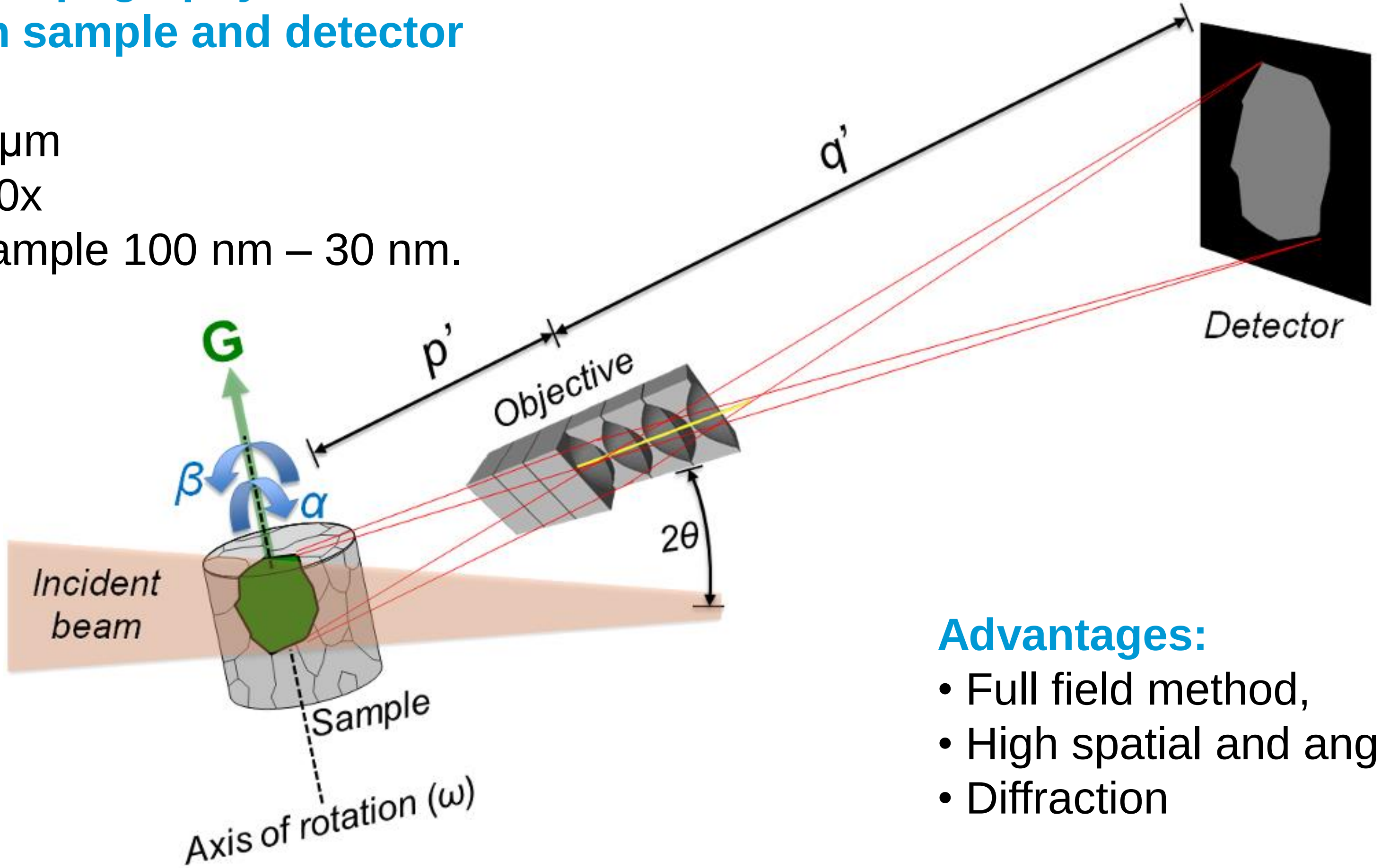
- Diffraction topography
- Rocking curve imaging

- X-ray microscopy

DARK FIELD X-RAY MICROSCOPY

Magnified diffraction topography using a lens between sample and detector

Detector resolution 1 µm
Magnification 10x – 30x
→Resolution at the sample 100 nm – 30 nm.



Advantages:

- Full field method,
- High spatial and angular resolution,
- Diffraction

COMPLEMENTARITY TO ELECTRON MICROSCOPY

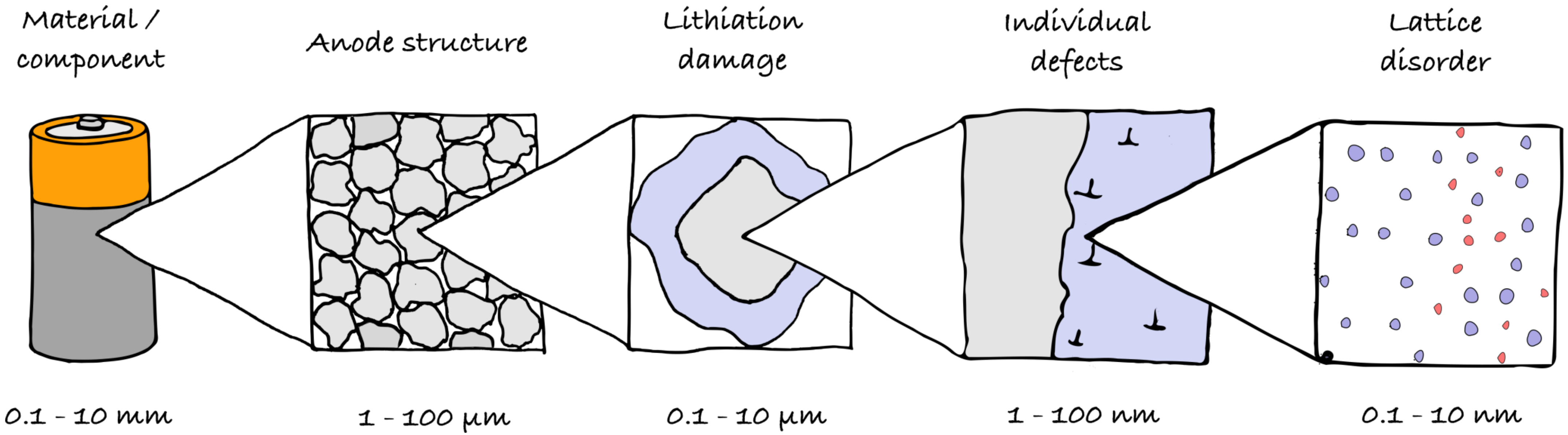
Complementary to electron microscopy (TEM, STEM)
→ both are diffraction based full-field imaging,
offering crystallographic sensitivity: phase, orientation, strain, …

	DXFM	TEM/STEM
Spatial resolution	~150 nm 😐	~1 pm 🙂
Sample preparation	<0.5 mm thick, no polishing needed 🙂	~100 nm thick, polished 😐
Strain resolution	~0.001% 🙂	~0.1% 😐
Sample environment	In-air, possibility of *in-situ* setups 🙂	Vacuum, limited sample environments 😐

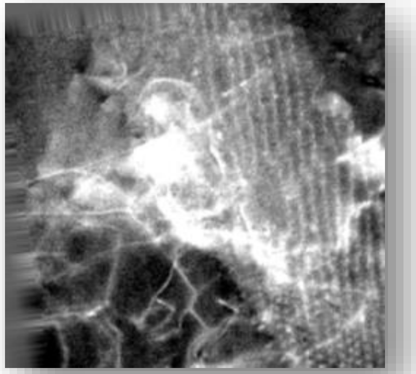
- **Main advantage, due to bulk samples sizes, is the capability to follow sample evolution *in-situ* during processing (temperature, electric field, mechanical strain…)**

Need for Multiscale Imaging

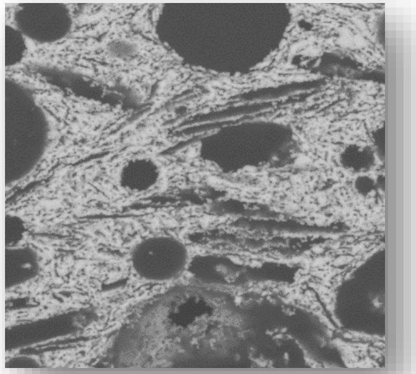
Materials properties are determined by hierarchically organized structures



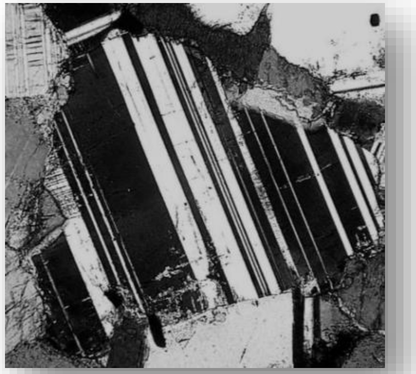
Metals



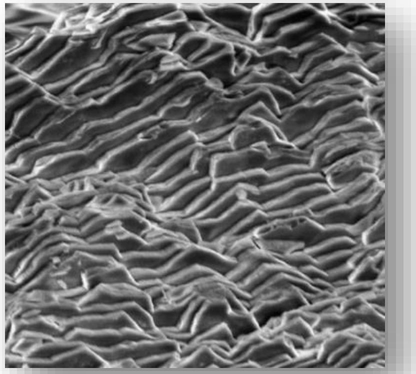
Ceramics



Minerals



Biomaterials



Courtesy of H. Simons

DARK FIELD X-RAY MICROSCOPY

Diffraction technique:

Bragg's law

$$\lambda = 2d \; \sin\theta$$

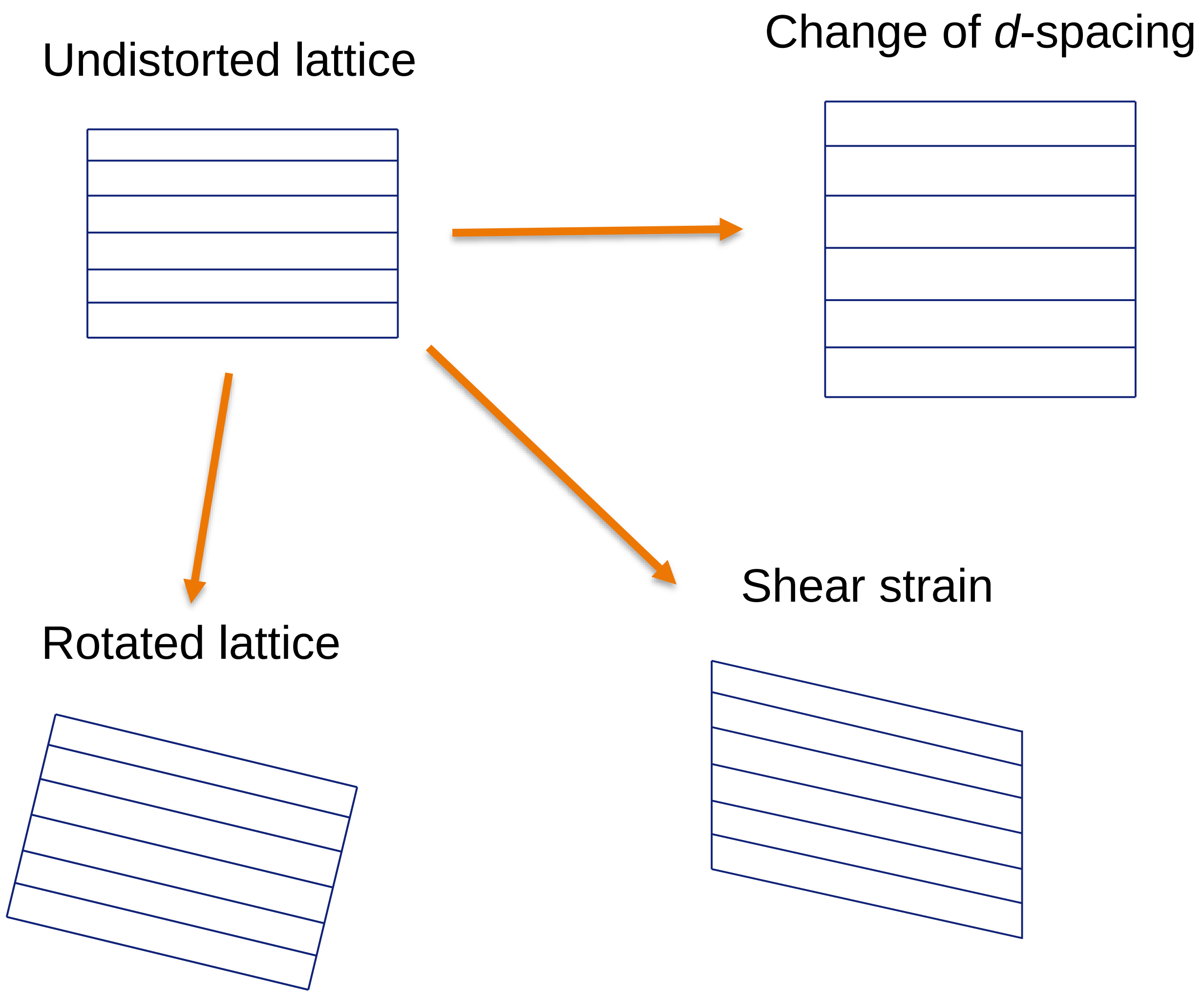
Sensitive to variations in the crystal lattice

- Phase
- Orientation of lattice planes
- *d*-spacing

→ Strain
→ Defects such as dislocations
→ Grain/domain boundaries

Measure diffracted intensity as function of crystal orientation and scattering angle

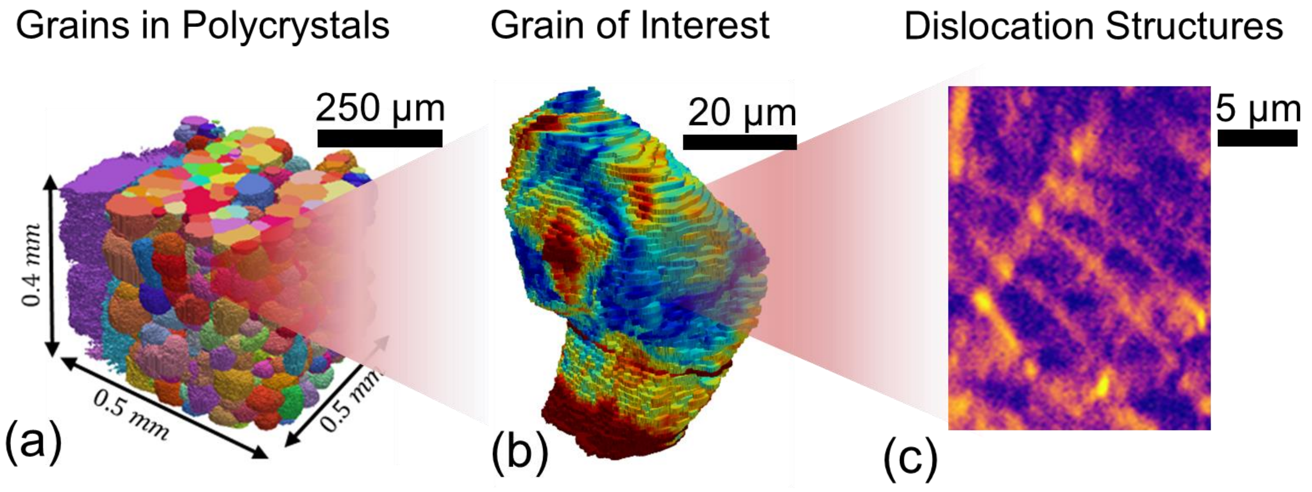
Record a microscopy image at each angle



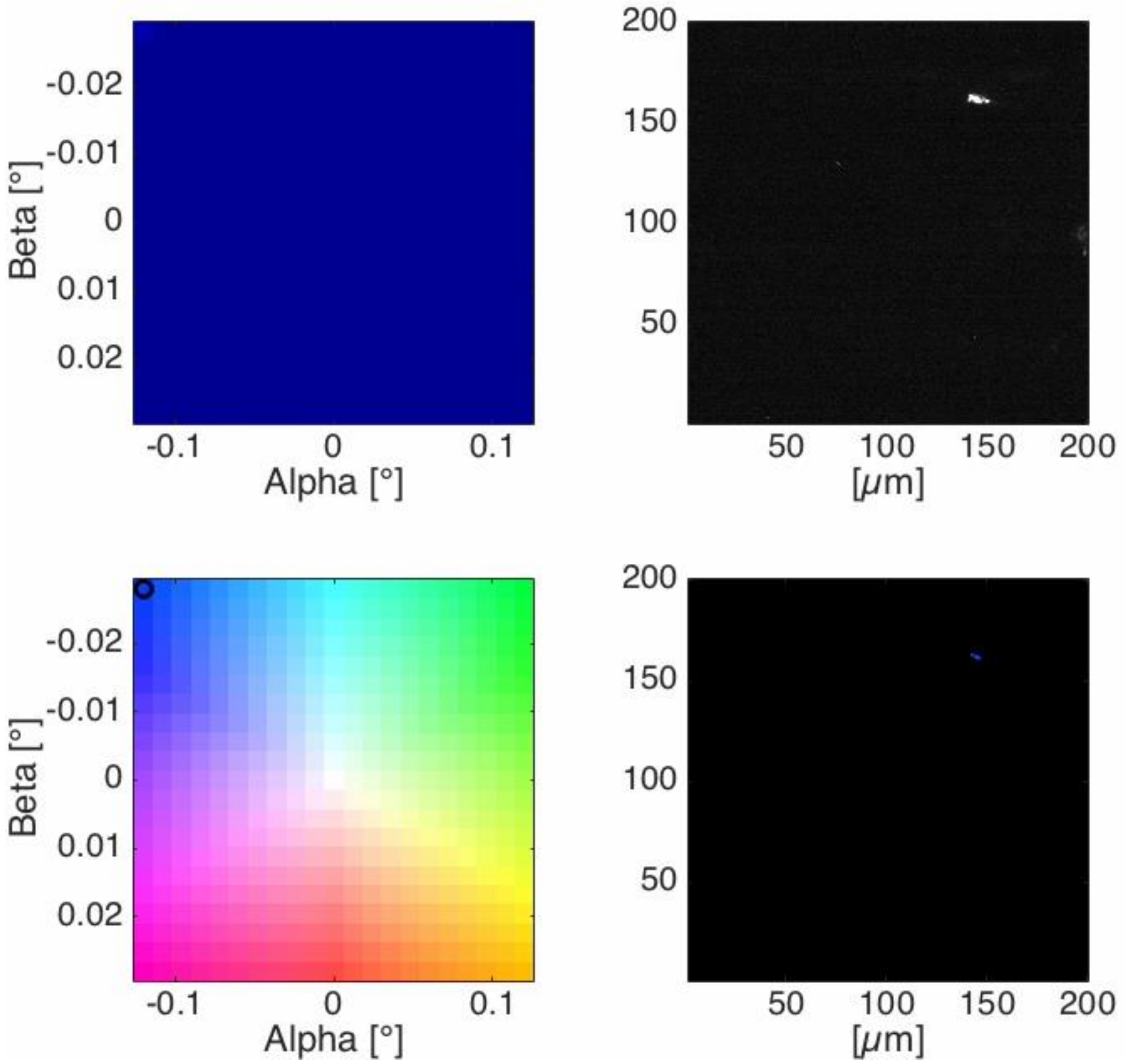
Dark Field X-ray Microscopy

A unique microscope to image embedded structures

- Mapping grain level 3D Lattice strain and orientation
- Can be coupled with 3DXRD/DCT
- Analysis package : *darfix*



Multi-scale Orientation/Strain Mapping from mm to nm range



Gustafson et al, Acta Mat. (2023)
Yildirim et al, Sci Rep. (2023), Garriga Ferrer et al, J. Synch Rad. (2023)

H. Simons et al, Nat. Com. (2015); Yildirim et al, MRS Bulletin (2020)

Courtesy of H. Simons

Key parameters:

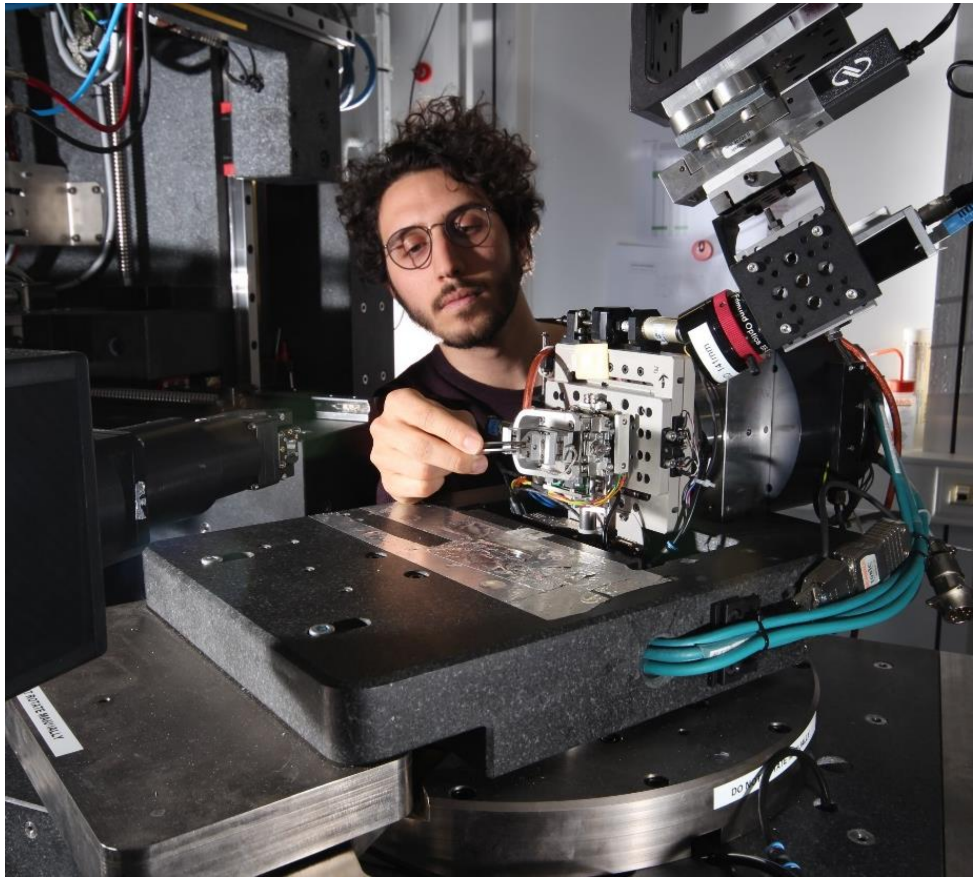
Working energy:

- typically 15-20 keV
- option for 33 keV

Focal length of the objective ~280 mm

Resolution:

- In practice ~150 nm
- Diffraction limit ~60 nm



Multi-scale operation:

(we are still working on integrating this)

- 3DXRD for identification of grains of interest
- DCT for identification of grains of interest
- Near field camera (~1-2 µm pixel size)

2D → 3D:

- Layer by layer with line beam
- Topo-tomo (in progress)

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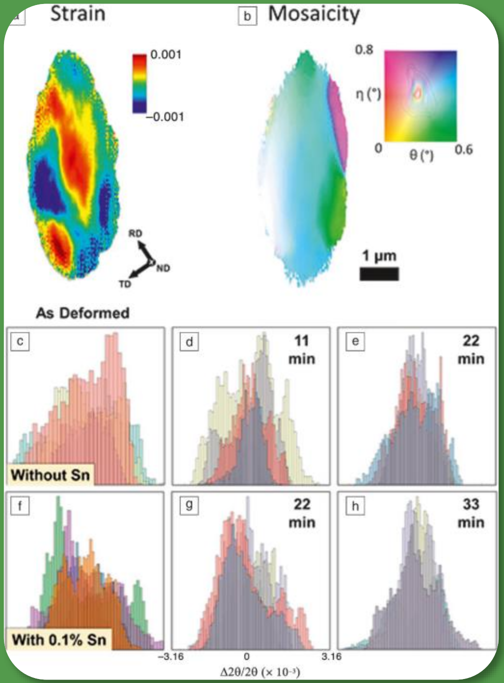
ID03: HARD X-RAY MICROSCOPY BEAMLINE

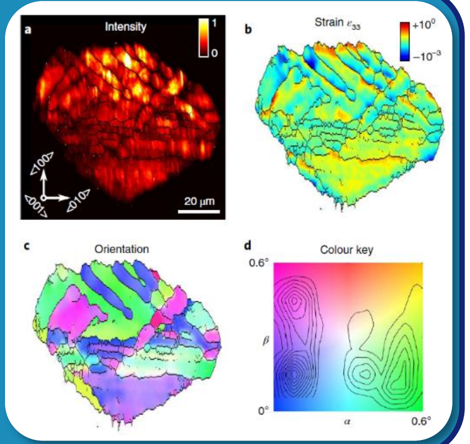
Metallurgy

- Pattern formation
- Materials fatigue
- Recovery and recrystallization



Functional materials

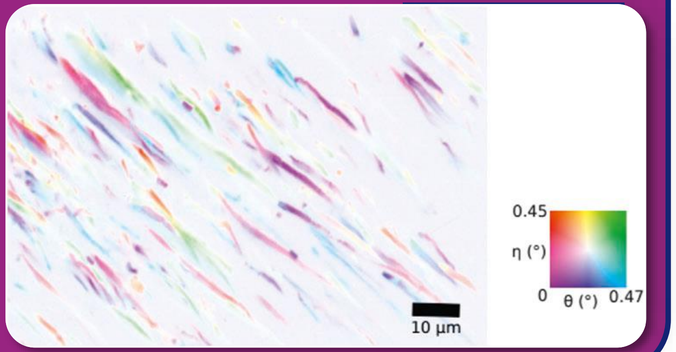
- Strain at grain boundaries
- Formation of domain patterns
- Dynamics of domain switching



3D strain maps with 150 nm spatial resolution

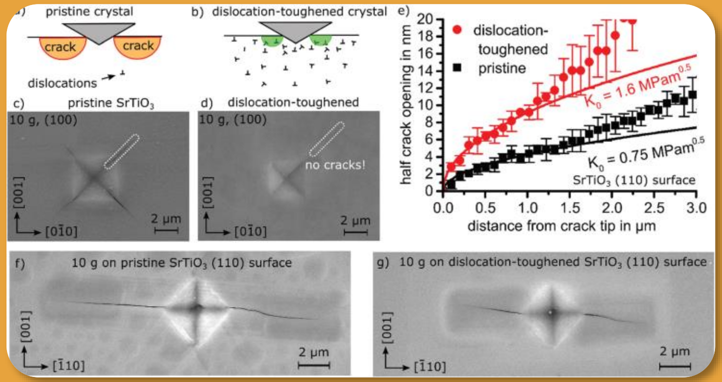
Biominerals

- Microstructure



Ceramics

- Dislocation-toughening
- Nano-twinning



STREAMLINE

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 870313.

The European Synchrotron | ESRF

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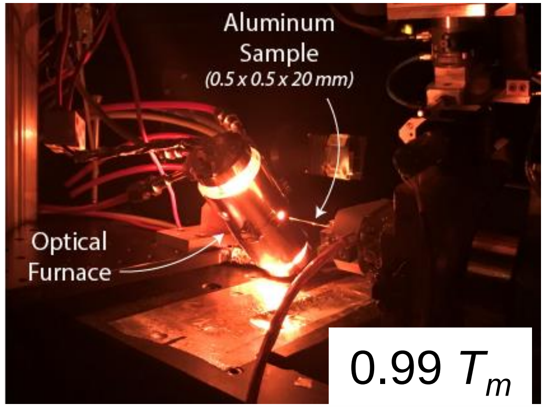
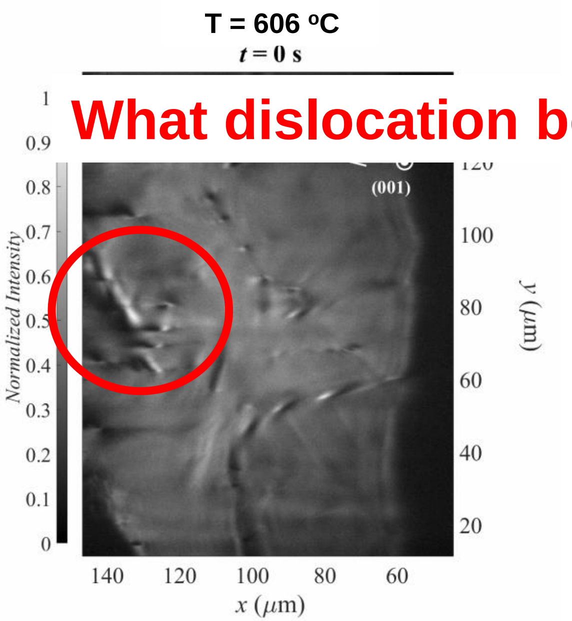
TIME RESOLVED THERMALLY DRIVEN DISLOCATION MECHANICS

Thresholded Dynamics of Dislocations as Mobility Increases, ~200-µm Below the Surface in Al single crystal



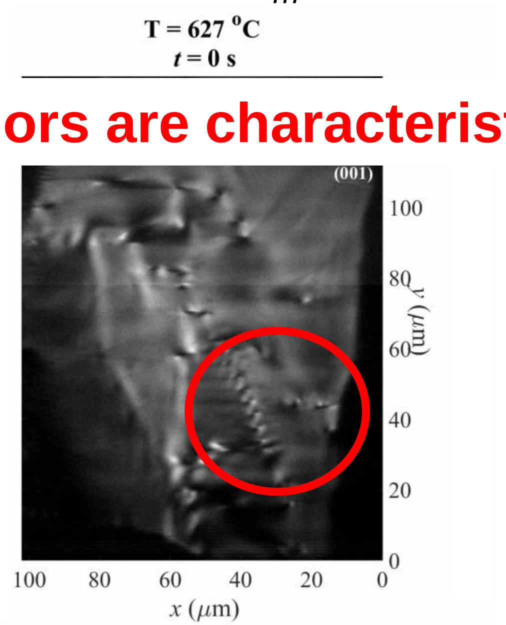
What dislocation behaviors are characteristic of these T regimes?

0.93 T_m



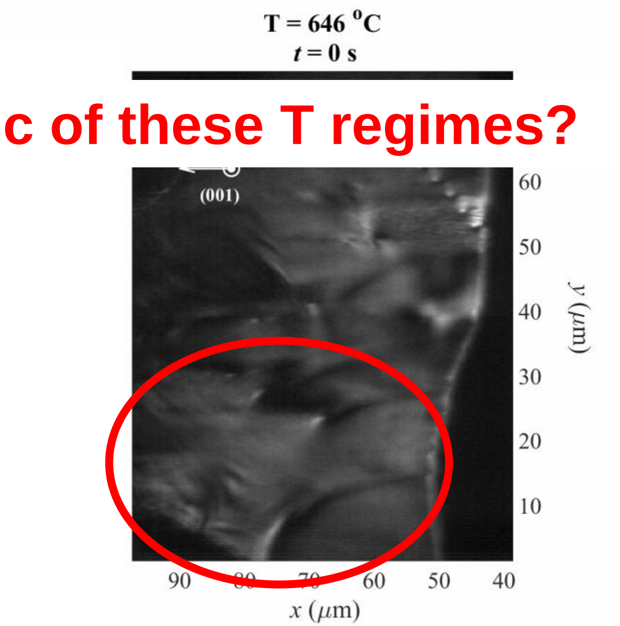
Breathing Motion

0.97 T_m



Interactions

0.99 T_m



Flow

13

Dresselhaus Marais et al. Science Advances 2021 – Yildirim et al. Rev. Sci Inst. 2020

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

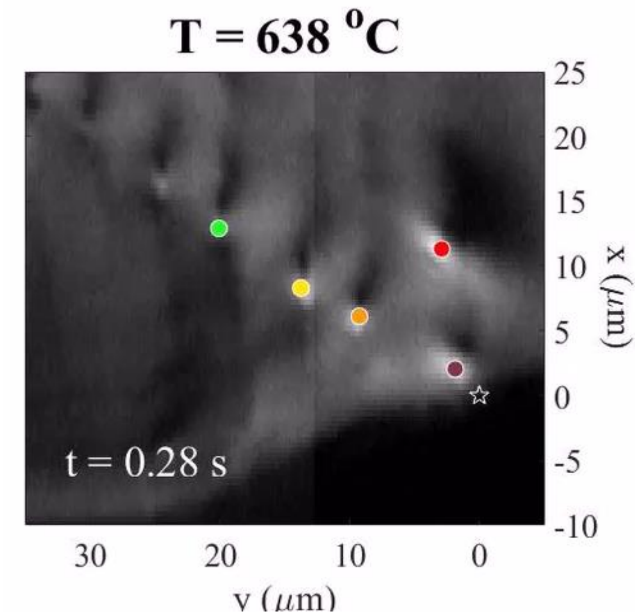
LLNL-PRES-818088

A NEW REGIME OF DISLOCATION BEHAVIOR

LLNL-PRES-818088

New View of Long-Range Interactions in Dislocation Structures

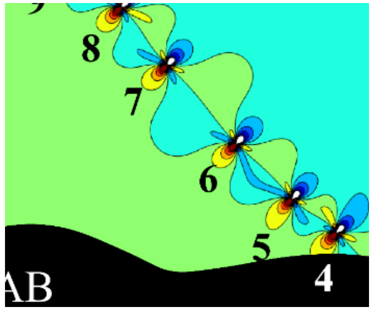
Classical Dislocation Dynamics



Thermal Forces Amplify

Thermal Effects Dominate Interactions

Insight into Regimes that Models & Experiments Struggle to Access



T_m

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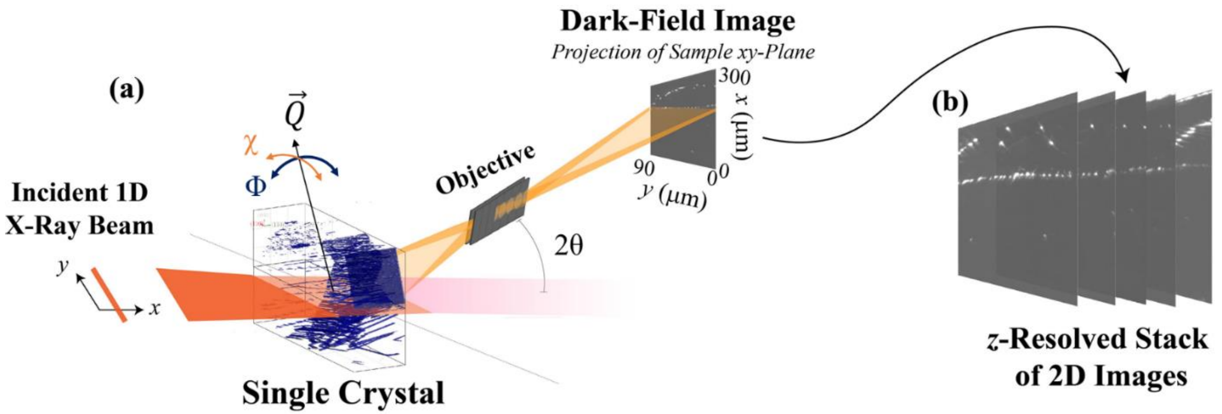
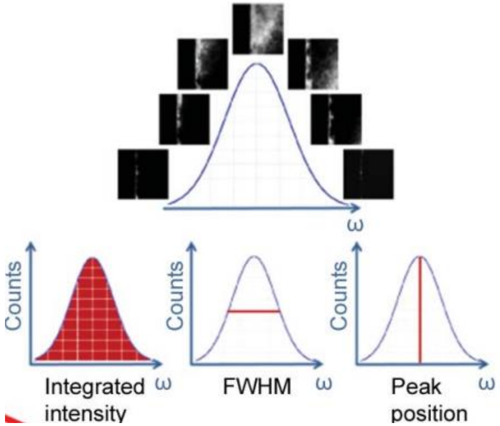
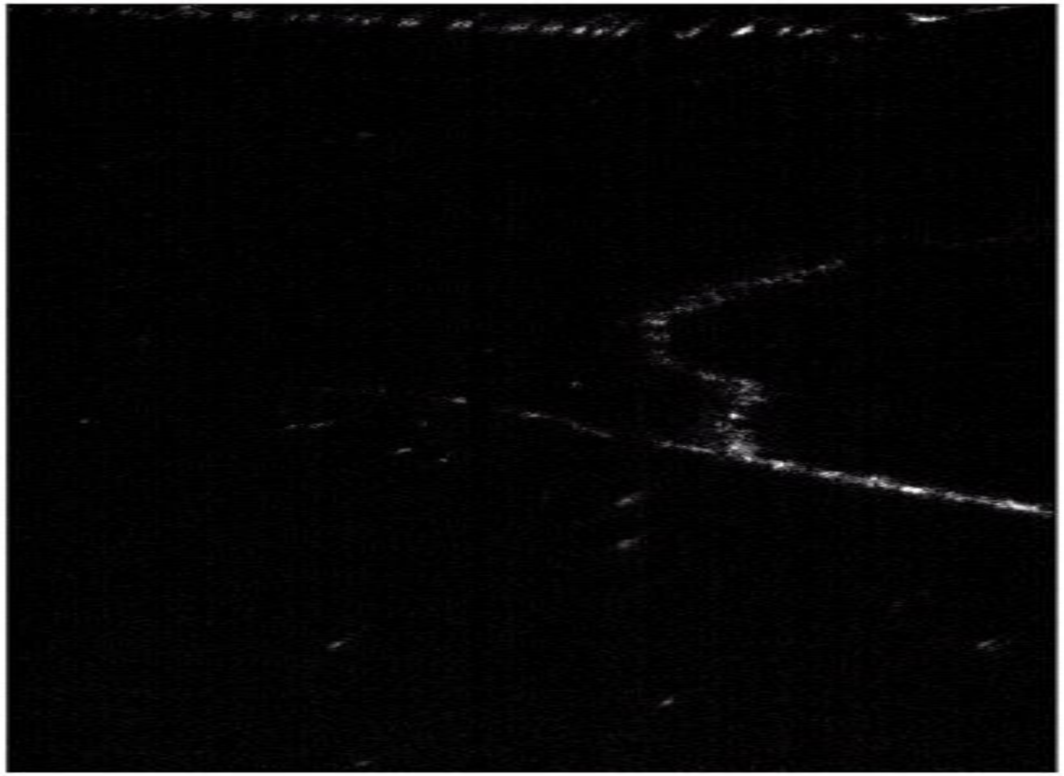
DISLOCATIONS IN ANNEALED AL SINGLE CRYSTAL



Al Crystal annealed 10h at 590C

Weak-beam Z scans shows dislocations in 3D space

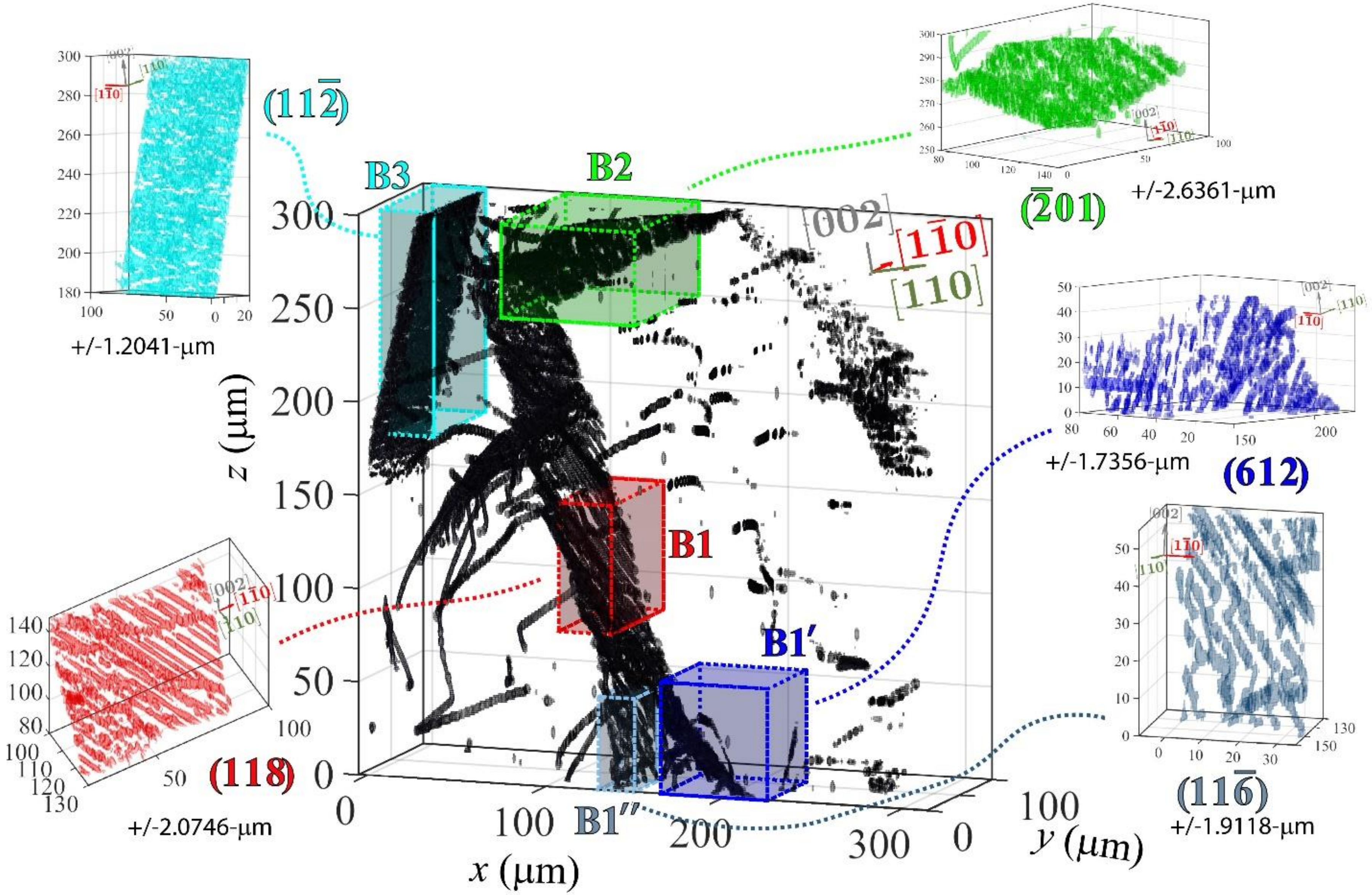




J. Appl. Cryst. (2017). 50, 561–569c

C. Yildirim, H. Poulsen, G. Winther, C. Detlefs, P. Huang and L. Dresselhaus- Marais, Sci Reports 13, 3834 (2023).

SELF ORGANIZATION OF DISLOCATIONS INTO BOUNDARIES



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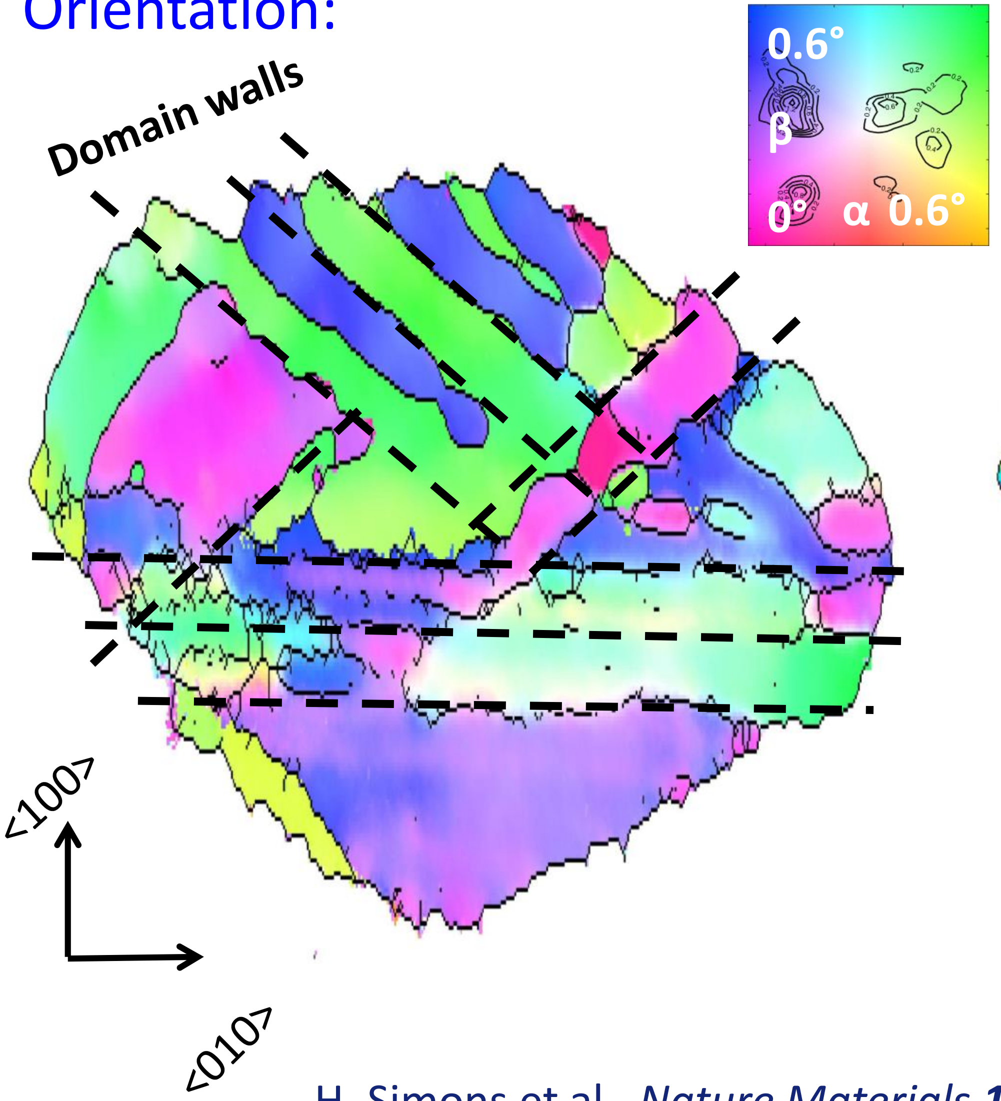
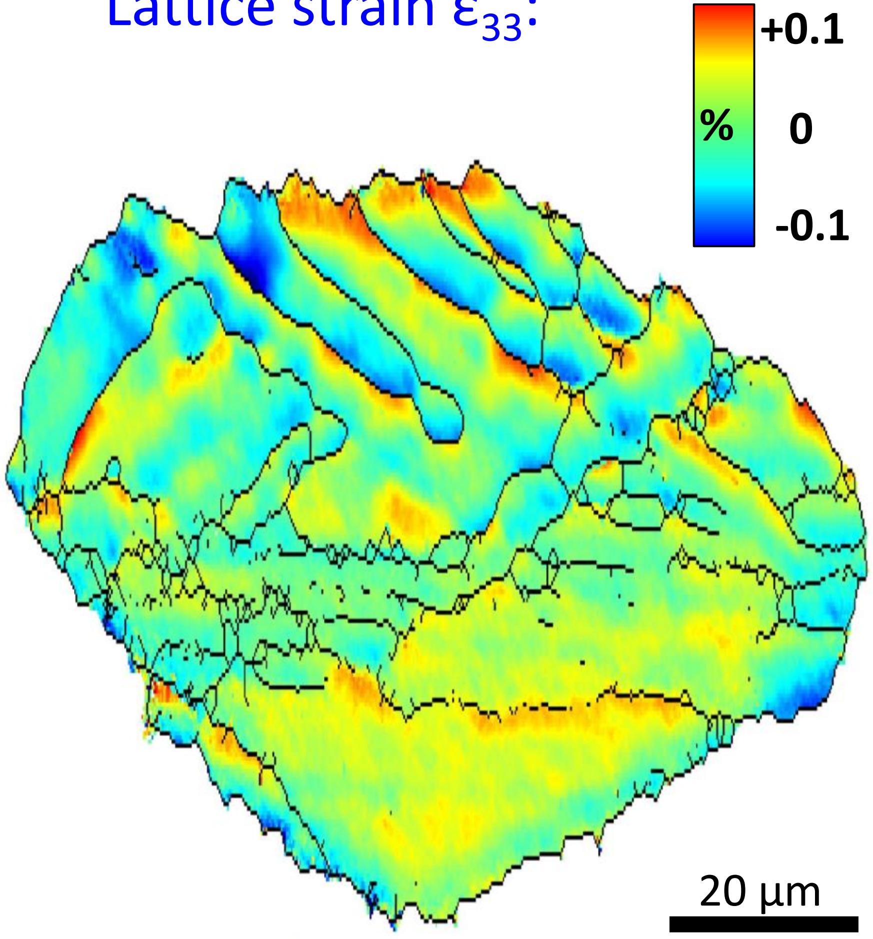
DOMAINS IN $BaTiO_3$

Orientation:



Lattice strain ε_{33}:



H. Simons et al., *Nature Materials* ***17***, *815* (2018).

DOMAINS IN $BaTiO_3$

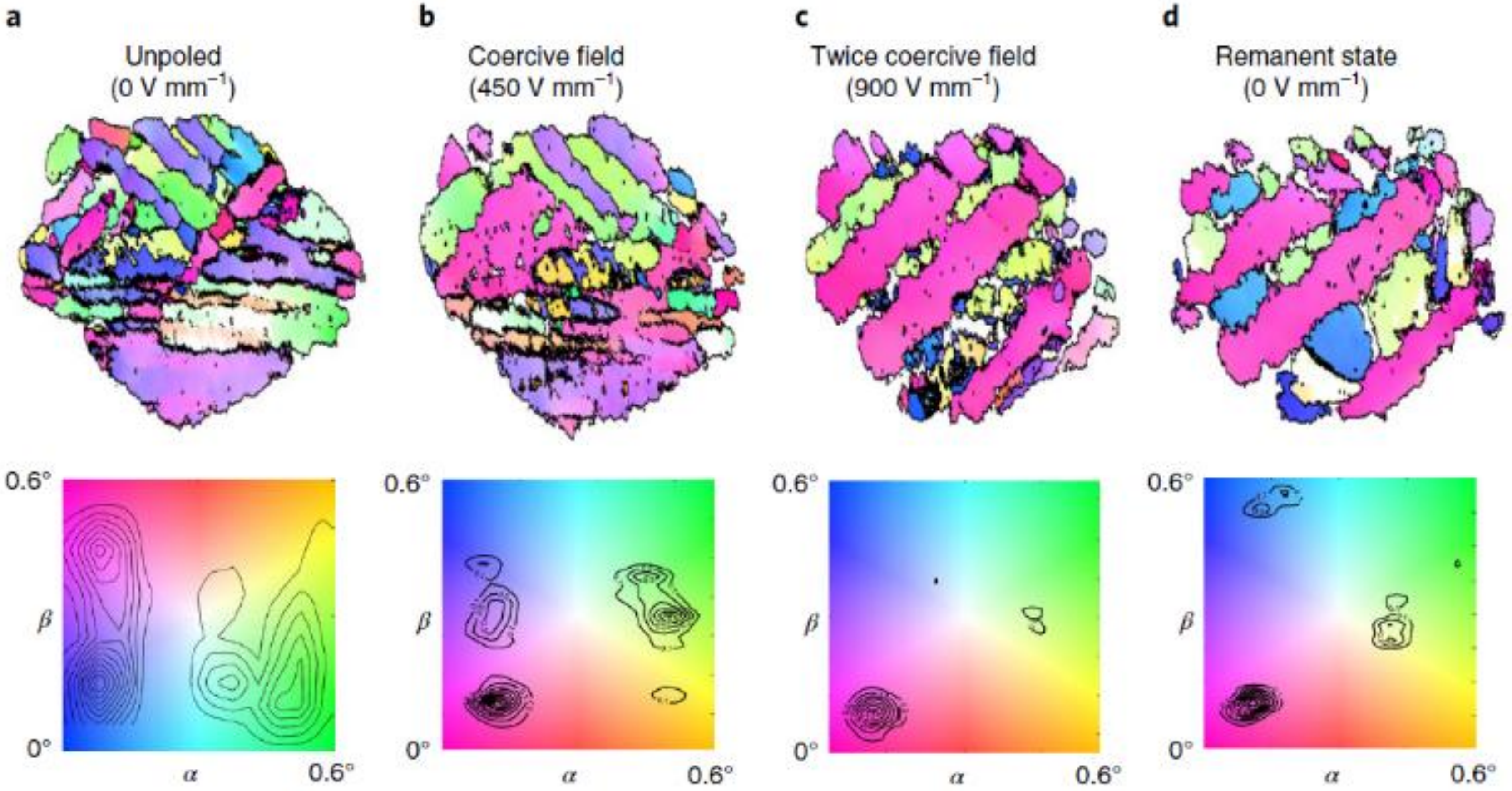


Fig. 4 | Changes to the domain topology and orientation distribution in the embedded $BaTiO_3$ grain during the in situ application of a unipolar electric field cycle along the <100> direction. a–d, Orientation maps (top) and diffraction intensity distributions (bottom) are shown for four points on a characteristic polarization (*P*) versus applied electric field (*E*) hysteresis curve: the initial zero-field state (**a**); at the coercive field, where most domain switching occurs (**b**); at twice the coercive field, where the induced polarization is saturated (**c**); and in the remanent state after the removal of the electric field altogether (**d**). Movies of the domain evolution as a function of field are available in the Supplementary Information.

H. Simons et al., *Nature Materials* ***17**, 815* (2018).

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SOFTWARE

Data analysis softw
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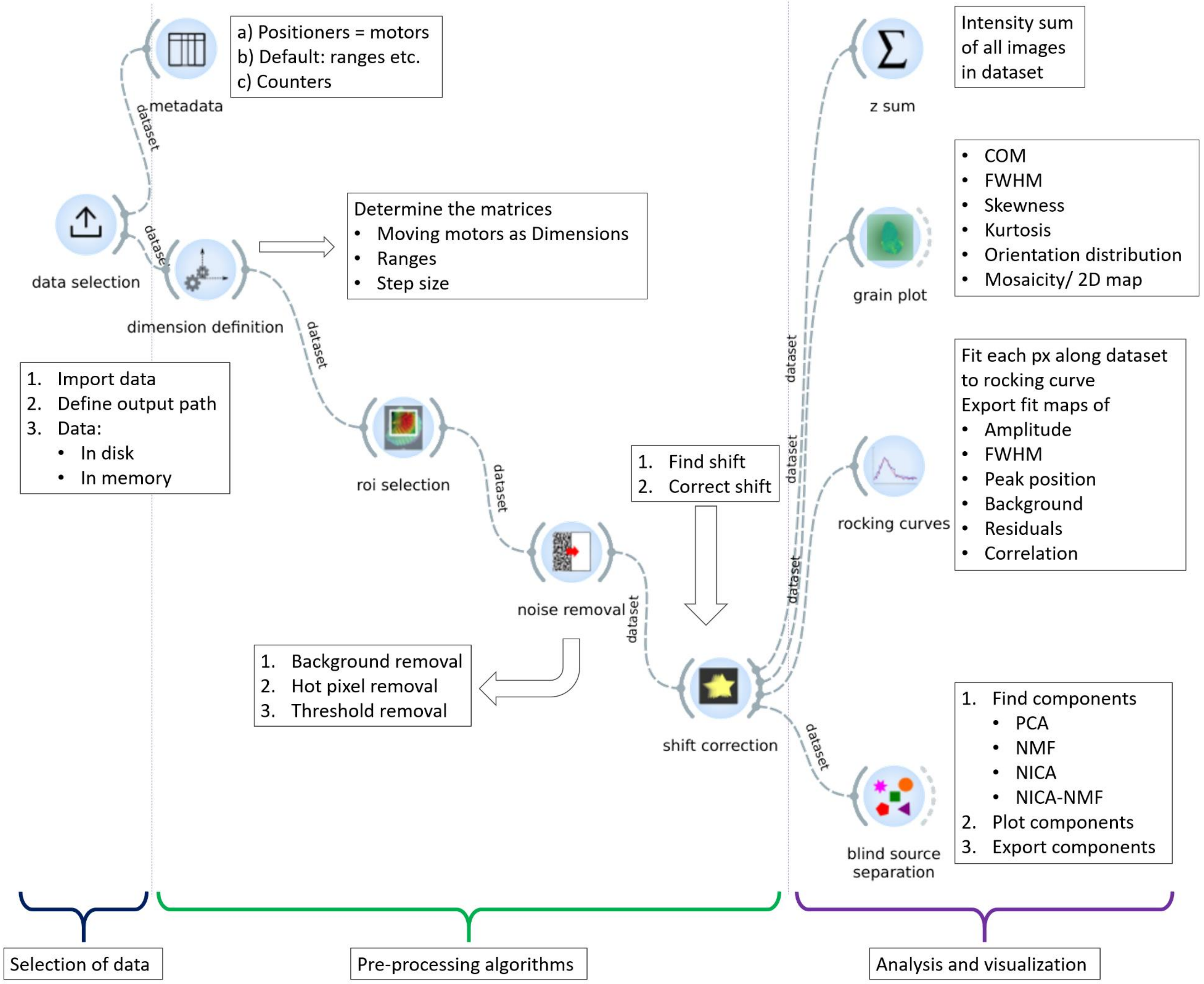
- Widgets for all ess
    - Data selection,
    - Pre-processing
    - Processing (CC
    - Blind source se



J. Garriga-Ferrer, R. Rodriguez-L
J. Synchrotron Rad. 30, 527 (202

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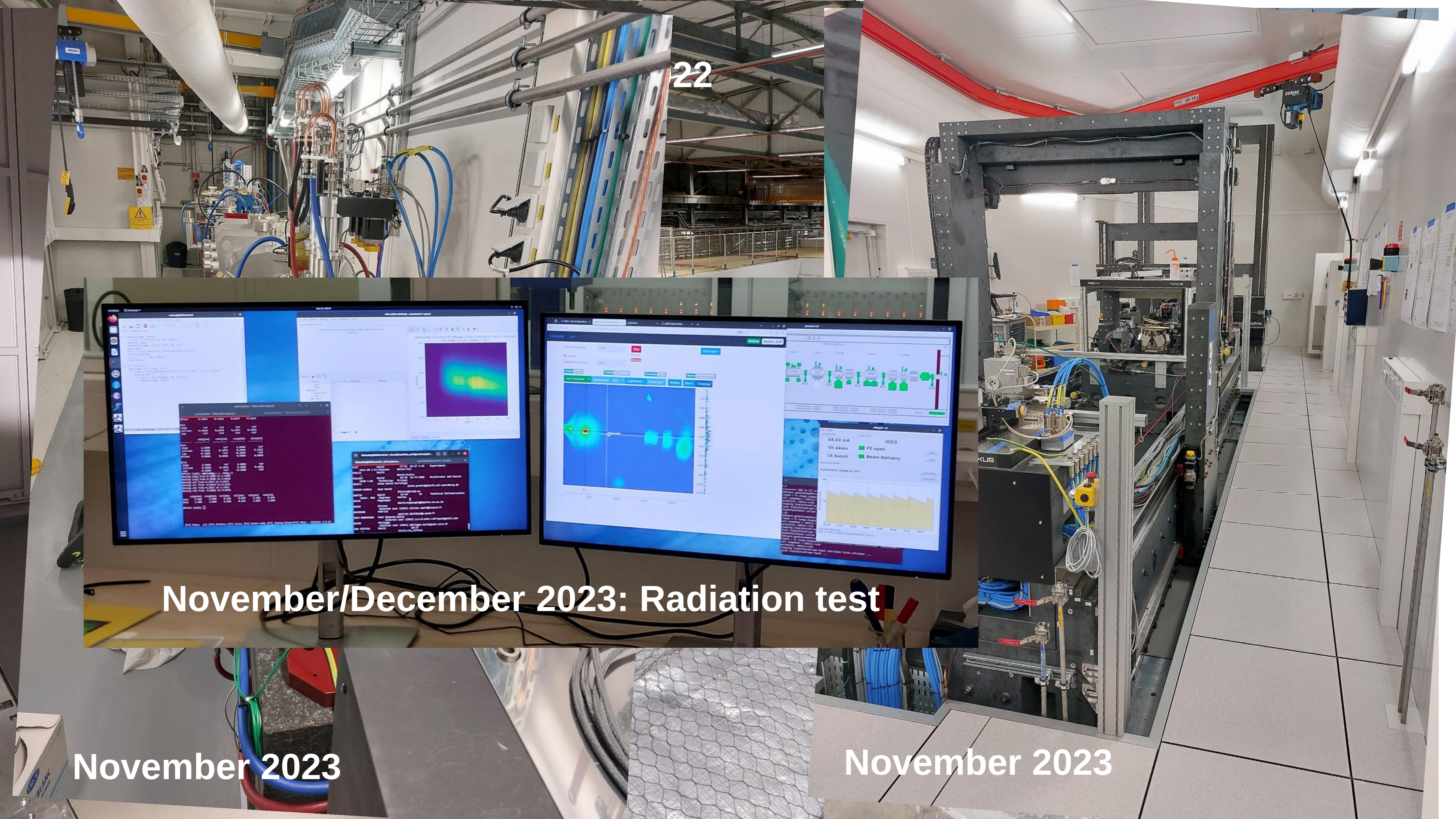
ID03 replaces ID06-HXM

- Re-use the DFXM end-station from ID06-HXM
- Rebuild the beamline from the ground up
- Build new, fully optimized optics

→ New, latest generation cryogenic undulator (CPMU)
→ Pink beam option, x100 flux
→ New sample goniometer optimized for topo-tomo
→ The beamline is fully dedicated to DFXM, therefore x2 more beamtime available


November/December 2023: Radiation test
November 2023
November 2023

THE NEW BEAMLINE ID03

Timeline