MAX-PLANCK-INSTITUT
FÜR EISENFORSCHUNG GmbH

LOCAL, ABSOLUTE RESIDUAL STRAIN MEASUREMENTS USING COMBINED RING-CORE MILLING AND CROSS-CORRELATION EBSD IN AN SEM

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Contents

- Residual stresses (RS) – why, what, how?
- Measurement of RS by cross-correlation CC-EBSD
- The measurement principles of ring core milling (RCM)
- Absolute measurement of RS by combination of CC EBSD and RCM
- Conclusions

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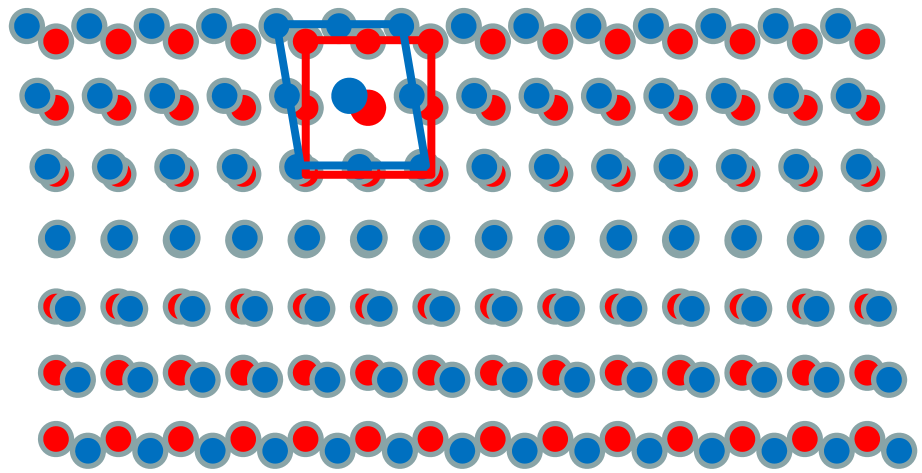
What are residual stresses?

- Elastic strains:
  - long range deviation of atom positions from their positions taken without presence of external forces while keeping their local neighborhood.

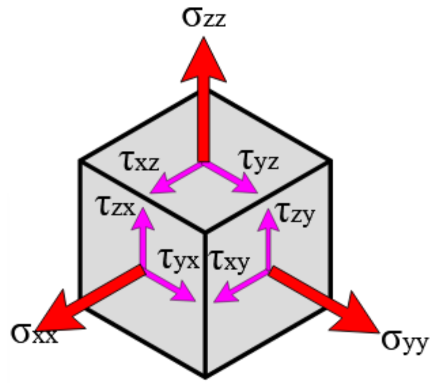


- Elastic stresses:
  - Forces that act to bring atoms back to their equilibrium positions according to Hook‘s law

Residual stresses, mathematical description

- Elastic strains:

$$\boldsymbol{\epsilon} = \begin{pmatrix} \epsilon_{11} & \epsilon_{12} & \epsilon_{13} \\ \epsilon_{21} & \epsilon_{22} & \epsilon_{23} \\ \epsilon_{31} & \epsilon_{32} & \epsilon_{33} \end{pmatrix}$$



- Hook‘s law:

$$\boldsymbol{\sigma} = \boldsymbol{E\epsilon}$$

- Elastic stresses:

$$\boldsymbol{\sigma} = \begin{pmatrix} \sigma_{11} & \sigma_{12} & \sigma_{13} \\ \sigma_{21} & \sigma_{22} & \sigma_{23} \\ \sigma_{31} & \sigma_{32} & \sigma_{33} \end{pmatrix}$$

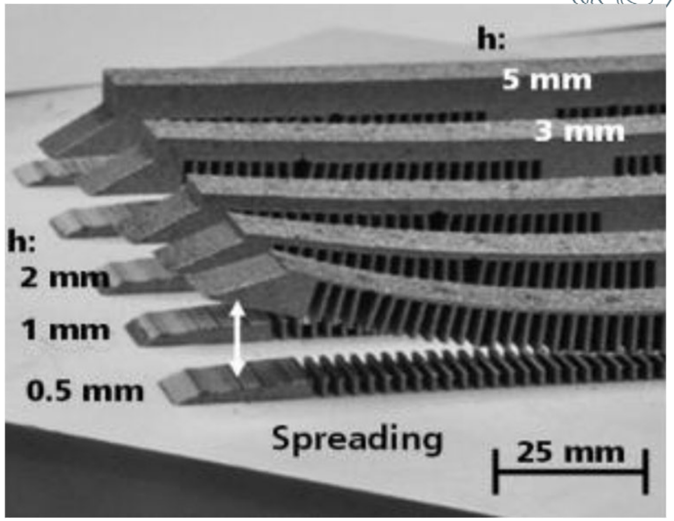
normal stresses
deviatoric stresses

- von-Mises equivalent stress (1 represents all)

$$\sigma_{vM} = \sqrt{\sigma_{11}^2 + \sigma_{22}^2 + \sigma_{33}^2 - \sigma_{11}\sigma_{22} - \sigma_{11}\sigma_{33} - \sigma_{22}\sigma_{33} + 3(\sigma_{12}^2 + \sigma_{13}^2 + \sigma_{23}^2)}$$

Residual stresses – why care?

- Shape accuracy during/after mechanical forming or additive manufacturing
- Fracture nucleation (e.g. in martensitic structures)
- Crack growth
- Mechanical properties (e.g. Bauschinger effect)
- Stress corrosion cracking
- Hydrogen embrittlement
- Electronic effects

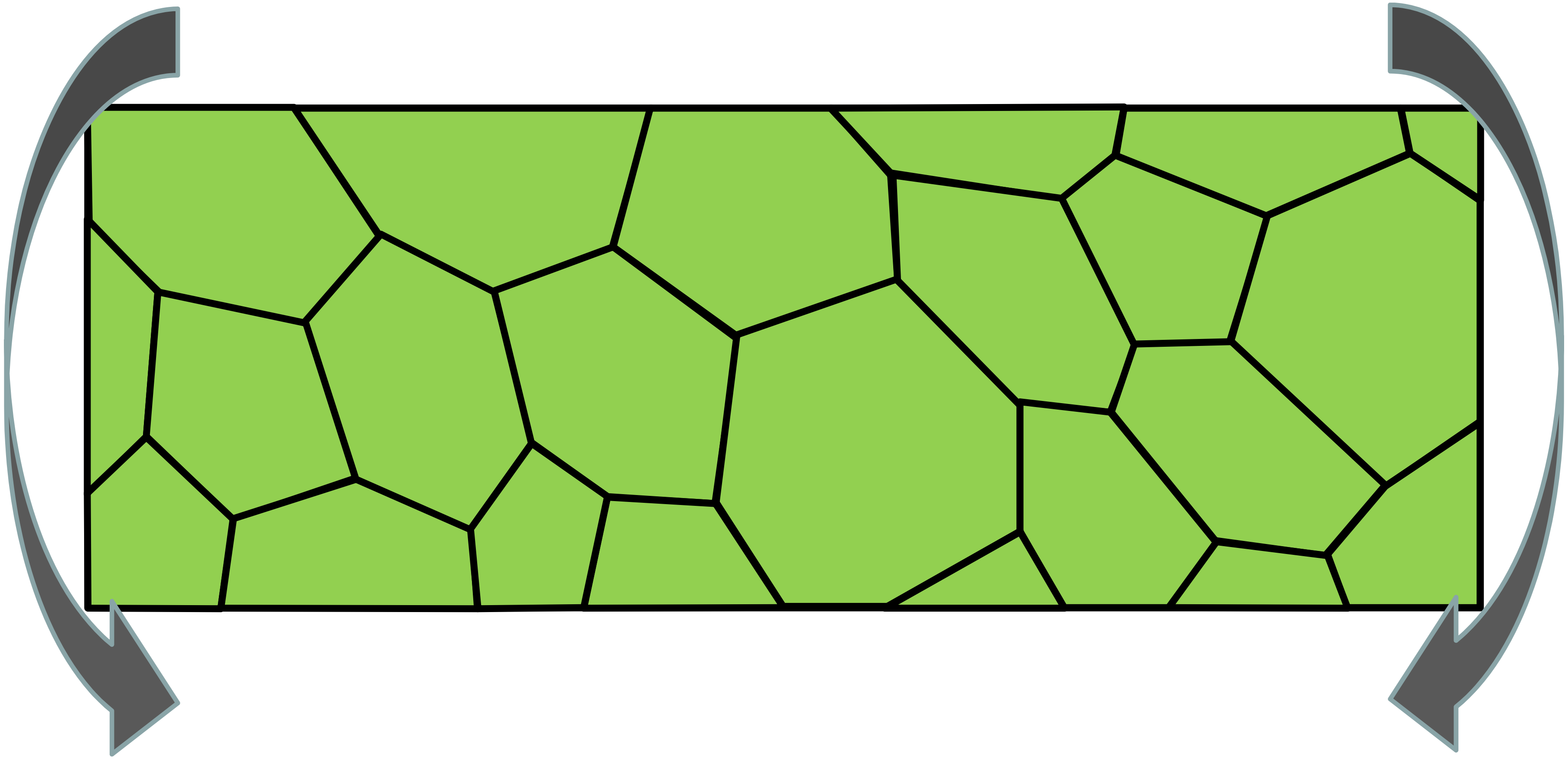


C. Li et al. Procedia CIRP 71 (2018) 348–353

S. Papula et al., Met. Mat. Trans. A 45 (2014), 5270
Review by Koyama et al: Int. J. Hydrogen Energy 42 (2017) 12706

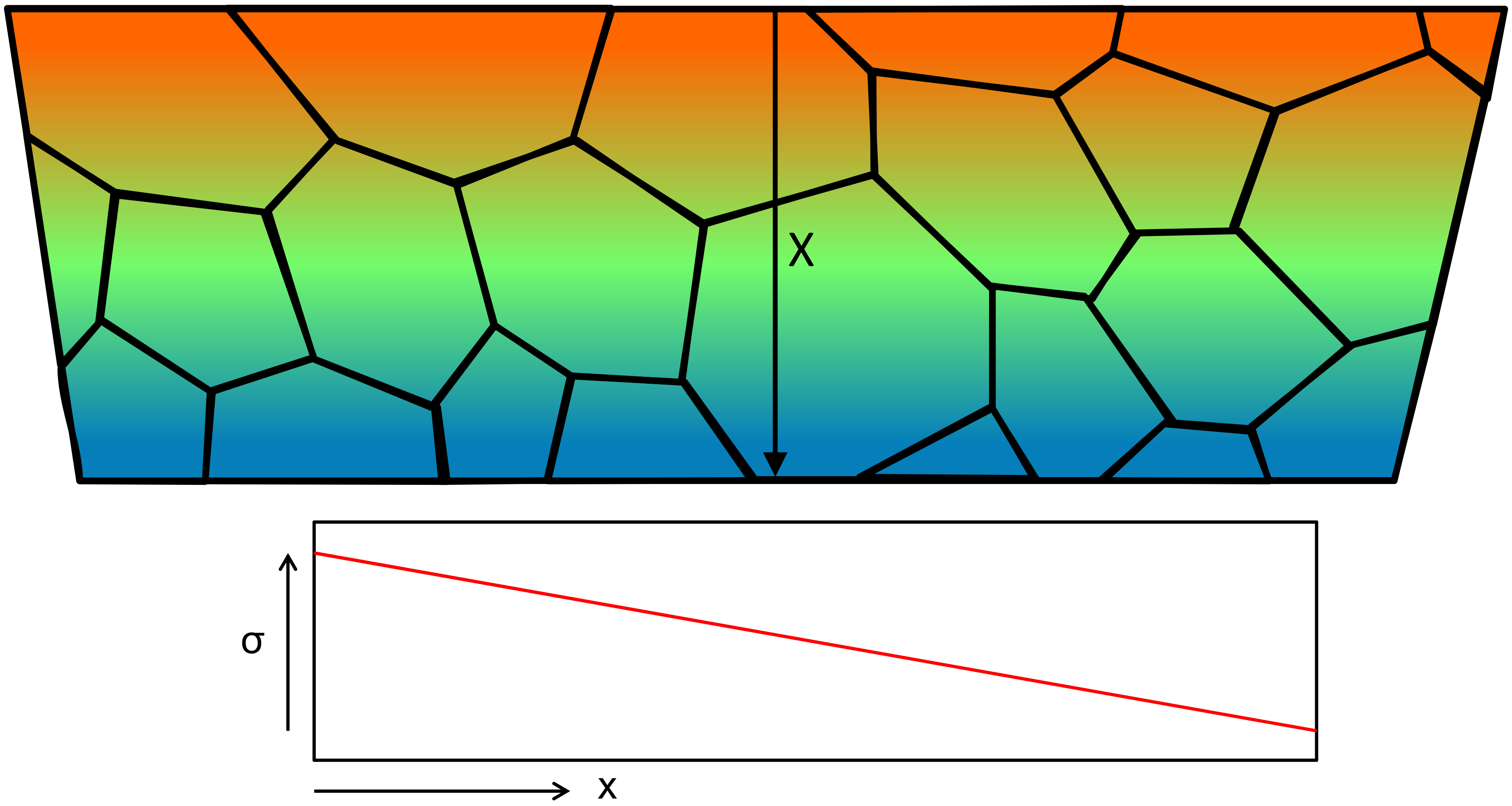
Classification of residual stresses

Stress-free



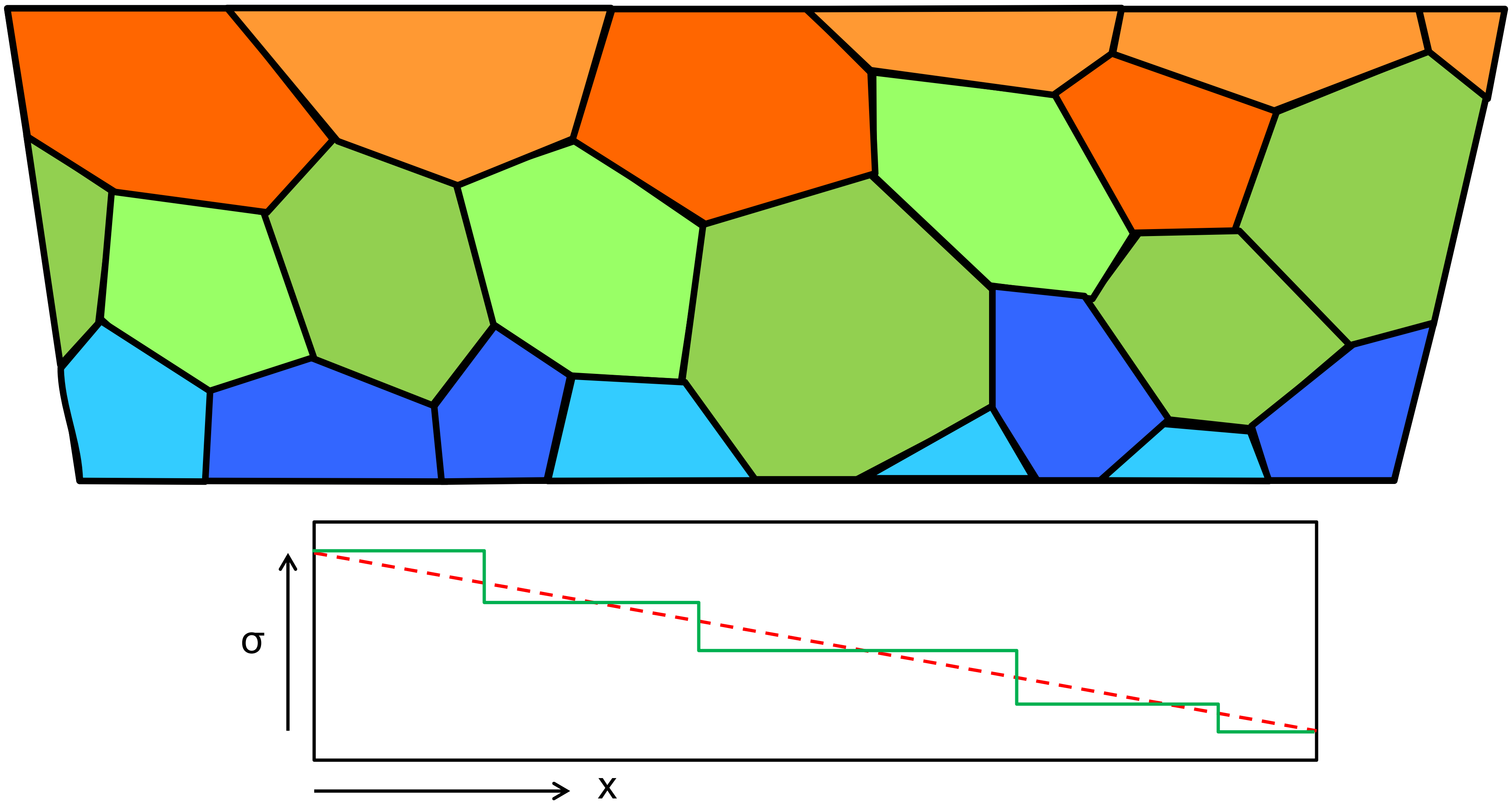
Classification of residual stresses

Stresses of 1st kind (macroscopic stresses)



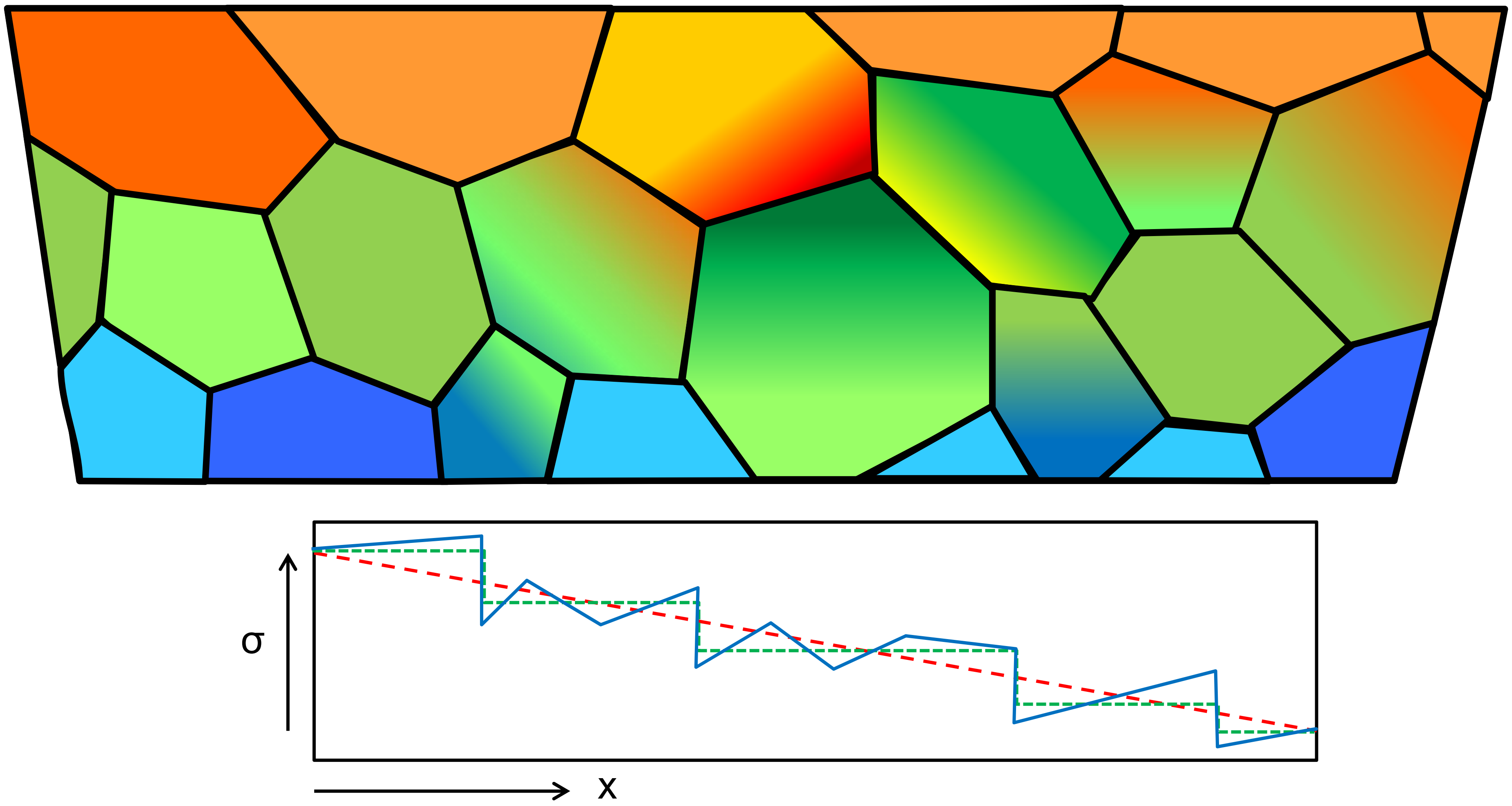
Classification of residual stresses

Stresses of 2nd kind (grain-to-grain stresses)



Classification of residual stresses

Stresses of 3rd kind (grain-internal stresses)



Measurement of residual stresses

Type	Techniques	Measured parameter	Measured quantities	Spatial resolution	Accuracy (limited by)	Type of evaluated stress	Comments
Mechan-ical	Beam deflection	Distortion caused by stress relaxation	Beam deflection vs. coating thickness	0.05 of thickness; no lateral resolution	Limited by minimum measurable curvature	Type 1	Measure in-plane stresses
	Slit / Hole drilling		Released strain	50 µm in depth	± 50 MPa (reduced sensitivity with increasing depth)		
	Ring core milling			**1 µm**	**10^{-3} ??**	Types 1, 2	
Diffrac-tion-based	X-rays $\sin^2 \psi$	Atomic strain gauge	Diffraction angles	1 mm laterally, 20 µm in depth	± 20 MPa (texture, surface condition	Peak shifts: types 1, 2	Sensitive to surface preparation
	Hard X-rays			20 µm lateral to incident beam; 1 mm parallel to beam	± 10^{-5} strain (grain sampling statistics)	Peak widths: types 2, 3	spotty powder patterns
	Neutrons			500 µm	± 5×10^{-5} strain (counting statistics and reliability of stress free ref.)	Types 1, 2	Difficult to access, costly
	Electrons (EBSD)			100 nm	10^{-4}	Type 3	
Ultraso-nic	Acousto-elasticity	Stress related changes in elastic wave velocity	Wave velocities	5 mm	10%	Types 1, 2, 3	Microstructu re sensitive
Magne-tic	Barkhausen noise	Variations in magnetic domains with stress	Magnetic Barkhausen noise	1 mm	10%	Types 1, 2, 3	

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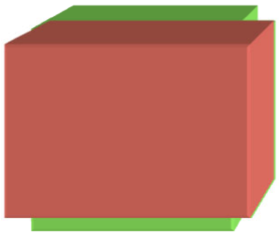
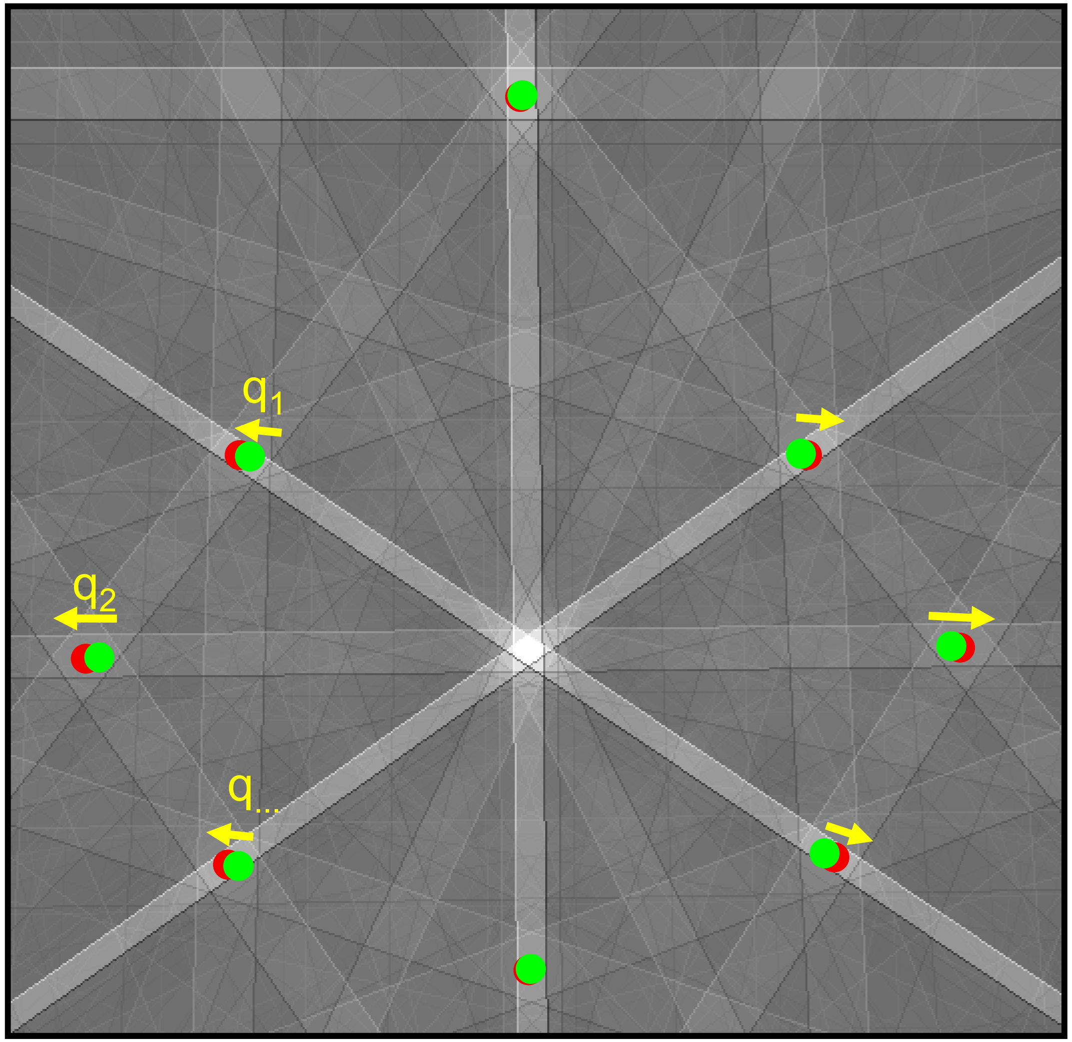
Cross-correlation EBSD pattern analysis

Basic idea:

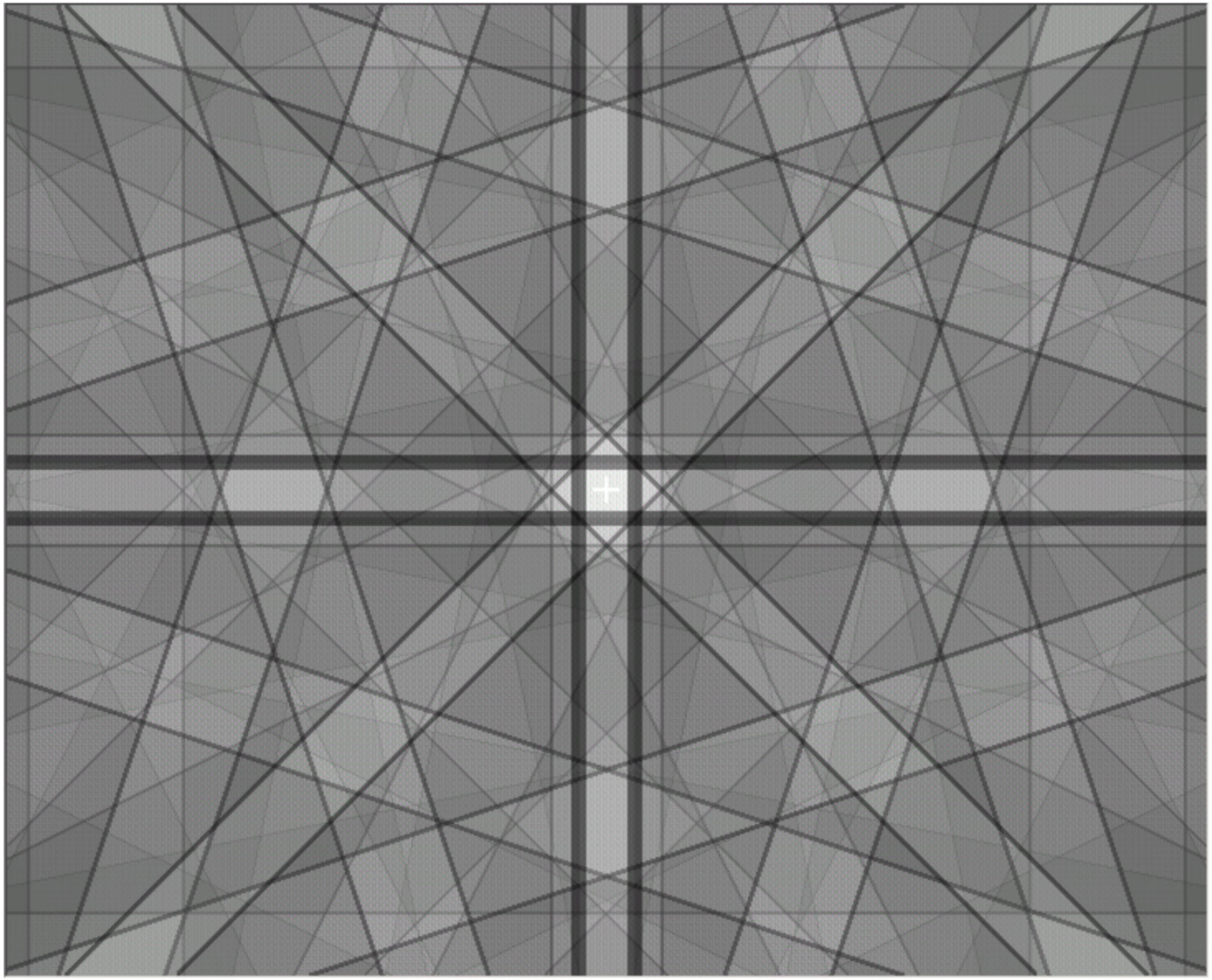
- Crystal lattice and diffraction patterns get distorted by the action of an external stress
- Note: hydrostatic part of stress tensor only changes width of Kikuchi bands → cannot be detected accurately
- only deviatoric part of stresstensor can be measured





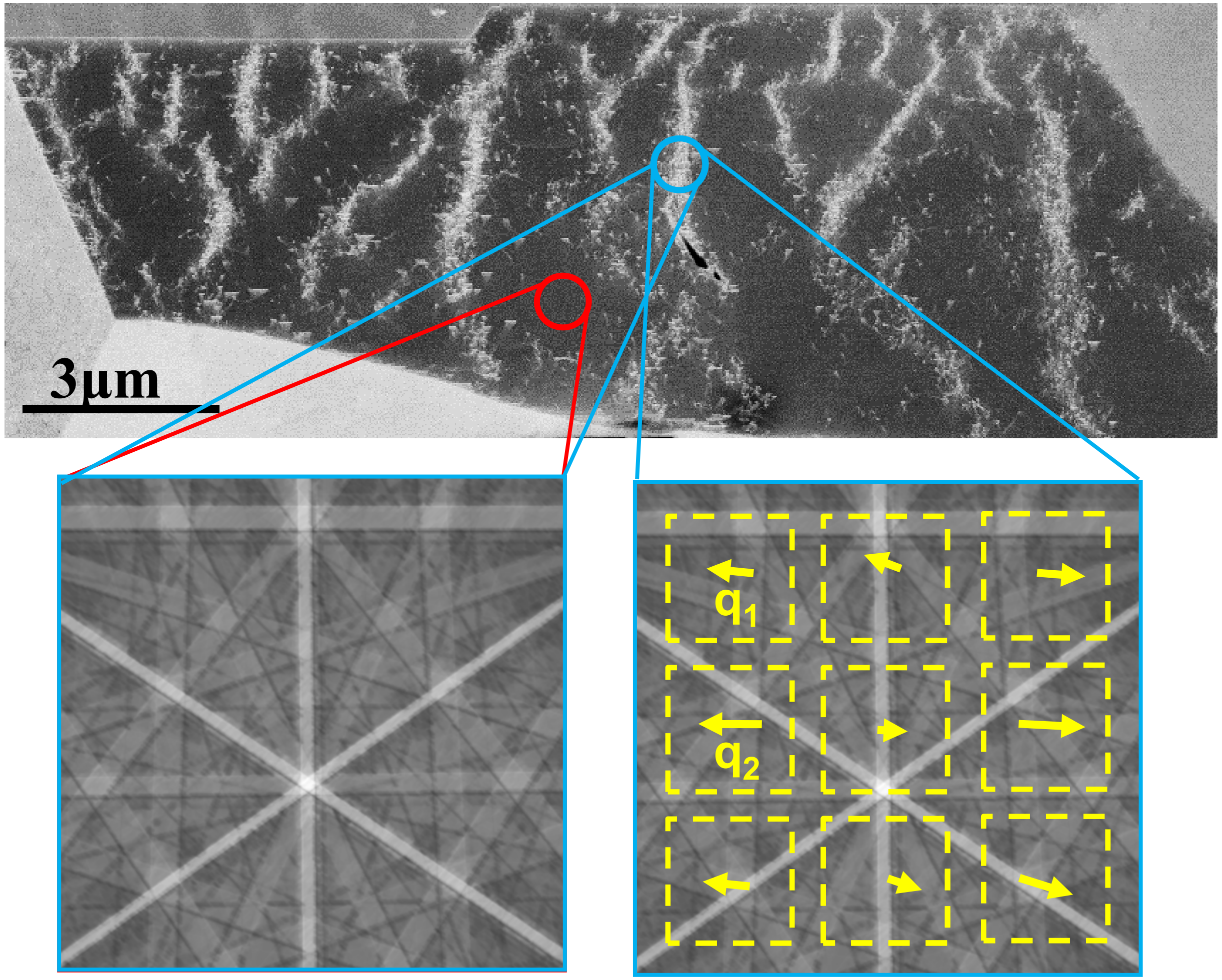
e.g. Wilkinson, Meaden, Dingley, Ultramicroscopy 106 (2006) 307

Determination of elastic strain by EBSD



Example: 0.2% of lattice strain (simulated on an fcc lattice)

Cross-correlation EBSD for stress and strain measurement



Calculation of displacement gradient tensor **A** from measured shift vectors **q***

$$\mathbf{A} = \begin{pmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{pmatrix}$$

elastic stretches
(symmetric part)

$$\boldsymbol{\varepsilon}_{shear} = \tfrac{1}{2}\left(\mathbf{A} + \mathbf{A}^{t}\right)$$

elastic stresses: **σ = E ε**

crystal rotations
(anti-symmetric part)

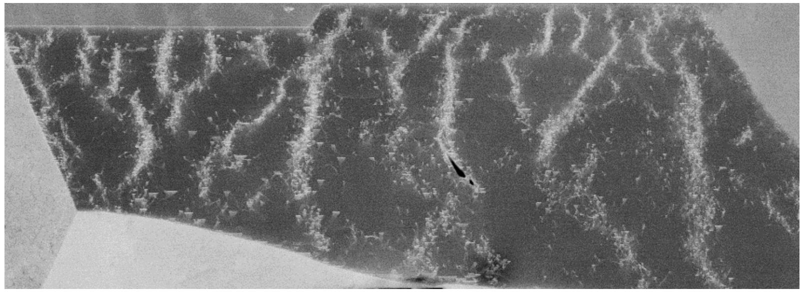
$$\boldsymbol{\omega}_{rotation} = \tfrac{1}{2}\left(\mathbf{A} - \mathbf{A}^{t}\right)$$

Accuracy:

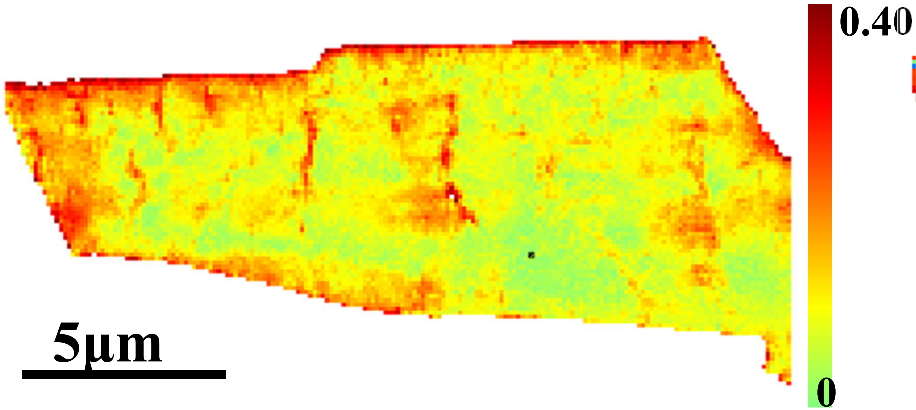
Rotations $\Delta\omega = 0.03°$, Distortions: $\Delta\varepsilon = 5 \times 10^{-4}$

Stresses: $\Delta\sigma_{ij} = \pm 12$ MPa (for TWIP steels)

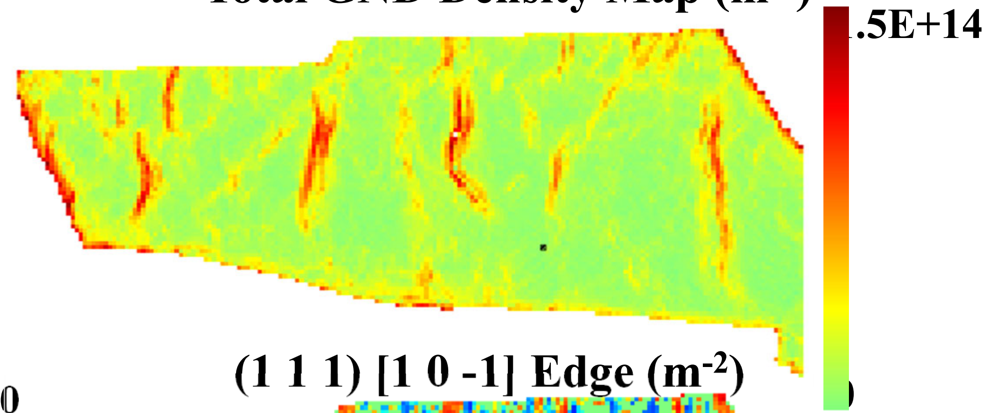
Stresses and GND densities



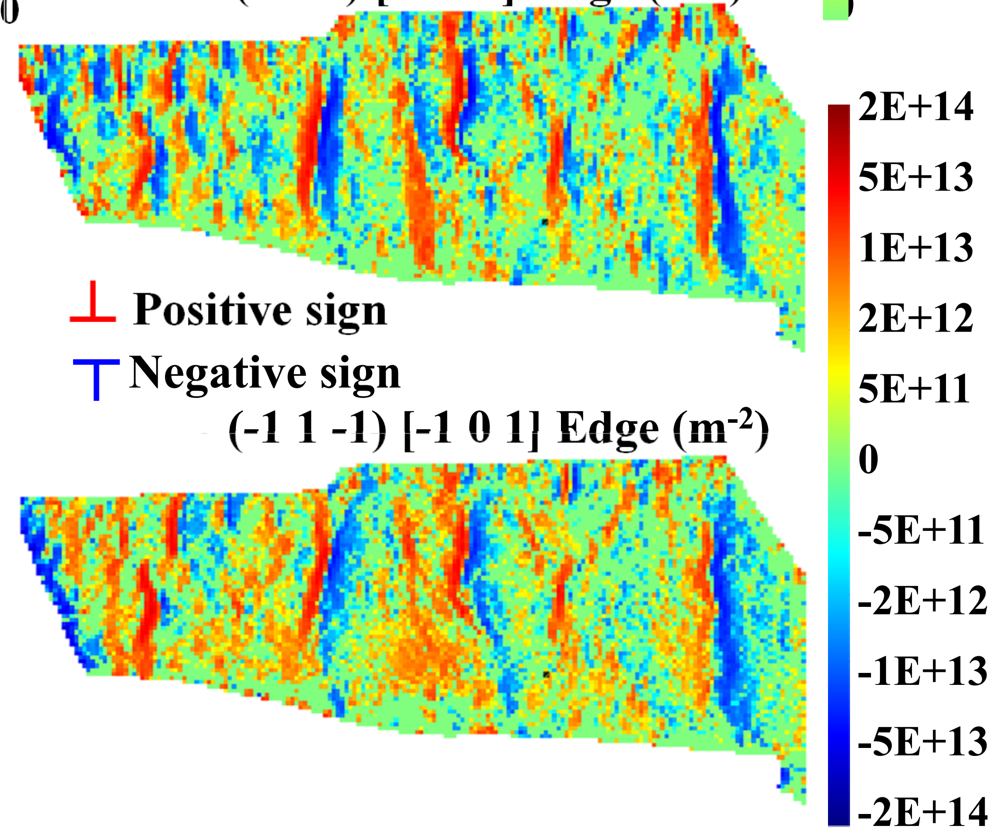
Von Mises stress [GPa]



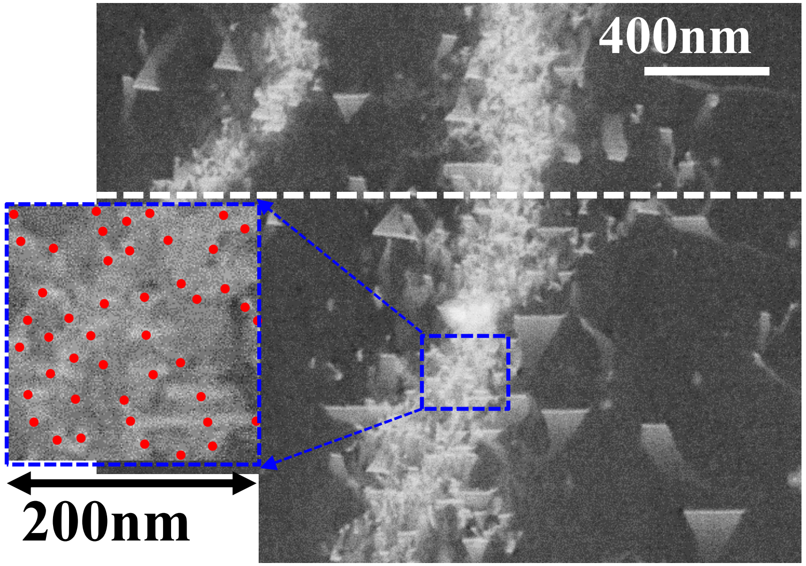
Total GND Density Map (m^{-2})

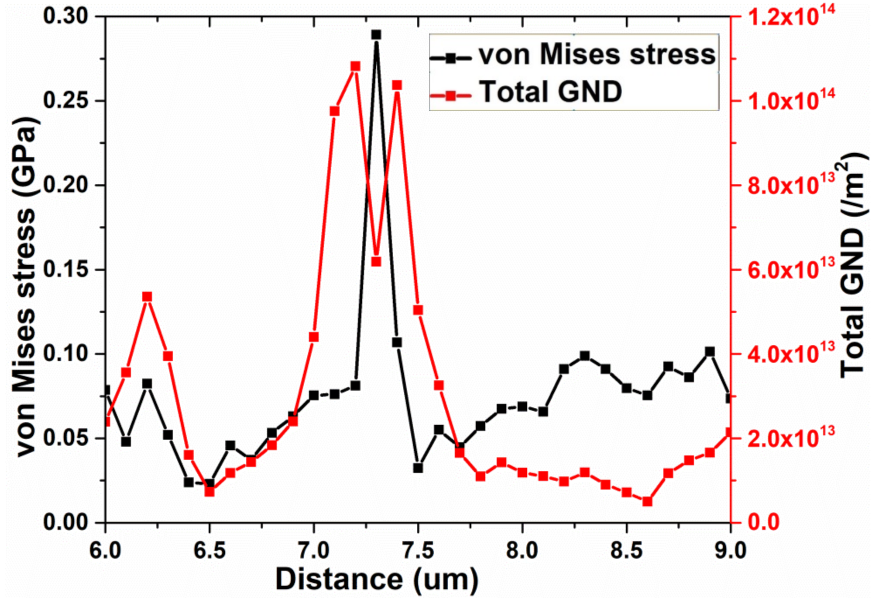


(1 1 1) [1 0 -1] Edge (m^{-2})



Dislocation wall structure (EBSD & ECCI)



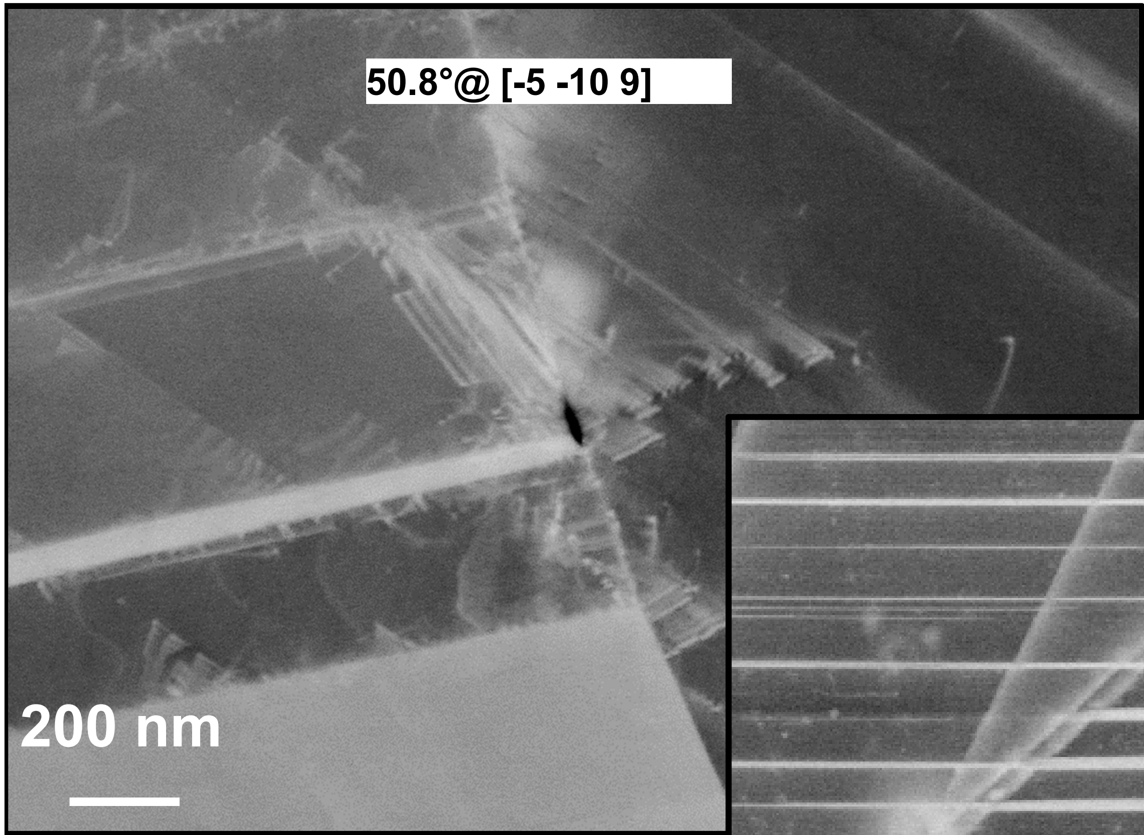


Dislocation densities:

ρ_{total} = 1.13 10^{15} m^{-2}
ρ_{GND} = 1.17 10^{14} m^{-2} ⇨ **ρ_{total} = 10 x ρ_{GND} !**

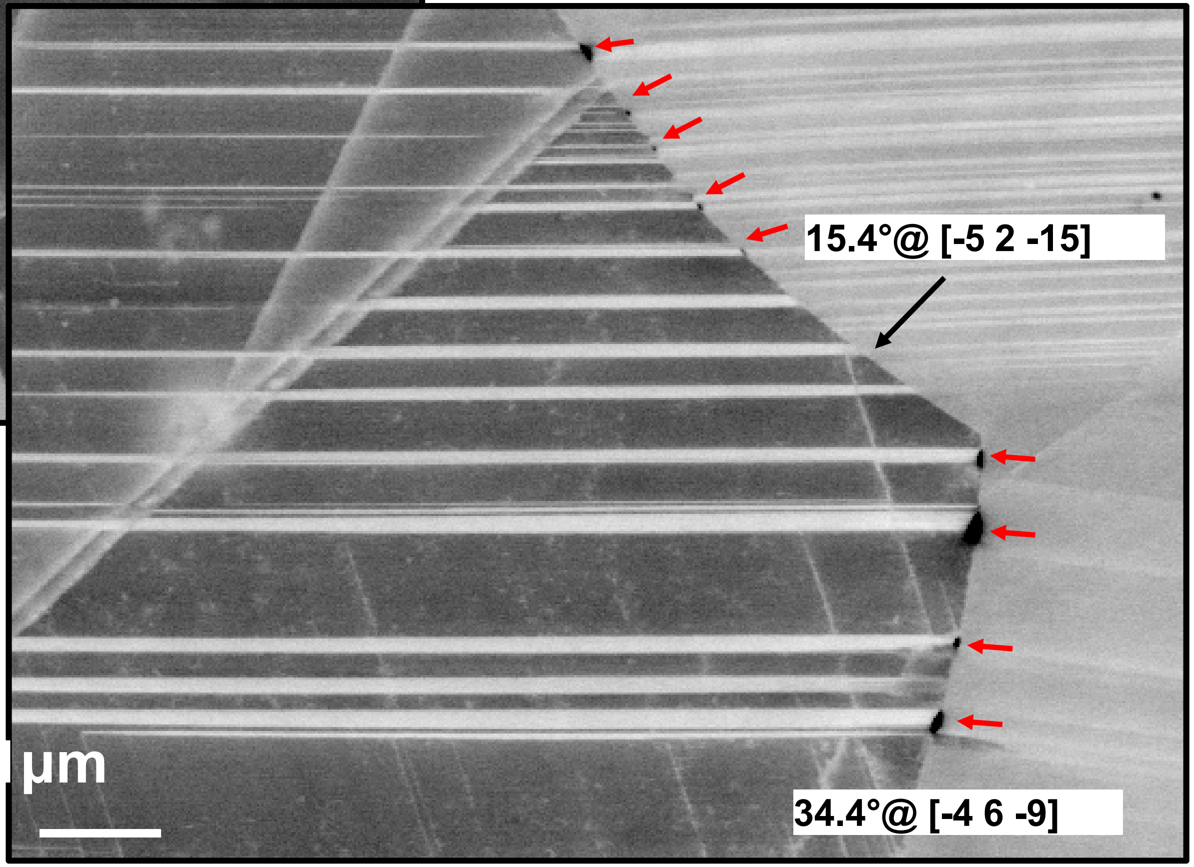
The main problem with CC EBSD: a reference pattern from same grain is needed, only strains of 3rd kind can be determined

Crack formation at grain boundaries



5 cycles, ε_a=1.1%

50 cycles, ε_a=0.9%



CC EBSD: pros and cons

- Mapping technique, potentially with high spatial resolution (EBSD: ~50...500 nm)
- Good strain accuracy (~10^{-4})
- Reasonably high rate of measurement (~1 Hz)
- In grains with larger misorientations (> 3°): use pattern remapping
- Only measurements of 3rd kind residual stresses
- Results usually in relatively large values → where do they come from?
- Which reference point should be selected?

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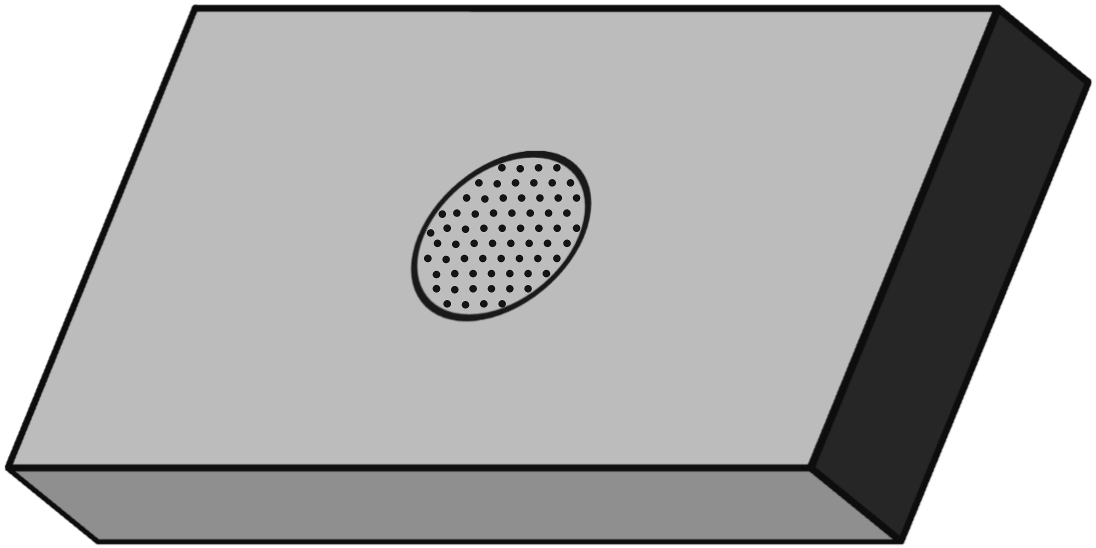
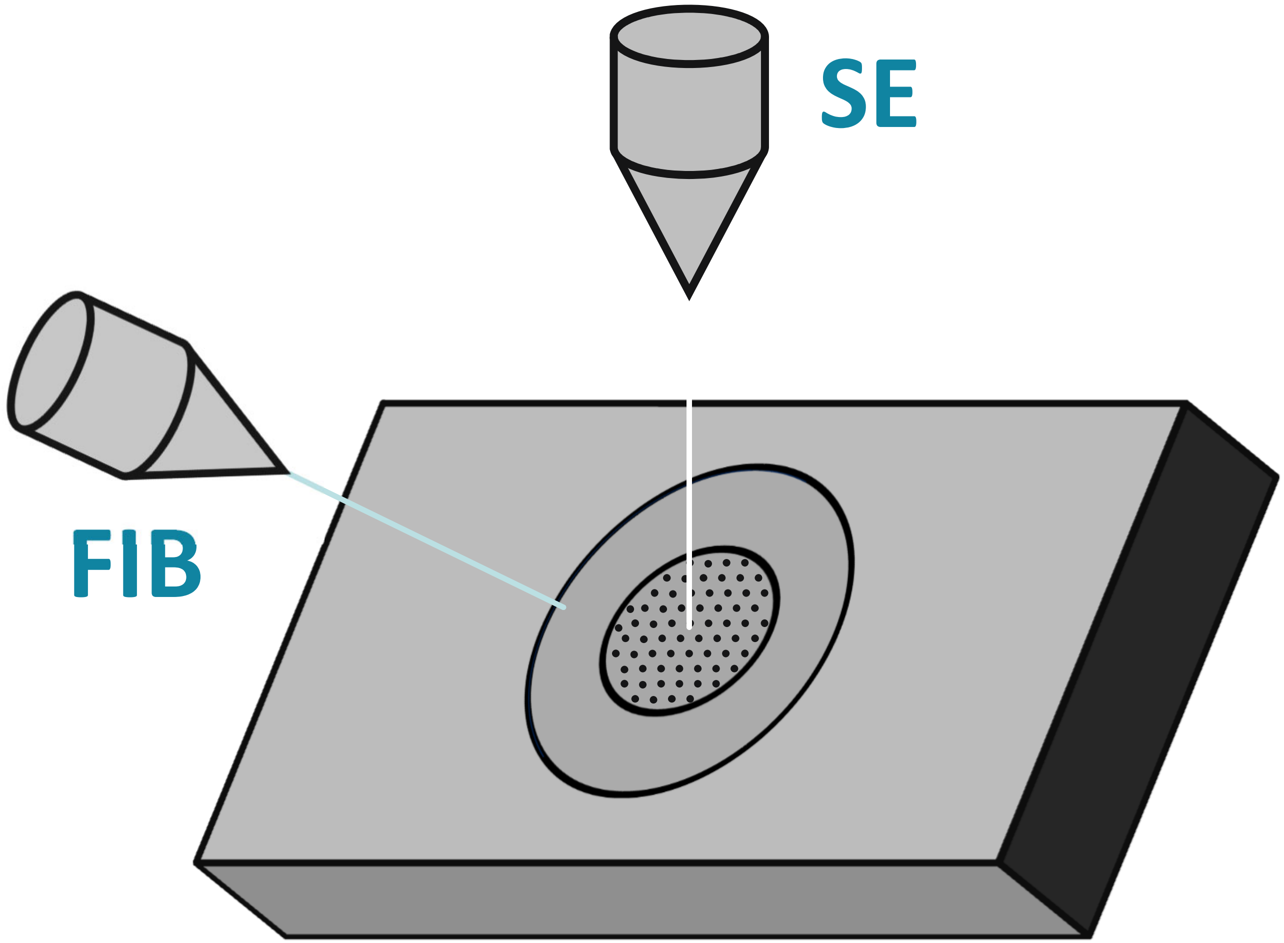
Principles of ring-core milling

Residual stress measurement by mechanical relaxation:
- cut-free a selected area and measure relaxation strains
- enables absolute stresss measurements also in heavily defected microstructures
- high spatial resolution if done by FIB sputtering



A. M. Korsunsky et al., Surface & Coatings Technology 205 (2010) 2393–2403
M. Sebastiani et al, Materials and Design, vol. 118, pp. 204-206, 2017.

Principles of ring-core milling



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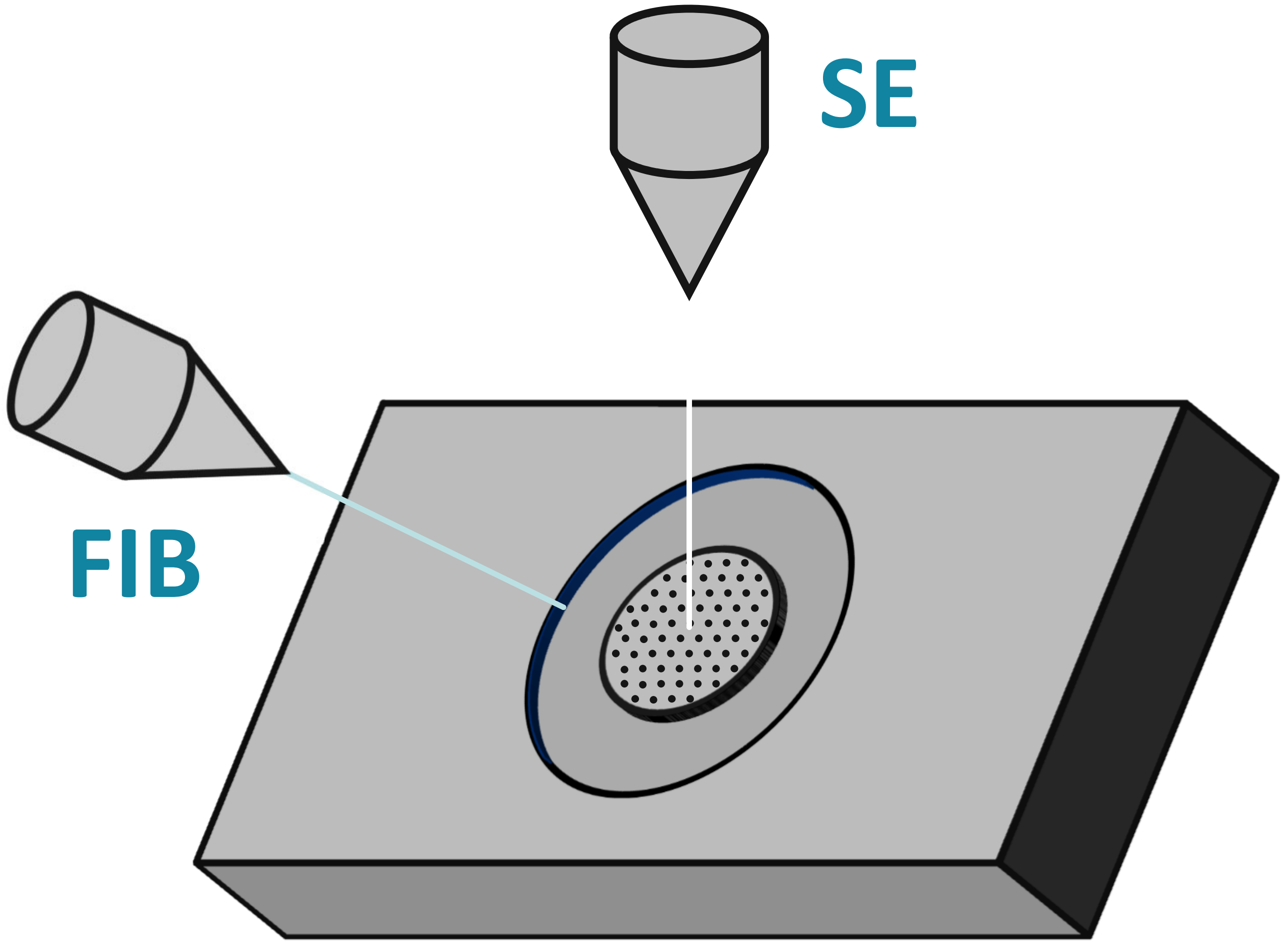

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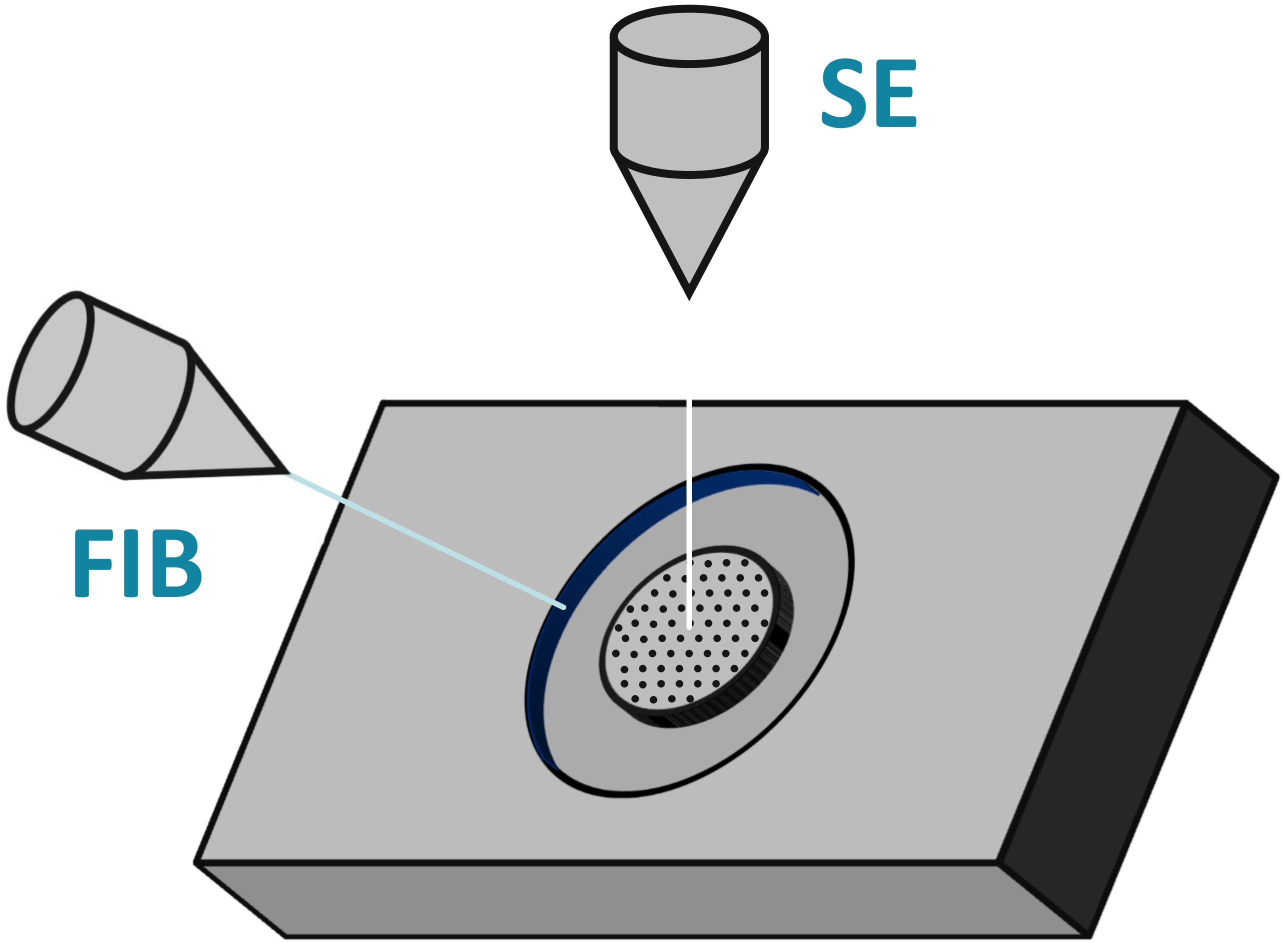

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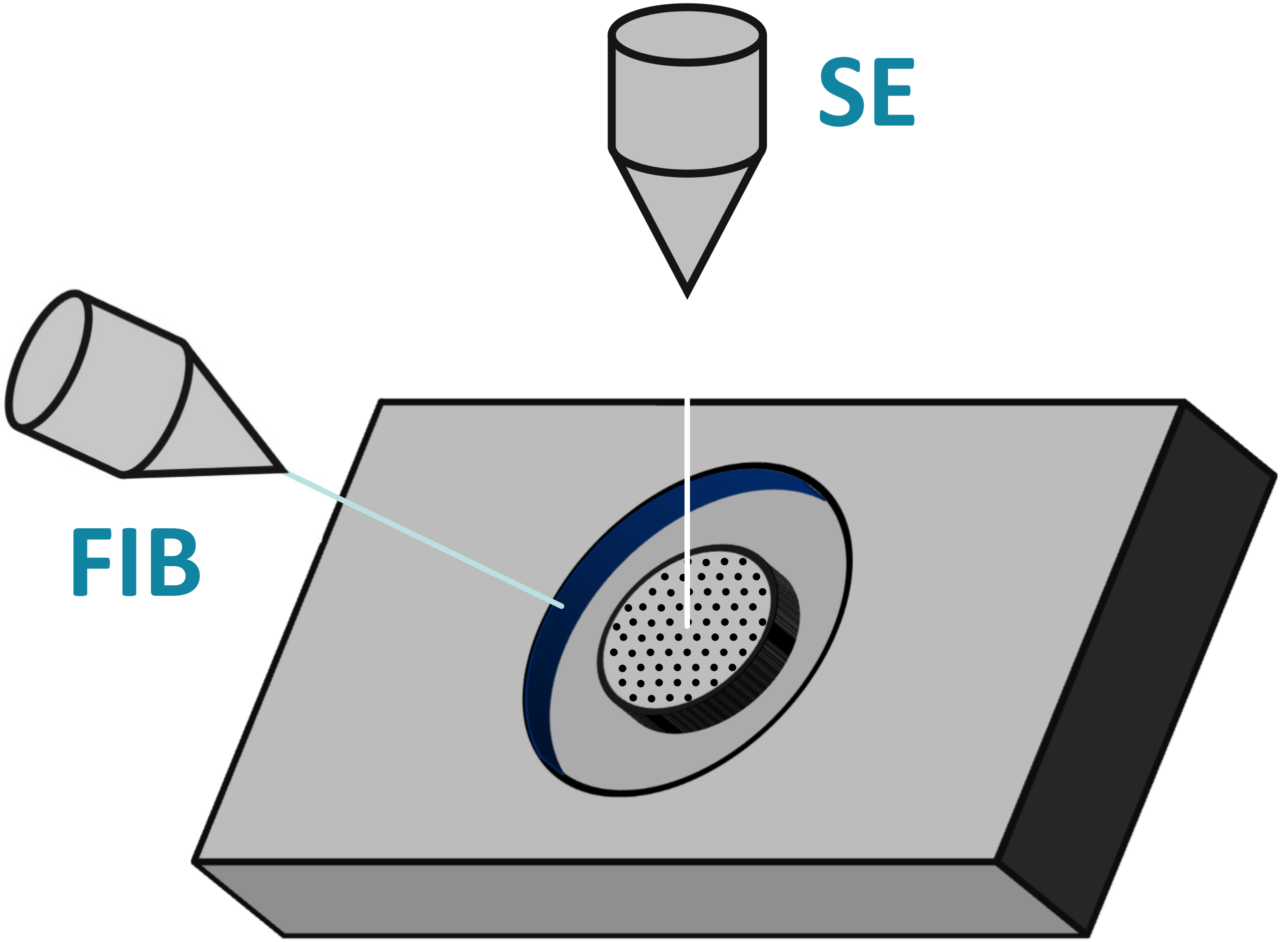
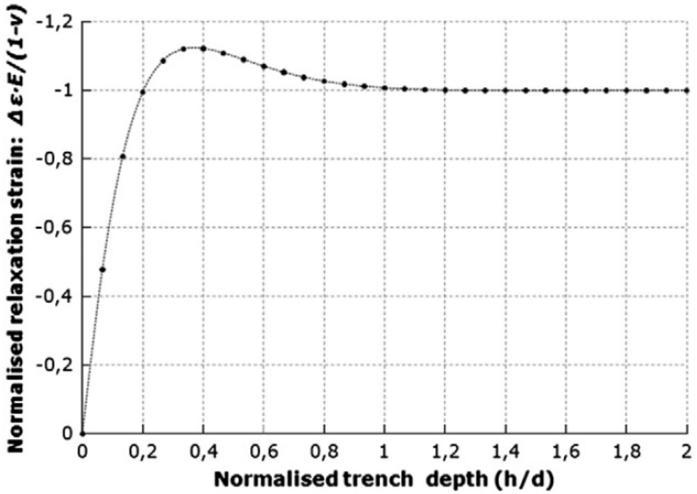
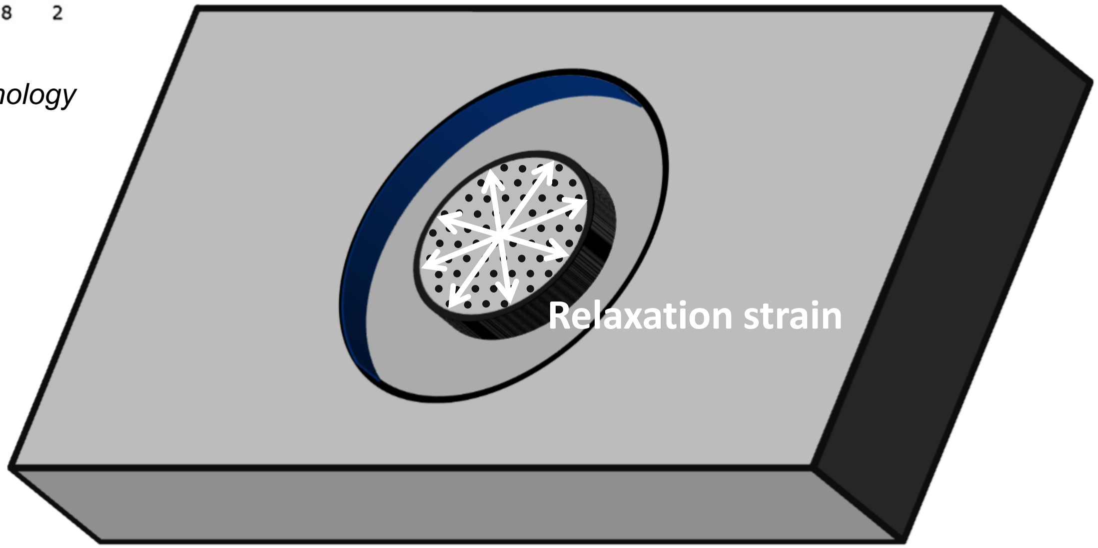


Principles of ring-core milling

Modelling by continuum mechancis

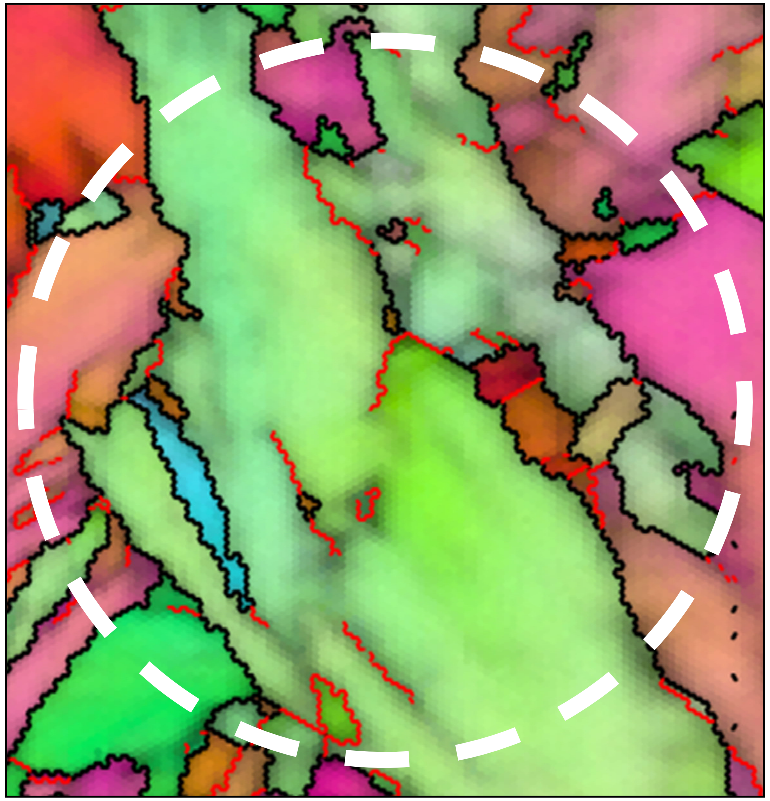


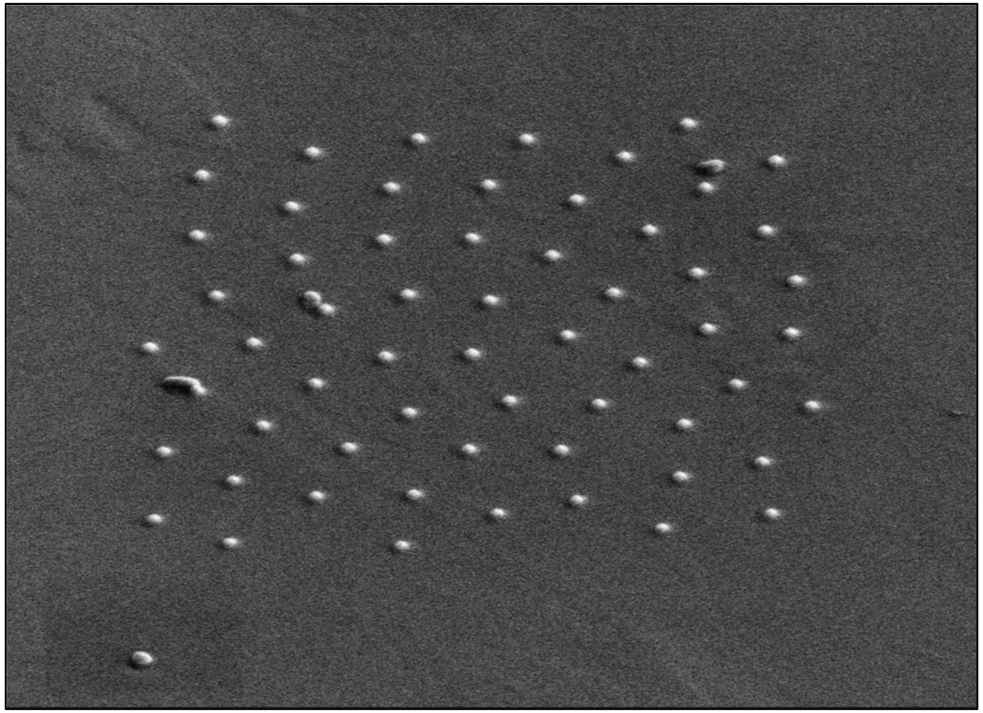
A. M. Korsunsky et al., Surface & Coatings Technology 205 (2010) 2393–2403



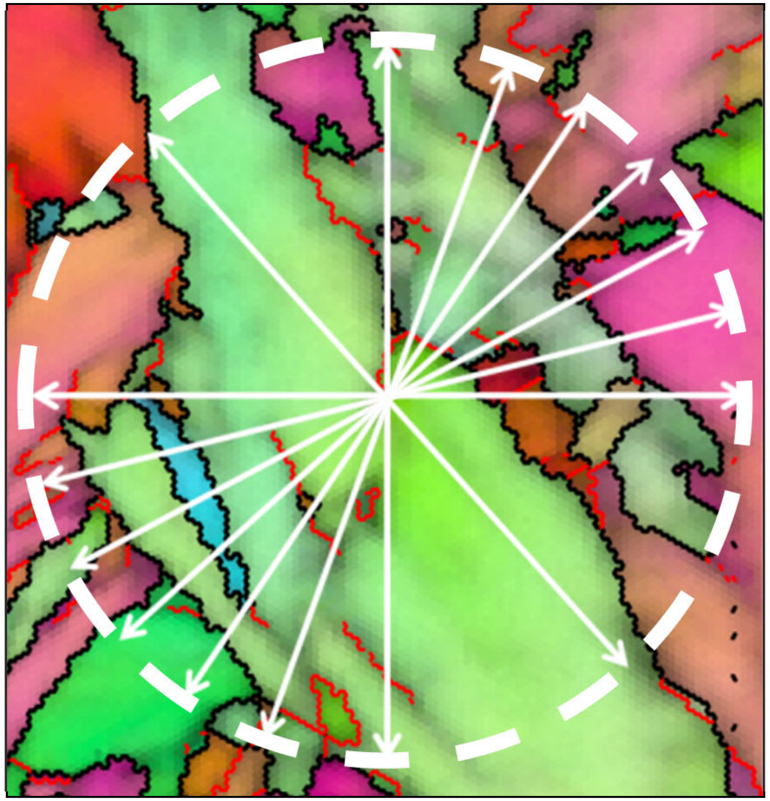
Full relaxation achieved when ring diameter equal ring depth!

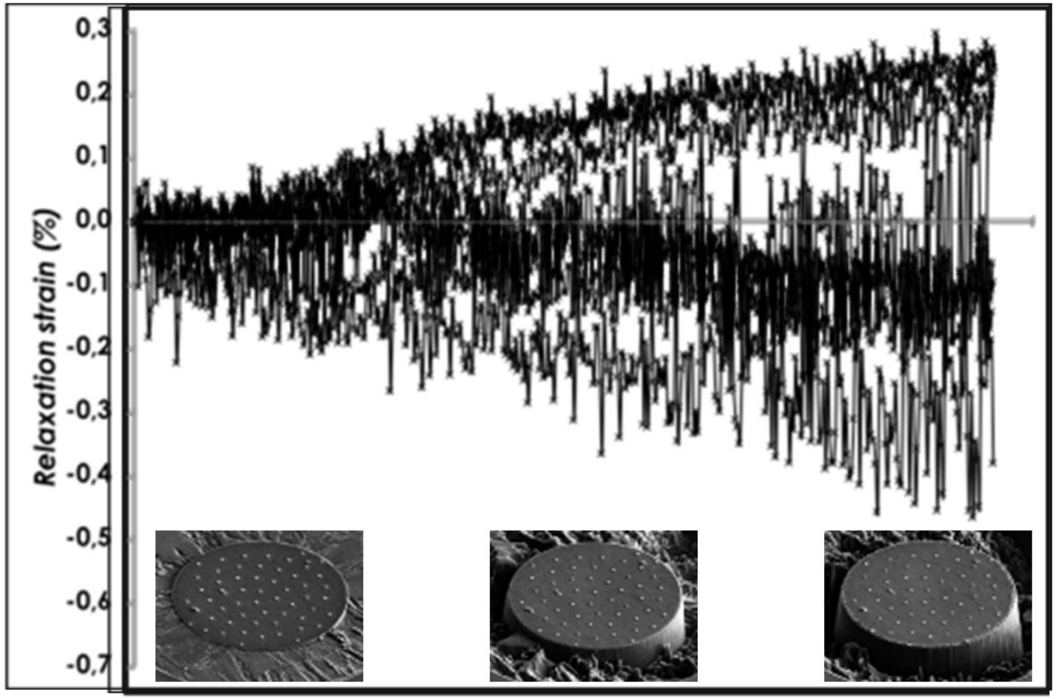
Measurement of relaxation strain



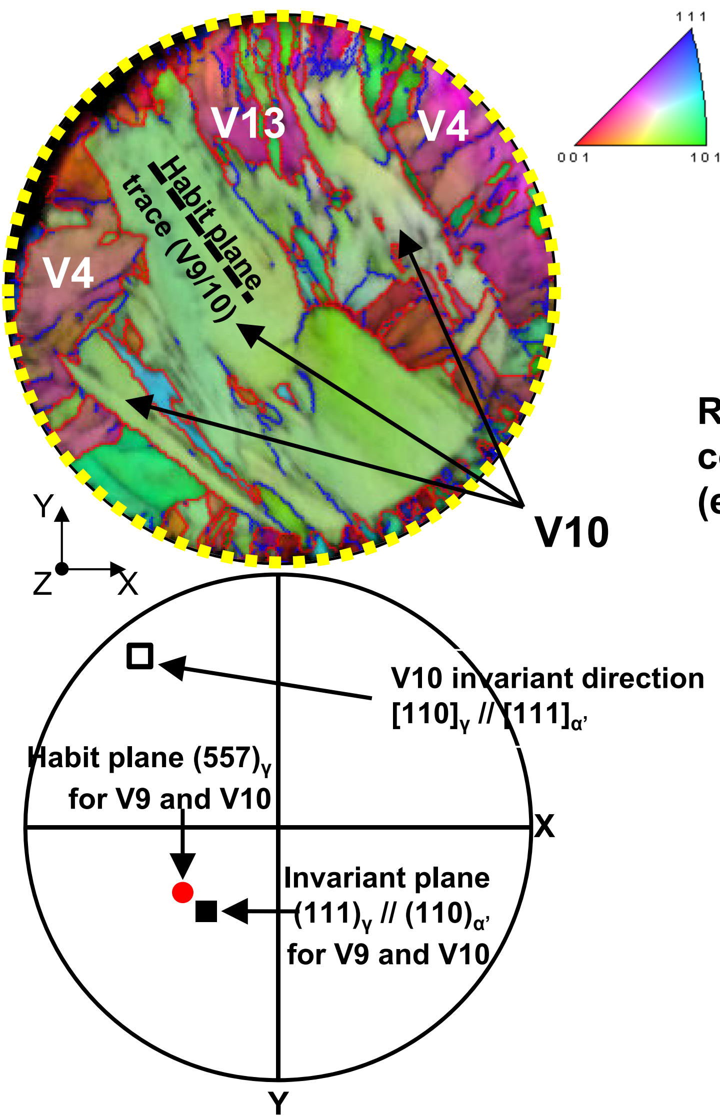


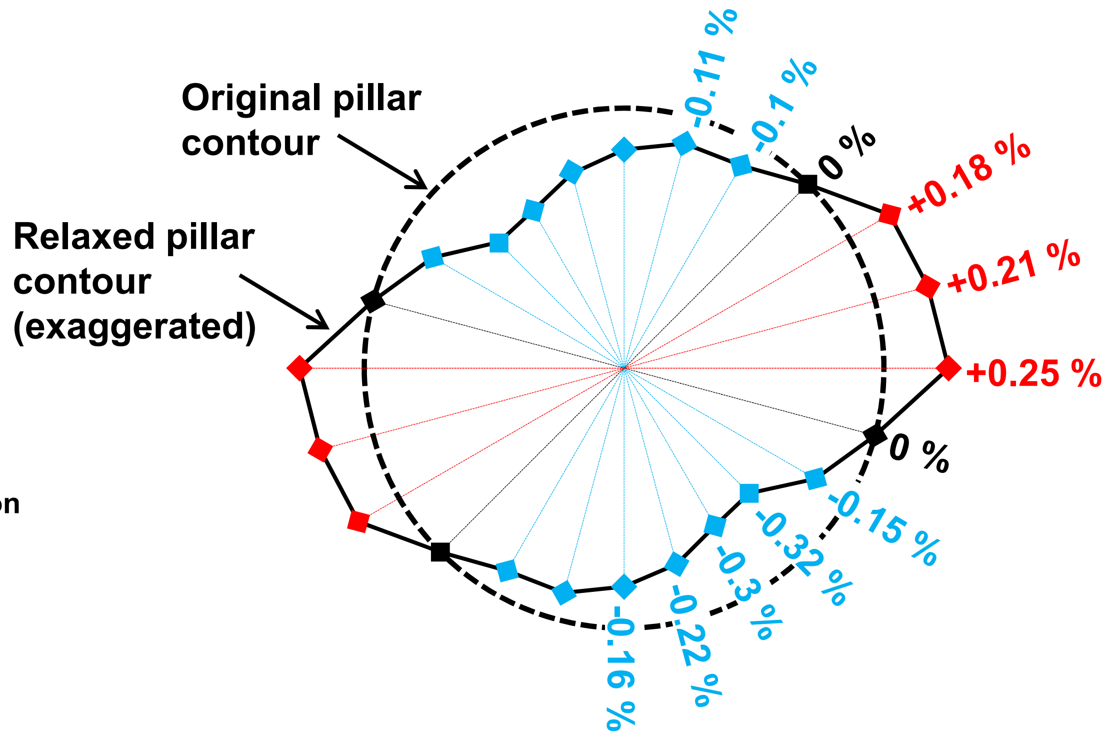
Anisotropy of strain relaxations





Strain relaxation and crystallography





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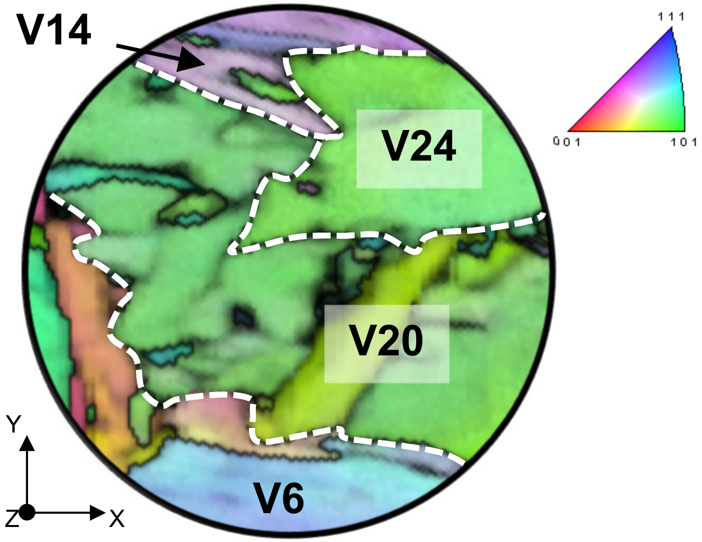
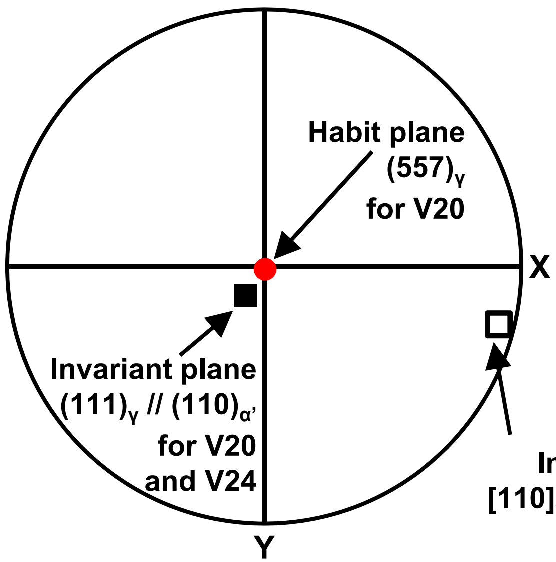
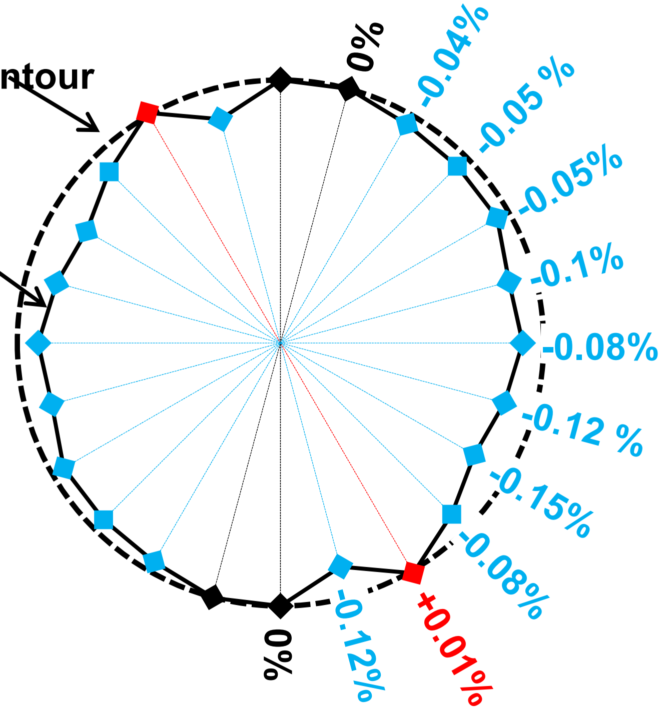
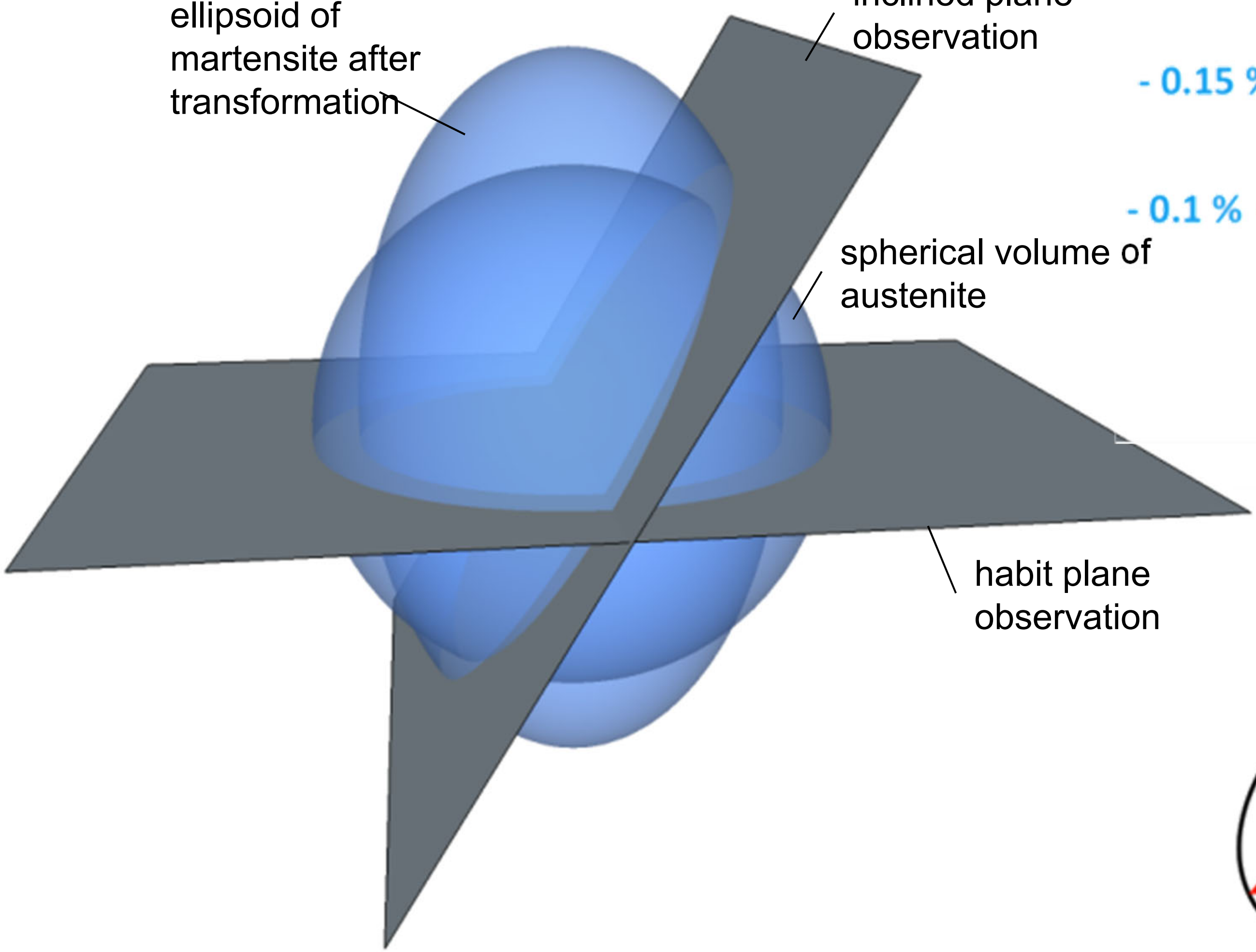
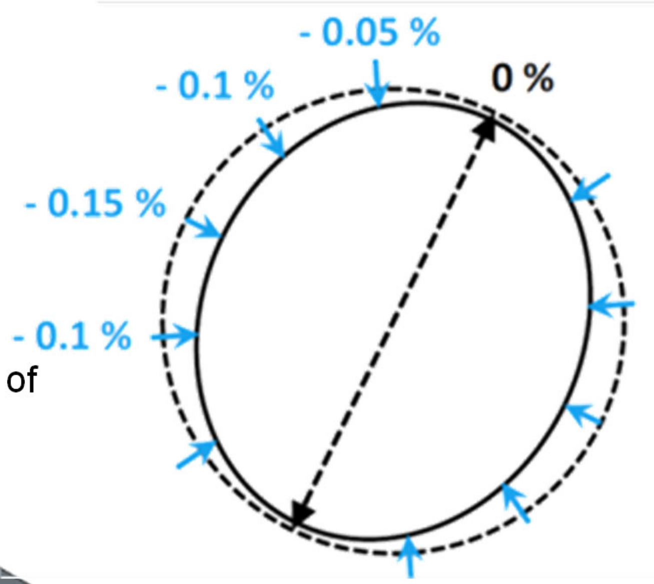


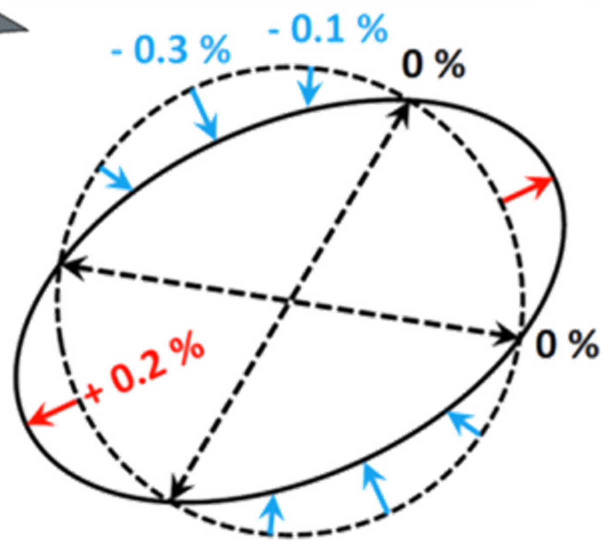




3-dimensional strain model







RCM: pros and cons

- Absolute strain measurement possible
- Micron-resolution (~ 5 µm)
- Creates its own reference at any place
- Applicable also to non-crystalline solids
- Laborious, time consuming process
- No mapping possible
- Tends to create artefacts during the imaging process
- Quality of DIC pattern determines quality of reference state

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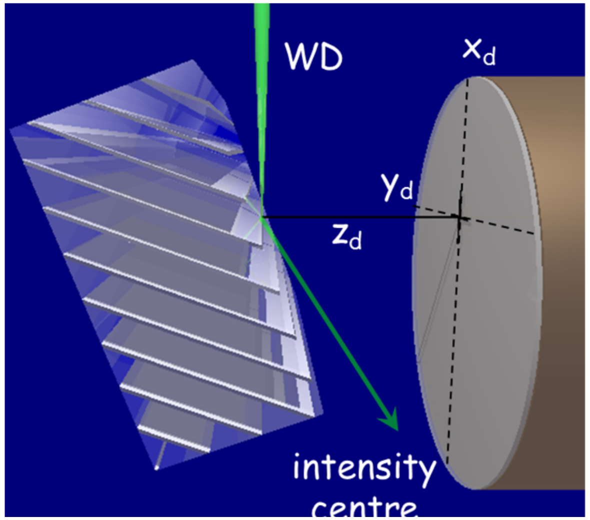
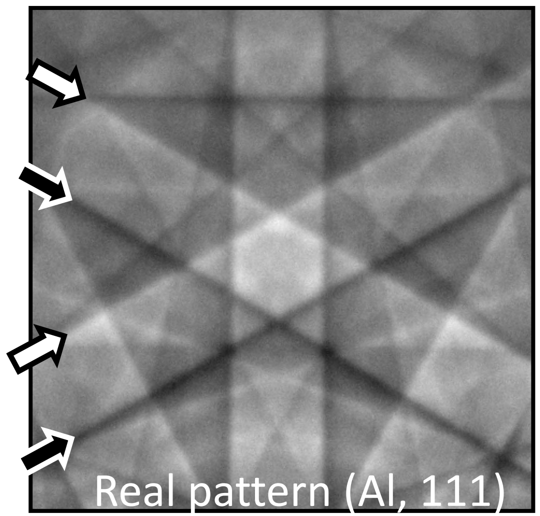
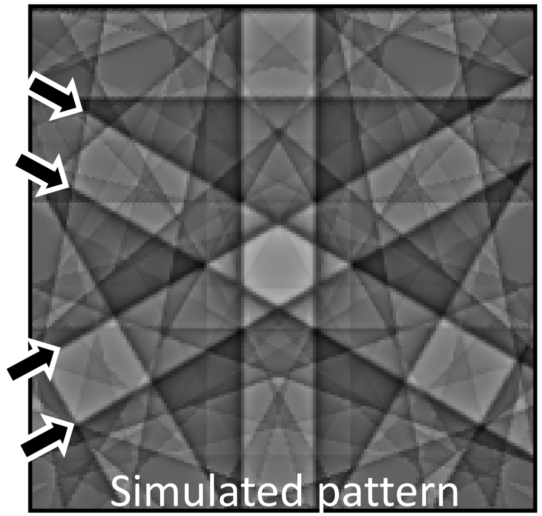
Absolute orientation measurements with EBSD

- What/where is the correct reference?
- For CC EBSD: Use a simulated diffraction pattern
  - What are the correct – relaxed – lattice constants?
    - ➢ Depends on local chemistry
  - How to obtain the correct pattern center?
  - How to deal with excess and deficiency lines?



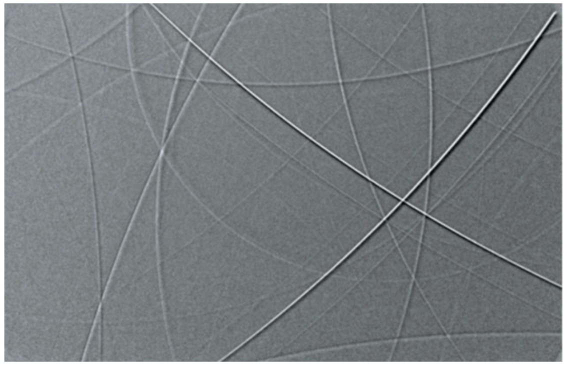




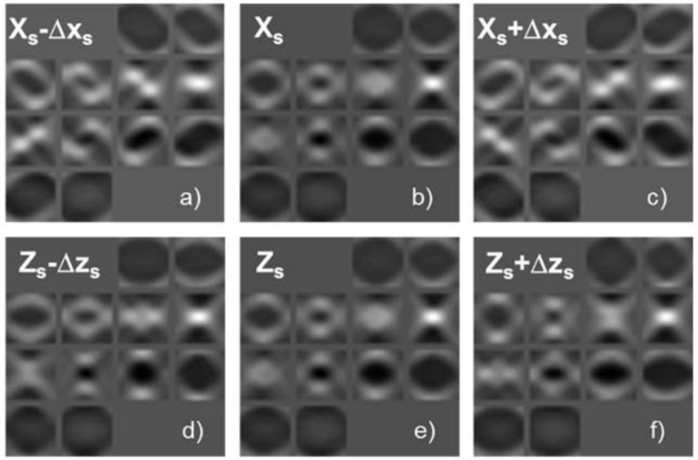
Create a fully relaxed local reference by ring core milling!

Alternative measurement techniques

- micro-Kossel diffraction *(E. Langer, or D. Bouscaud et al, J. Appl. Cryst.* **47**, 1699
  - Highest possible strain accuracy (10^{-5})
  - Absolute measurements possible
  - Mapping possible
  - Moderate spatial resolution (10 µm)
  - Limited applicability (suitable x-ray wavelength required)

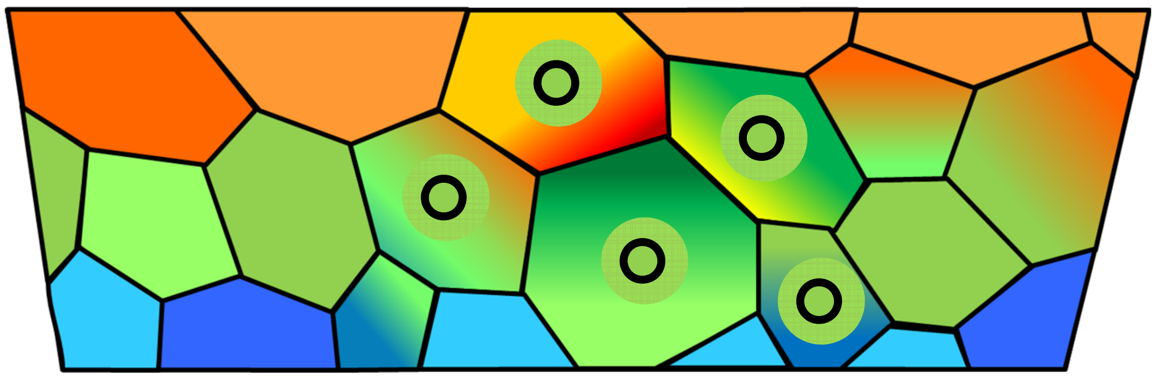


- 3D Hough transform *(Fortunier & Maurice,* Microsc. Microanal. 19 (Suppl 2), 2013*)*
  - High spatial resolution (50...500 nm)
  - Absolute measurements principly
  - Moderate accuracy for absolute measurements
  - Pattern center accuracy sufficient?
  - 9 independent variables, but only 3 fitting parameters

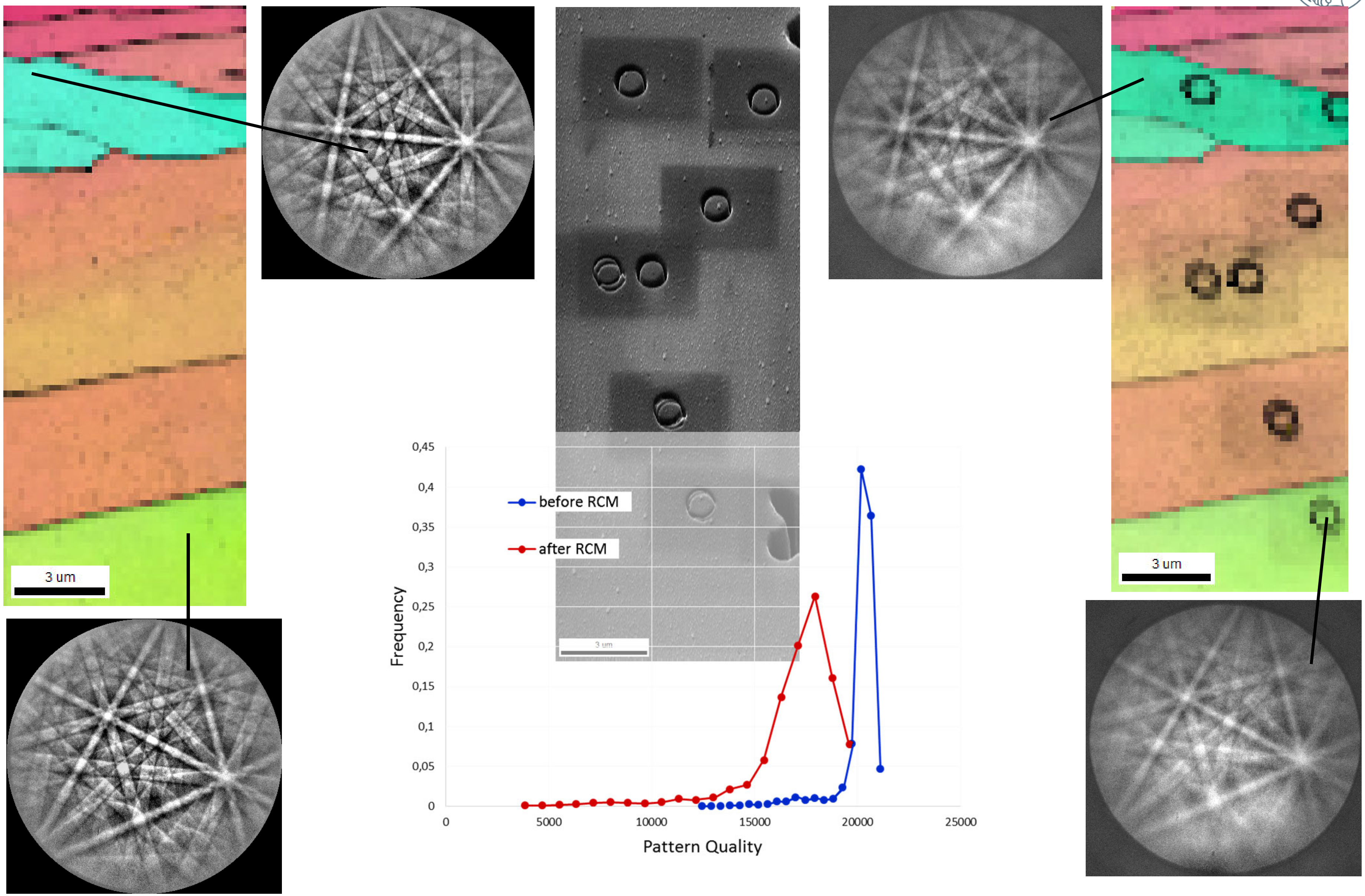


Combination of RCM and CC EBSD – general principle

- Create a fully relaxed reference point by RCM at every relevant position
  - no need to perform DIC for RCM
  - use center of RCM as reference of CC EBSD
  - no need of accurate pattern center determination (linear extrapolation from reference pattern for every grain)
  - high resolution strain mapping possible



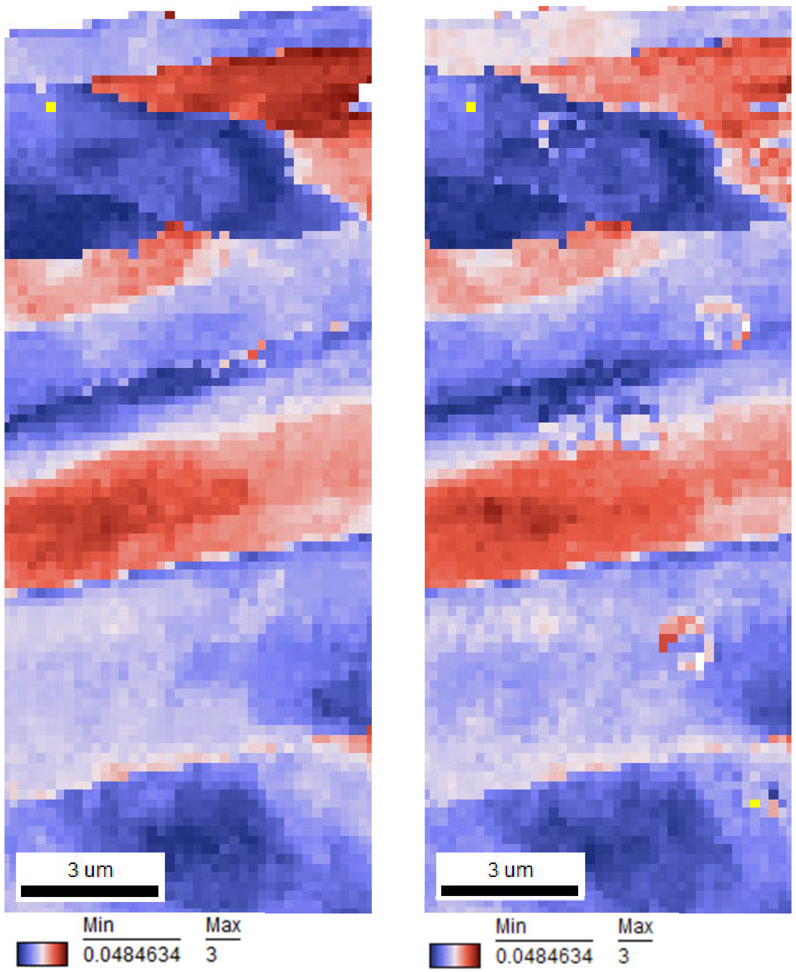
RCM - CCEBSD Experimental procedure



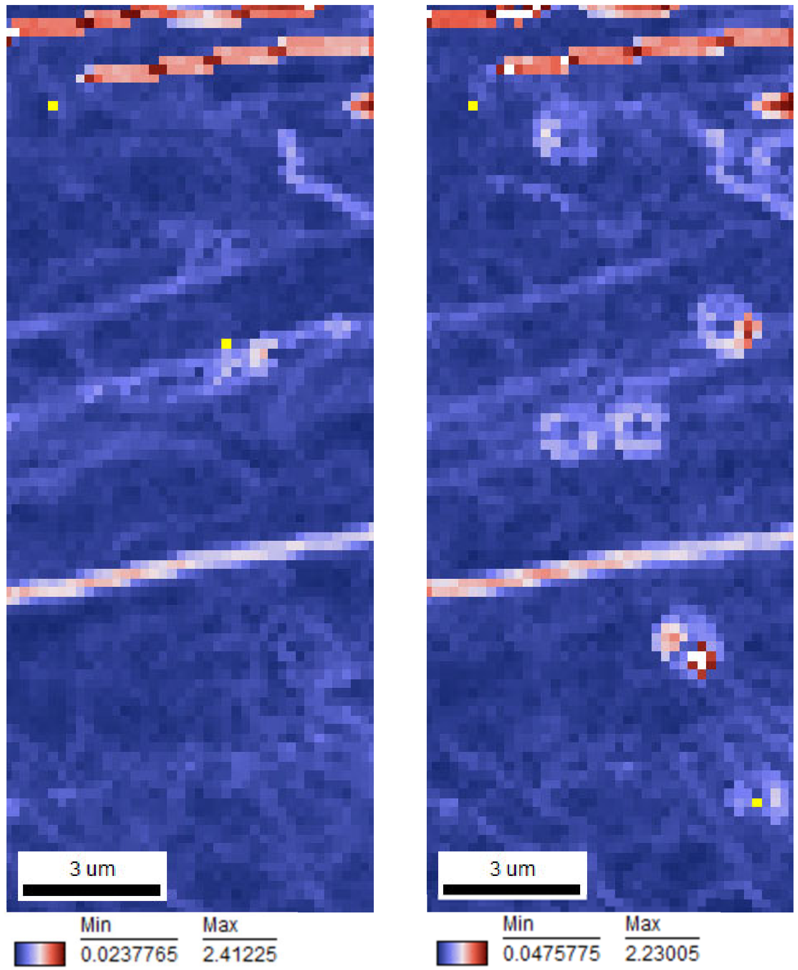
Reduction of pattern quality due to extensive electron observation

Influence of milling on lattice defect density

GROD



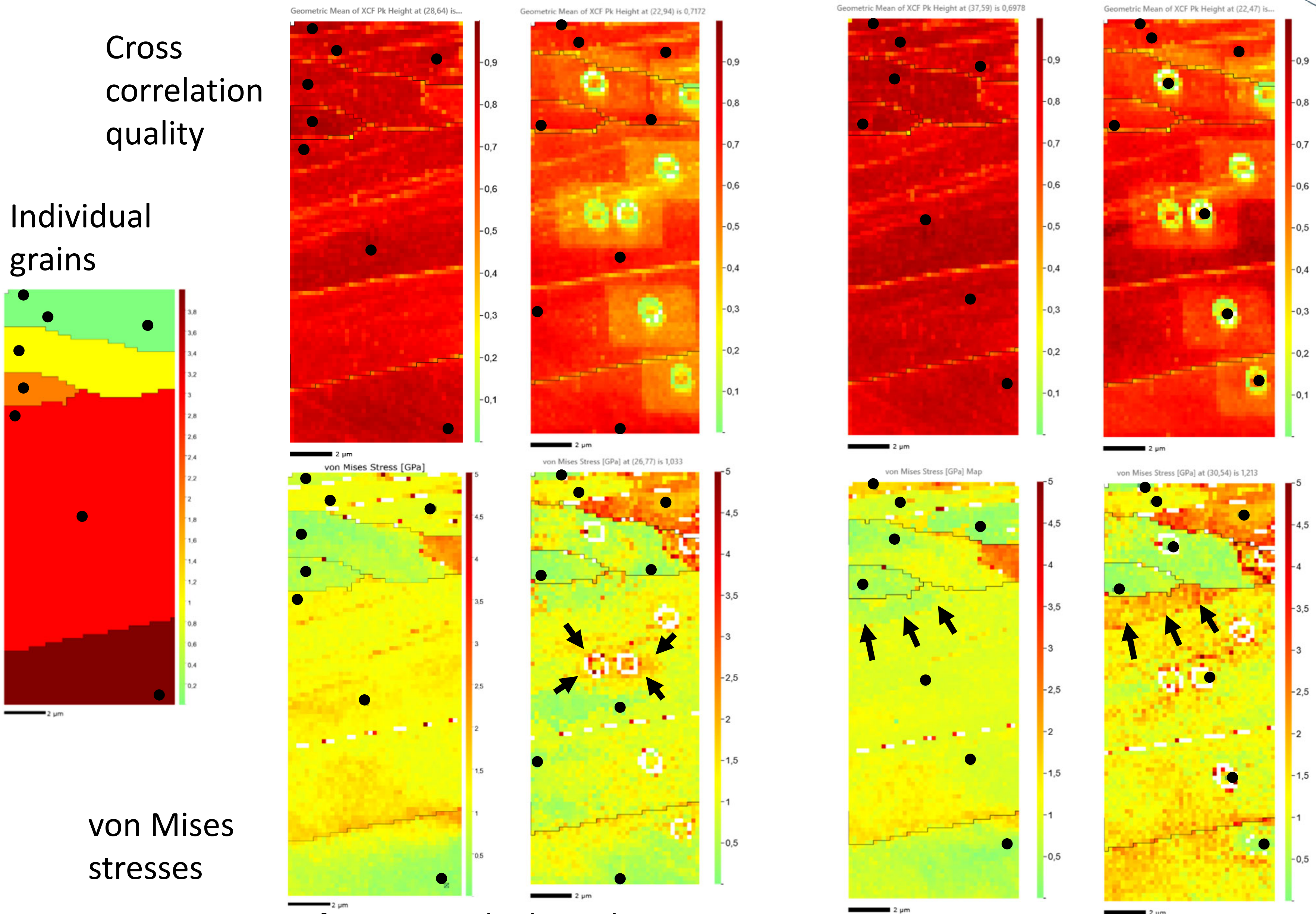
Local Orientation Spread



Influenced area larger than milling area, center of ring cores not affected

Optimum ring size: 1.5...2 µm, affected area not much larger than ring

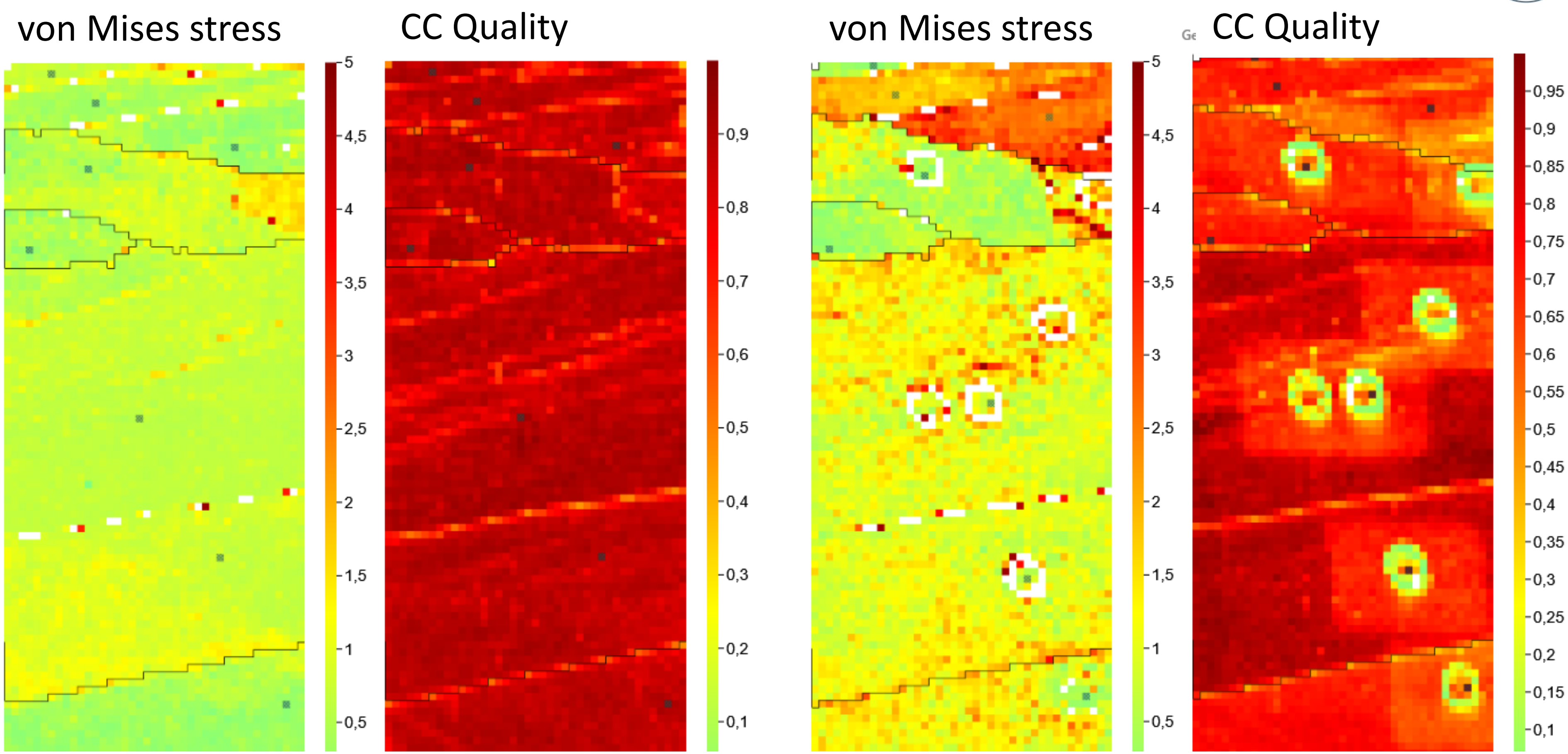
Reference patterns outside and inside of ring cores



References at high quality sites

References inside ring cores

References in the ring cores with pattern remapping



- Current (preliminary) results: RCM references changes the stress state without affecting the lattice defect density
- 1st kind stress mapping is possible without further reference!
- Electron illumination must be kept minimum,
- ring diameter ~1.5 µm appears optimum

RCM & CC EBSD: pros and cons (preliminary)

- 1st kind residual stresses are measured
- Internal references – no need to know cell parameters
- Mapping with EBSD resolution
- Much simpler procedure than RCM
- No scanning artefacts
- No need for accurate pattern center determination
- FIB instrument required
- More complicated and laborious than only CC EBSD
- Grain size must be larger than ~ 5 µm

Contents

- Residual stresses (RS) – why, what, how?
- Measurement of RS by cross-correlation CC-EBSD
- The measurement principles of ring core milling (RCM)
- Absolute measurement of RS by combination of CC EBSD and RCM
- Conclusions

Conclusions

- Local residual stress measurements via CC EBSD
  - high lateral and stress resolution
  - only stresses of 3rd kind can be measured
- Ring core milling: very valuable local stress measurement method
  - 1st and 2nd kind stresses measured
  - lateral resolution 1 µm
  - strain resolution not fully established
- RCM & EBSD: the best of both worlds!
  - 1st, 2nd and 3rd ind stresses are measured
  - lateral resolution 0.1 µm, strain resolution 10^{-4}

IMC20

The 20th International Microscopy Congress

10-15 September 2023, BEXCO, Busan, Korea

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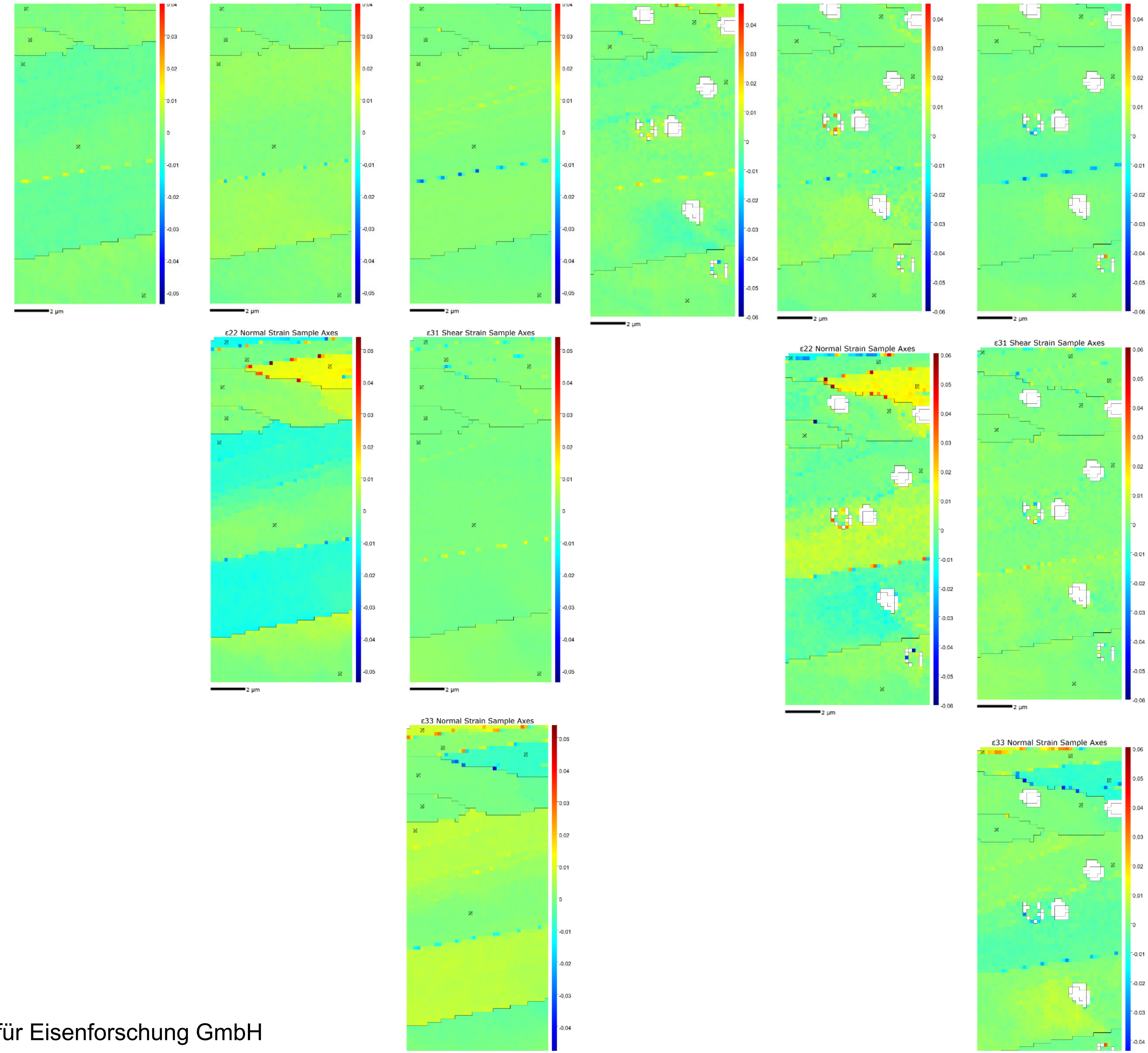
AS-09 Advances in scanning electron microscopy and focussed ion beam microscopy ×

This symposium aims to address issueScanning electron microscopy (SEM) is the most common and established electron microscopy technique. Reasons for this are its enormous versatility of observation techniques enabling the study of a vast amount of materials and material states, its high degree of automation, and its relative ease of operation. SEM based techniques span observation of materials from soft biological matter all the way to metals, semiconductors, minerals and ceramics. They cover high resolution imaging with secondary electrons, spectroscopic methods like x-ray spectroscopy for elemental composition measurement, Raman spectroscopy for strain measurements, cathodoluminescence for opto-electronic property measurements and diffraction techniques like electron backscatter diffraction (EBSD) techniques and electron channelling contrast imaging (ECCI) for the investigation of crystalline matter.

The focussed ion beam (FIB) scanning microscopes are usually built in combination with SEMs although they display an independent class of instrumentation. Mostly FIBs are used for target preparation of samples for transmission electron microscopy and atom probe tomography where one profits of simultaneous observation and analysis with the joint SEM. Nevertheless, they are also used for micromachining of samples for SEMs (e.g. for local mechanical testing) or for exposing features for further investigation with SEM e.g. by serial sectioning to perform 3D materials analysis. The combination with mass spectroscopy also allows high resolution elemental analysis. Lately the combination of FIB and SEM with laser beams enables the controlled removal of large amounts of material which offers further powerful preparation and investigation means.



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