3D and 4D sub-grain mapping of lattice strains and orientations in polycrystals using Diffraction Contrast Tomography (DCT)

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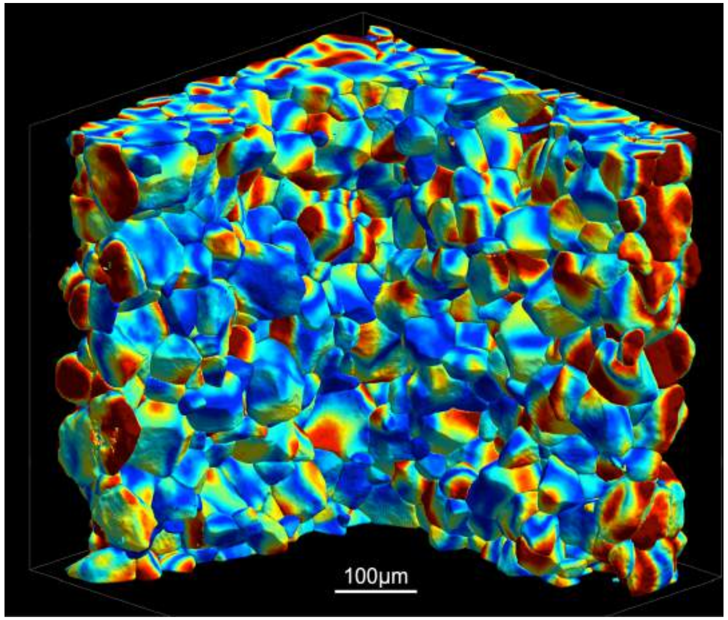

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ESRF DFXM online workshop
06 May 2021

Motivation:

simplicity, efficiency, speed, statistics

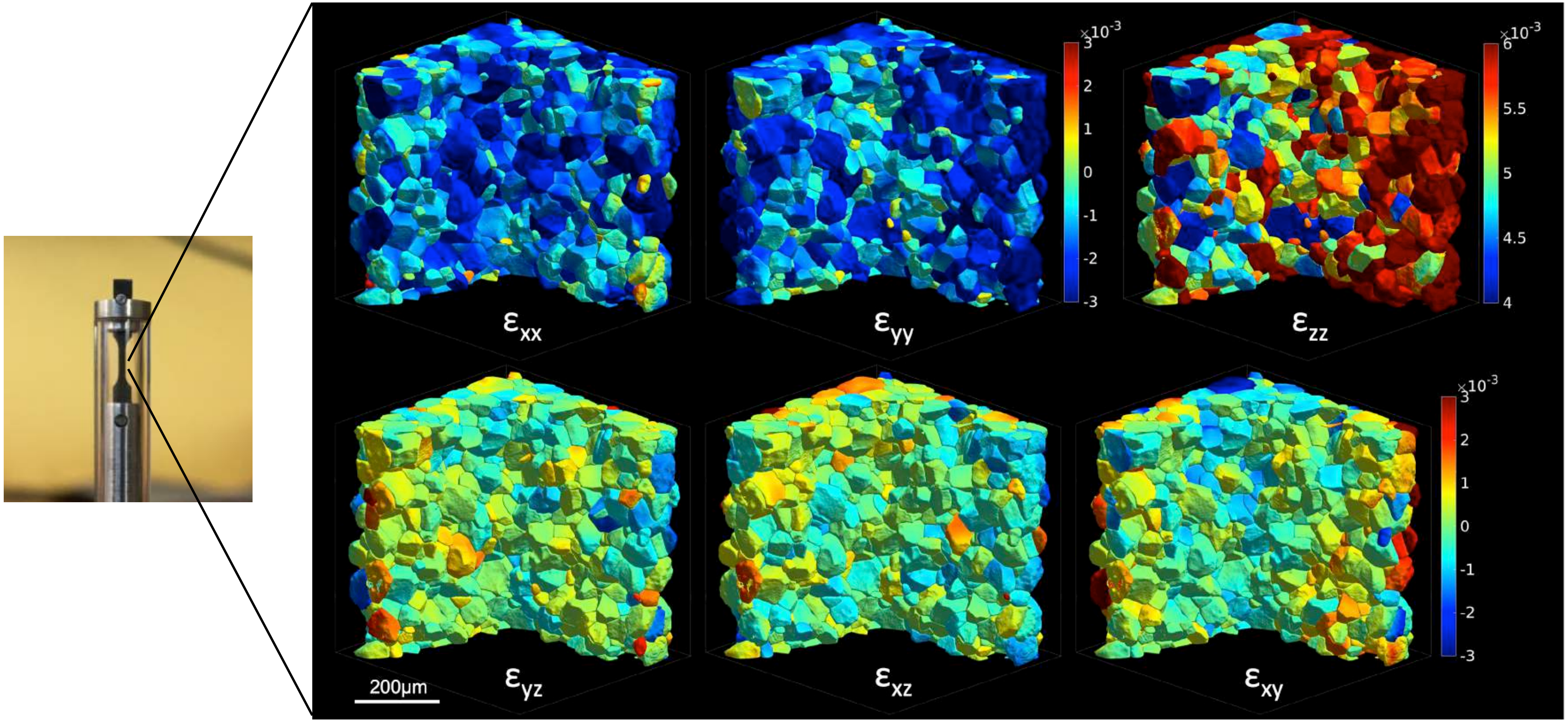
Sub-grain 3D strain maps in polycrystals by DCT

Inferring locally in a complete 3D section:

- crystallographic phase
- orientation: *3 parameters*
- strain: *6 parameters*
  - complete strain/stress tensor
  - local unit cell

Performance goals:

- spatial resolution: 1…5 um
- orientation & strain resolution: 1…5 x 10^{-4}
- scanning times: minutes to hours



Strain tensor components in the Sample reference (Gum Metal at 365 MPa)

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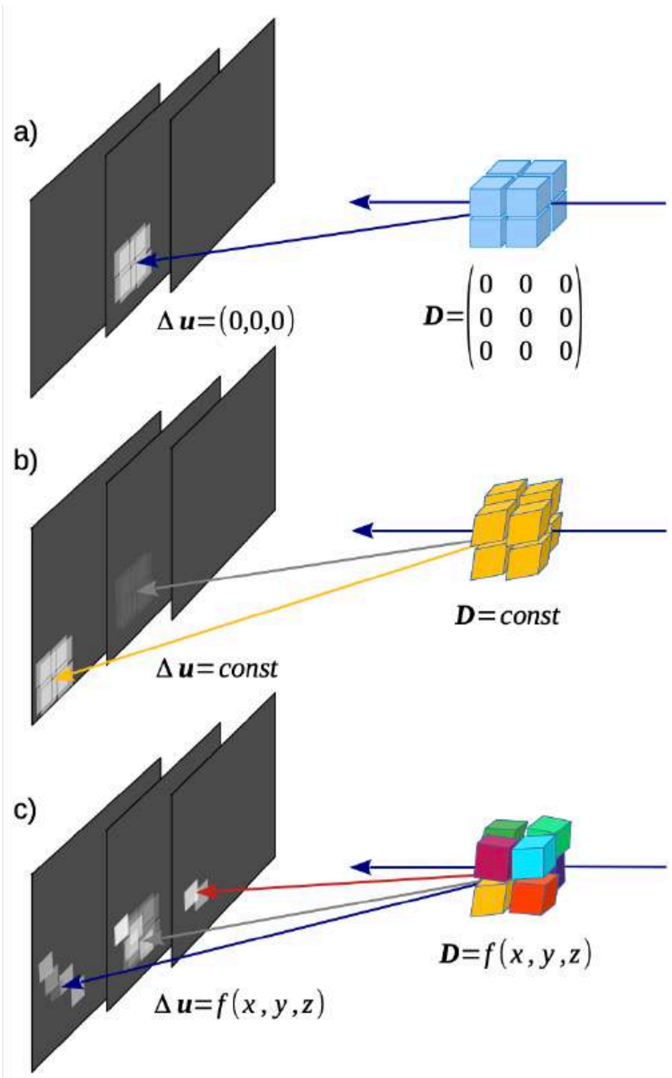
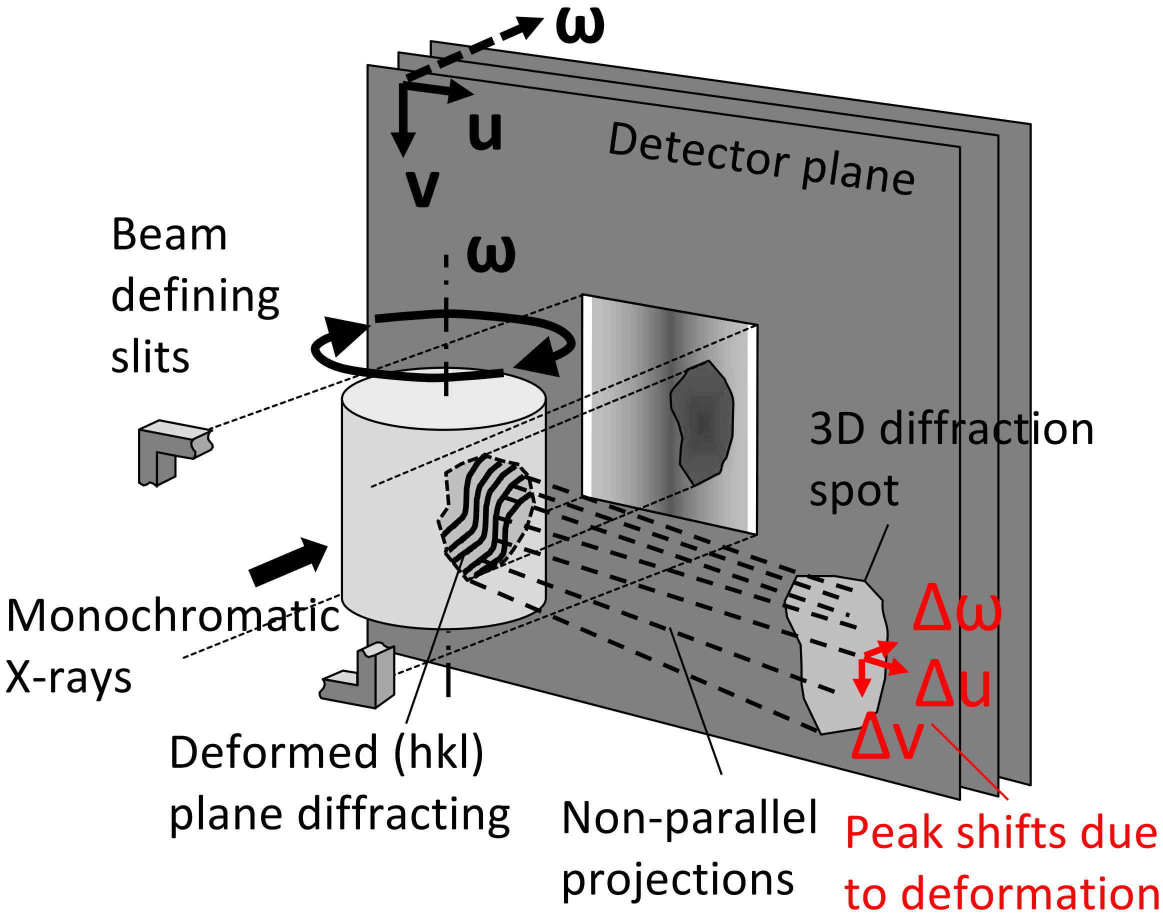
Diffraction Contrast Tomography (DCT) setup

- full beam approach
- pixel size: 0.7 .. 5 um
- monochromatic X-rays: 15 .. 60 keV
- single axis scan: continuous **ω** rotation
- small **ω** steps: 0.005 ... 0.1 °





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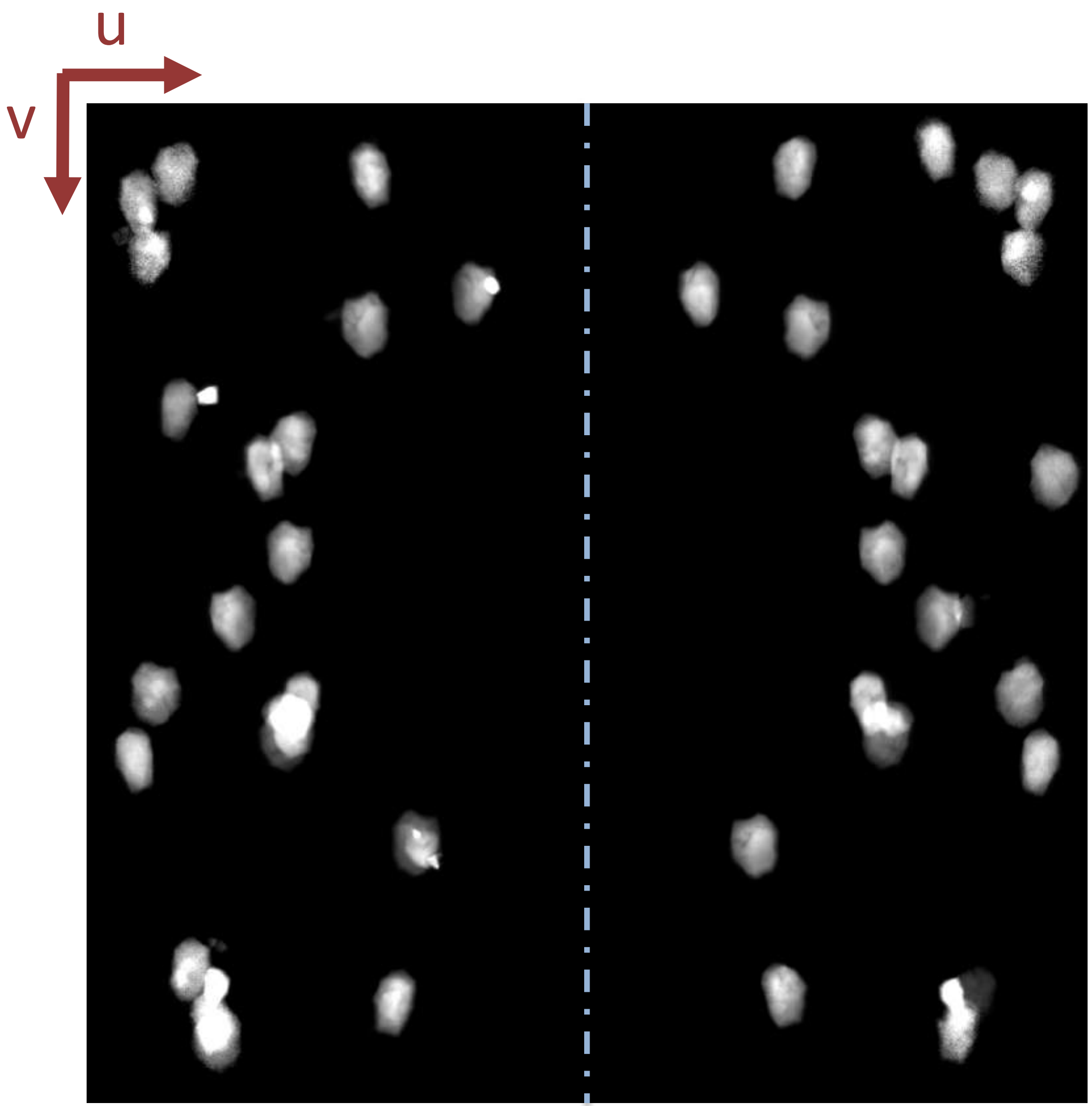
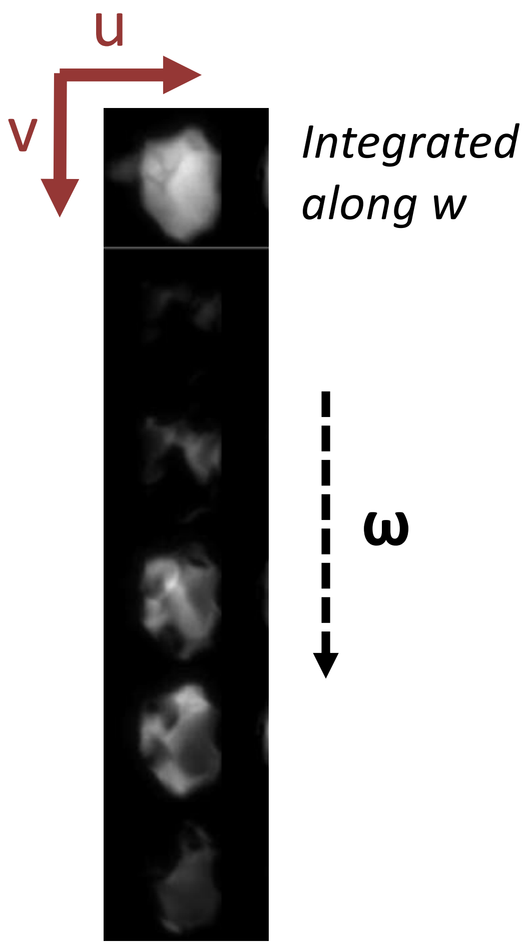
Combination of (hkl)-s reflections



Composite frame of all indexed and summed (hkl) diffraction spots of Grain #1



3D (u,v,w) diffraction spot

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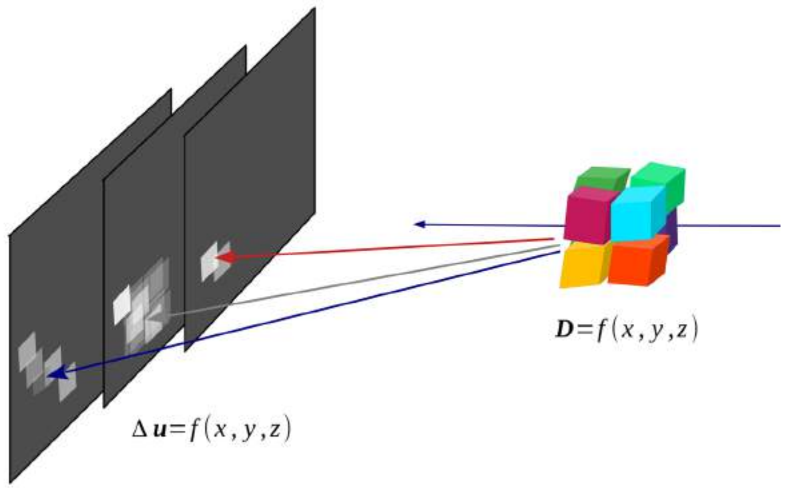
Diffraction model &
Iterative Tensor Field Reconstruction

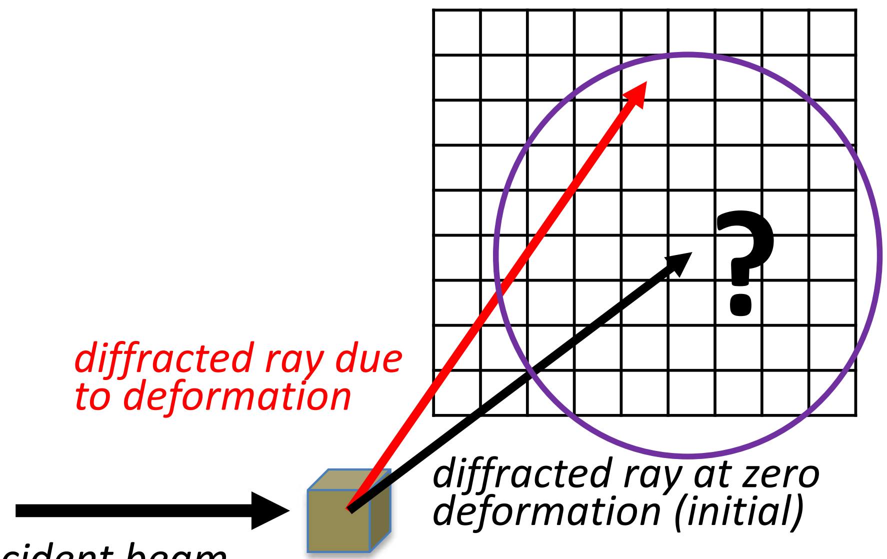
Model:

- kinematical model: intensities add up
- grain-by-grain
- seeking:
  - 3D shape
  - 9D deformation field:
    - 3 misorientation
    - 6 strain
- ray tracing from voxellated volume
- solving a single load step



Recontruction challenge:

- projection of a voxel moves across several pixels
- projection geometry unknown
  → large non-linear problem
- underdetermined (ill-posed)



Solver:

- iterative non-linear
- locally linearised large scale optimisation
- smooth deformations

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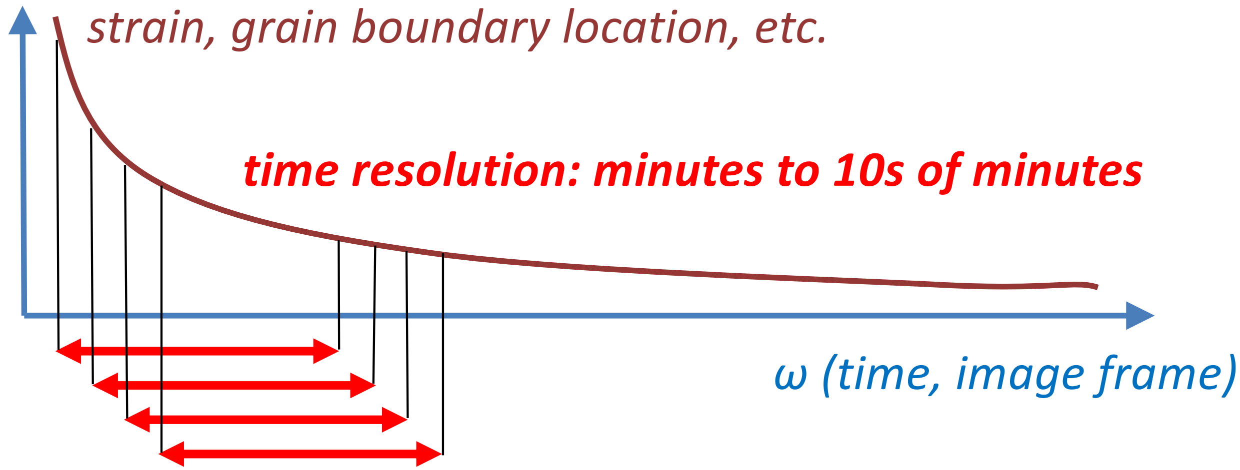
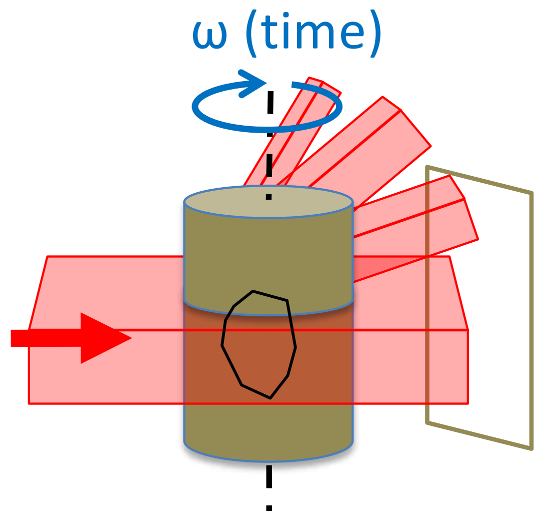
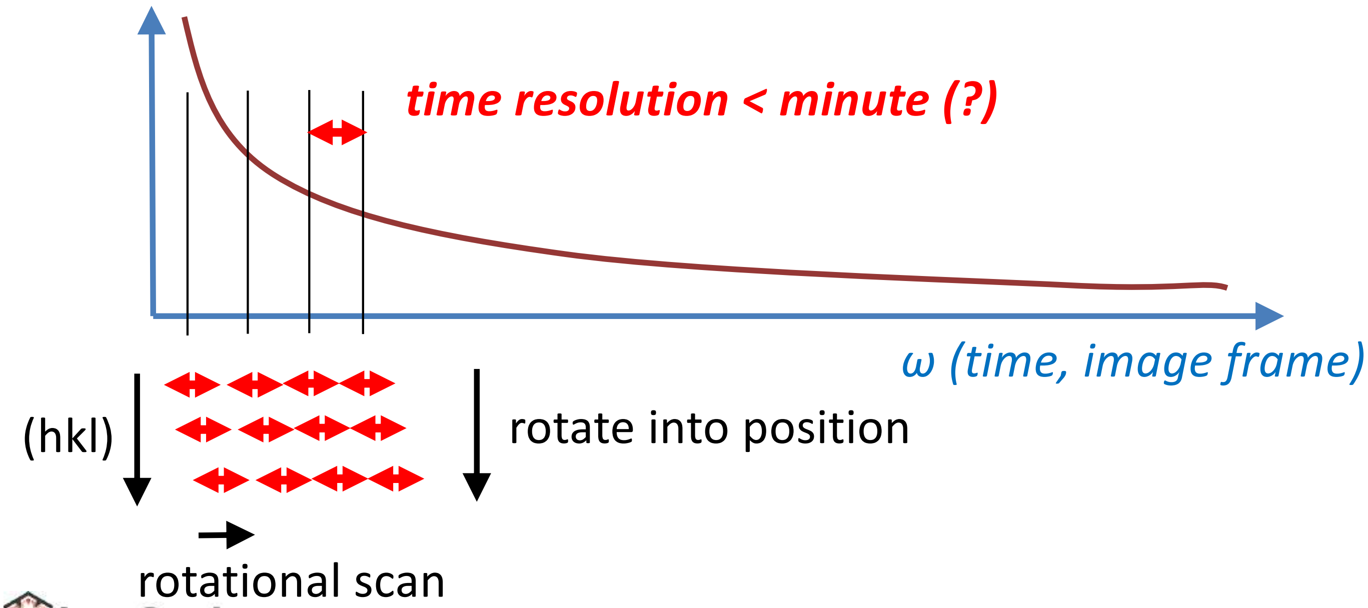
4D scan – Time resolution via sliding window

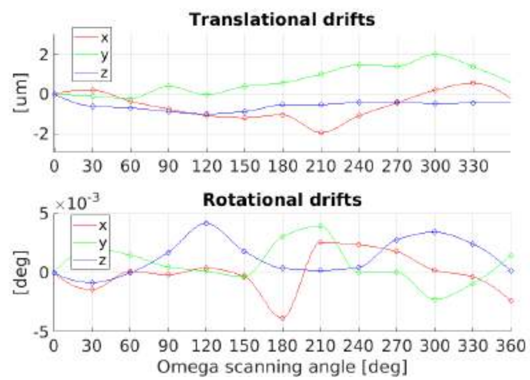
Full volume scans





Single grain scan

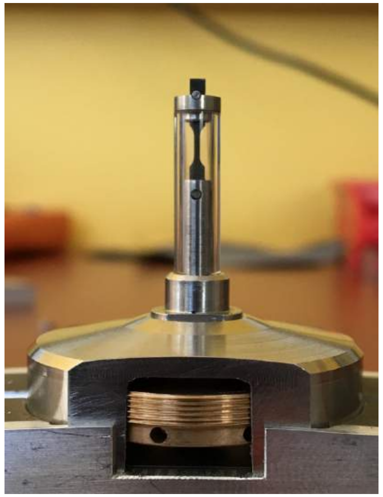


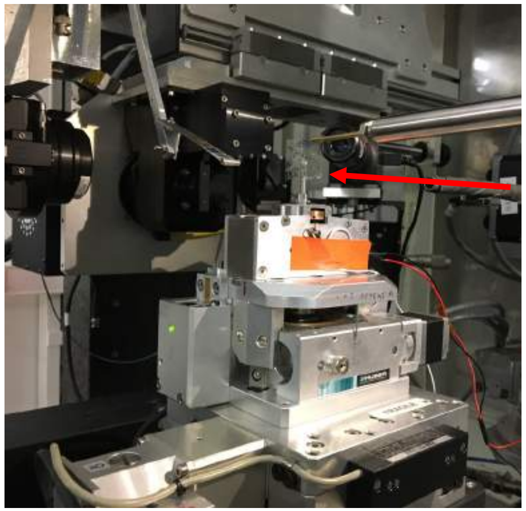


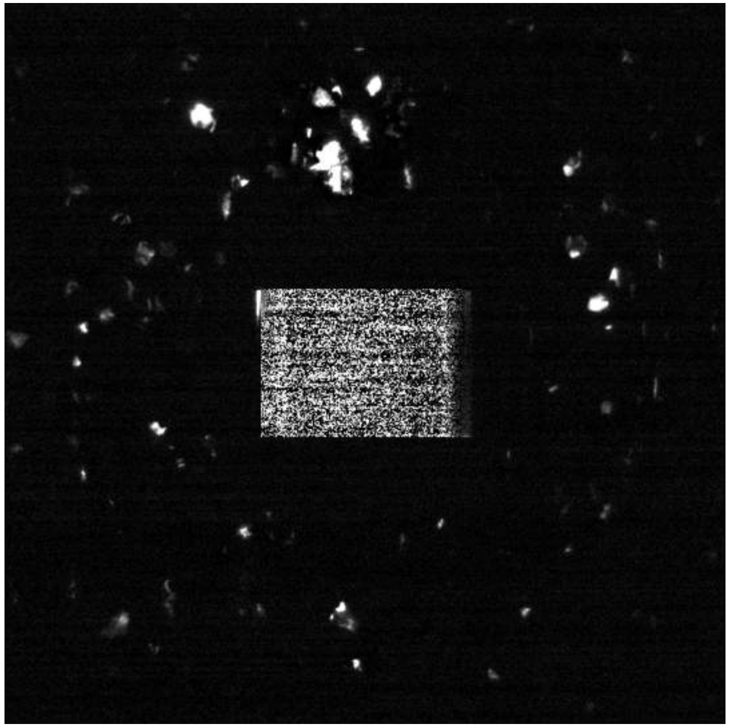
Sycnhrotron experiment on Gum metal

Experiment: Gum Metal under tensile strain

- material: Gum Metal (Ti - 36Nb - 2Ta - 3Zr - 0.3O wt%)
  sustains elastic strains up to 1.5 .. 2 %
- sample: ~0.6 mm thick dog bone; ~1500 grains in gauge volume
- beamline: ID11 ESRF
- beam: 40 keV, monochromatic bandwidth dE/E = 10^{-3}
- ω rotation: continuous, gap-free, 0.05 ° steps, 7200 images
- exposure time: 1.5 sec / frame *Now with brand new ESRF EBS source: 20x faster !*
- pixel size: 1.4 um
- distance: 7 mm
- load levels: 1) 33 Mpa
  2) 365 Mpa - Presented







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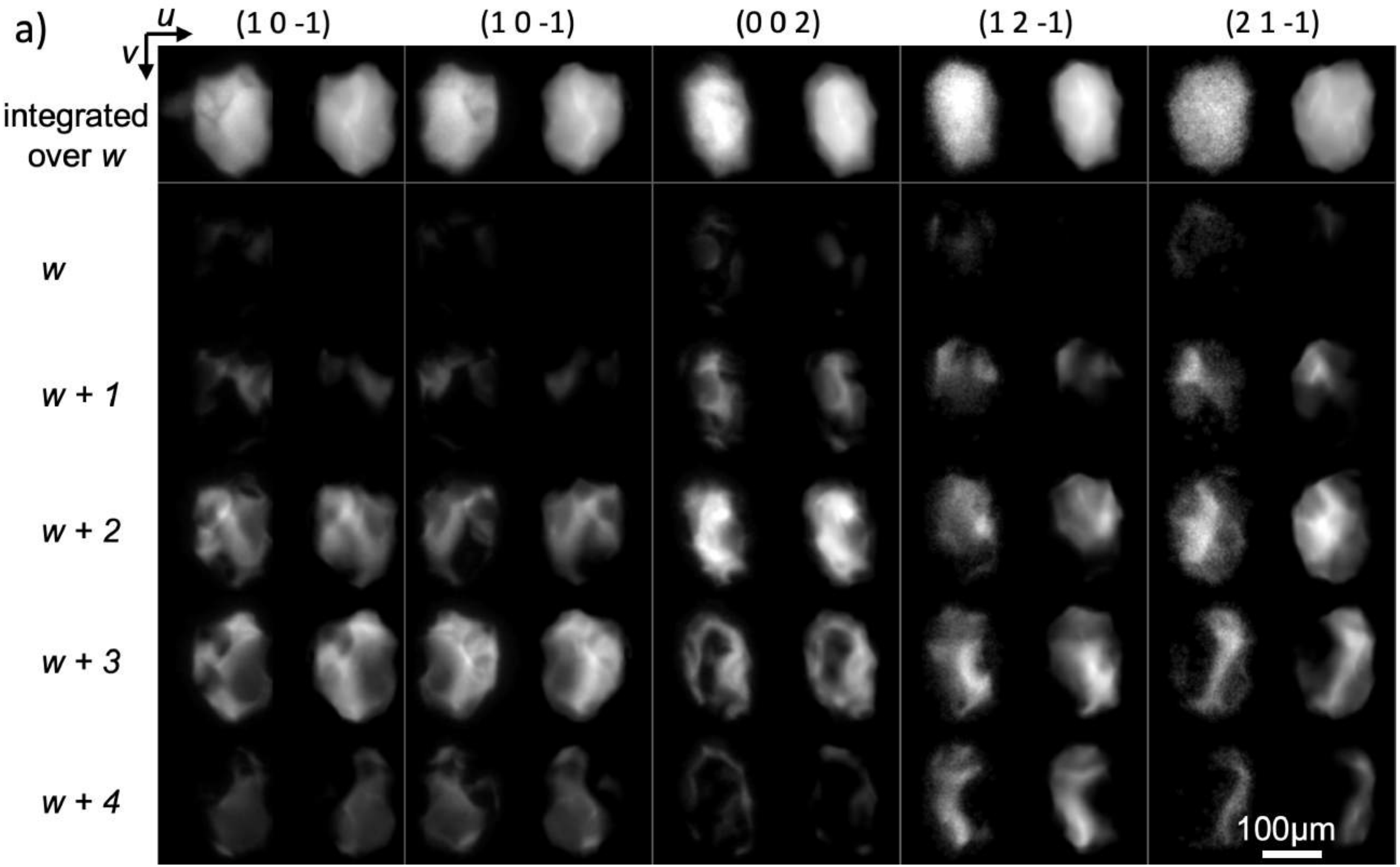
Simulated vs observed diffraction spots

Gum metal at high load (365 MPa): 1500 grains in grain map



Left: Measured   Right: Simulated

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Simulated vs observed diffraction spots

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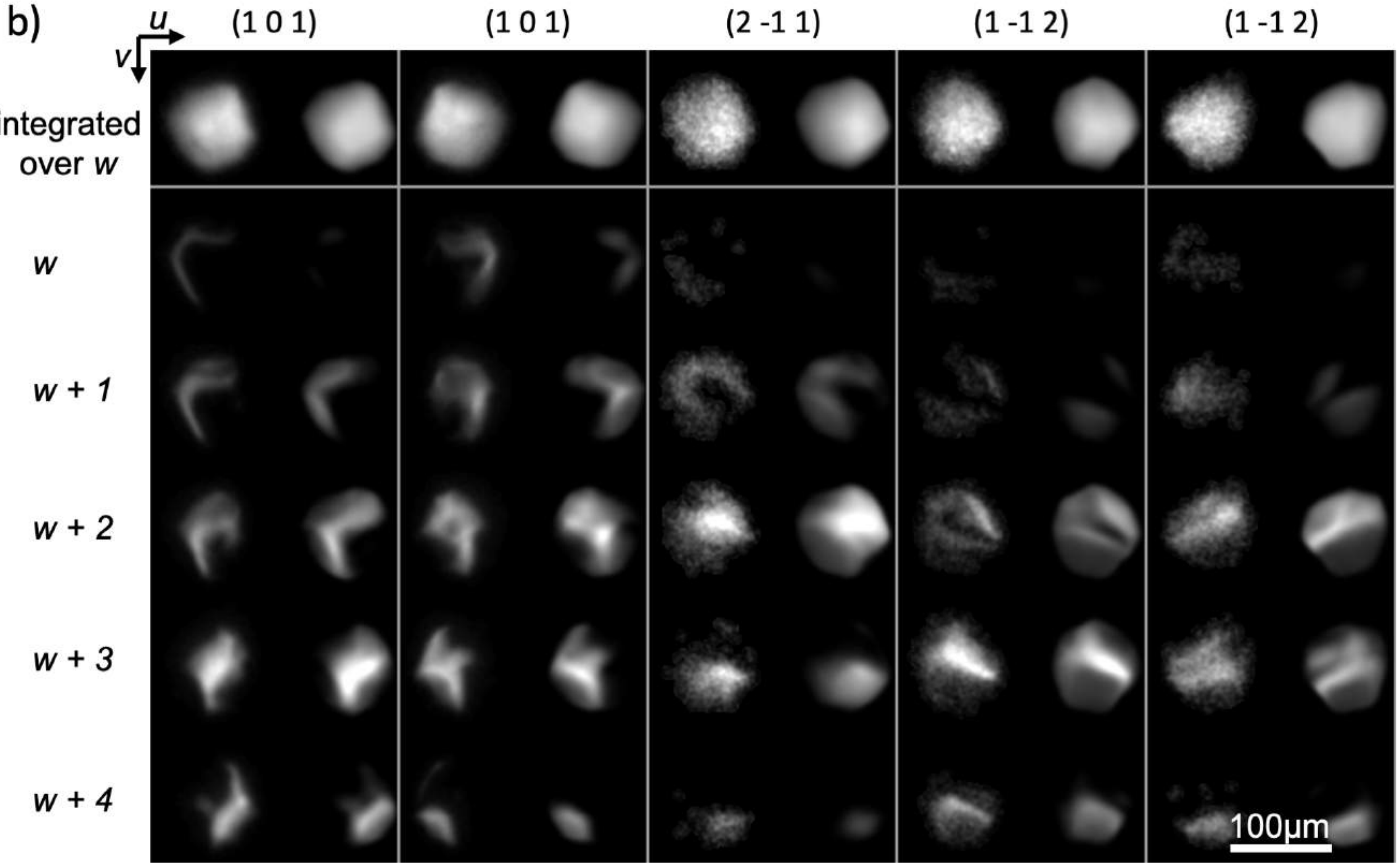

Left: Measured *Right: Simulated*

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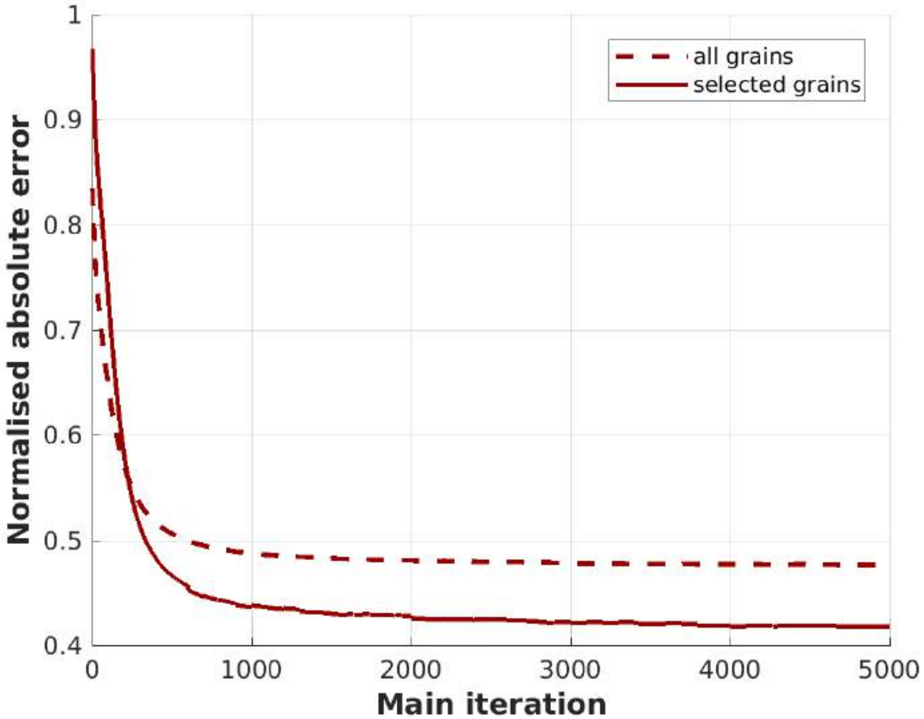
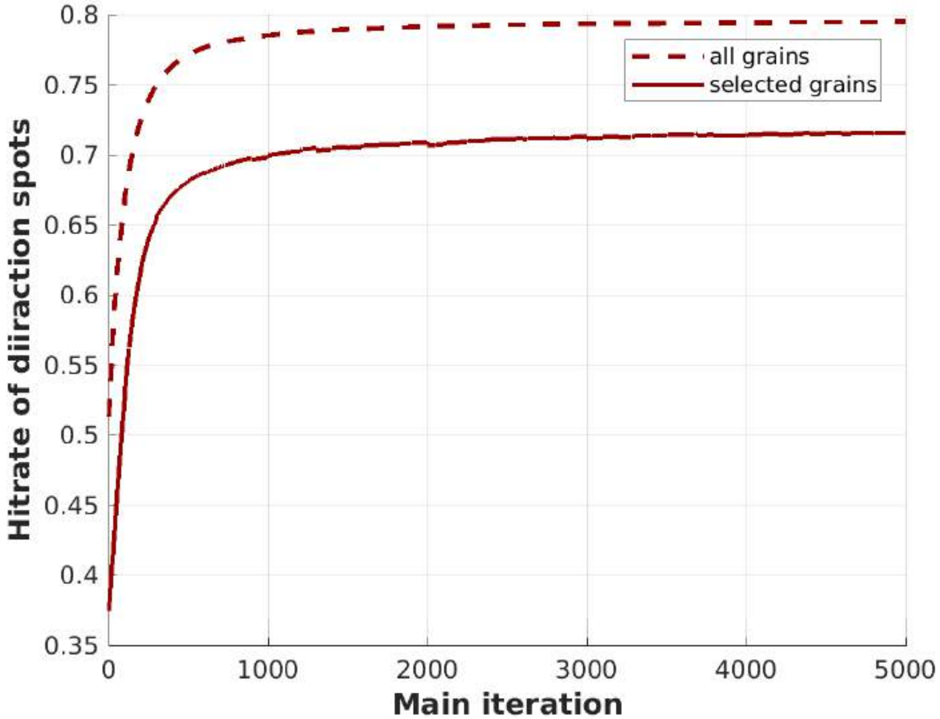
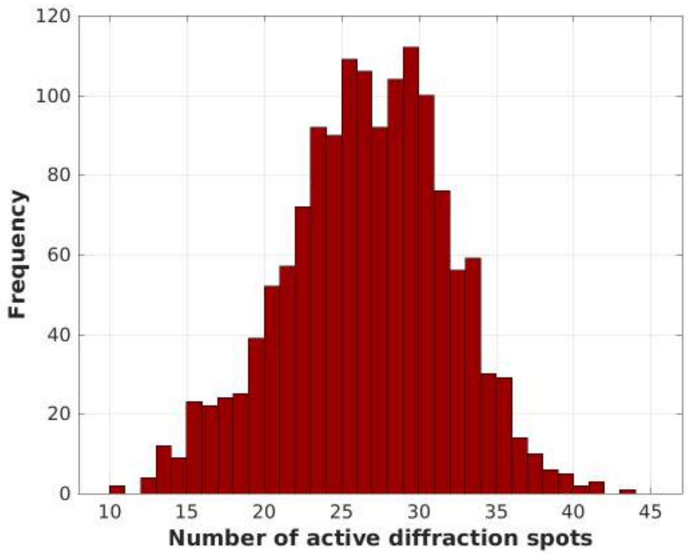
Deformation solver – Convergence (high load)







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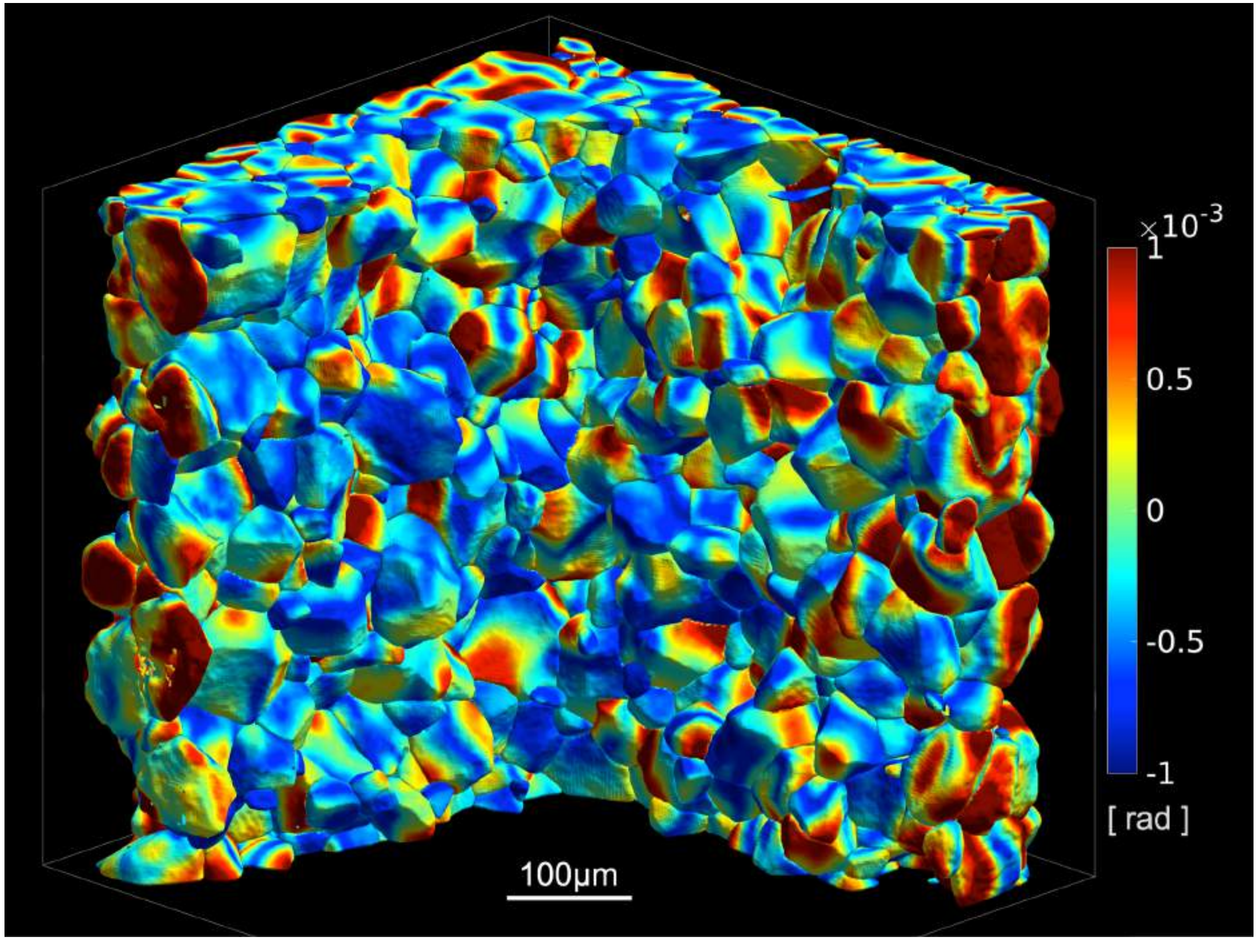
Sub-grain misorientation

- Orientation: 3-component Rodrigues vector or 3 Euler angles
- Misorientation angle: scalar

Misorientation from grain mean



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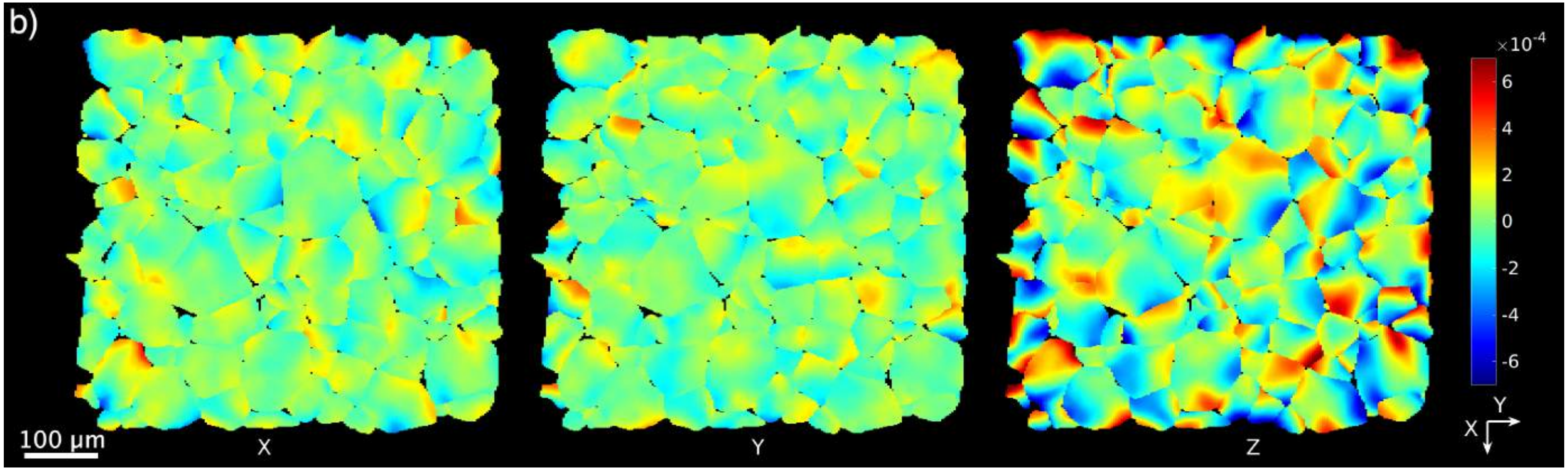
Sub-grain misorientation

Rodrigues vector components deviation from the grain mean



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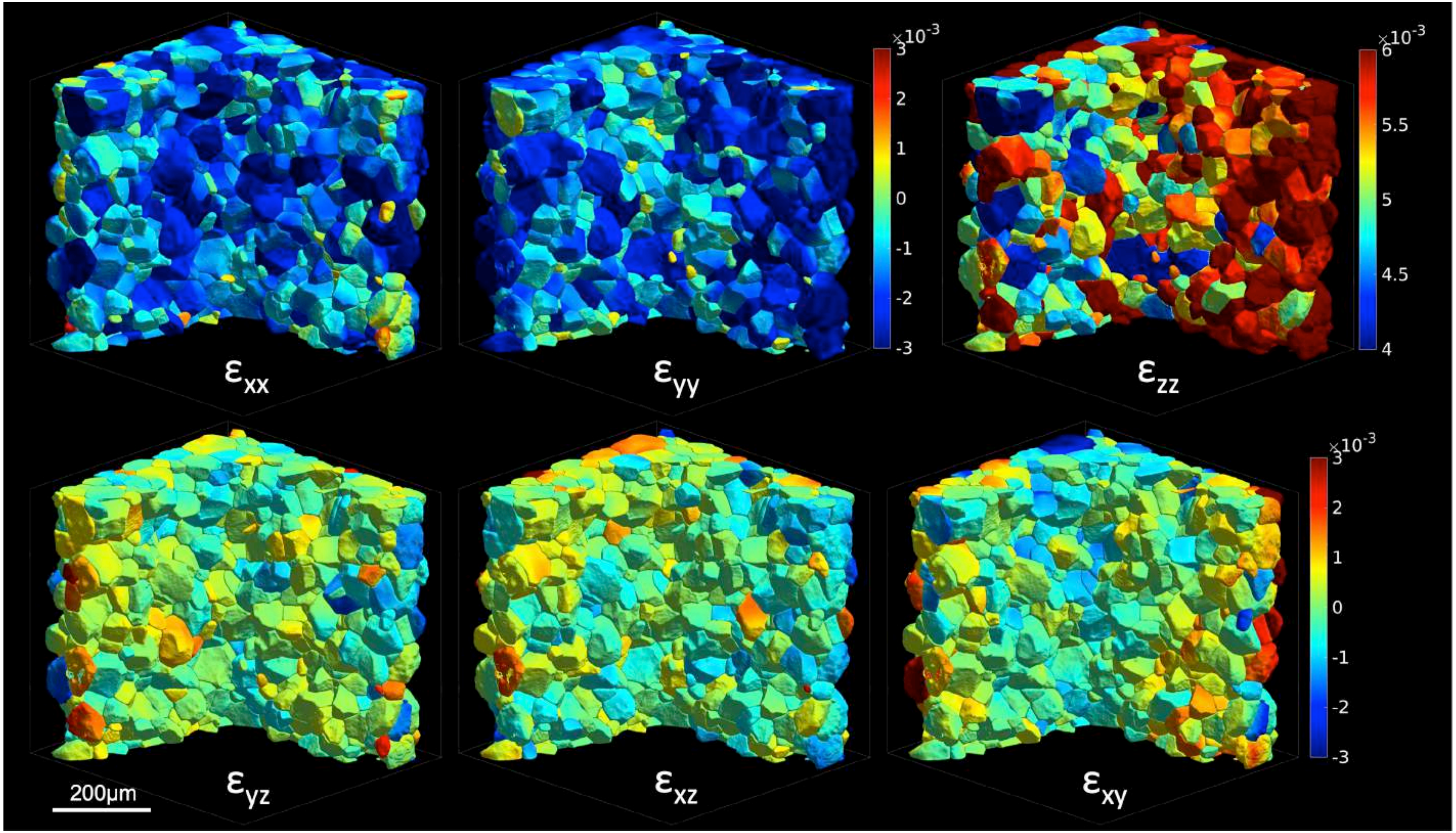
Sub-grain strain maps

Strain tensor components in the Sample reference



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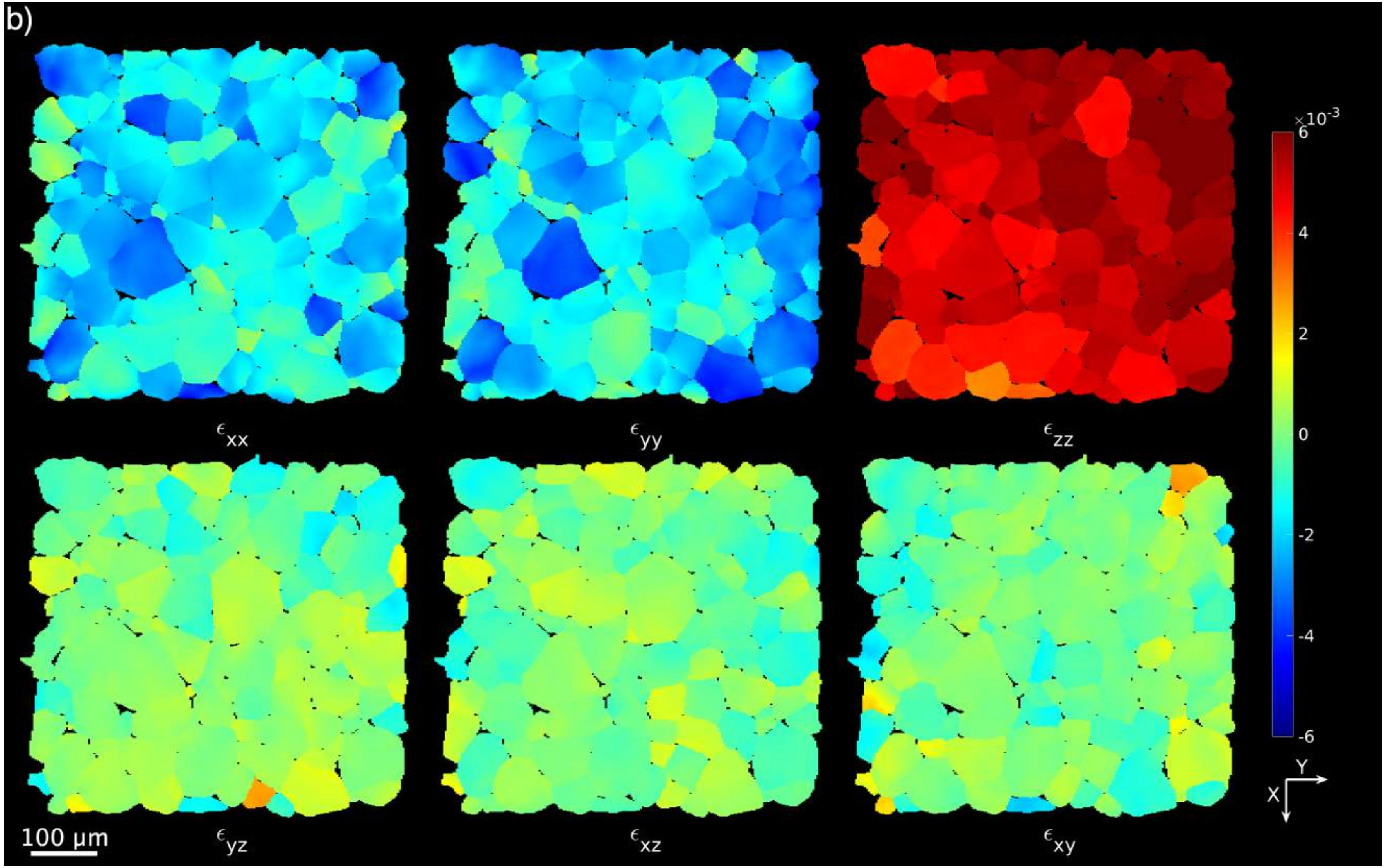
Sub-grain strain maps

Strain tensor components in the Sample reference



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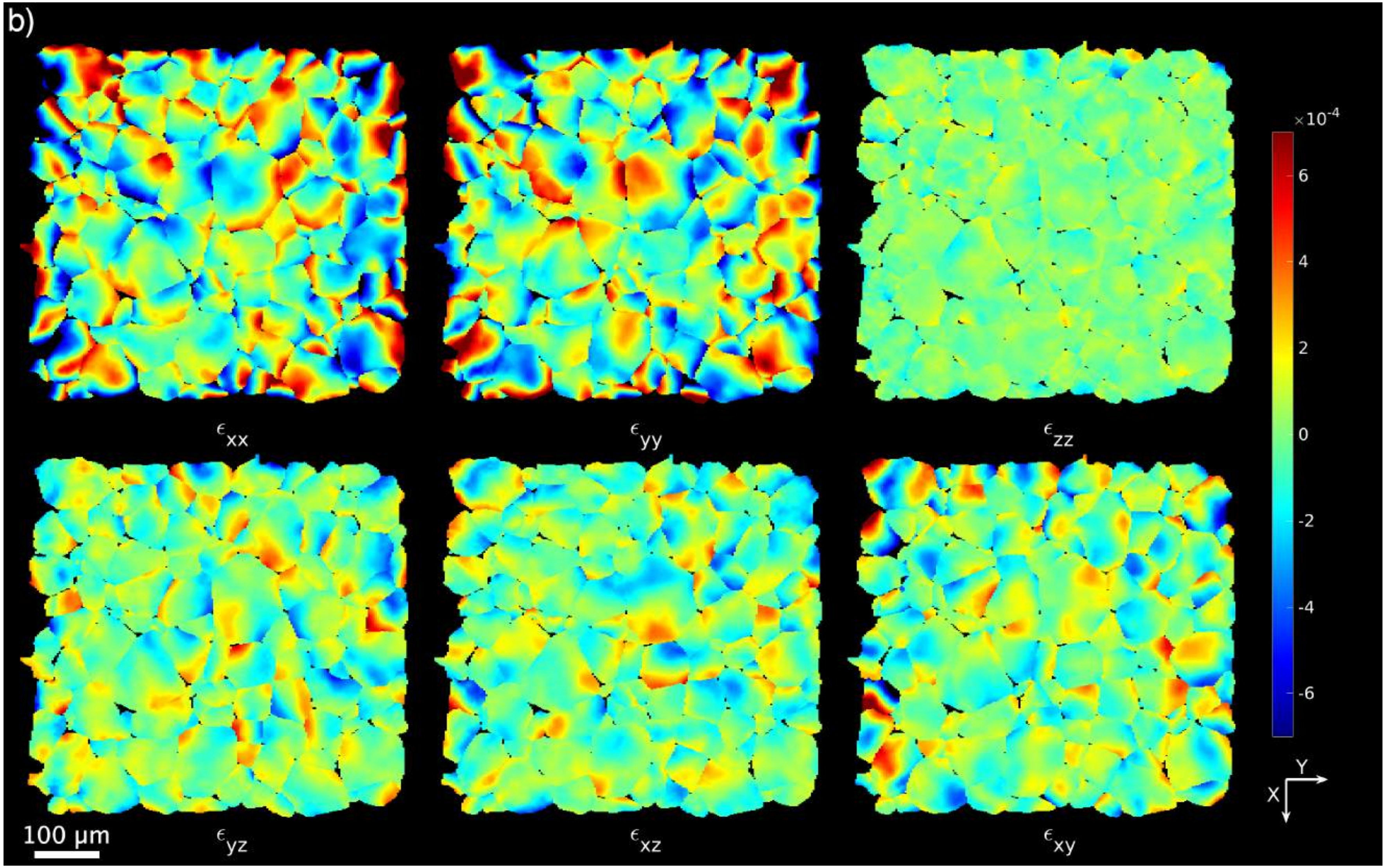
Sub-grain strain maps

Strain versus grain average



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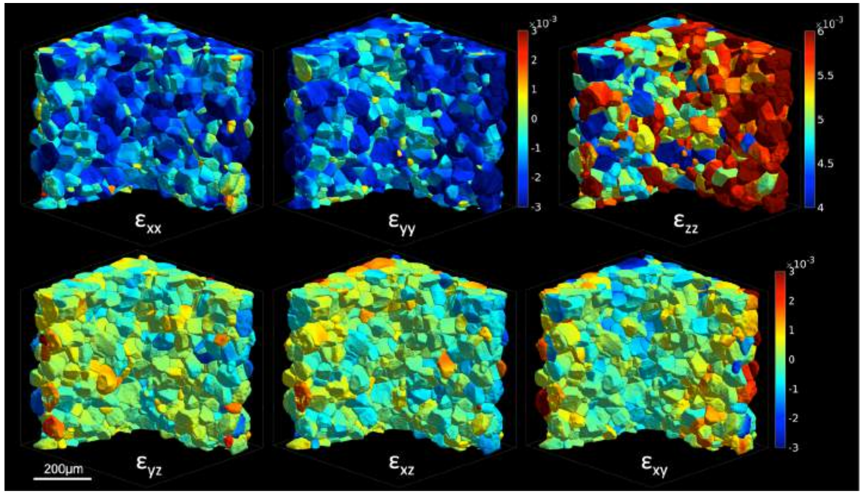
Fitting of single crystal elastic constants from DCT or 3DXRD data

Fitting of elastic constants from strain maps

Conclusions

- DCT: simple setup but challenging reconstruction
- efficient mapping of polycrystals at sub-grain level:
  - (mis)orientation & strain in the bulk in 3D
  - potential for 4D time resolved scans
  - non-destructive, in-situ
  - spatial resolution: 1…5 um
  - orientation & strain resolution: possibly 1…5 x 10^{-4}
  - scanning times: minutes to hours
- **direct comparison to digital twins in modelling**
- can provide the single crystal elastic moduli
- can handle slow sample or energy drifts
- best adapted to:
  - coarse grains (> 10 um)
  - limited deformation (few %)



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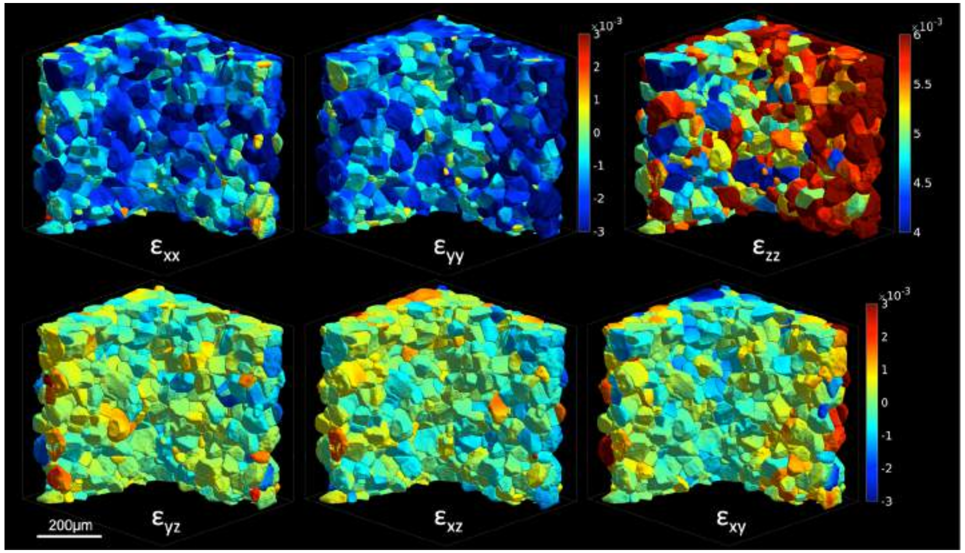

3D reconstruction of intragranular strain and orientation in polycrystals by near-field X-ray diffraction
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