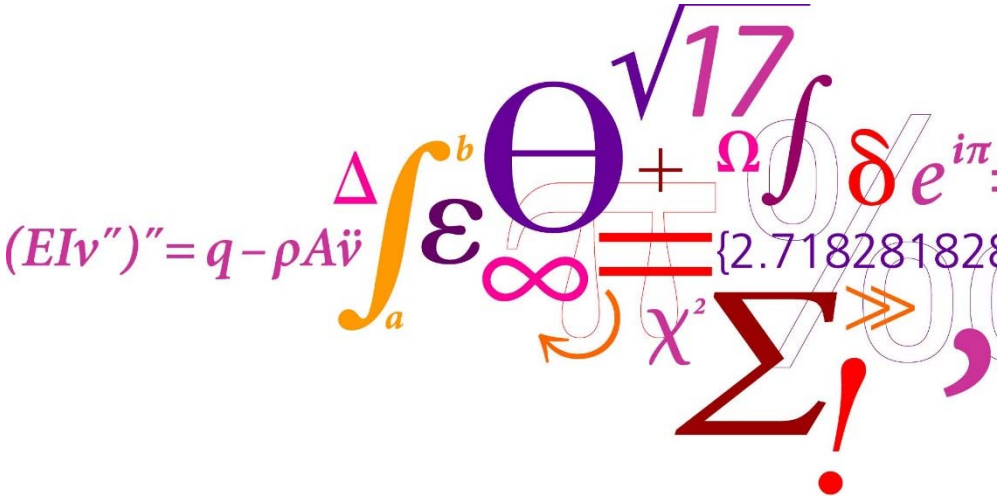
Cyclic deformation of Aluminium: In-Situ monitoring of microstructural changes

Annika Martina Diederichs, Wolfgang Pantleon

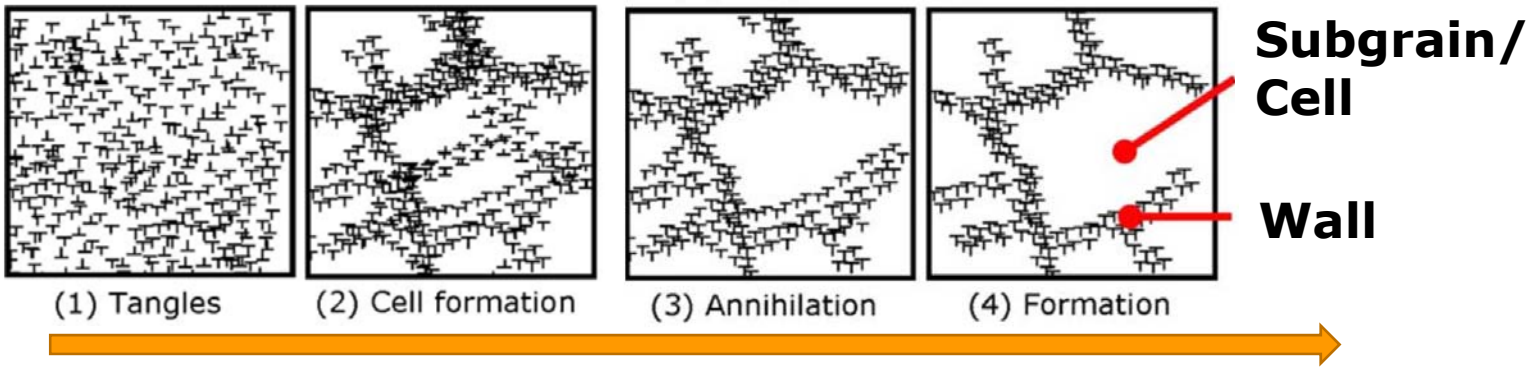
DMS Vintermøde
24.1.2019



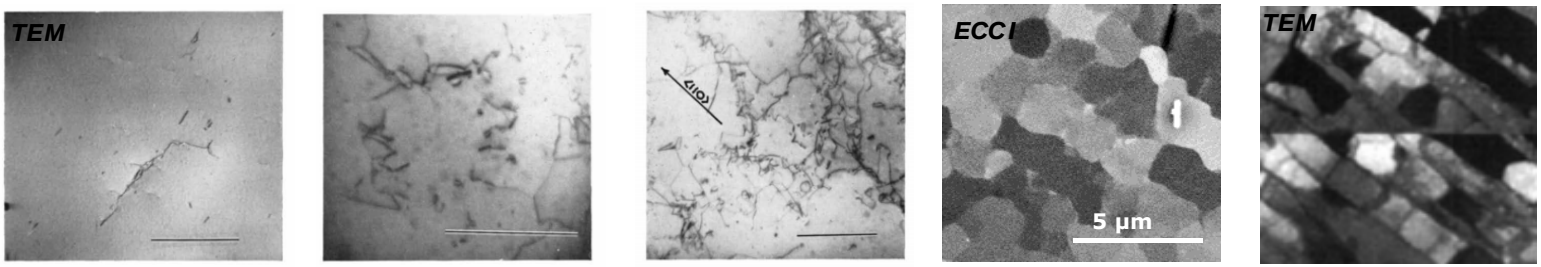
DTU Mechanical Engineering
Department of Mechanical Engineering

Cyclic Deformaton in fcc metals

Generation of subgrains by dislocation clustering



pure Al



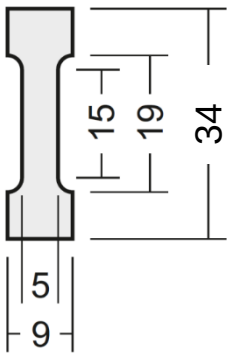
Grosskreutz et al. (1963) Snowden et al. (1963) Li et al. (2010)

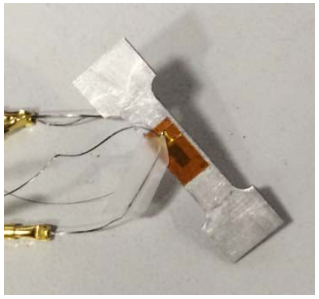
But: understanding of interaction/ stresses between grains/subgrains on the microscale is still insufficient

Small misorientations not visible with EBSD, only surface studies!
In-situ studies challenging and destructive

Material

- commercially pure aluminium AA1050
- 90% cold-rolled sheets (thickness 1 mm),
- dog bone specimens with 15 mm gauge length
- annealed (2 h, 600˚C), 30-100 µm grain size
- two strain gauges





Why Aluminium?

fcc

high SFE

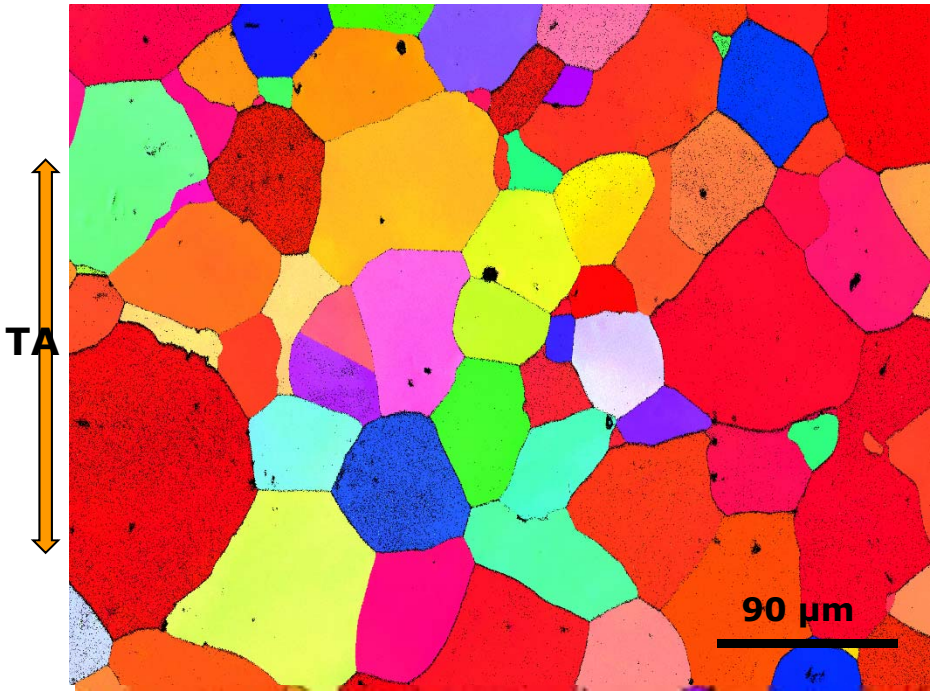
develops deformation structures with relatively large cells/subgrains (~2.5 µm - 5 µm)

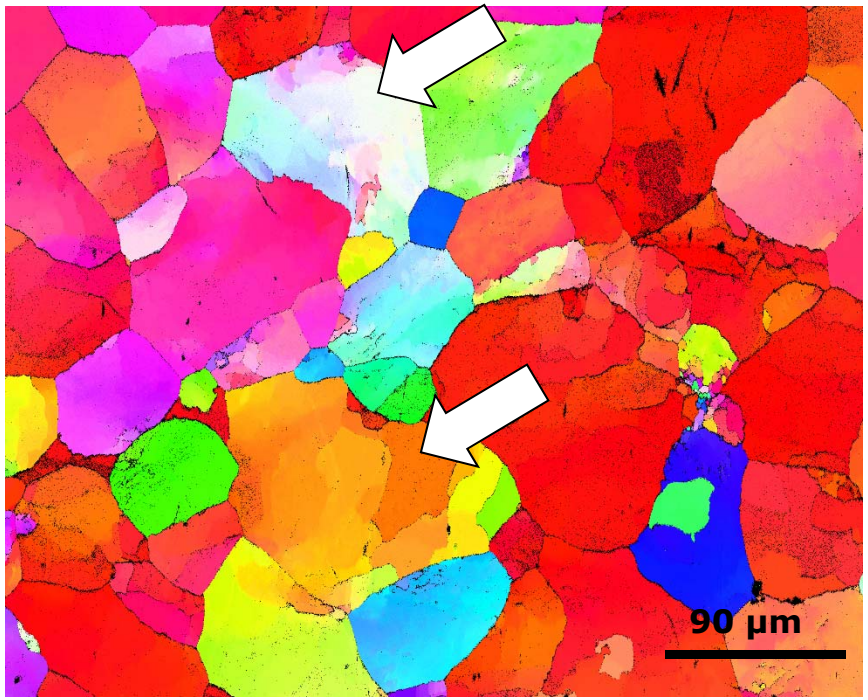
Cyclic Deformation

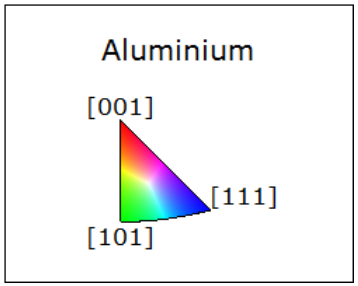
DTU: MTS Acumen 3kN Electrodynamic Test System

Microstructural changes in aluminium during cycling deformation

EBSD Orientation Maps





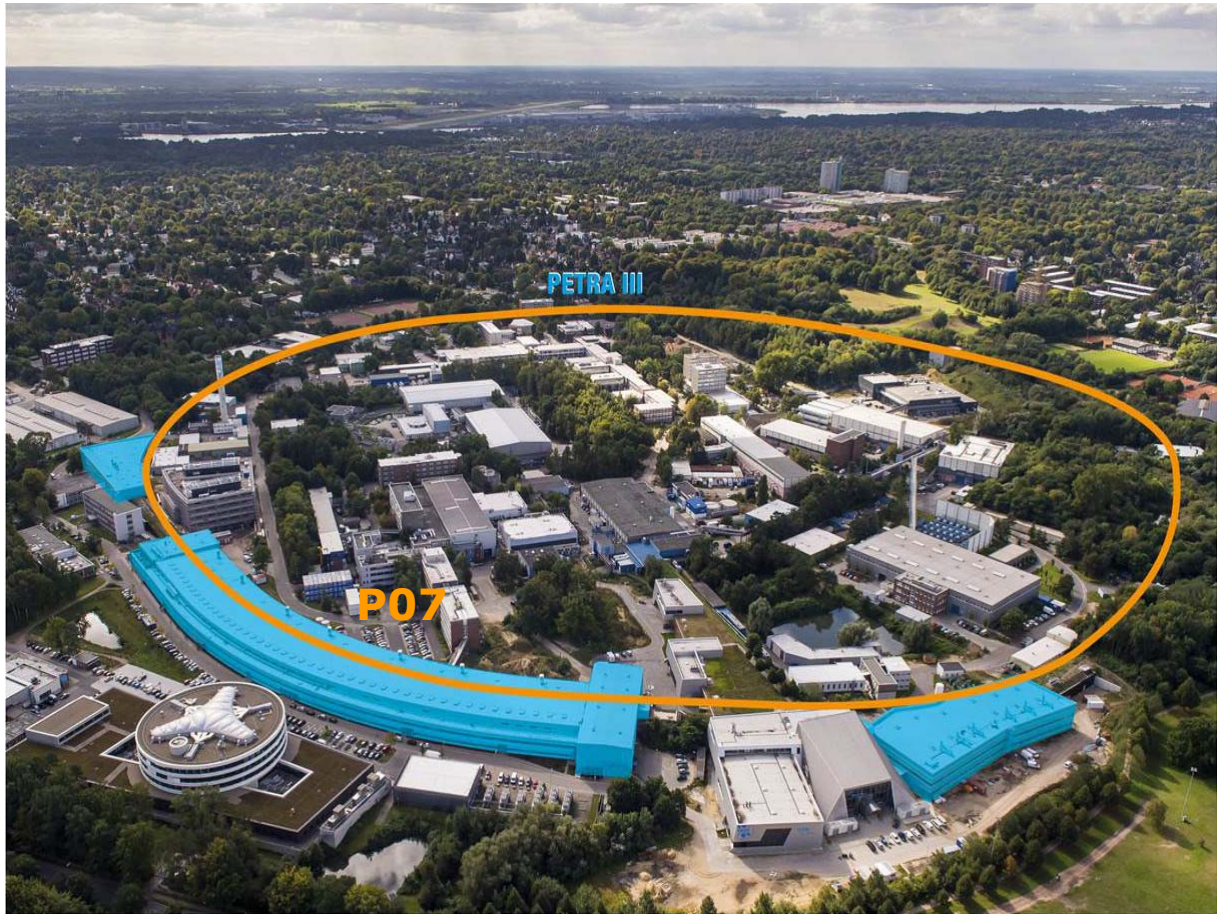


annealed

60 000 cycles

High Resolution Reciprocal Space Mapping

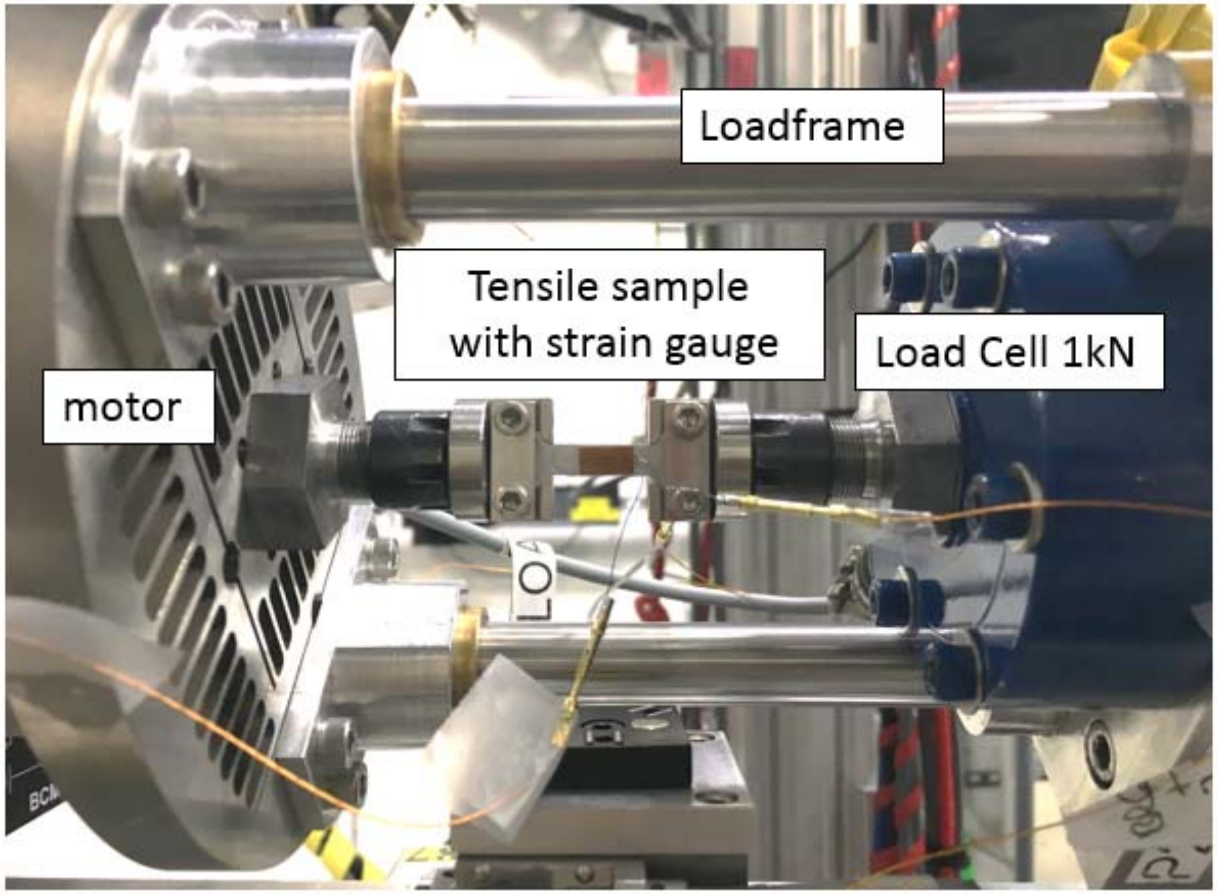
APS, Argonne National Laboratory, Chicago (US)



HRRSM
(Jakobsen et al., 2006)

- *In-situ* during deformation
- High angular resolution
- Illumination of single grains in bulk sample with focussed narrow beam
- Diffraction technique

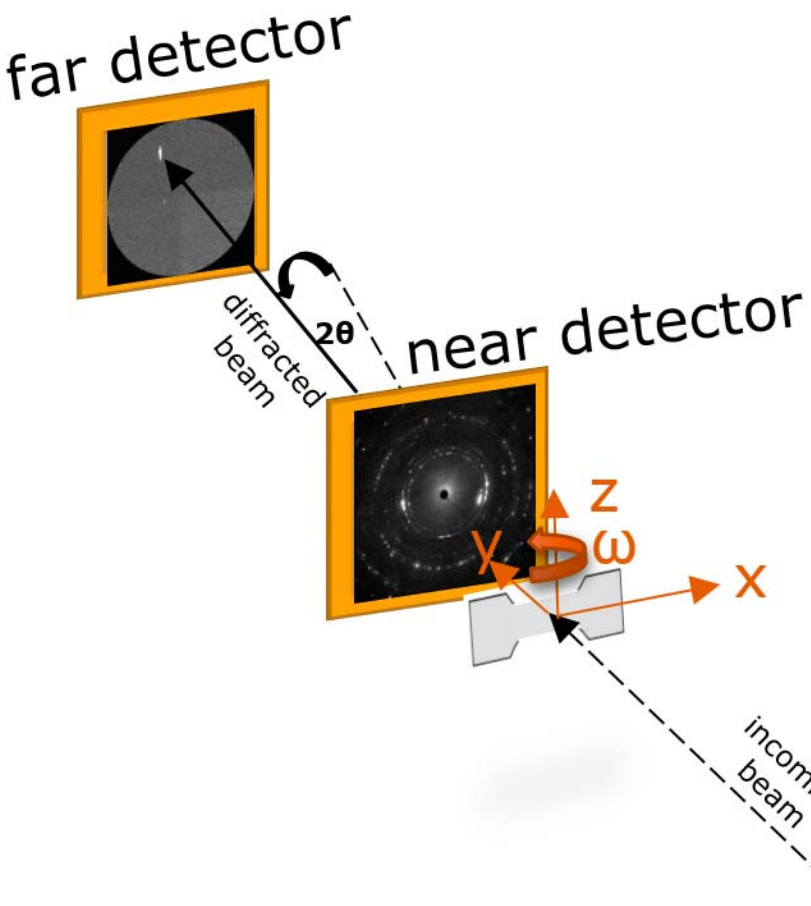
Cyclic Deformation

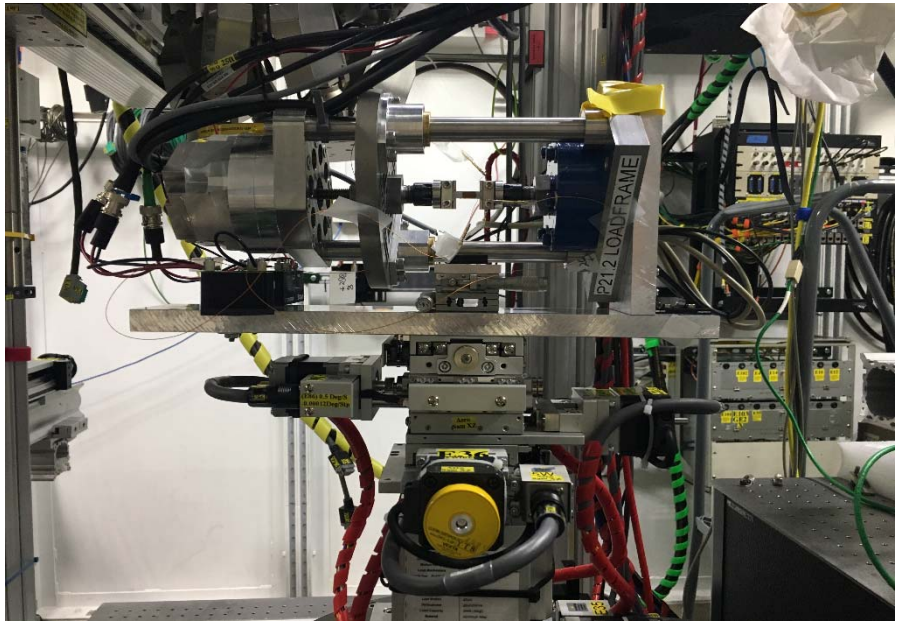


APS 1-ID: HRRSM
Load frame (Petra III, P21, Hamburg)

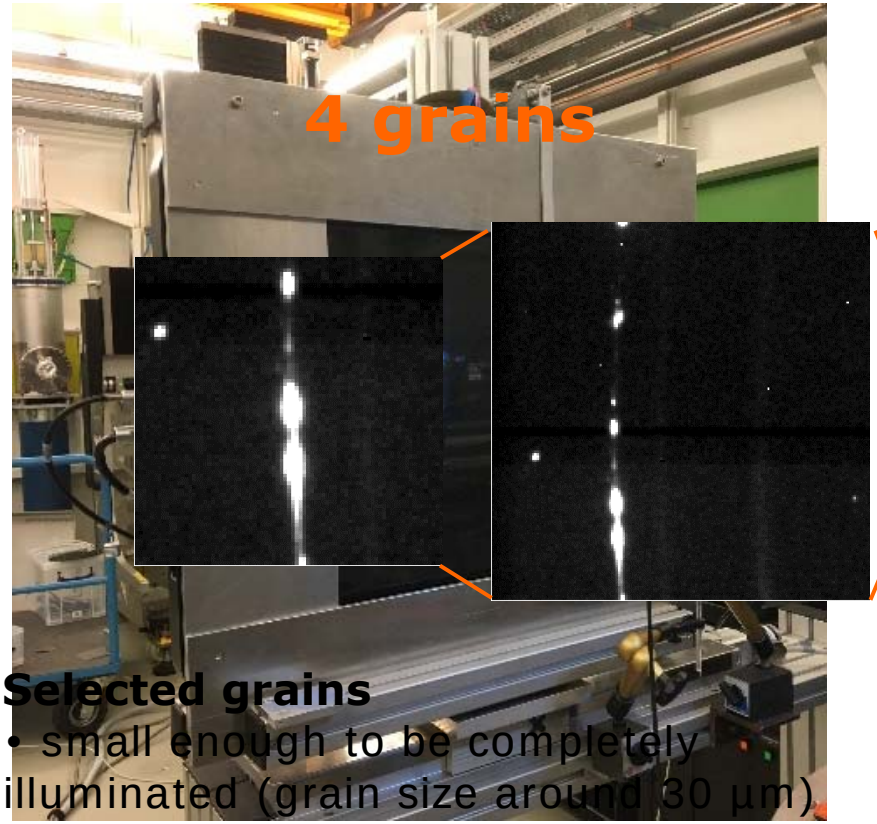
Set-up at APS

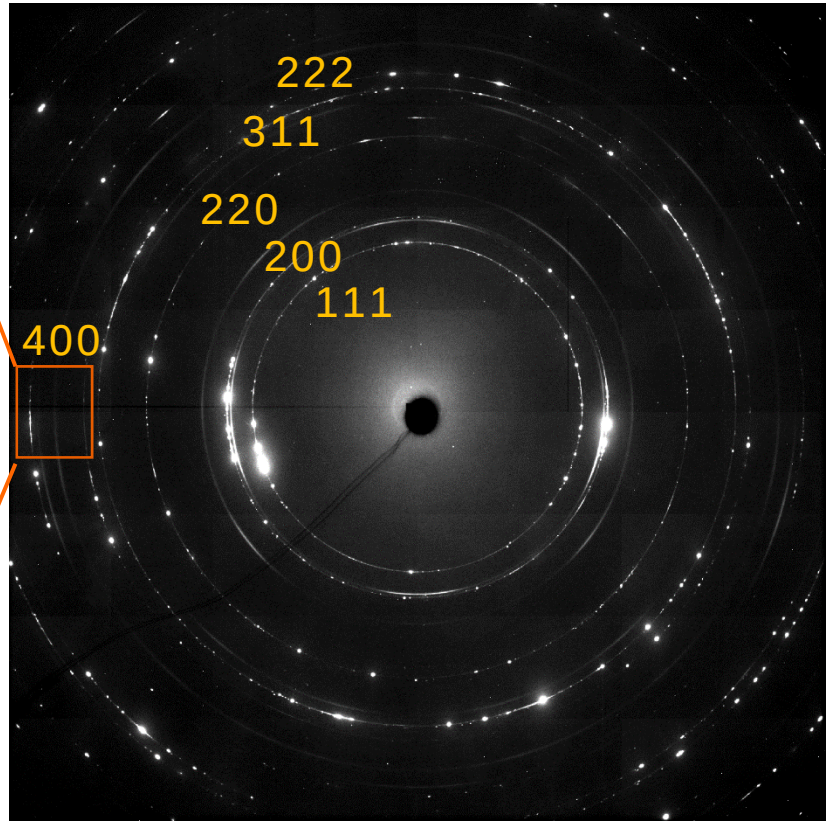
Beam line 1-ID-E, Argonne National Laboratory, Illinois





Near Detector
Grain selection





Selected grains
- small enough to be completely illuminated (grain size around 30 µm)
- 400 reflection (with g nearly parallel tensile axis)
- isolated reflection (no neighbours)

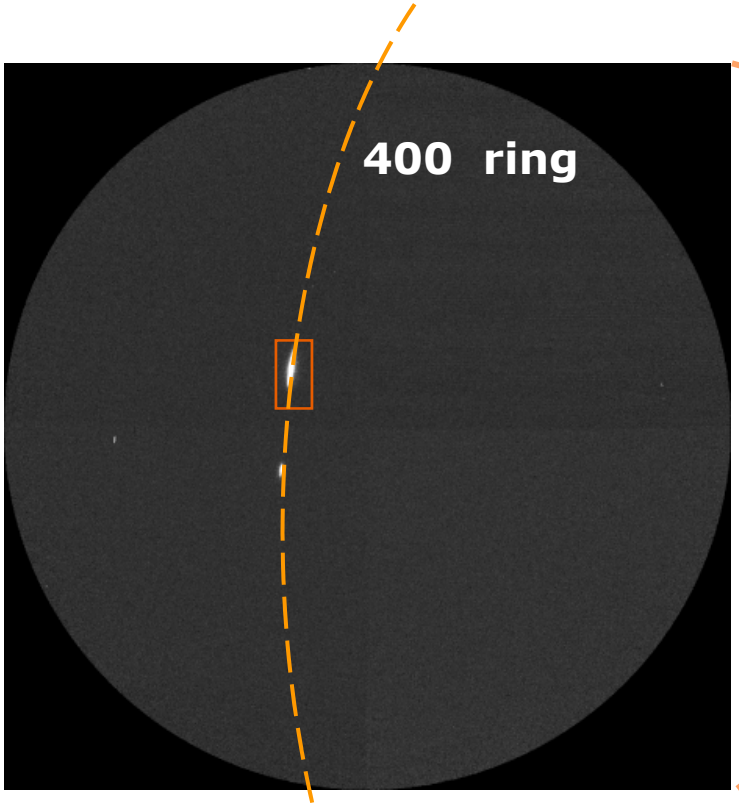
→ bringing spot on far detector

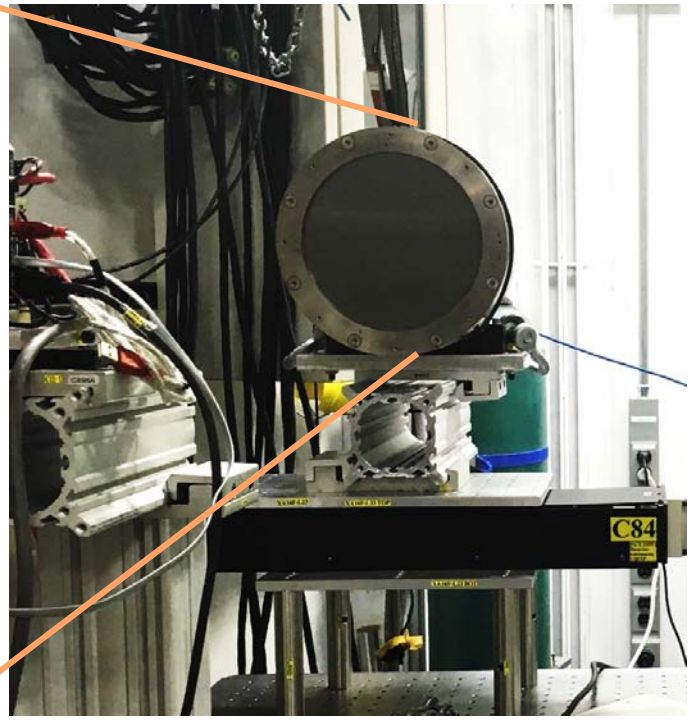
Far Detector
Mapping

- Grain centered for narrowed beam
- 2D acquisition of diffraction peaks while rocking

Bragg equation:

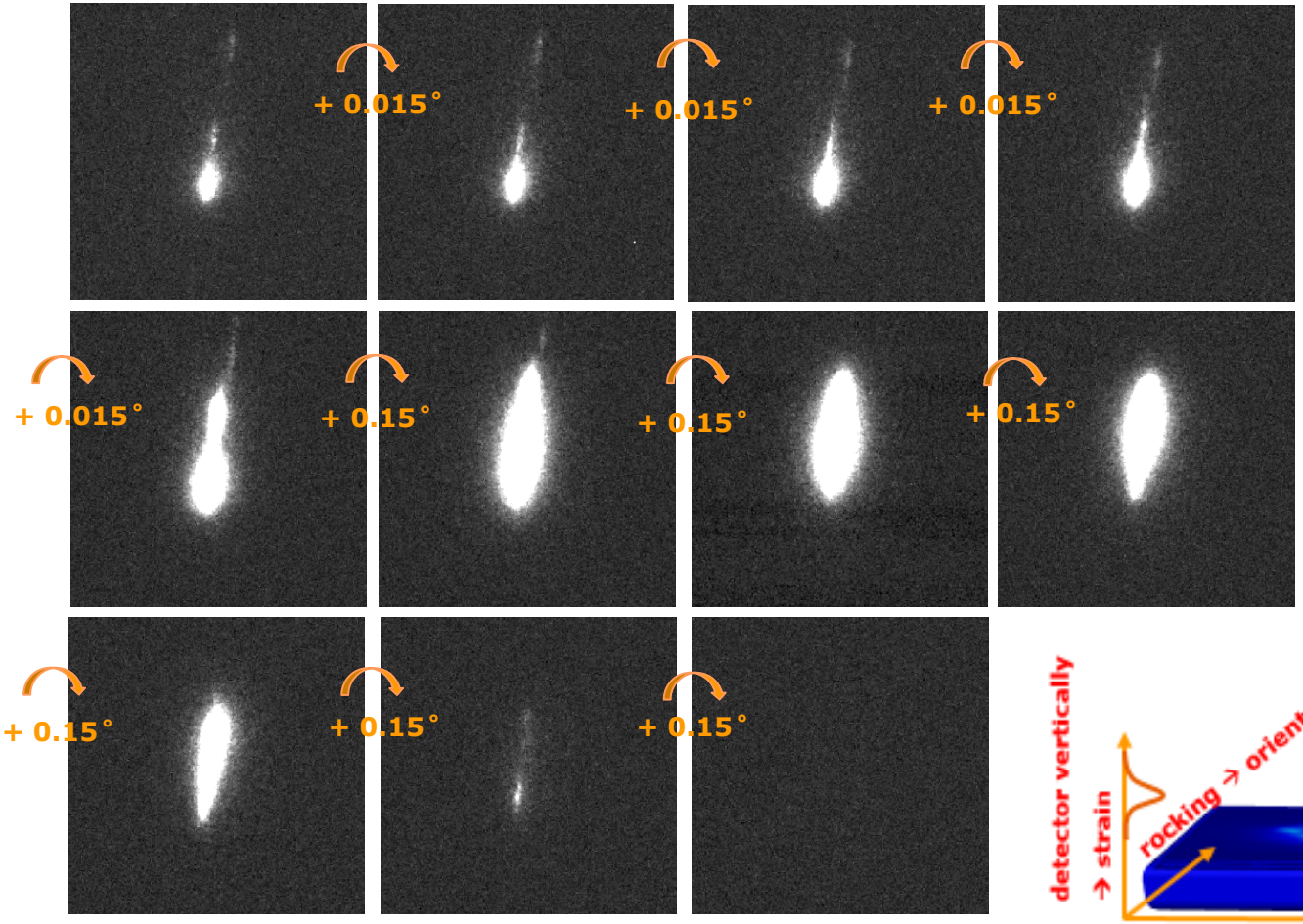
$$2d_{400}\sin\theta = \lambda$$

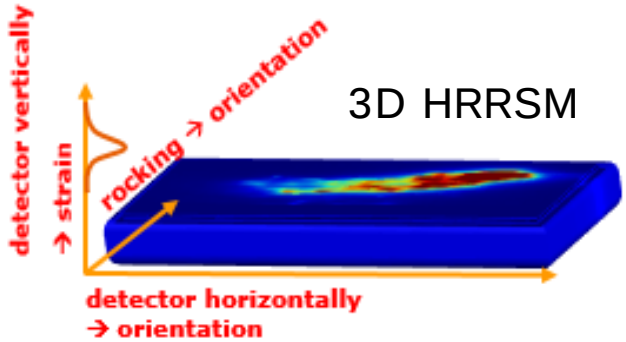




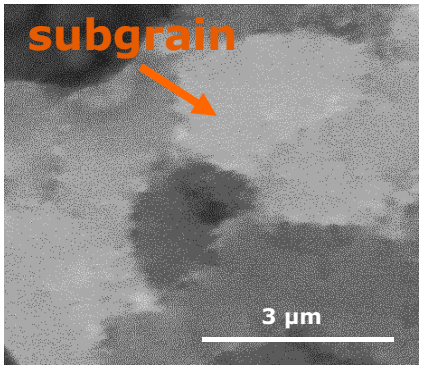
Data aquisition
Mapping by rocking

70 rocking intervals (Δω=0.015°)

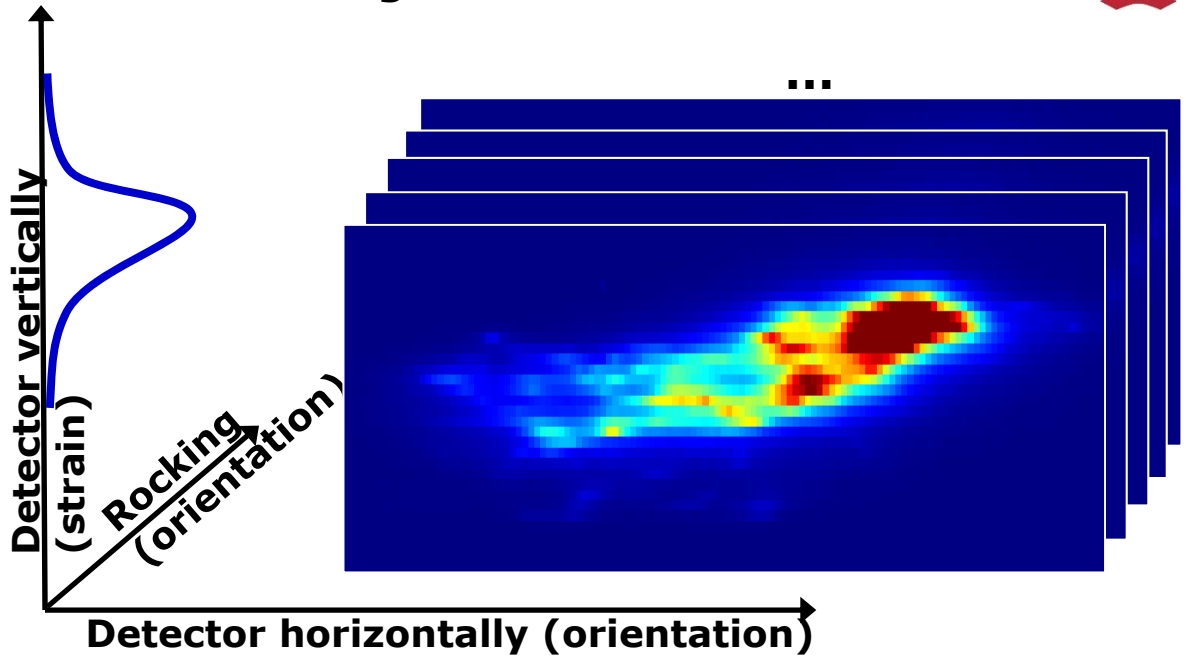


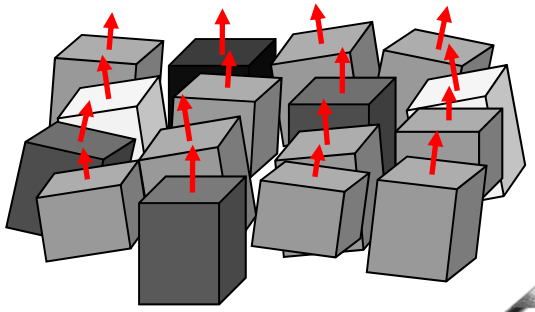


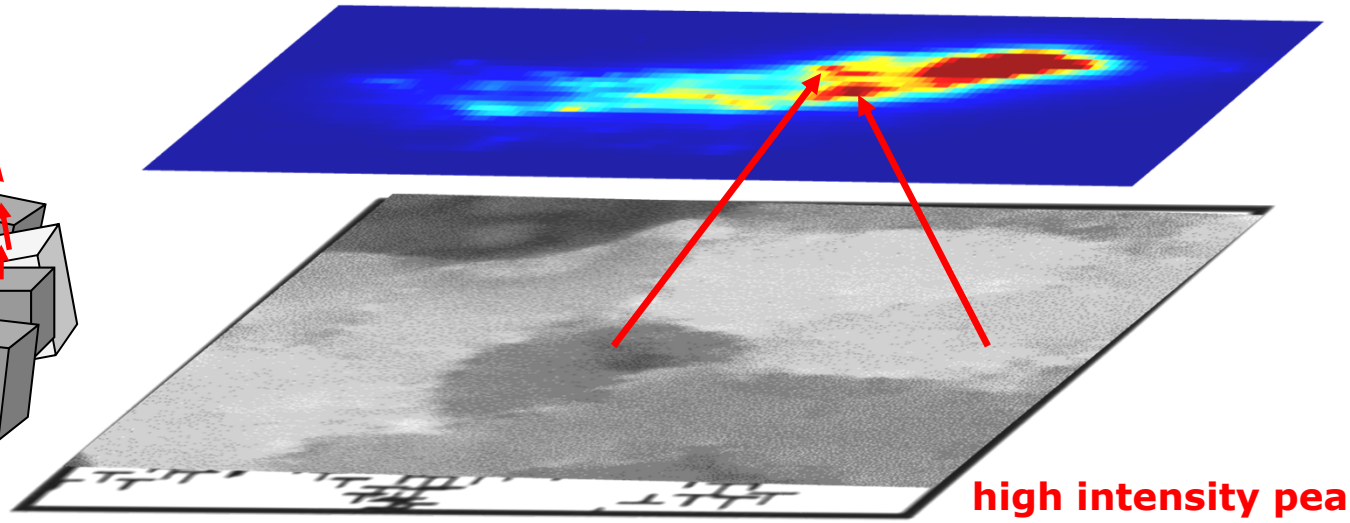
3D HRSSM: Azimuthal Projection



SEM ECCI, AA1050

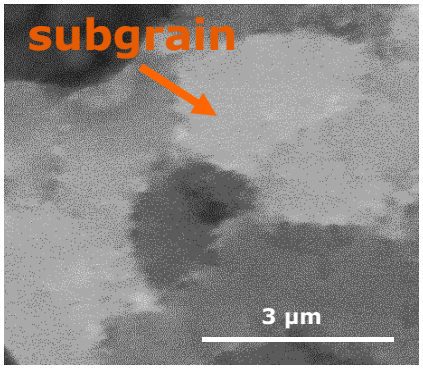




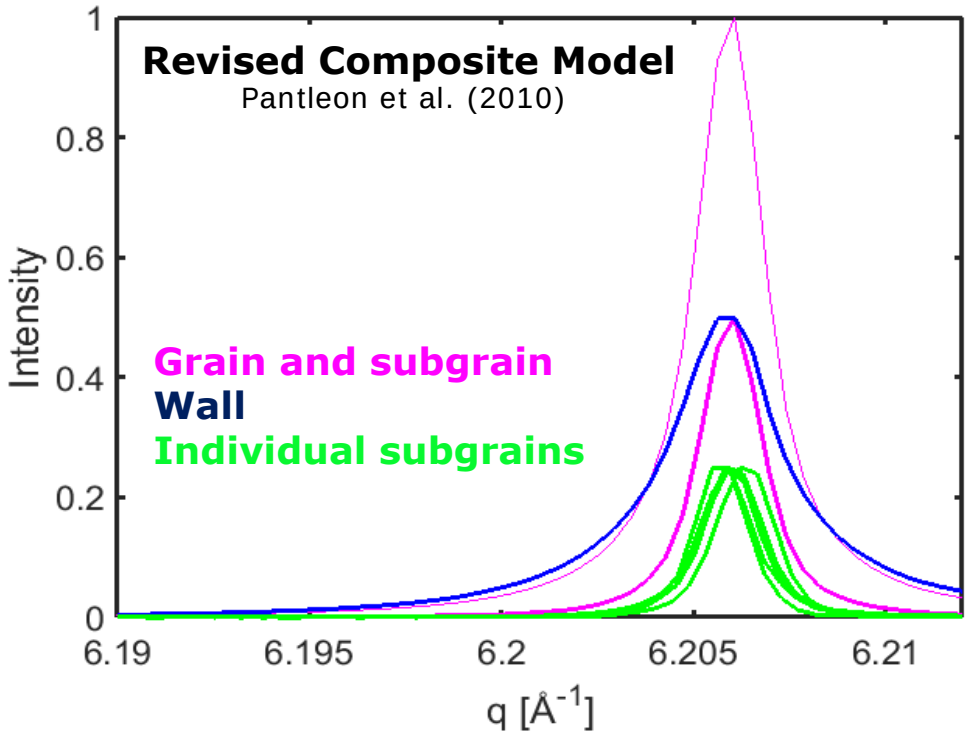


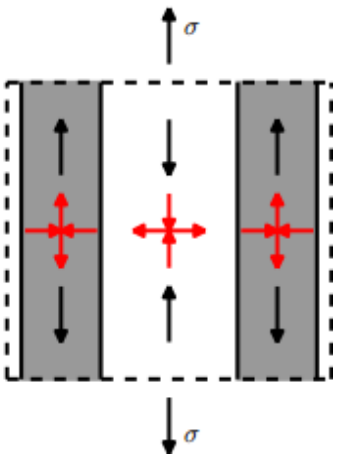
high intensity peaks from cells

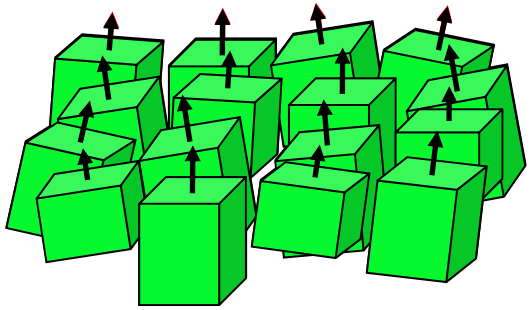
3D HRSSM: Radial Projection



SEM ECCI, AA1050







Experiments

X-Ray High Resolution Reciprocal Space Mapping

- Tension-tension cycling
- Tension-compression cycling
- Individual tension-compression load cycles

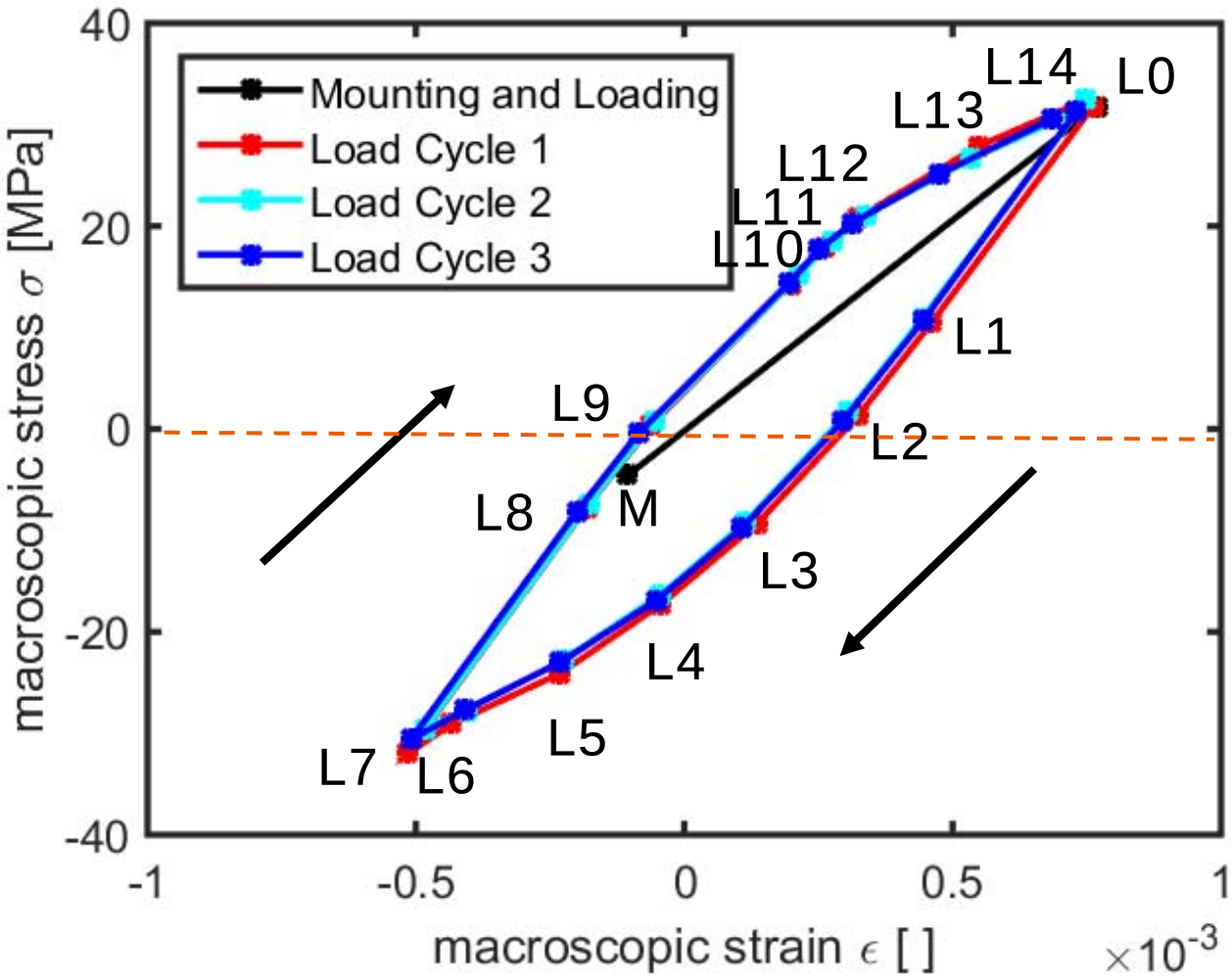
- Different strain amplitudes
- **Subsequent load cycles**

Complementary analysis

- Light Optical Microscopy: Sample surface
- Electron Microscopy: SEM (SE, ECCI) , EBSD for grain structure
- X-Ray Dark Field Microscopy (ESRF, Grenoble): spatial information

Tension-Compression Load Cycles

after 10 000 cycles with ε = 10^{-3}



Radial profiles

Grain

$$q = \frac{4\pi}{\lambda}\sin\theta$$

→ **Peak position**

elastic strain (average)

q_{mean}, q_{max},

→ **Peak width**

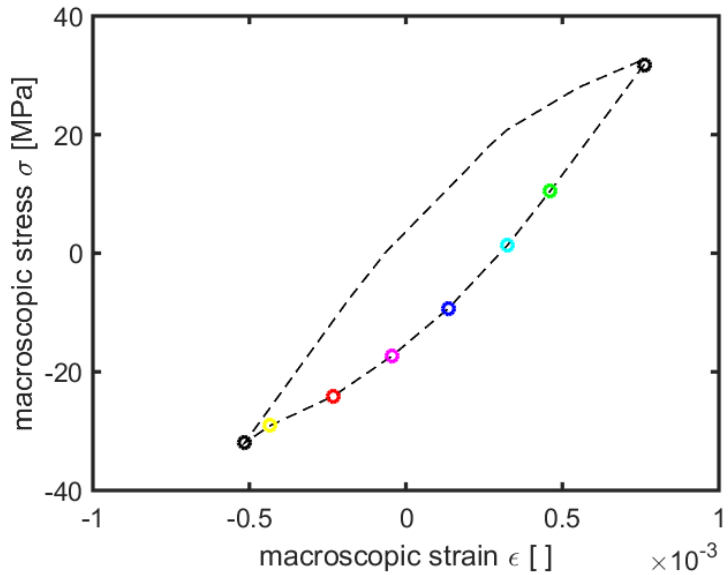
dislocation density

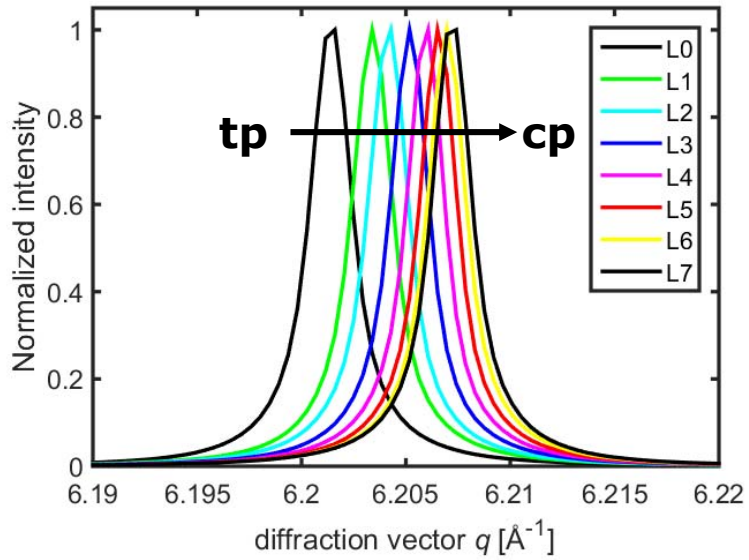
integral width, FWHM

→ **Asymmetry**

ordered structures

absolute, relative





Radial profiles

Grain

$$q = \frac{4\pi}{\lambda}\sin\theta$$

→ **Peak position**

<u>elastic strain (average)</u>

q_{mean}, q_{max},

→ **Peak width**

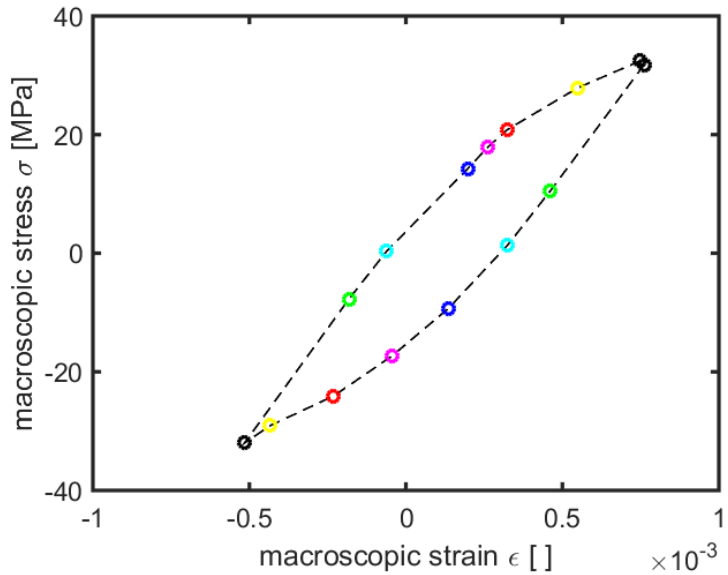
<u>dislocation density</u>

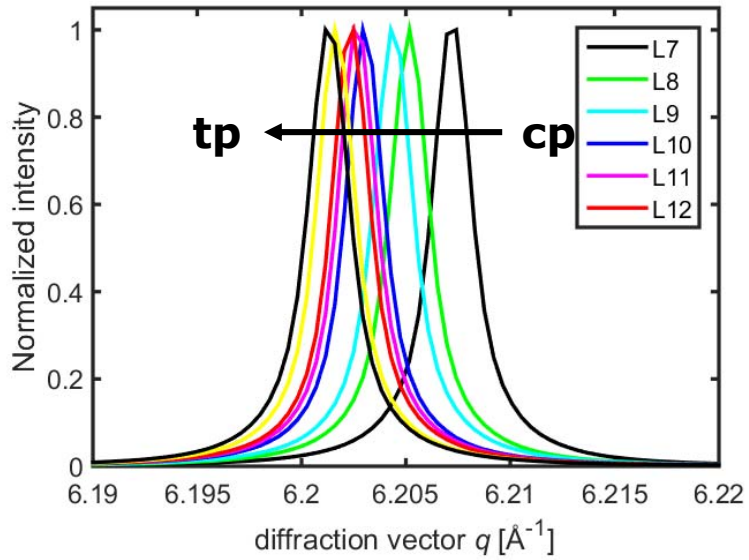
integral width, FWHM

→ **Asymmetry**

<u>ordered structures</u>

absolute, relative





Radial profiles

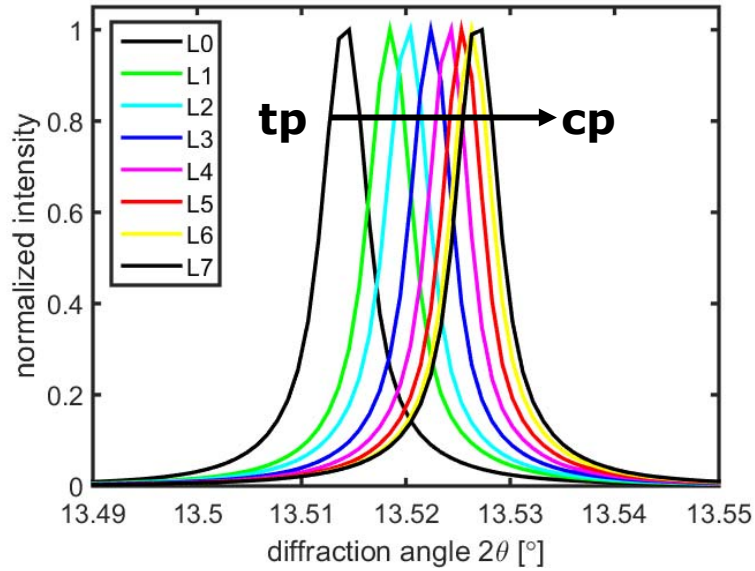
Grain

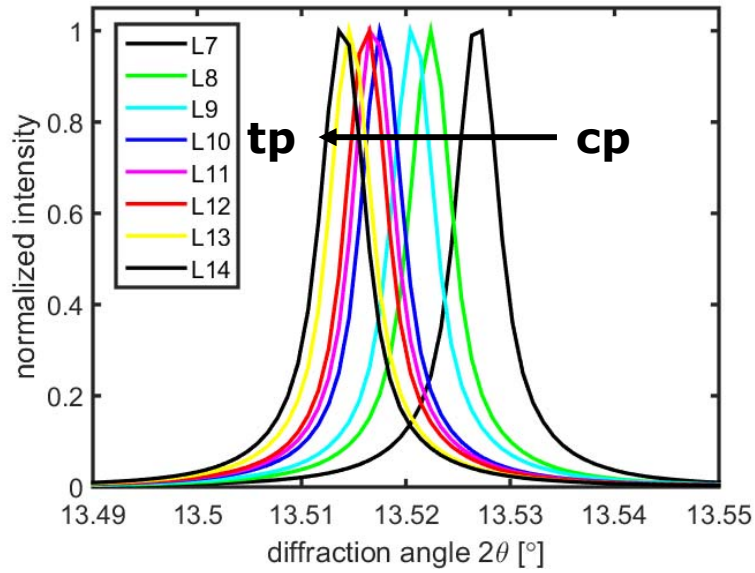
$$q = \frac{4\pi}{\lambda}\sin\theta$$

→ **Peak position**
elastic strain (average)
q_{mean}, q_{max},

→ **Peak width**
dislocation density
integral width, FWHM

→ **Asymmetry**
ordered structures
absolute, relative





Radial Profiles
Analysis

→ **Peak position**

q_{mean}, q_{max},
elastic strain (average)

$$q_{mean} = 2\pi / d_{400}$$

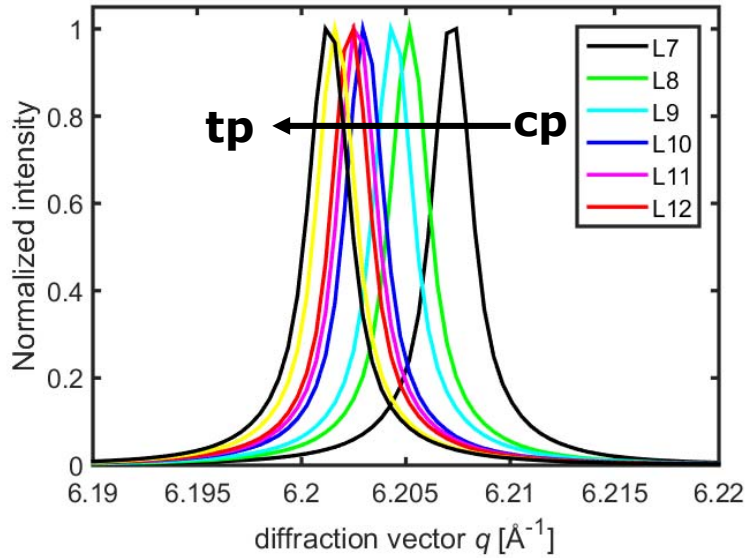
$$\varepsilon = \frac{d - d_0}{d_0} = \frac{q_{mean,L0}}{q_{mean}} - 1$$

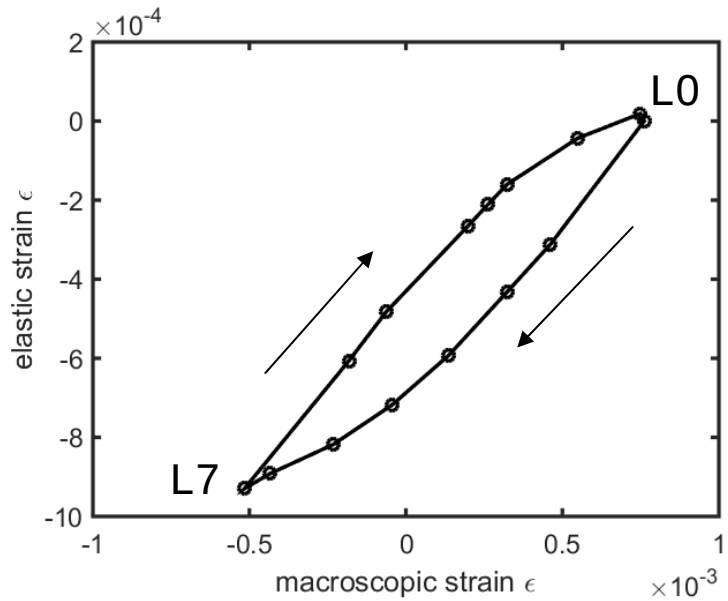
→ **Peak width**

integral width, FWHM

→ **Asymmetry**

absolute, relative

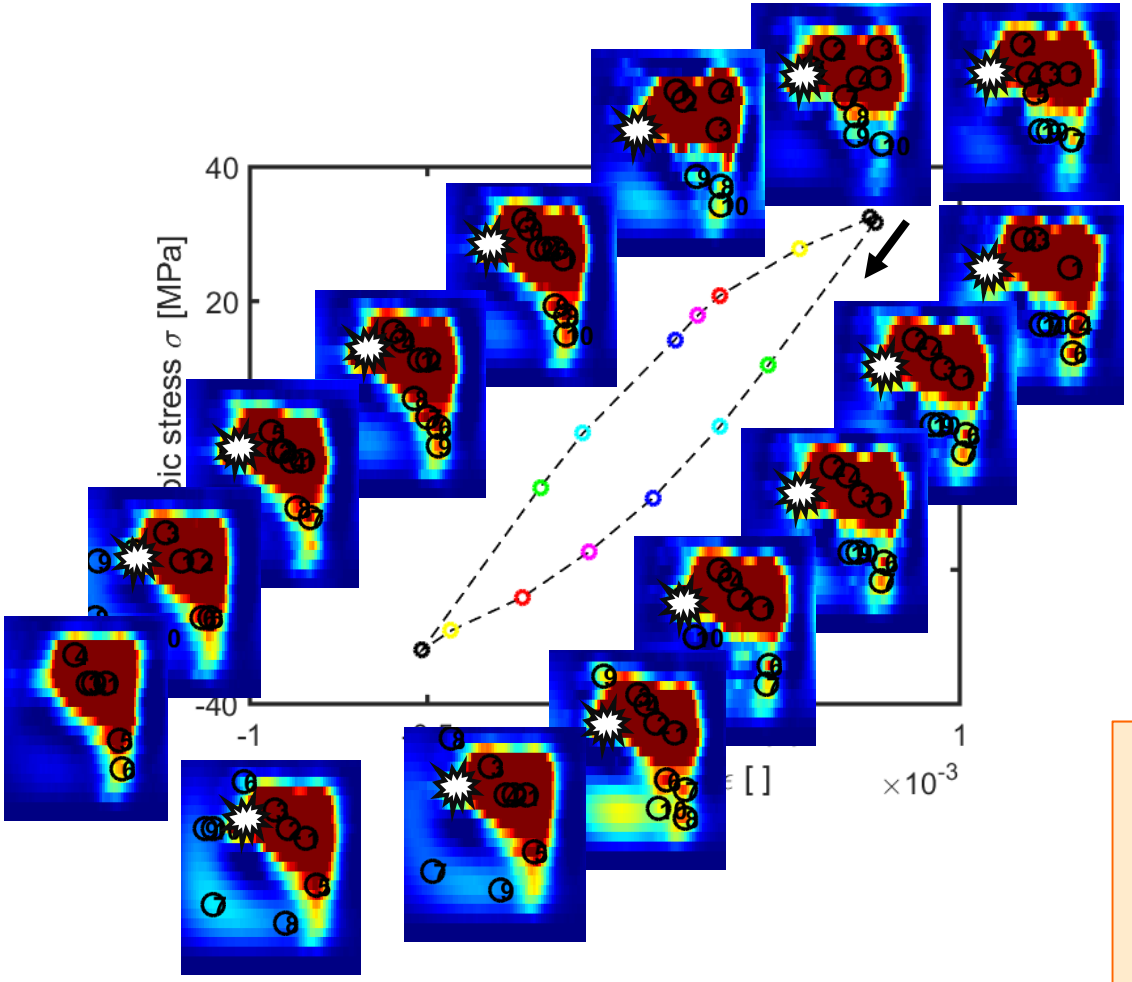




Peak position shifts
to lower q during tensile loading
and to higher q during compressive loading

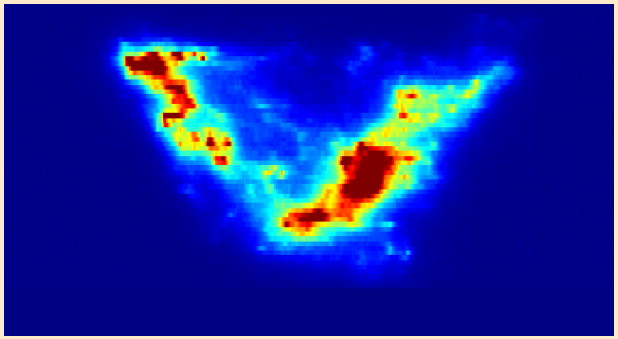
Identification of Subgrains

Radial Profiles



No predeformation

1% tensile predeformation



Radial profiles

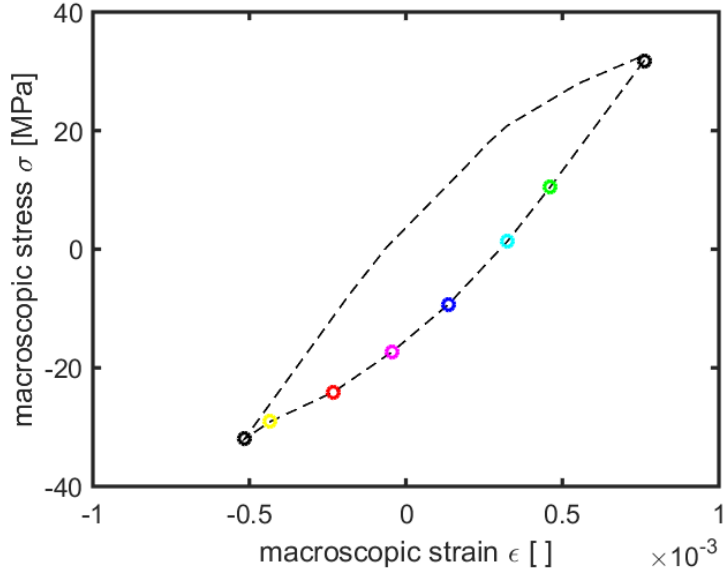
Subgrain

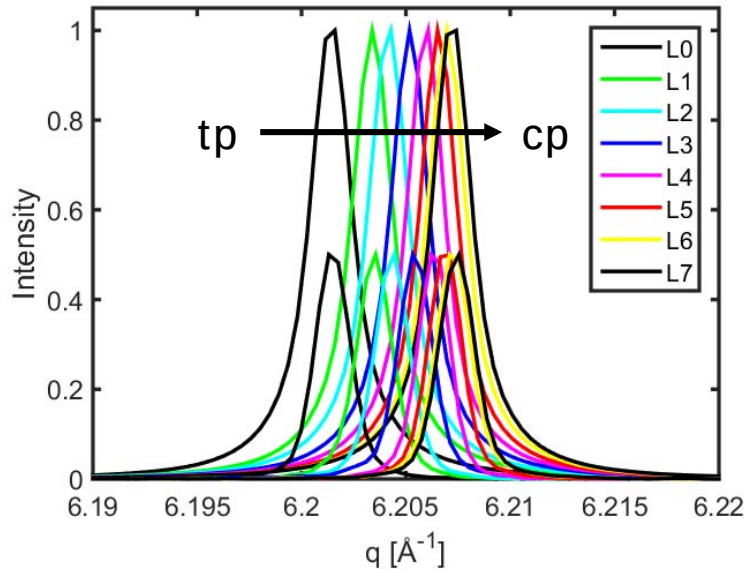
$$q = \frac{4\pi}{\lambda}\sin\theta$$

→ **Peak position**

q_{mean}

elastic strain





Radial profiles

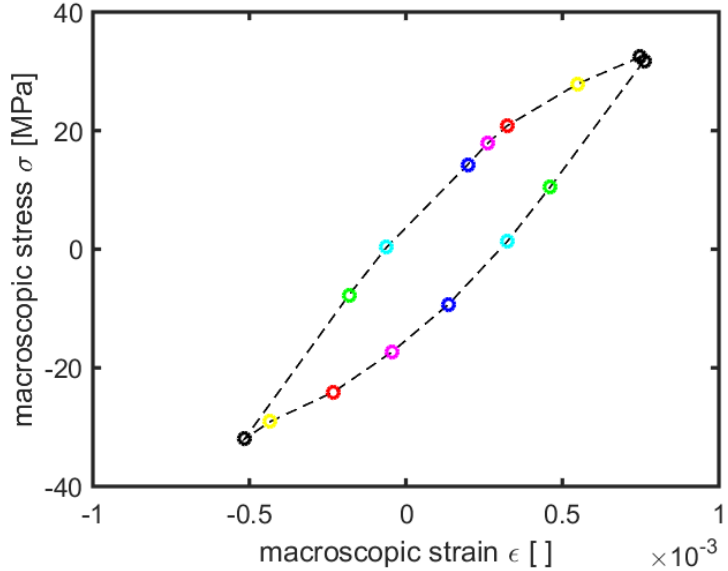
Subgrain

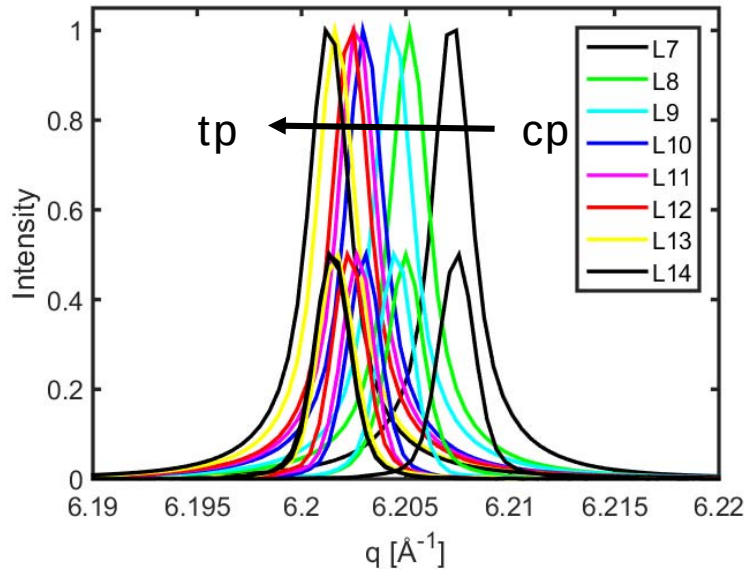
$$q = \frac{4\pi}{\lambda}\sin\theta$$

→ **Peak position**

q_{mean}

elastic strain





Summary and Outlook

- Various deformation regimes are possible, individual cycles (e.g. tension-tension, tension-compression)
- Follow individual grains and subgrains during a loading/cycling/ single cycles
- Identification of individual subgrains in selected grains

High Resolution Reciprocal Space Mapping as useful technique to follow microstructural evolution and local strains during cyclic deformation

- Extremely local behavior of grains and subgrains
- Leading to increased understanding and improved models for mechanical behavior and future design tools
- Application on other composite-materials (e.g. harmonically structured materials)