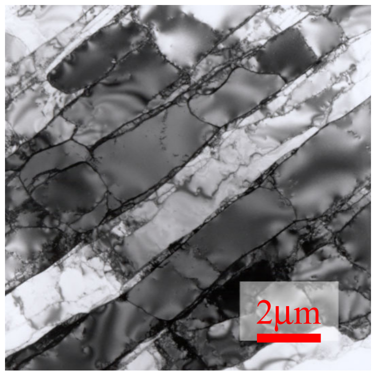
Wolfgang Pantleon
Technical University of Denmark

In-situ monitoring of evolving deformation structures by high-resolution reciprocal space mapping

Deformation structures by EM

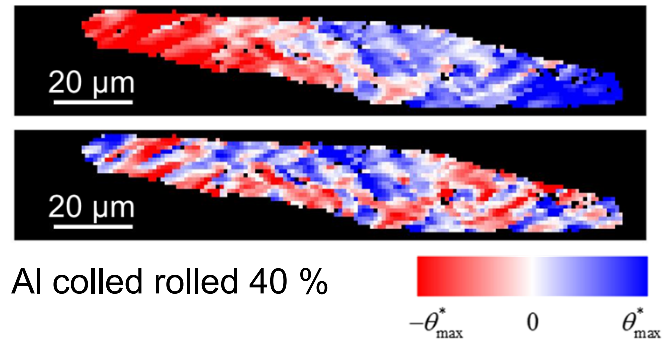
- TEM



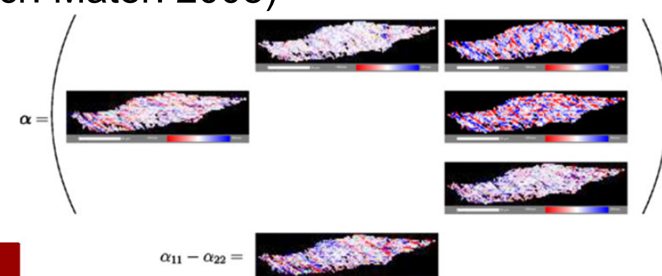
- Observation of dislocation structures

- Post-mortem investigations
- Specimen preparation artefacts

- SEM EBSD

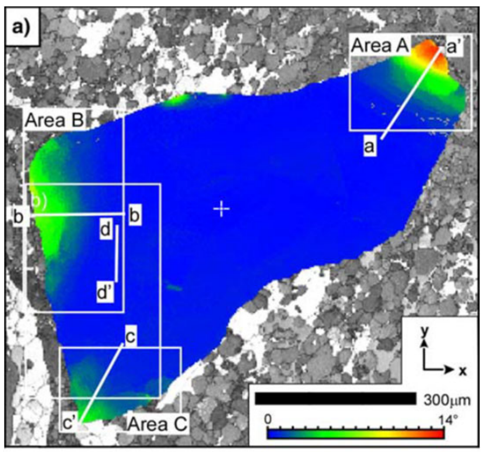


- Revealing orientation heterogeneities (Pantleon, Mater. Sci. Techn. 2005, Pantleon et al., Mater. Sci. Eng. A 2008)
- Resolving dislocation densities (Pantleon,. Scr. Mater. 2008)



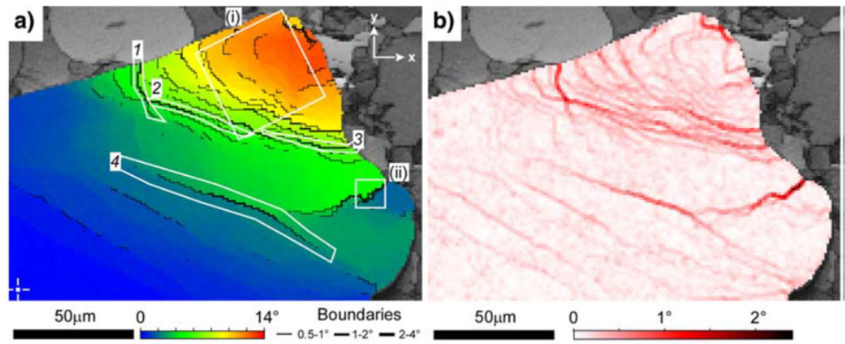
Resolving deformation structures in zircon

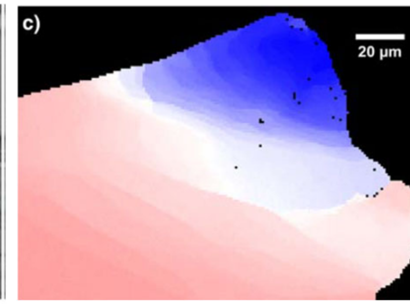
- Zircon from ocean drilling program
- Single zircon grain



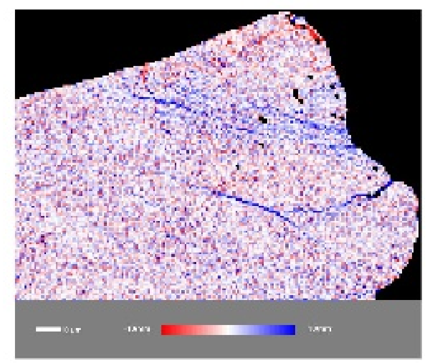
(Reddy, Timms, Pantleon, Trimby, Contrib. Mineral. Petrol. 2009)

- Orientation differences



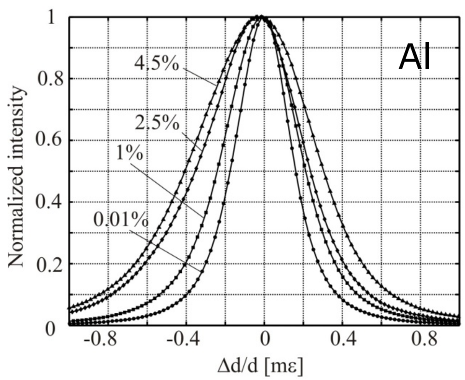


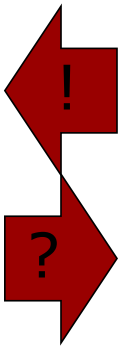
- Geometrically necessary dislocation density (Pantleon, Scr. mater. 2008)



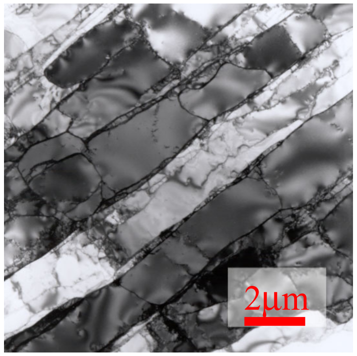
Deformation structures by XRD

- Traditional line profile analysis

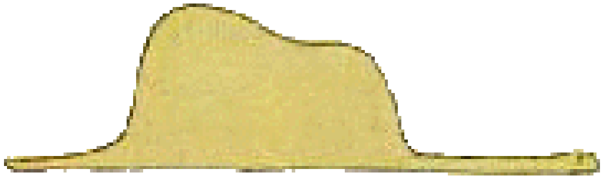




- Link to dislocation structures



- 'Le Petit Prince'
  (de Saint-Exupery 1943)



High-resolution reciprocal space mapping

B. Jakobsen, H.F. Poulsen, U. Lienert, W. Pantleon (2007)

High-resolution reciprocal space mapping

Reciprocal space maps
(peak shapes)
- of individual grains
- embedded in bulk
- in-situ during deformation

- high angular resolution
  0.004°=10″=$7 \cdot 10^{-5}$ rad

- no real space information
- high energy 50 keV

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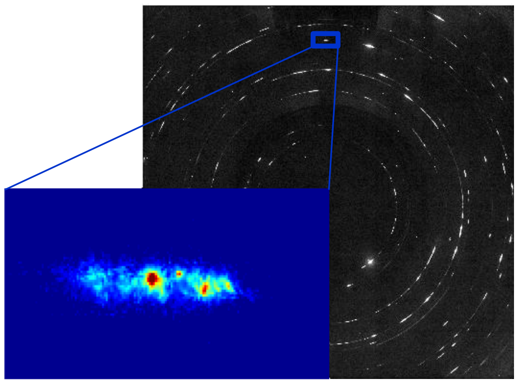
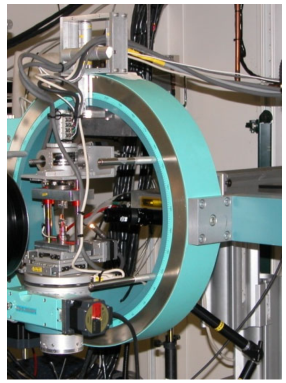
1-ID

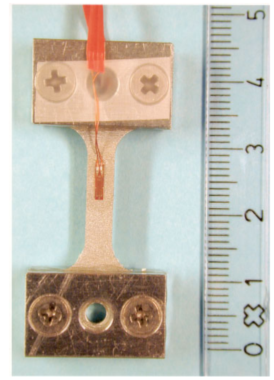
DESY

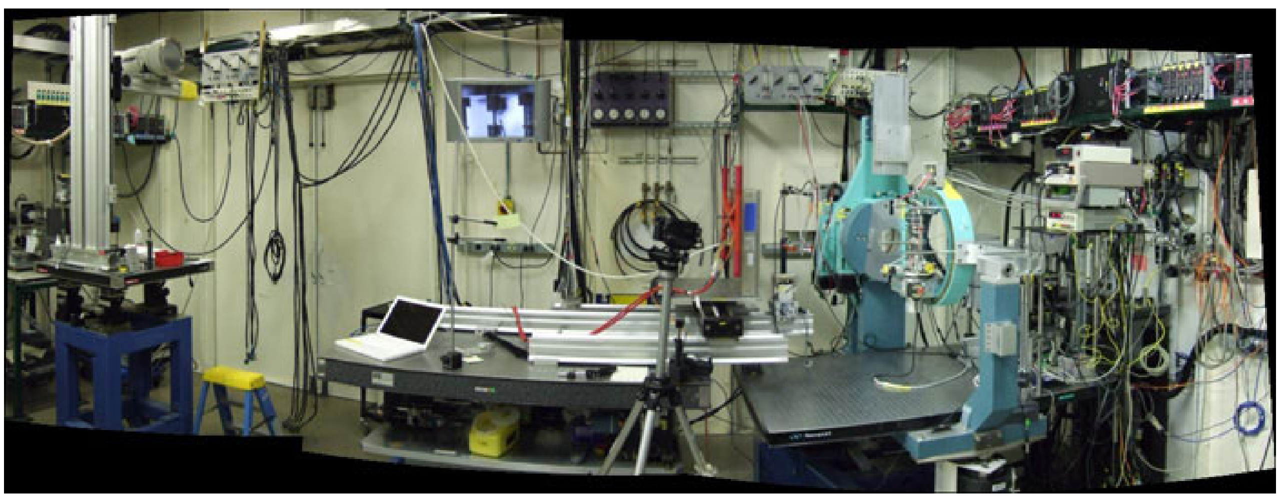
P07
P21

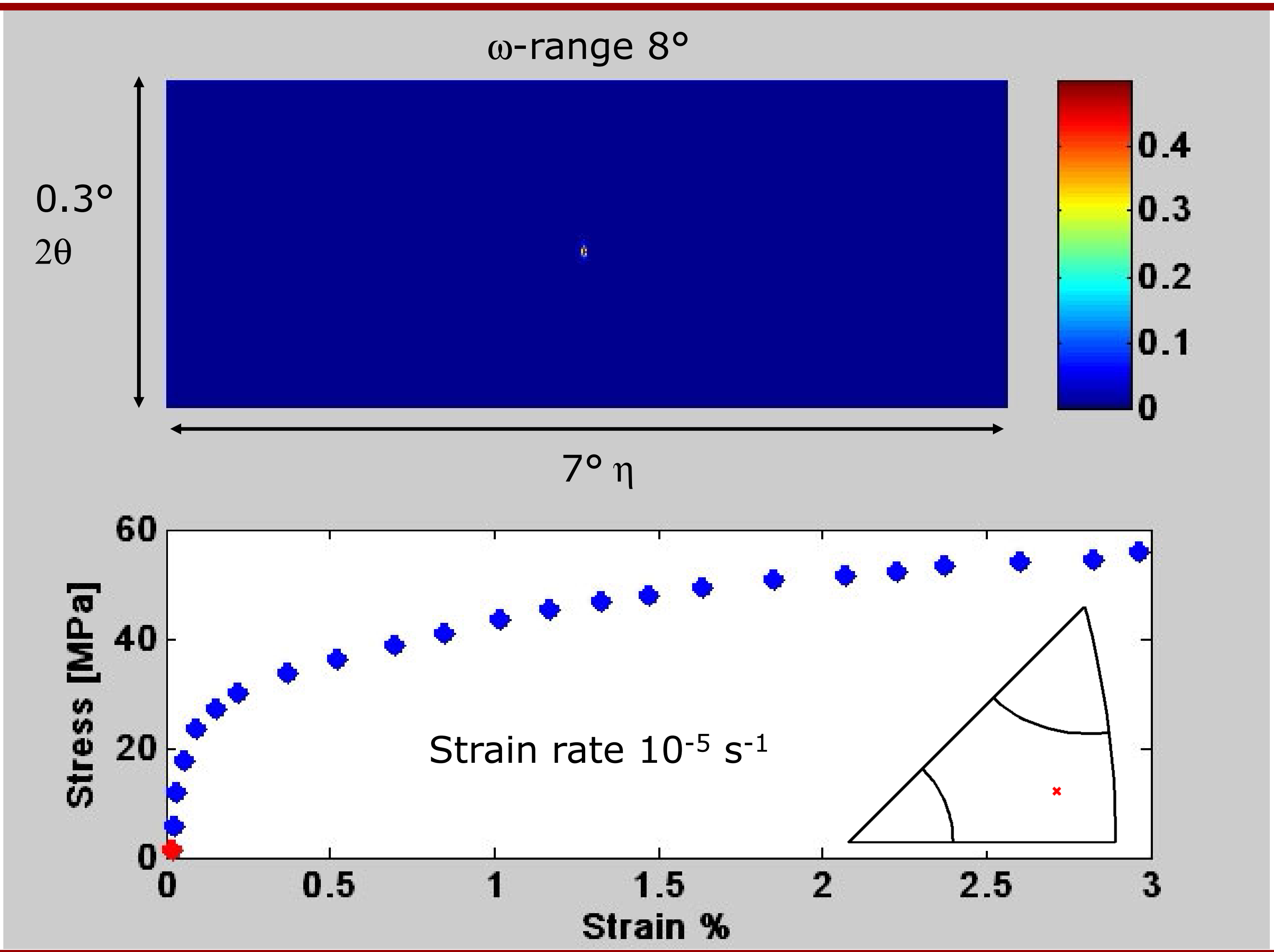
High-resolution reciprocal space mapping APS Beamline 1-ID



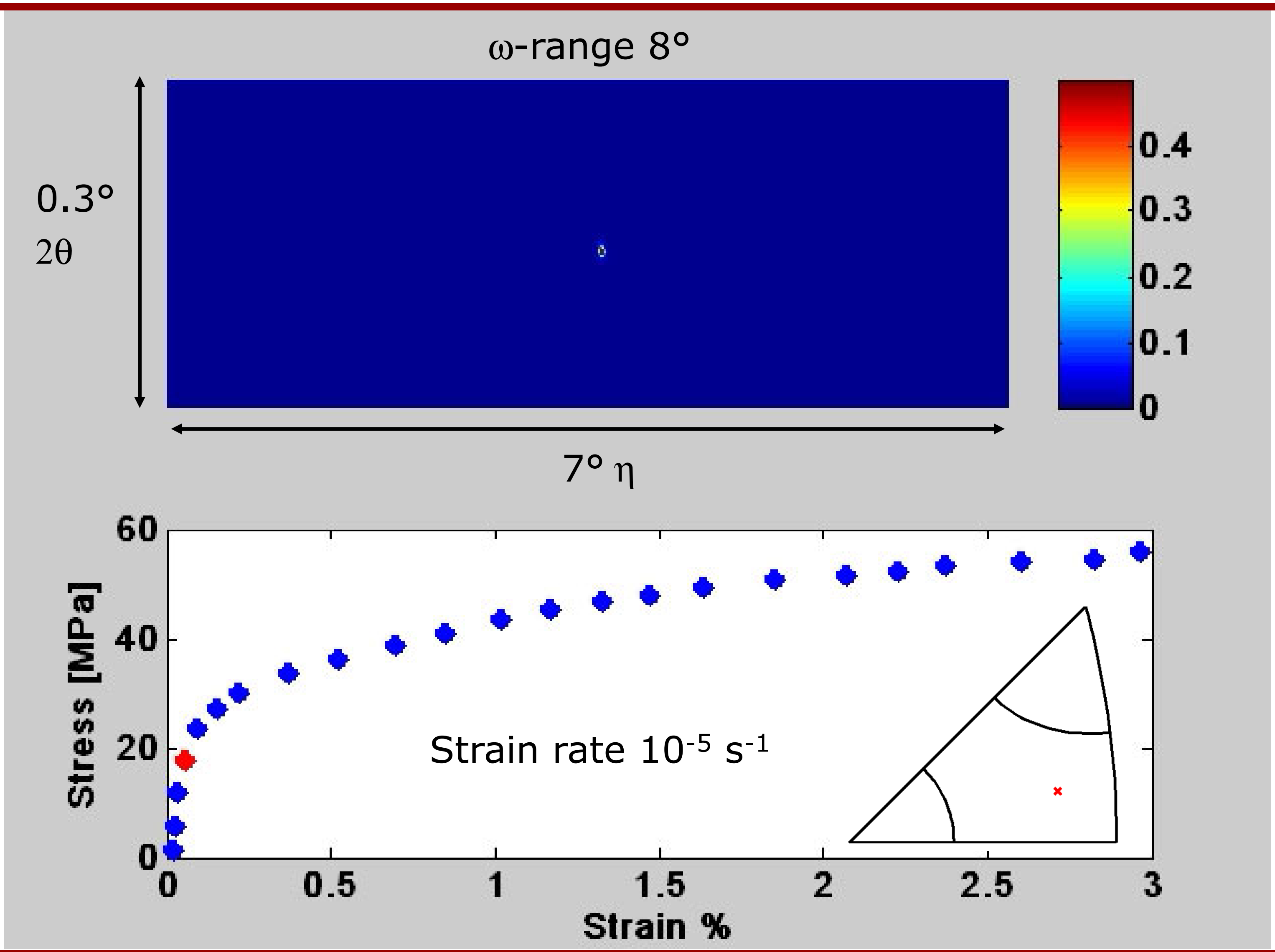








ω-range 8°
0.3°
2θ
7° η
0.4
0.3
0.2
0.1
0
60
40
20
0
Stress [MPa]
Strain rate 10-5 s-1
0
0.5
1
1.5
2
2.5
3
Strain %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7° η
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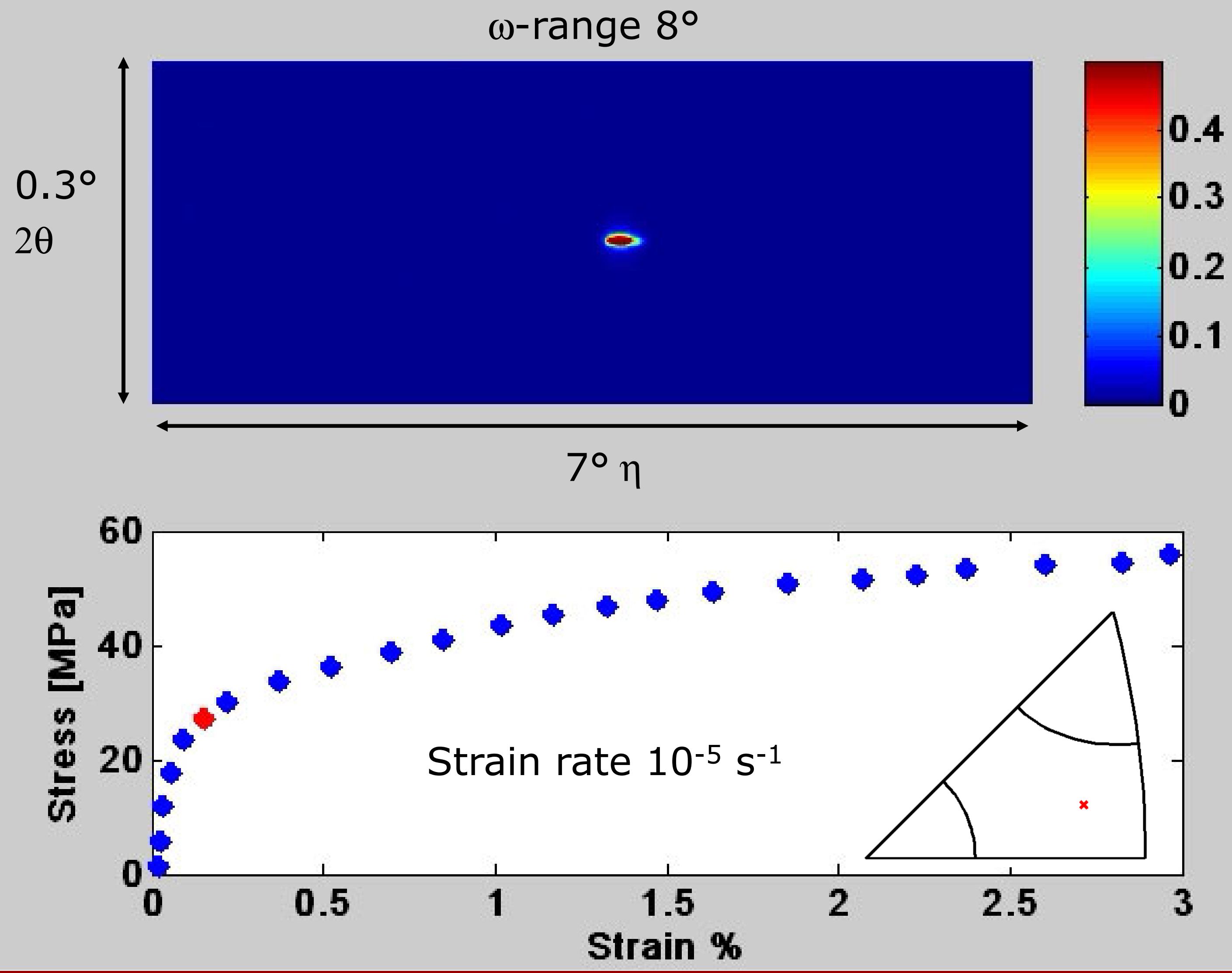
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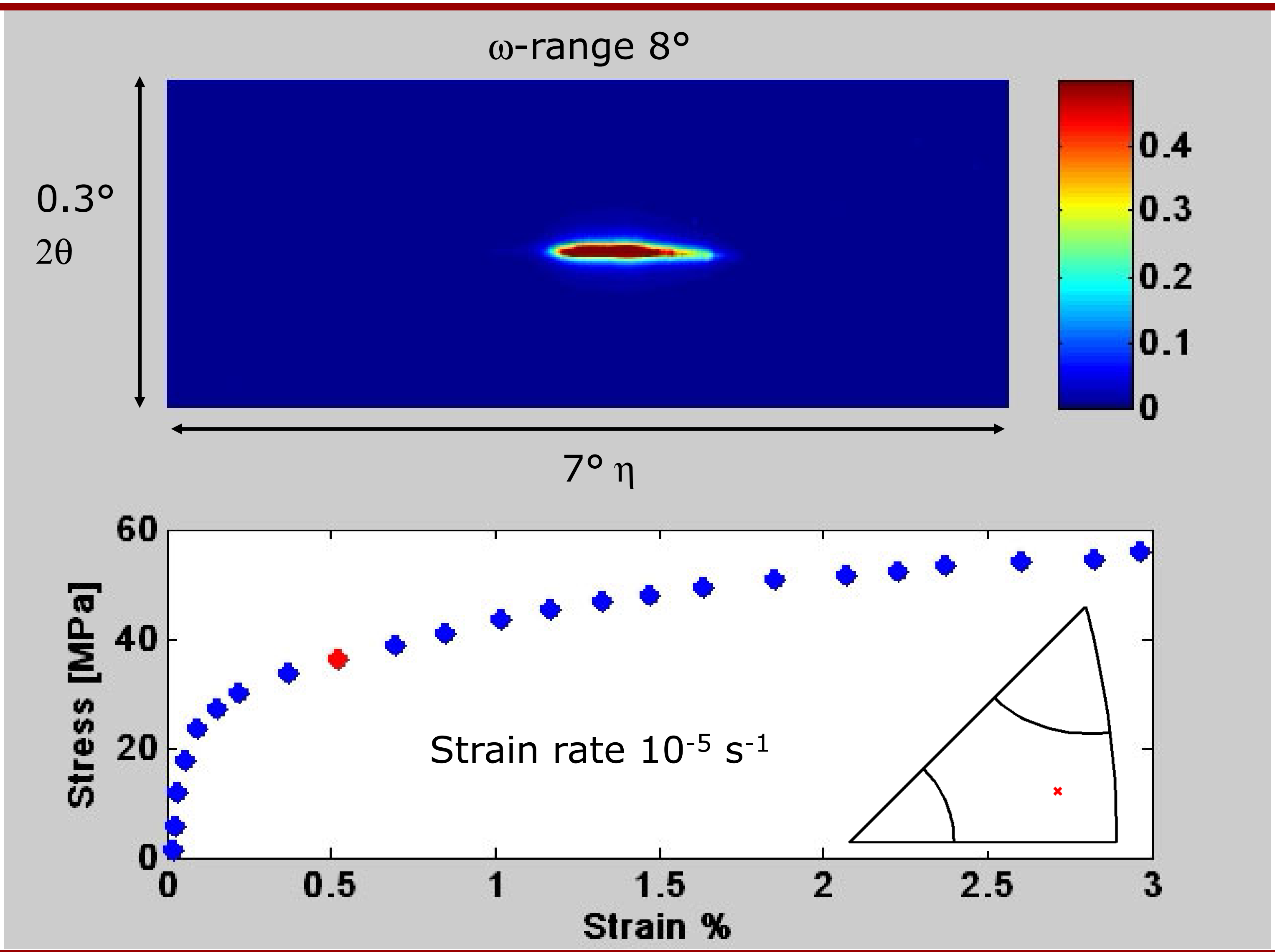
ω-range 8°
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Stress [MPa]
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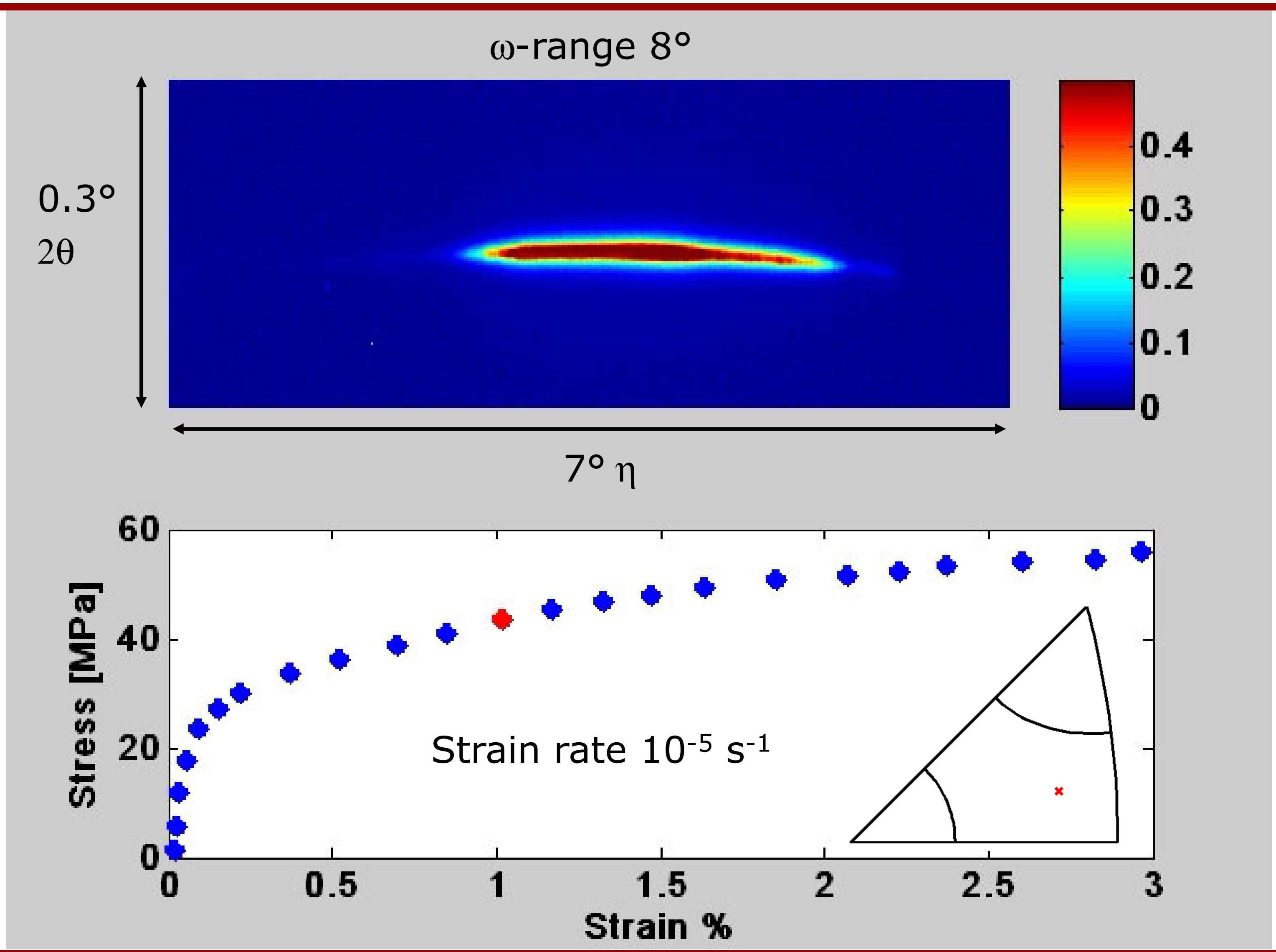

ω-range 8°
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Stress [MPa]
Strain %
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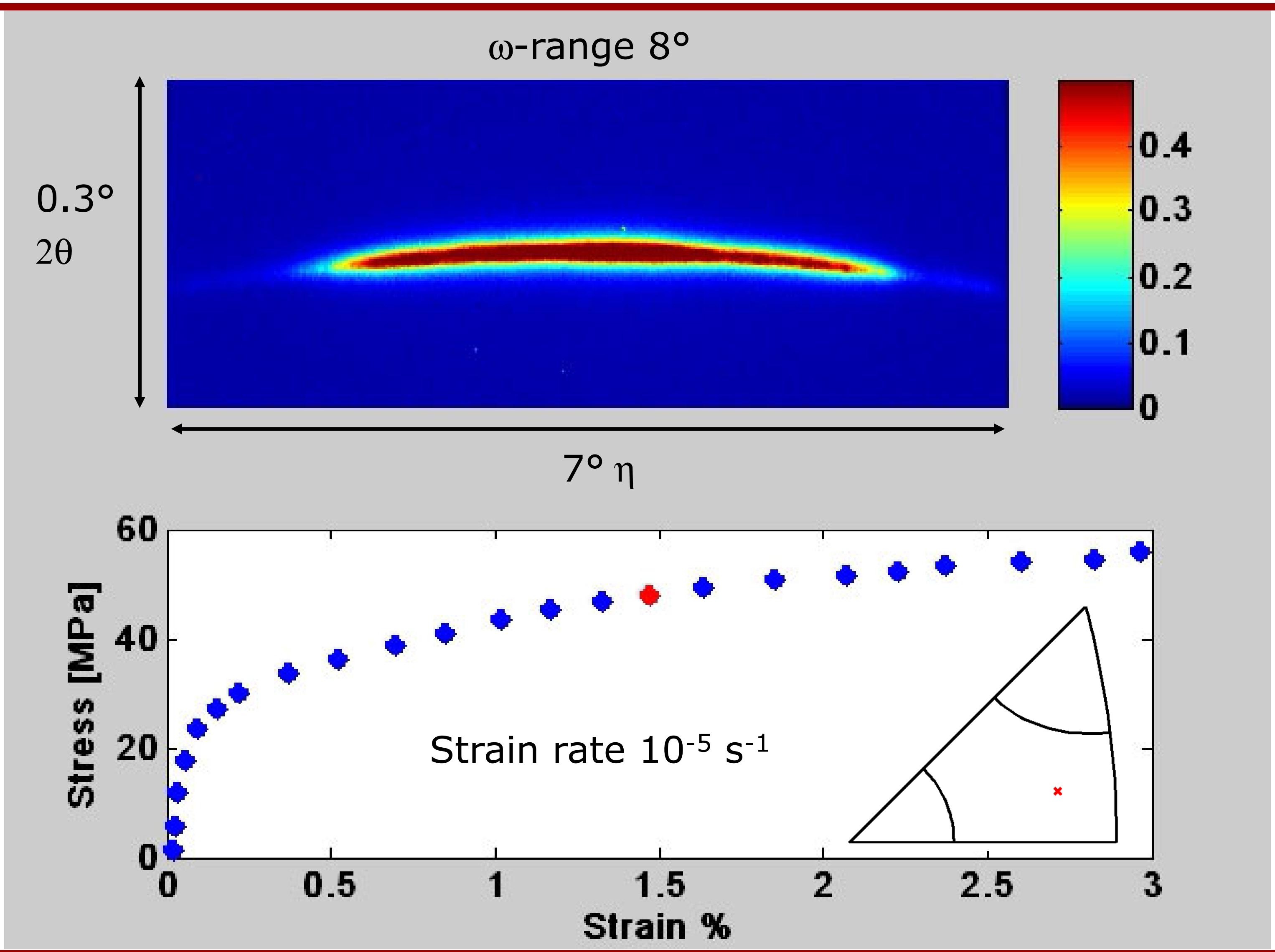
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Stress [MPa]
Strain %
Strain rate 10-5 s-1
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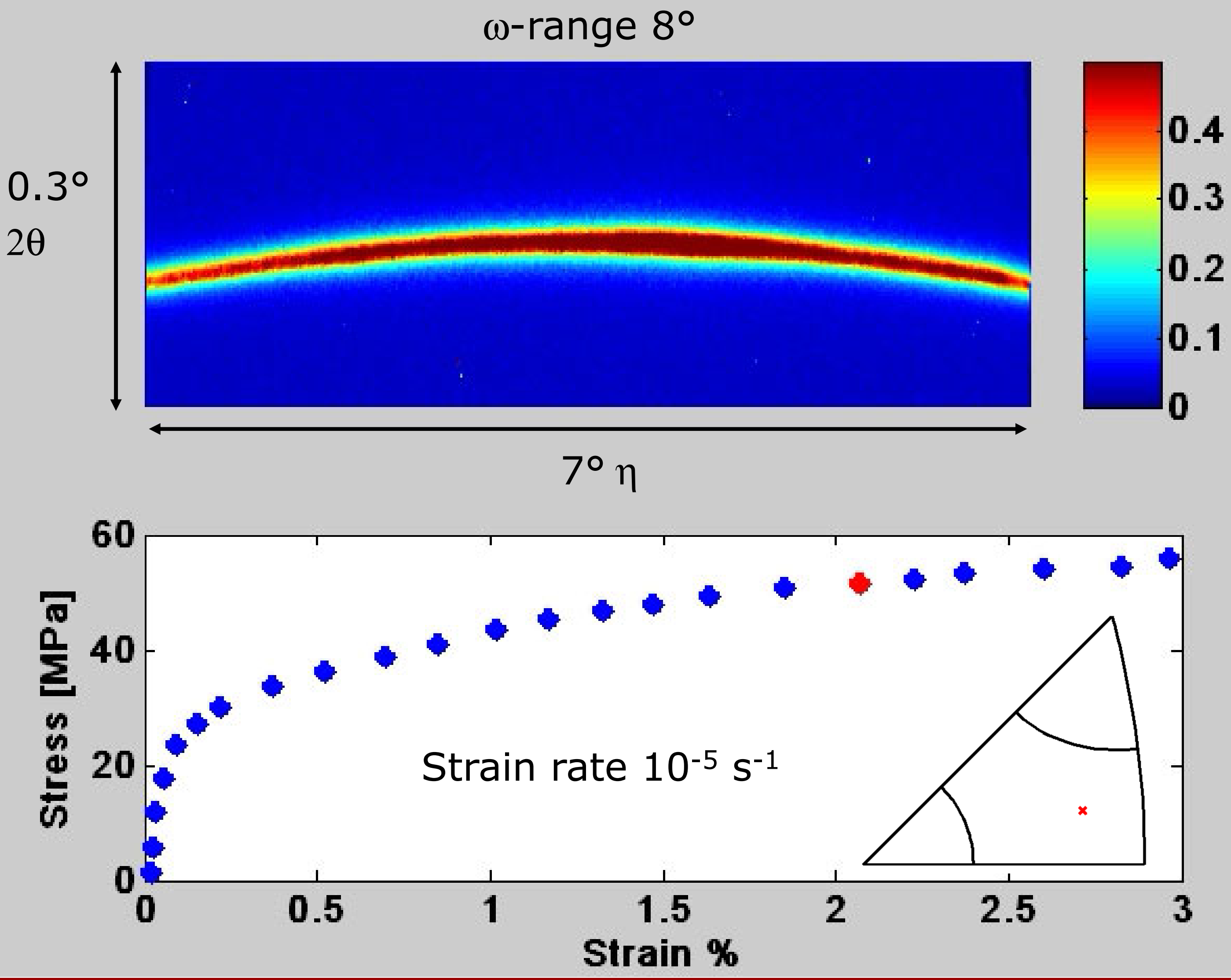
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20
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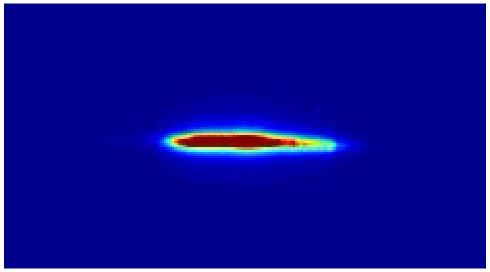
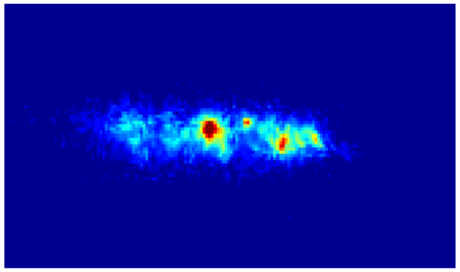
Stress [MPa]
Strain rate 10-5 s-1
0
0.5
1
1.5
2
2.5
3
Strain %

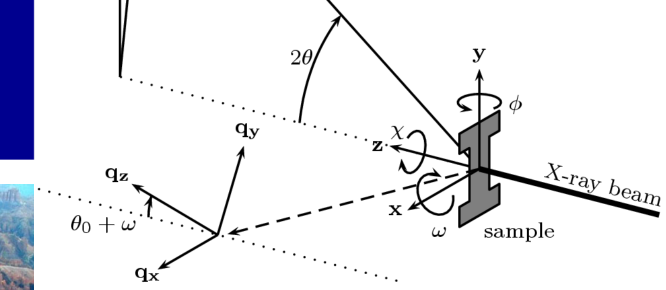
High-resolution reciprocal space mapping

In-situ monitoring

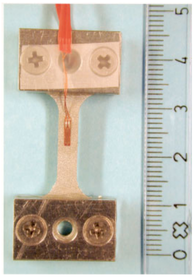
- of individual reflections
- during varying loads



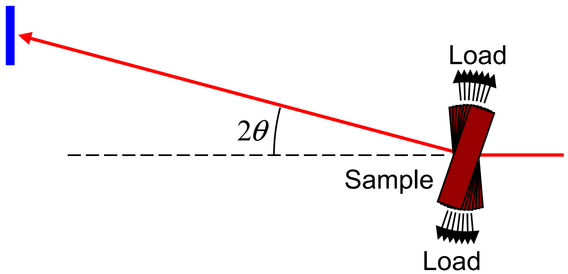


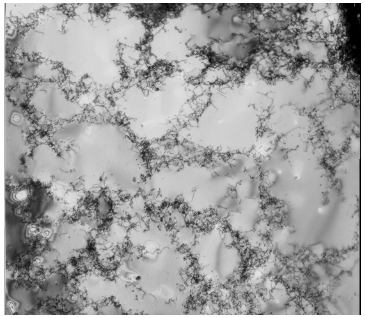


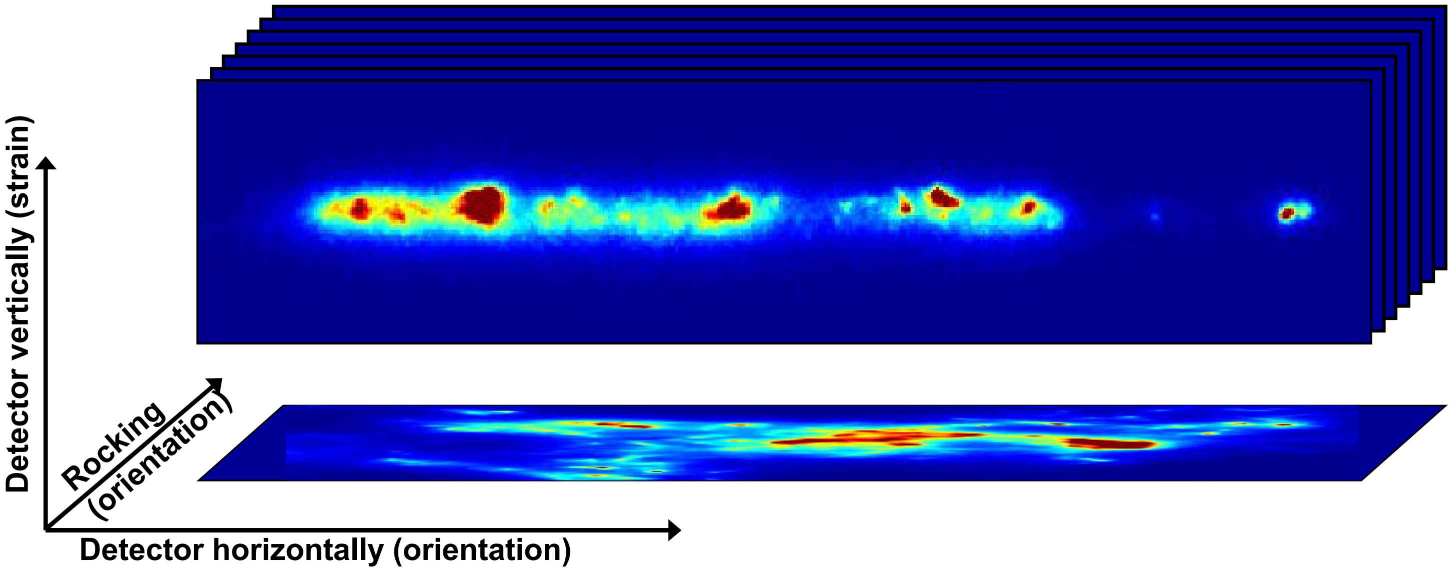
thickness 300 µm



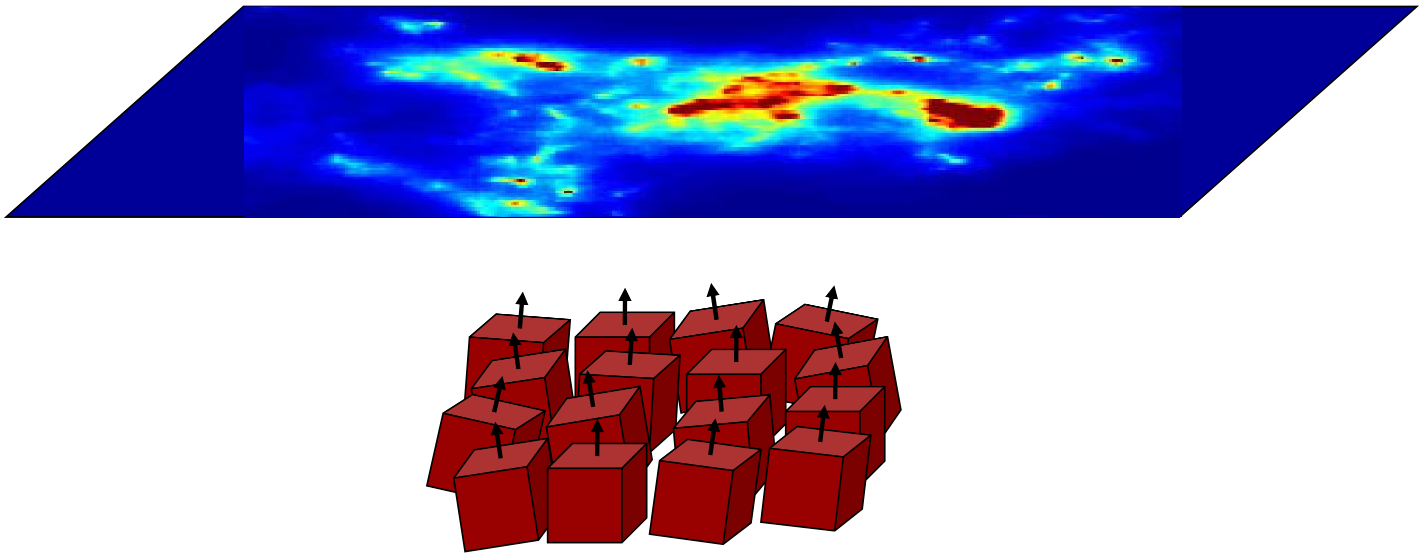
3D reciprocal space maps





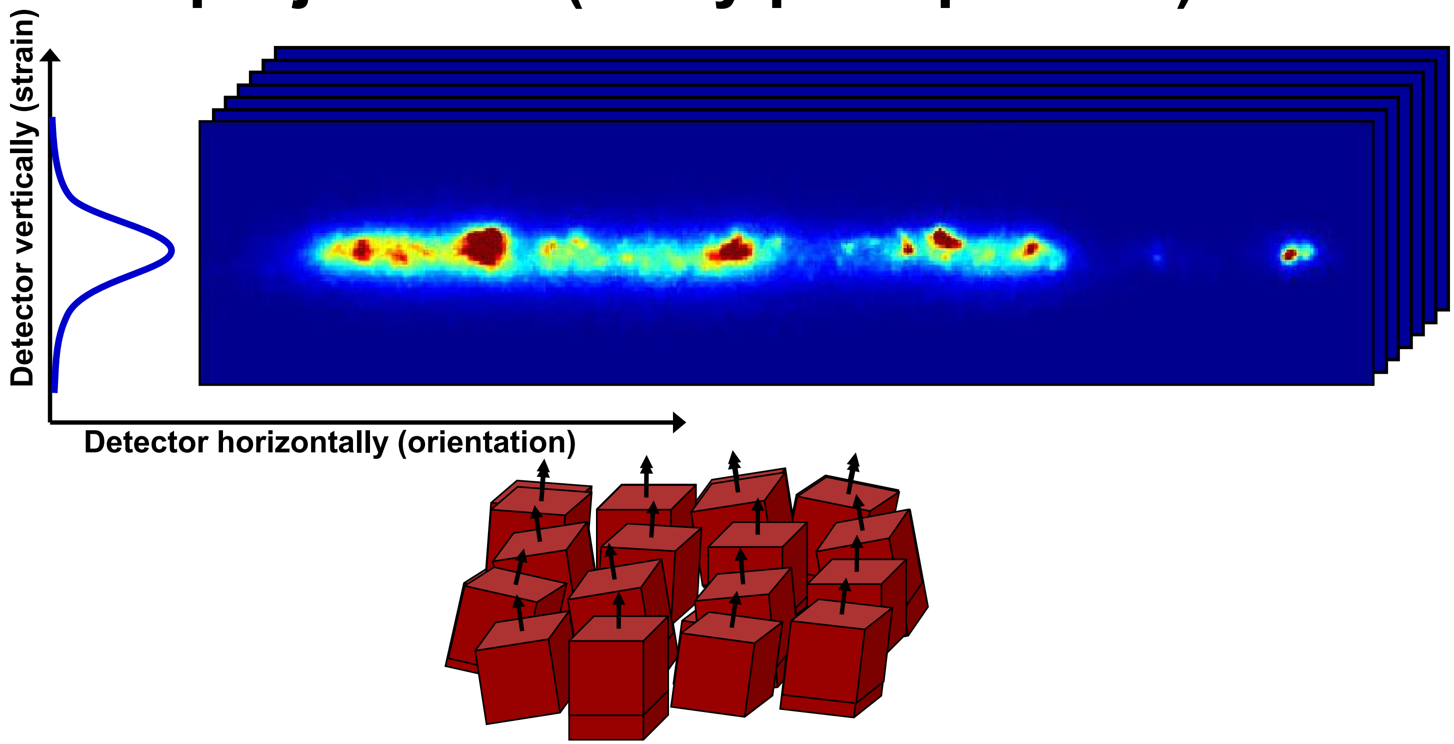


Azimuthal projection of 400 reflection



- Distribution of crystallographic [100] directions in one particular grain
- 100 pole figure (zoom in)

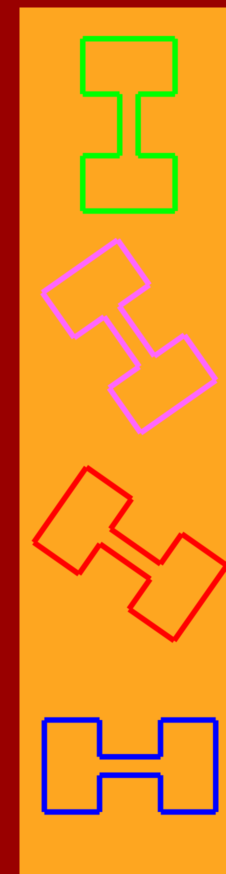
Radial projections (X-ray peak profiles)



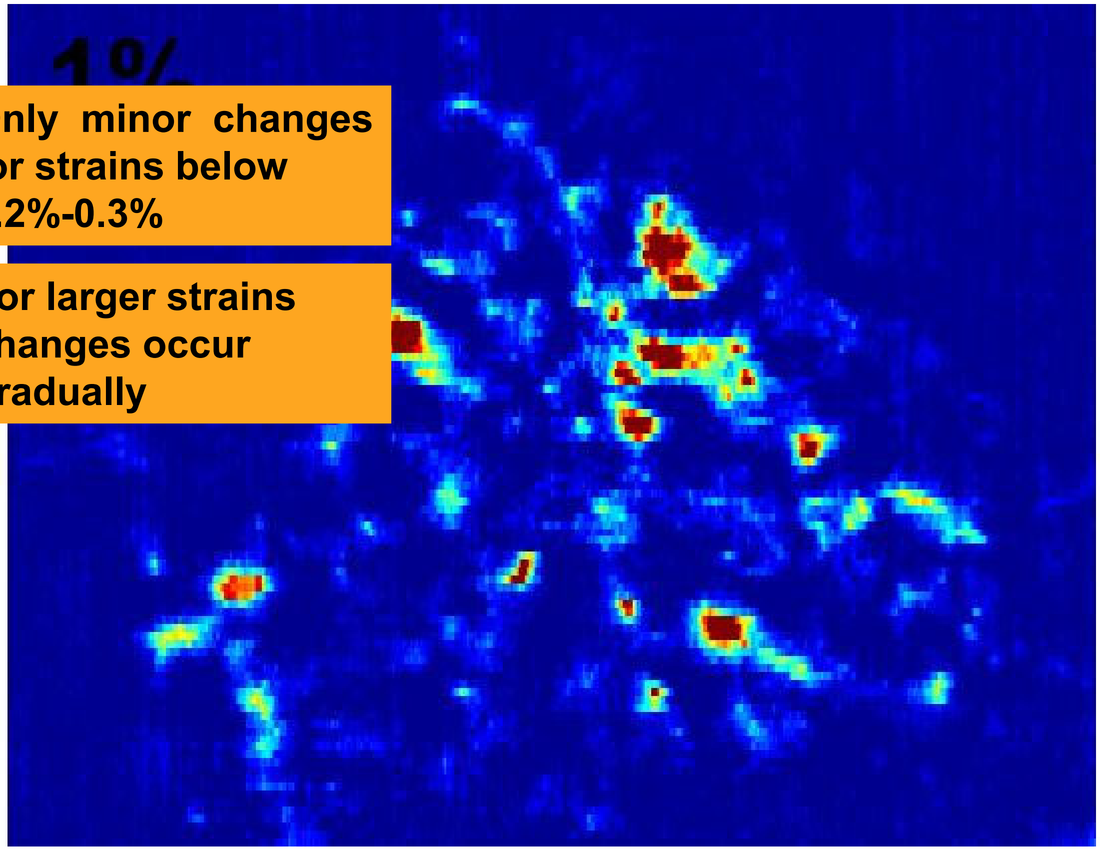
- Distribution of (100) lattice plane spacings or elastic strains along [100]

Strain path changes

C. Wejdemann, U. Lienert, W. Pantleon (2010)



Dynamics – strain path change

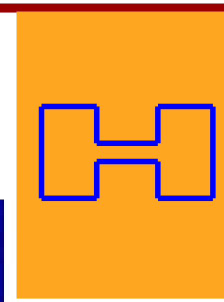


Only minor changes for strains below 0.2%-0.3%

For larger strains changes occur gradually

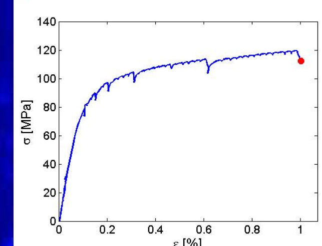
Rocking (orientation)

Detector horizontally (orientation)

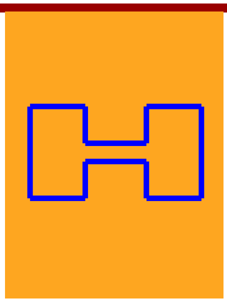


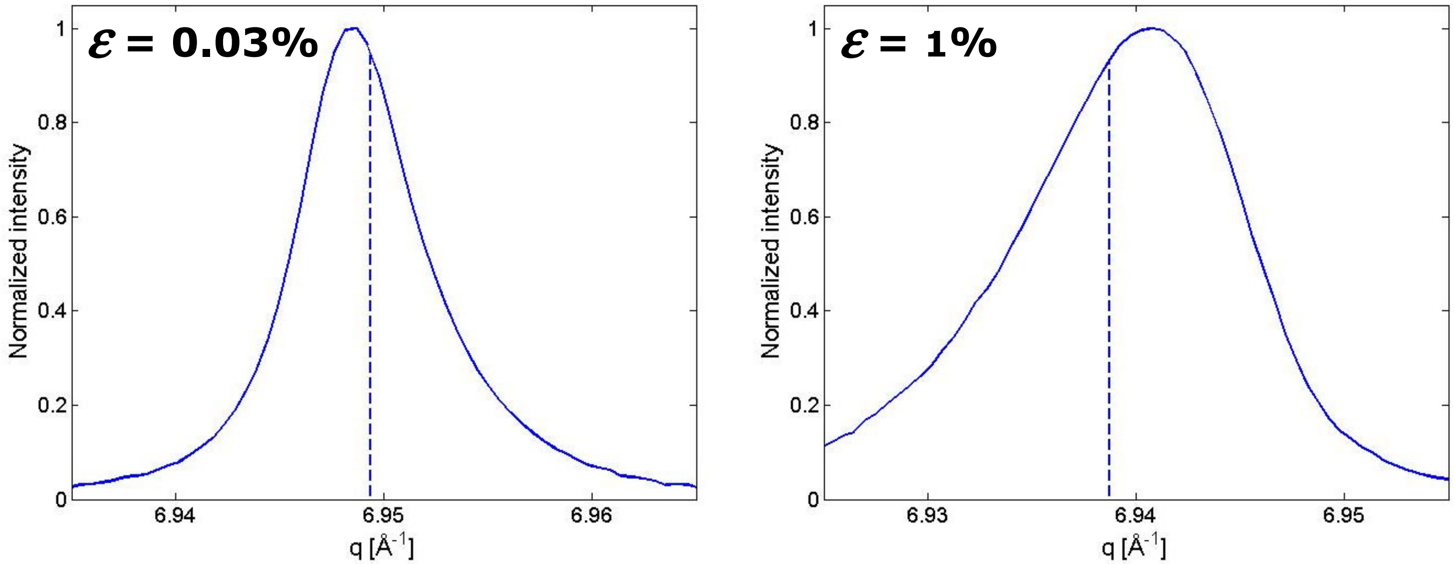
$\psi = 90^\circ$

$\alpha = -\frac{1}{2}$



Radial profiles





$\psi = 90°$

$\alpha = -½$

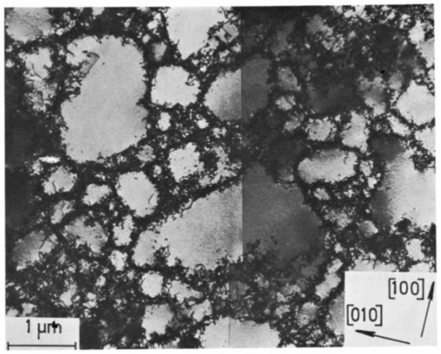
Composite model for dislocation structures
(Mughrabi 1983)

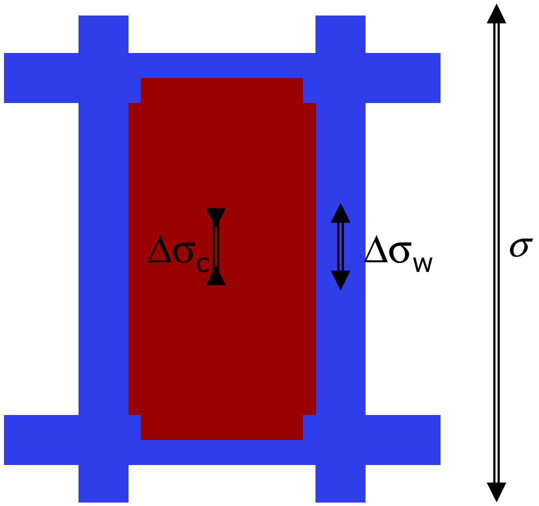
- hard dislocations walls (w)
- soft cell interiors (c)
- interiors deform plastically
- compatibility
- local axial elastic stresses
  - forward in walls
  - back in cell interiors

$$\Delta\sigma_w > 0 > \Delta\sigma_c$$

$$d_w > \bar{d} > d_c$$

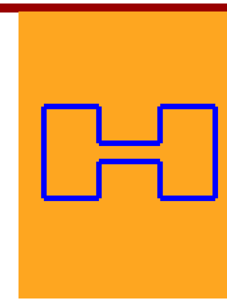
$$2\hat{\theta}_w < 2\bar{\theta} < 2\hat{\theta}_c$$

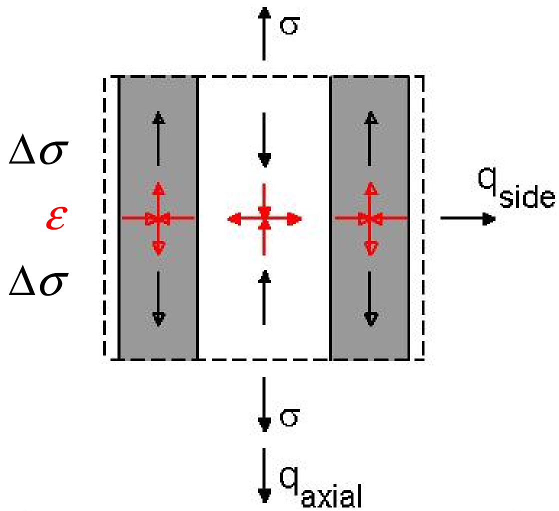


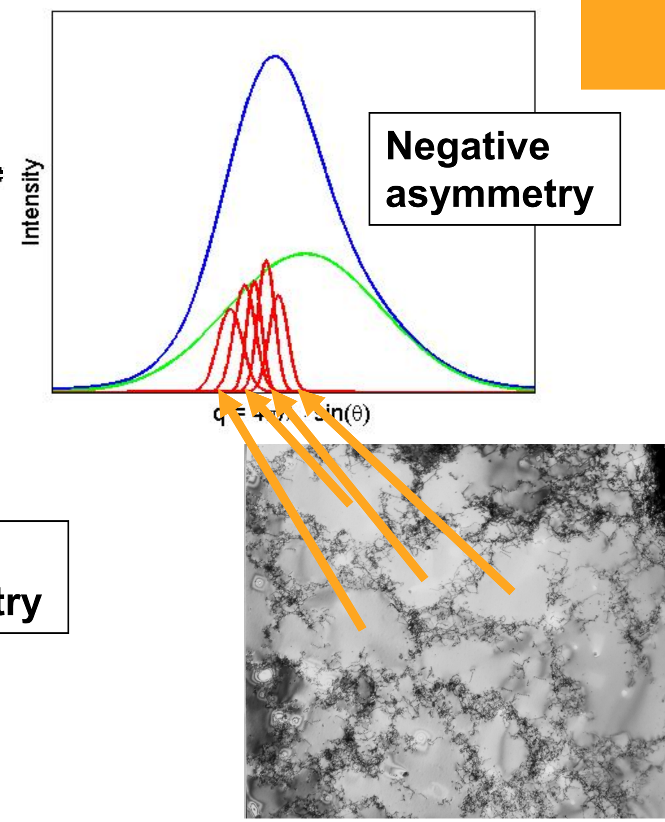


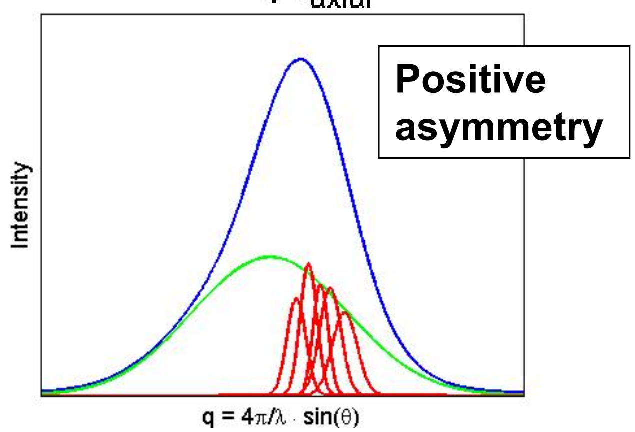
Composite model – refined

(Ungár, Mughrabi 1984) **(Pantleon 2011)**

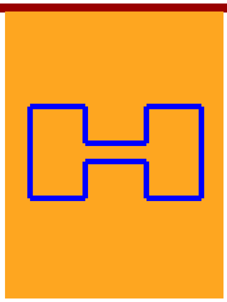


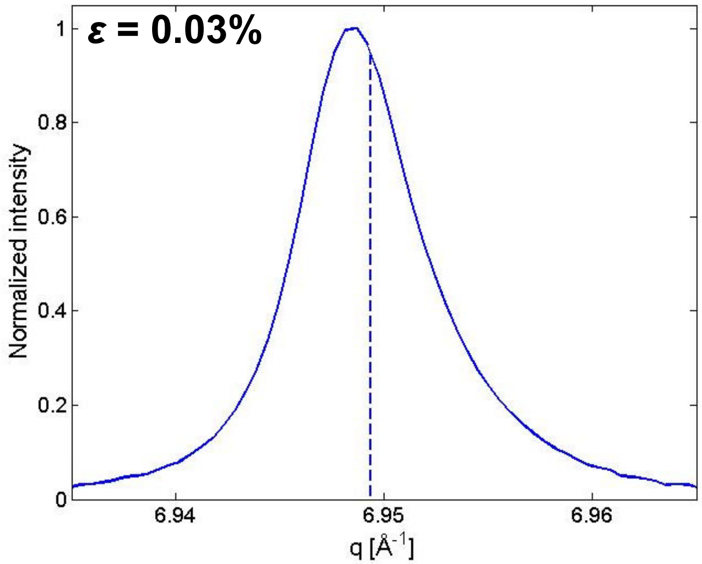


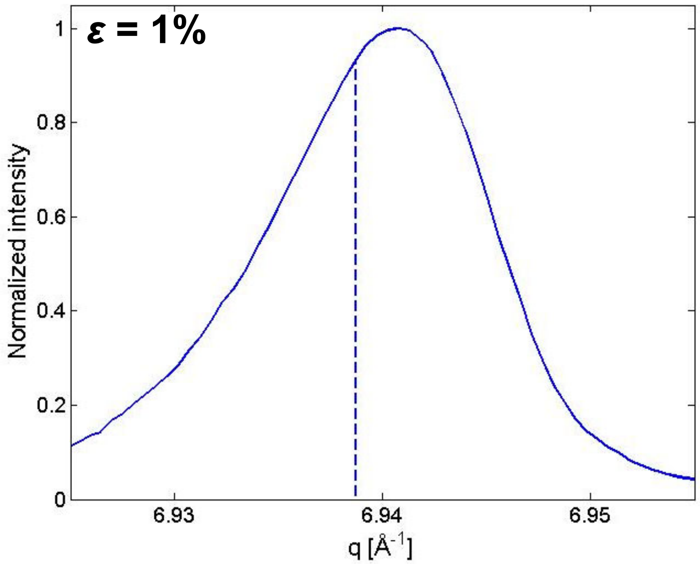




Radial profiles





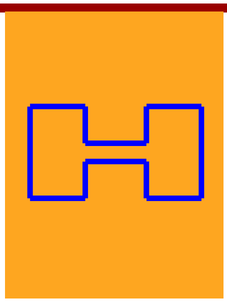


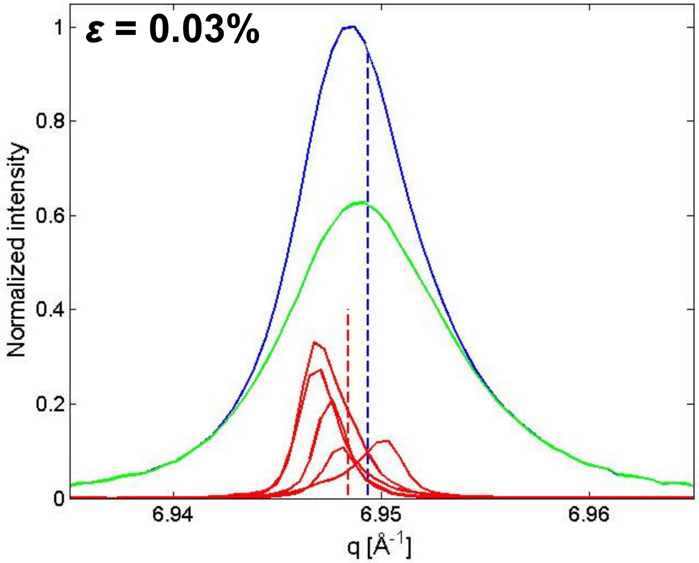
$\psi = 90°$

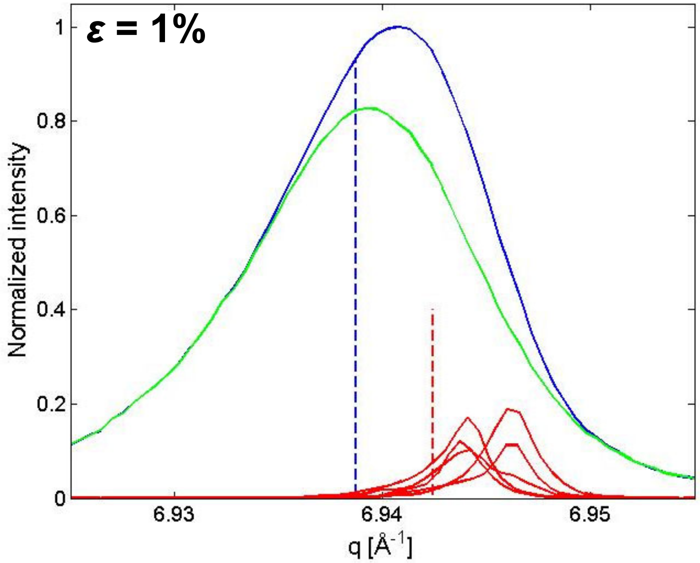
$\alpha = -½$

Radial profiles

Elastic back-strain:
Changes from tensile
to compressive







ψ = 90°

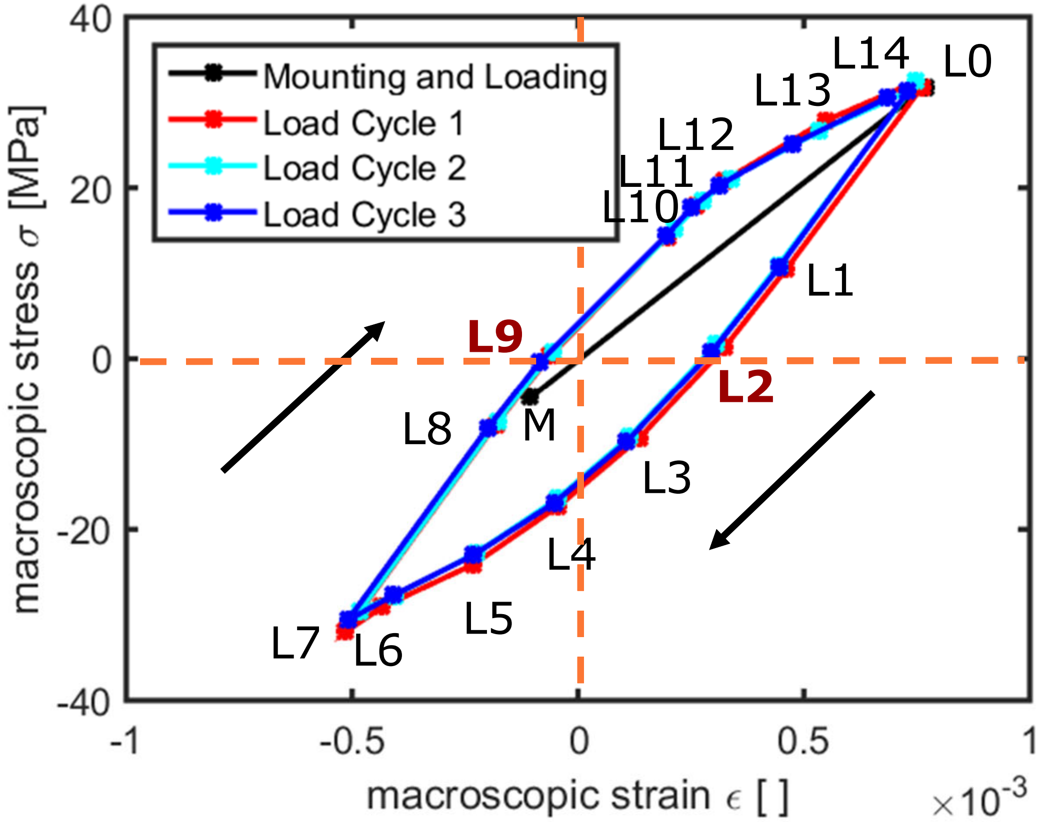
α = -½

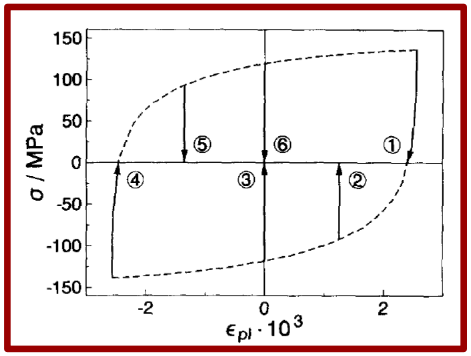
Elastic back-strain:
Significant variation
between subgrains

Cyclic deformation

A. Diederichs, F. Thiel, U. Lienert, W. Pantleon (2018)

In-situ mapping during consecutive tension-compression cycles



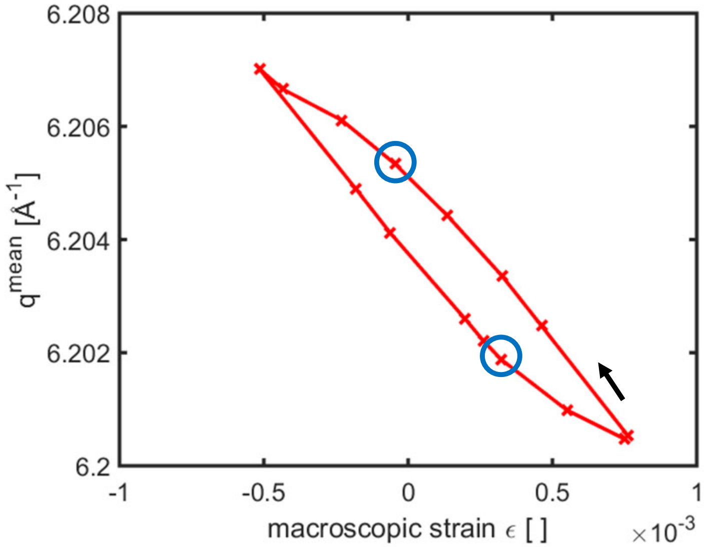


(Biermann et al. 1993)

Tension-compression cycle

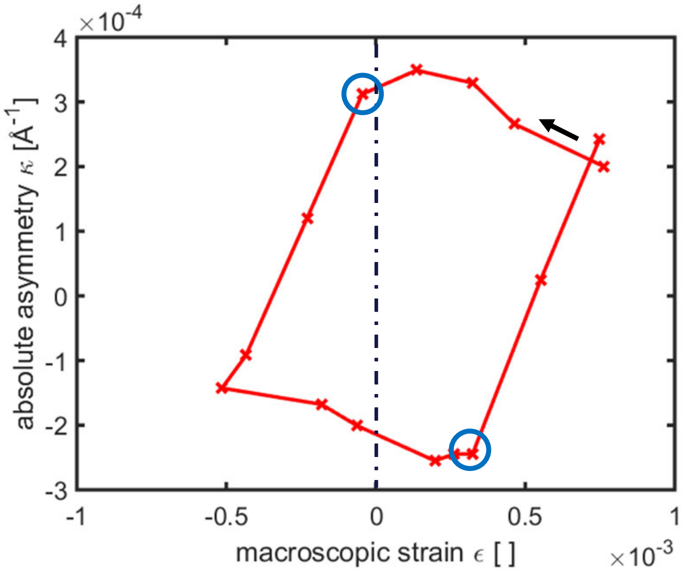
apparent plastic yielding

- Mean peak position



- Follows macroscopic hysteresis

- Asymmetry

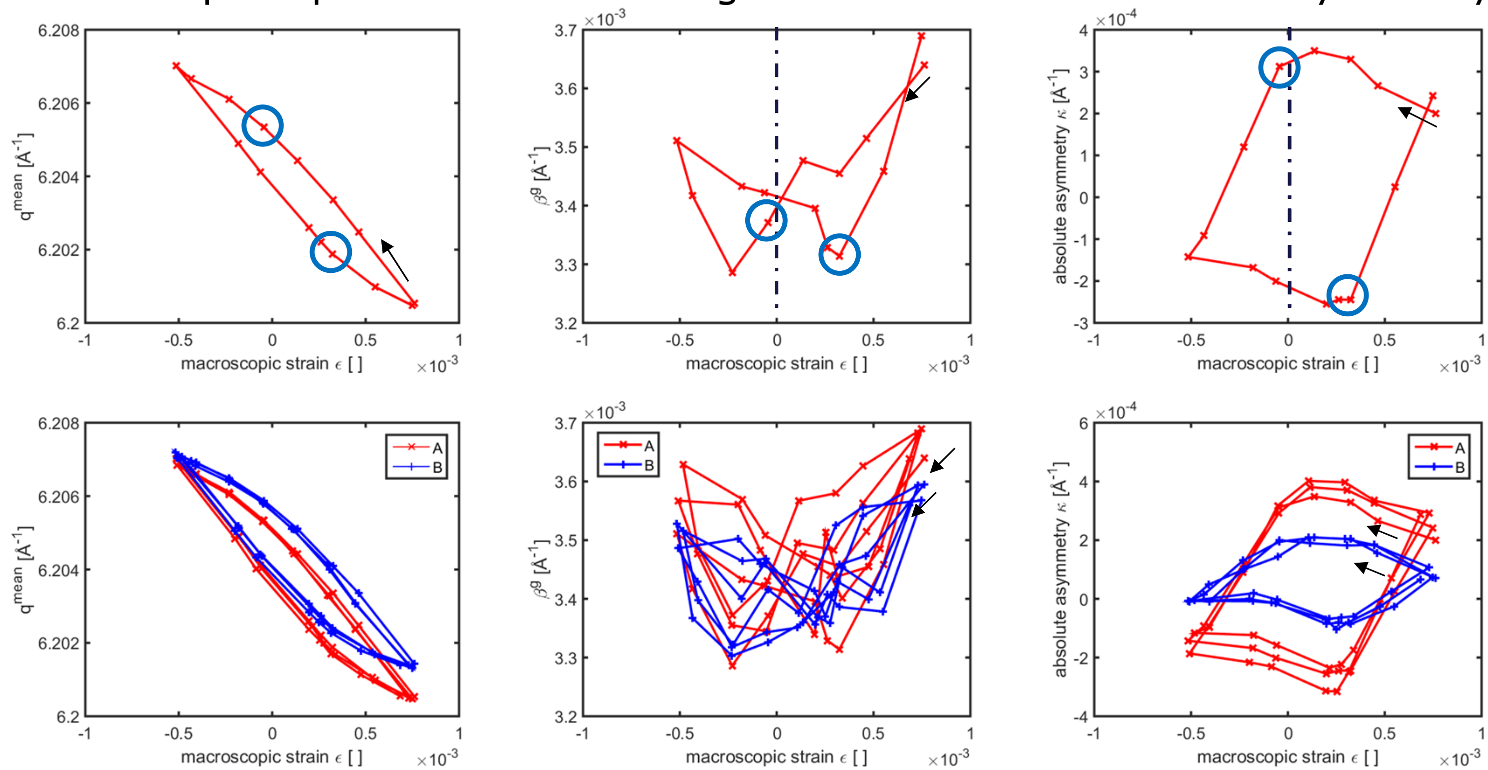


- Increases by 50% during apparent elastic unloading
- Changes sign during reversed plastic loading

apparent plastic yielding

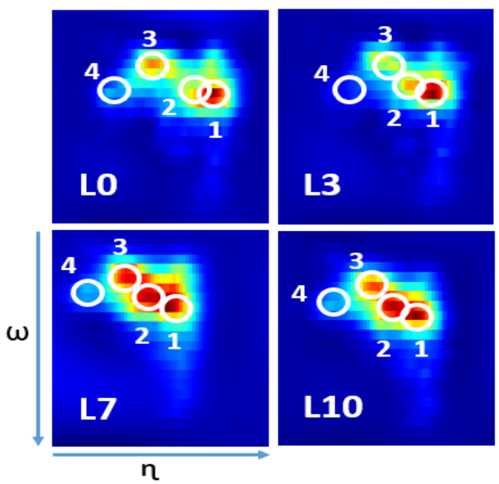
Tension-compression cycle

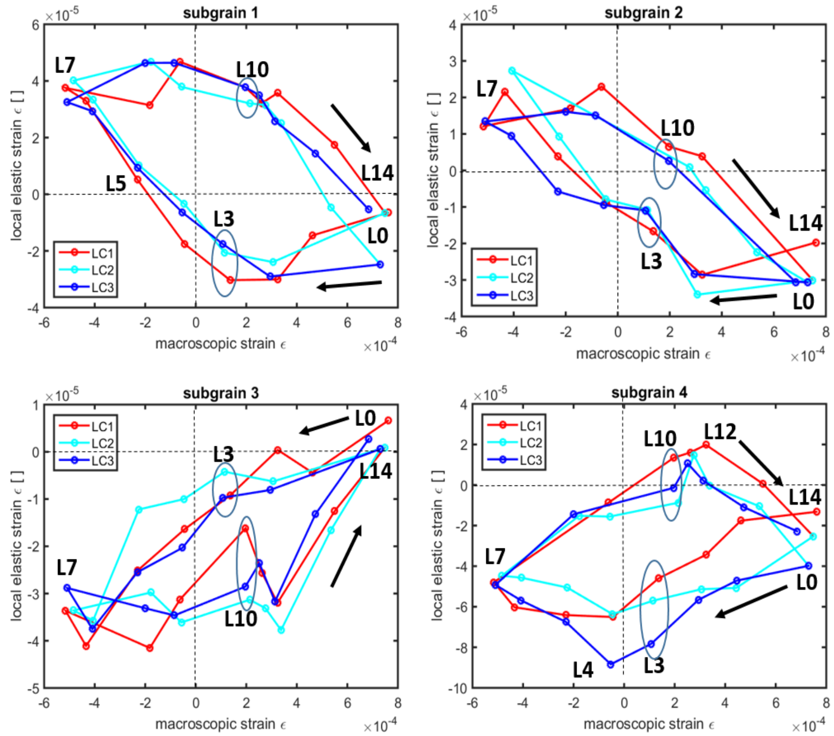
Mean peak position | Integral width | Absolute asymmetry



Behavior of individual subgrains

4 subgrains



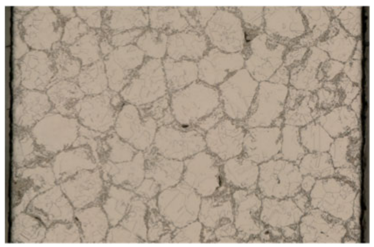


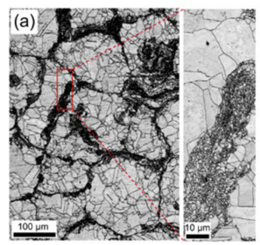
Harmonic structures

Tension of harmonic structured Ni

(Sjøgren-Levin, Pantleon, Ahadi, Hegedüs, Lienert, Tsuji, Ameyama, Orlov 2022)

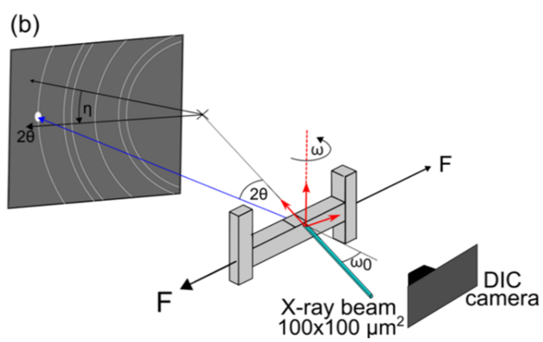
- Harmonic structures



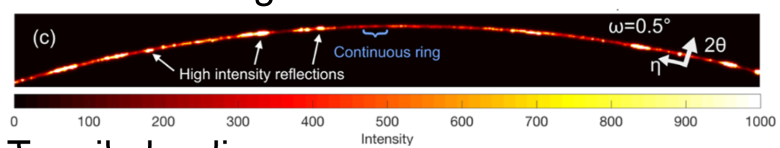


Coarse grains embedded in skeleton of ultrafine grains

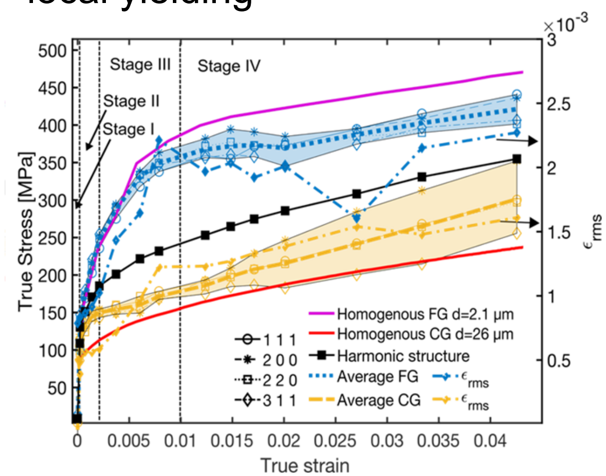
- Tensile loading



- Observation: high intensity peaks on continuous rings



- Tensile loading
  - load partitioning
  - local yielding

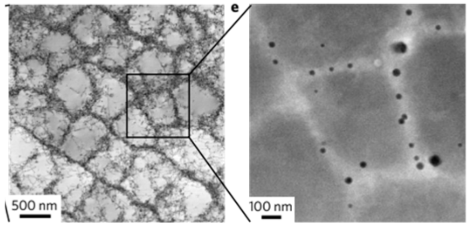


Dislocation reorganization during thermal cycling: additive manufacturing of 316L

Additive manufacturing of 316L SS

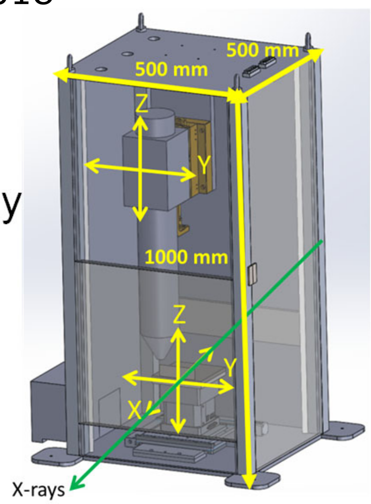
(Gaudez, Abdesselam, Gharbi, Hegedüs, Lienert, Pantleon, Upadhyay 2022)

- Dislocation cells after AM

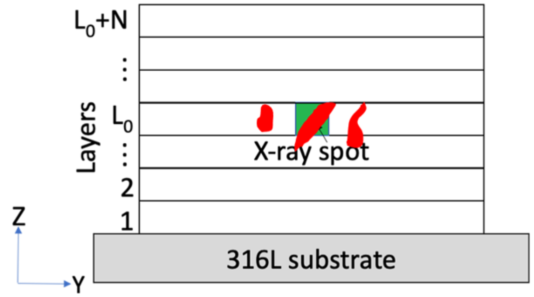


Morris et al. 2018

- mini-LMD machine for directed energy deposition (LDED)



- Printing of wall



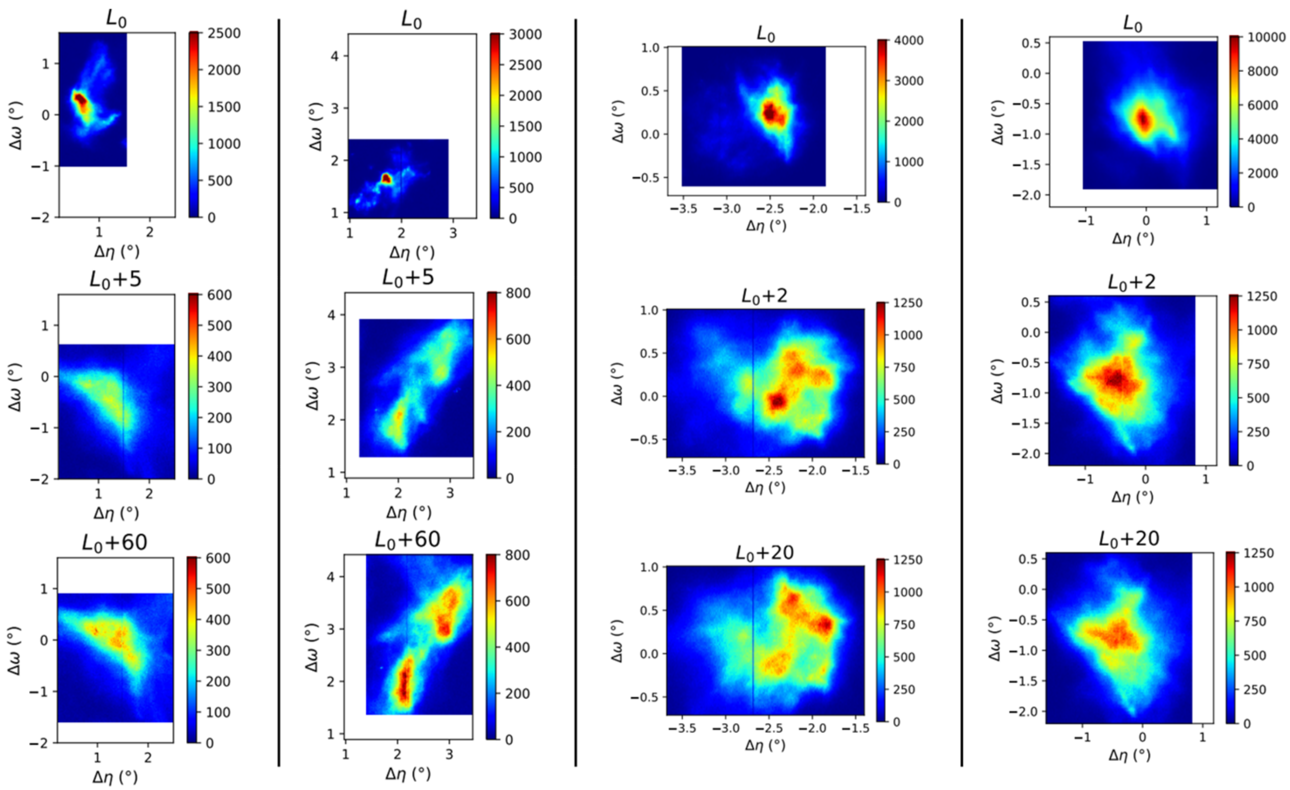
- Print 20 layers
- Identify grains in top layer with [400] along print direction
- HRRSM on 400 peak before and after adding layers
- Role of solid-state thermal cycling

Additive manufacturing of 316L SS

(Gaudez, Abdesselam, Gharbi, Hegedüs, Lienert, Pantleon, Upadhyay 2022)

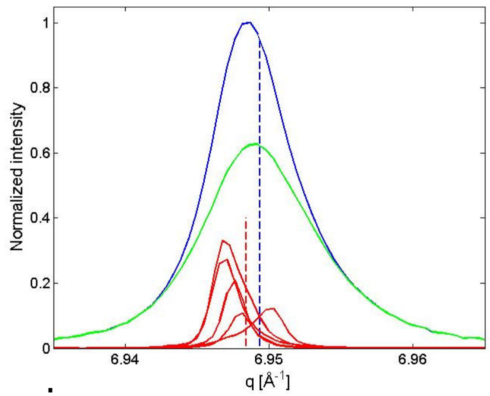
- Observation: dislocation structures evolve during SSTC

- Evolution of orientation domains (cells not resolved)



High-resolution reciprocal space mapping

- Allows disclosing of information hidden below smooth diffraction peaks
- Versatile tool to monitor evolution of deformation structures
  - during thermo-mechanical treatments
  - for individual grains in polycrystals (metals)



- Realized at 1-ID@APS and P21@PETRA

- Acquisition time for 1° orientation spread initially 40 min now 2 min
  - Improved resolution achieved by photon counters with negligible down time