3D orientation microscopy based on FIB-EBSD tomography: Potentials and limits.

S. Zaefferer

Contents

- The need and methods for 3D characterization of crystalline matter
- Principle of 3D characterisation by FIB-EBSD tomography
- Application example:
  - Coupling of 3D measurements with 3D modelling
- Material restrictions:
  - beam induced material changes
- Conclusions

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The need for 3D observations

Pre-condition: crystallographic information must be accessible to investigate the microstructure of crystalline matter

2D Stereology

statistical observations:

- grain size distribution
- grain shape (from 2 sample sections)
- volume fraction and distribution of 2nd phase constituents
- texture-microstructure relations

3D Destructive

static observations:

- comprehensive morphology information
- 3D connectivity of features
- grain boundaries
- input data for modelling
- 3D deformation structures

3D Non-destructive

process observations:

- recrystallization (e.g. nucleation, grain growth)
- deformation (e.g. texture formation)
- phase transformation (e.g. variant selection)

Many problems can be solved by 2D statistical observations but for some 3D observations are essential

Serial sectioning methods

Sectioning:

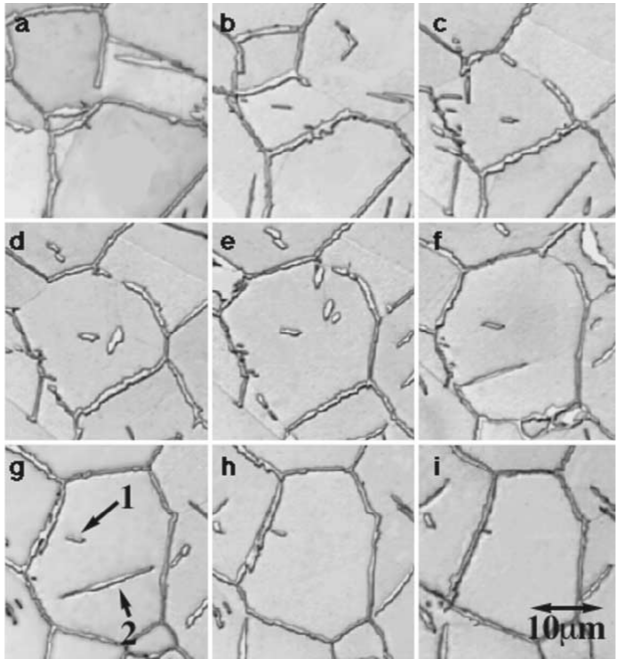
- mechanical or chemical polishing
- FIB milling....

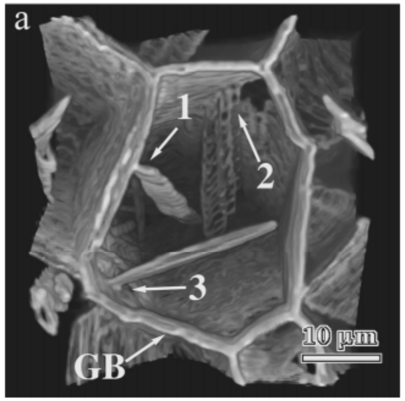
Observation:

- BSE-Microscopy, EBSD, optical microscopy....

Problems:

- depth definition
- contrast definition for segmentation
- very laborious





M.V. Kral & G. Spanos, Acta Mater. 47 (1999), 711

serial sectioning and reconstruction of allotriomorphic cementite by mechanical polishing

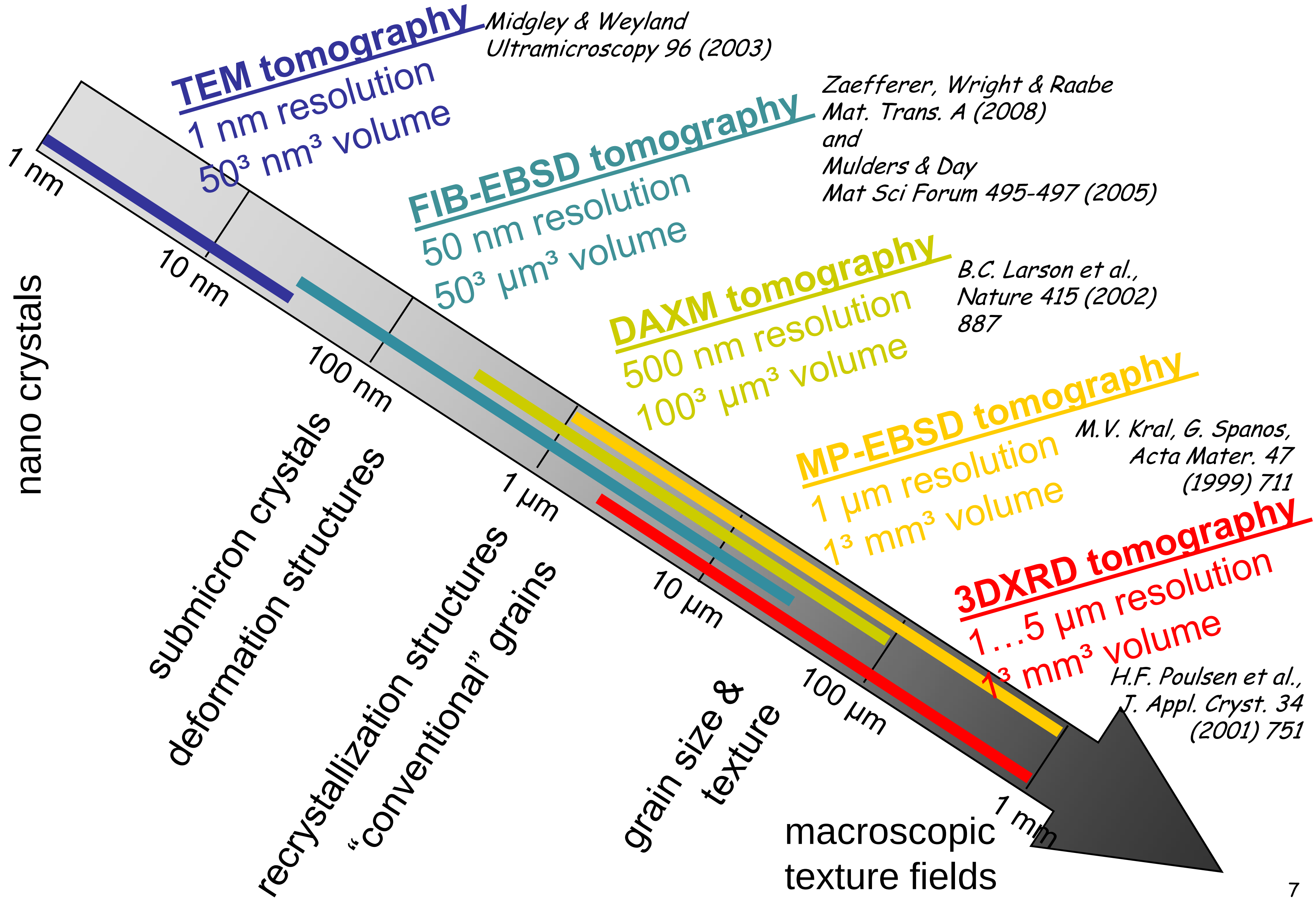
Advantages of FIB-EBSD tomography

- Sectioning by FIB
  - accurate depth definition
  - flat and parallel sections (< 1° deviation)
  - high resolution (< 50 nm)
- Observation by EBSD
  - well-defined contrast on crystalline material
  - ideal for reconstruction of grains in 2D and 3D
  - quantitative description of microstructure
  - high resolution (~ 50 nm)
- Combination of FIB and EBSD
  - table-top instrument
  - "high" measurement speed
  - fully automatic

Recent reviews:

- *Uchic et al., MRS Bulletin 32 (2007) 408-416*
- *Zaefferer et al., Met. Mater. Trans. 39A, (2008) 374-389*

Length scale of tomographic measurements



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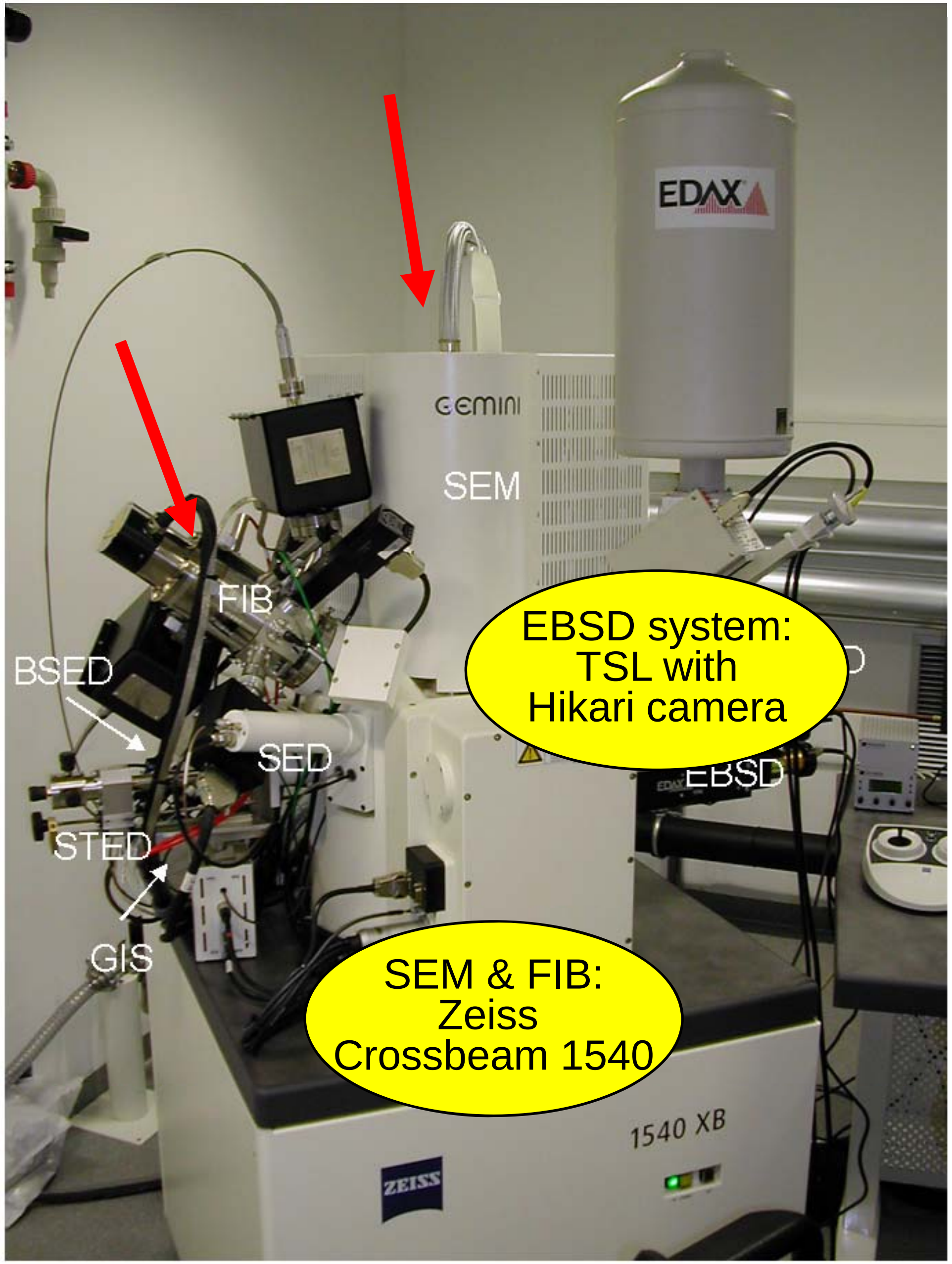
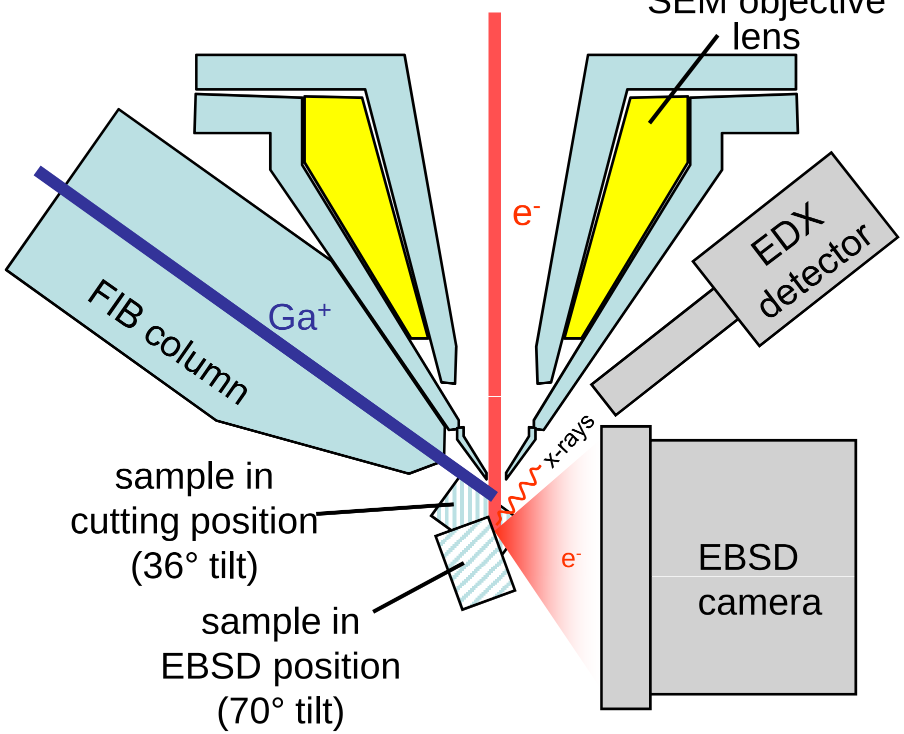
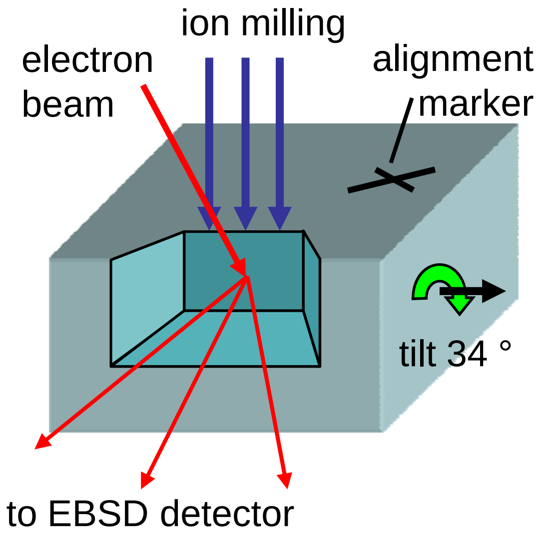
Instrument overview

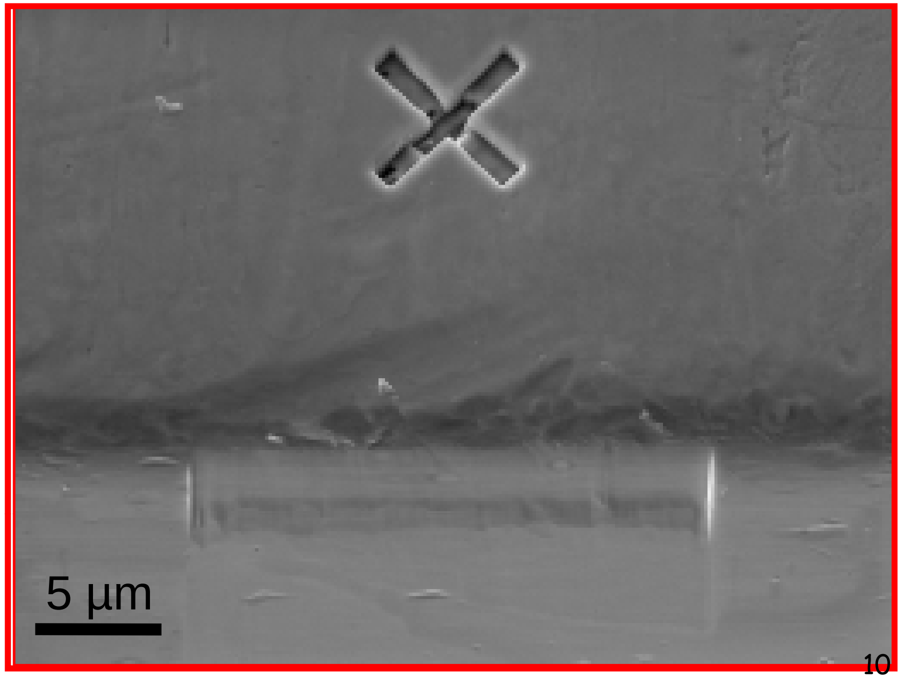


- Scanning electron microscope (SEM)
  - observation of microstructure
- Scanning Ga^{+}-ion microscope (FIB = focused ion beam)
  - sputtering of material for serial sectioning
- Quantitative images with EBSD and EDX
  - quantitative characterisation of microstructure

Principle of serial sectioning & orientation microscopy

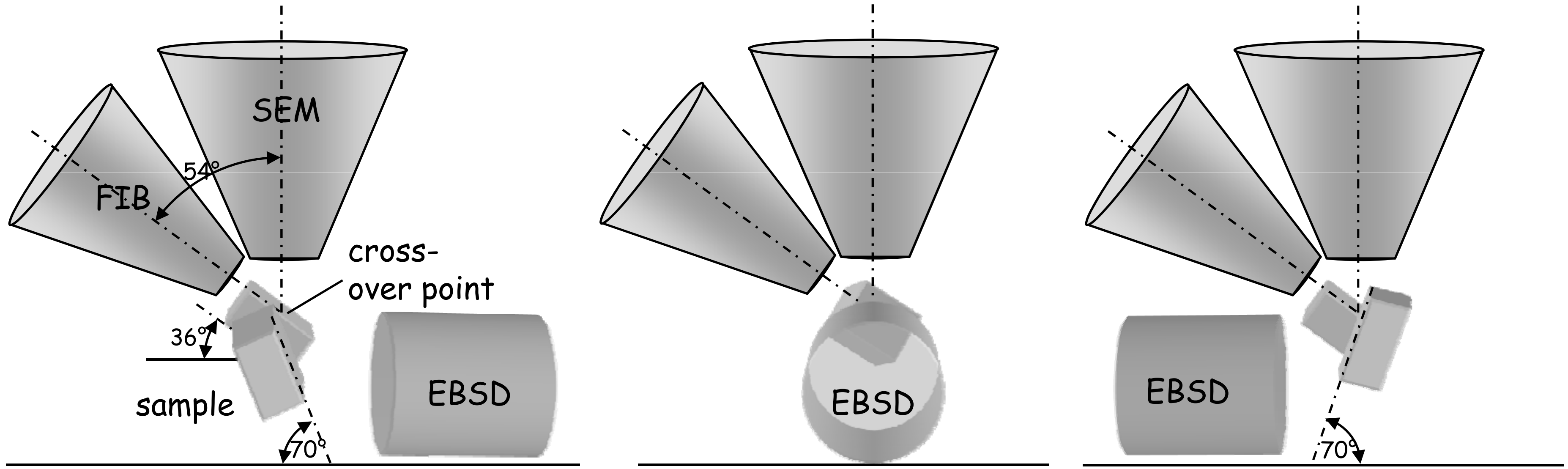






"tilt set-up"
Zaefferer, Wright, Raabe,
Mat. Trans. A (2008)

Geometrical set-up alternatives for FIB-EBSD



tilt set-up

- +/- medium tilt positioning accuracy
- + tilt inaccuracies create linear distortions
- + simple software correction possible
- + freely selectable milling position

Zaefferer et al., Met. Mater. Trans. 39A, 374-389 (2008)

static set-up

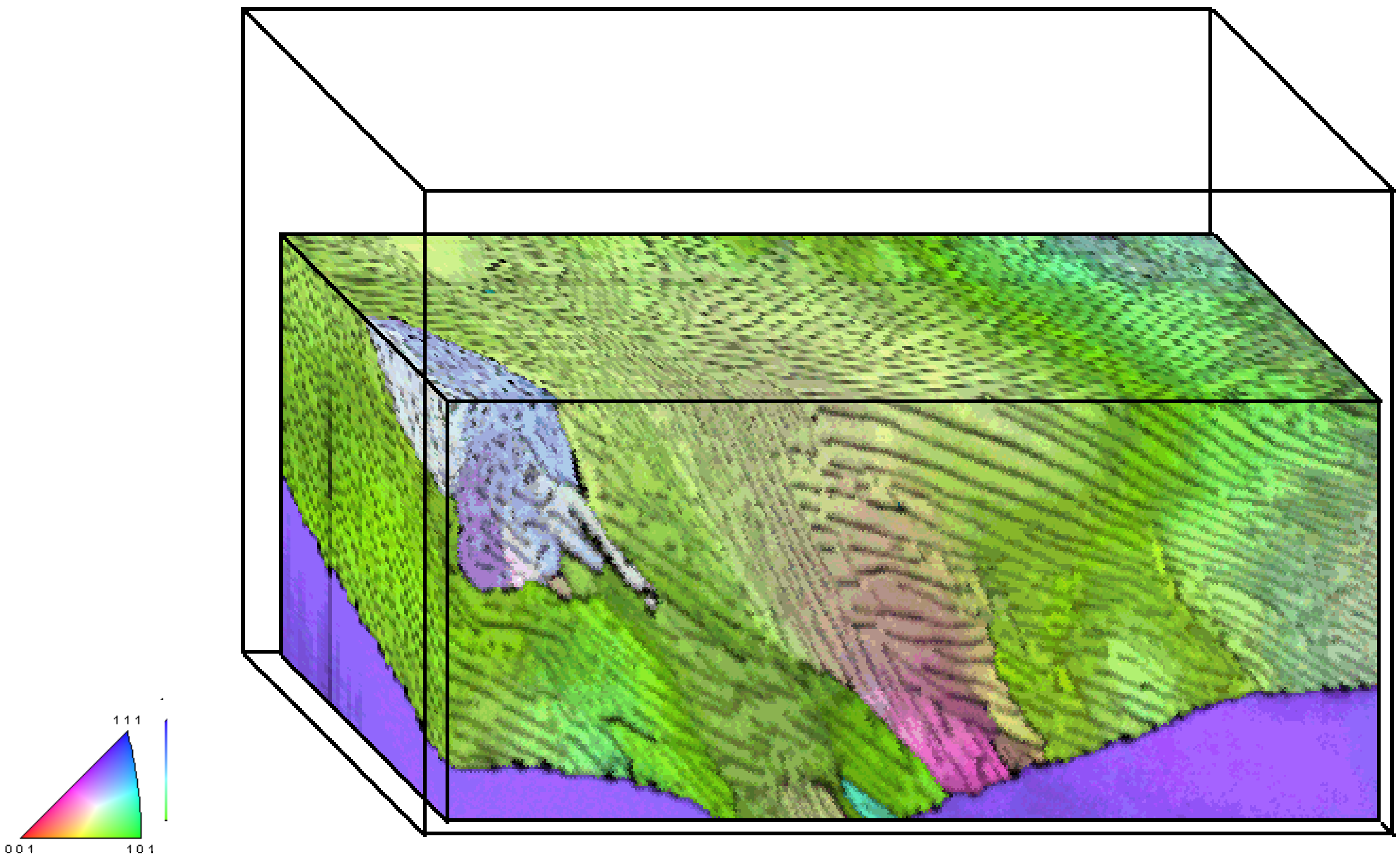
- + no stage movement required
- + highest possible positioning accuracy
- + unconventional but non-problematic EBSD set-up
- + high measurement speed

rotation set-up

- + high stage positioning accuracy
- +/- rotation inaccuracies create shear distortions
- +/- software correction more complex
- +/- every milling position requires a different holder

Mulders, Day, Mat. Sci. Forum 495-497, 237-242 (2005)

EBSD & FIB-sliceing: 3D microstructure of pearlite



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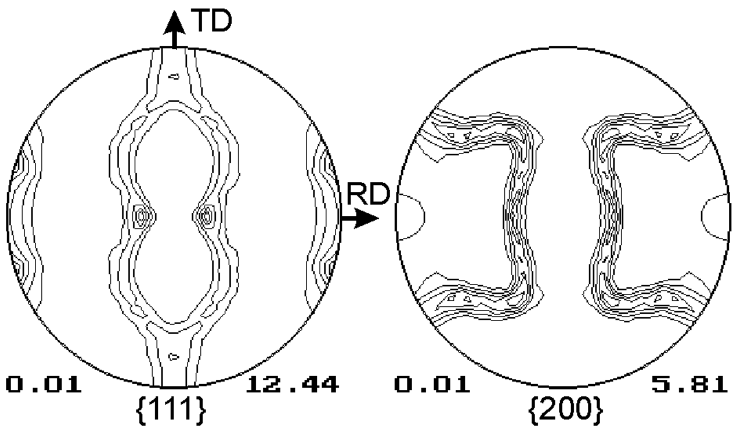
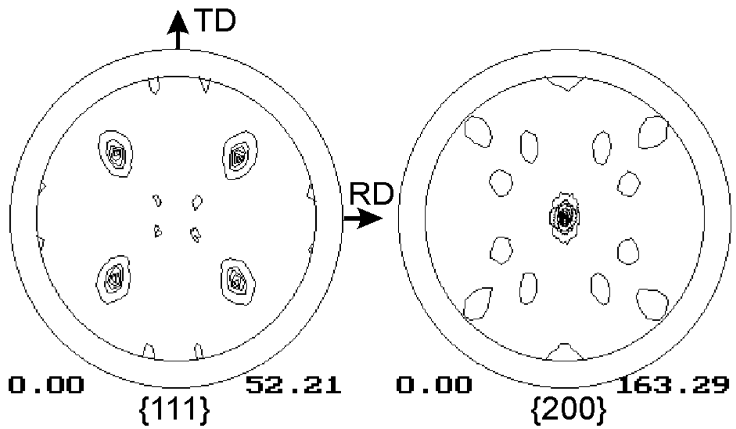
The cube texture in Fe 36% Ni



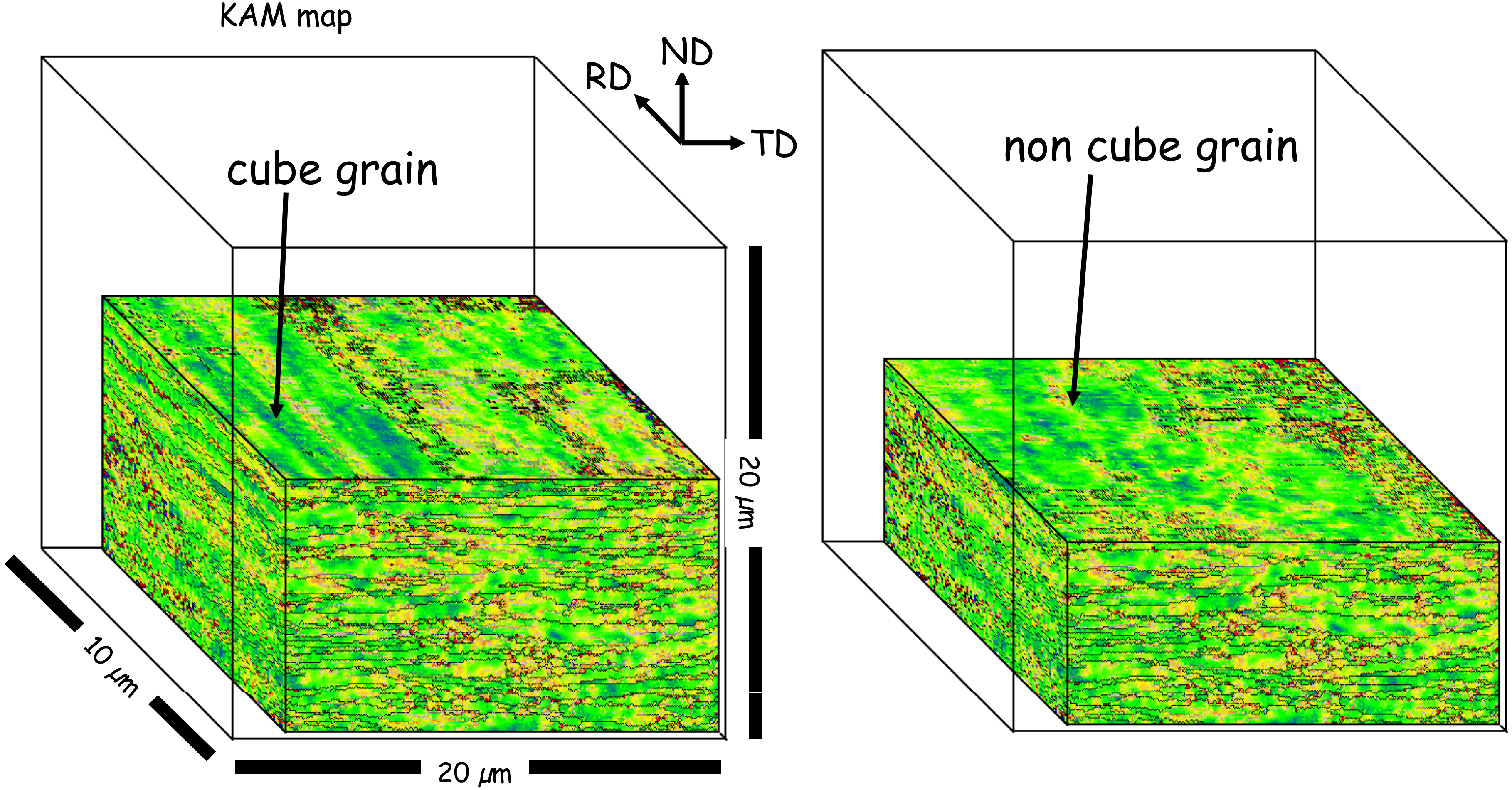
cold rolled material:
Cu-type rolling texture



recrystallized material:
sharp cube texture

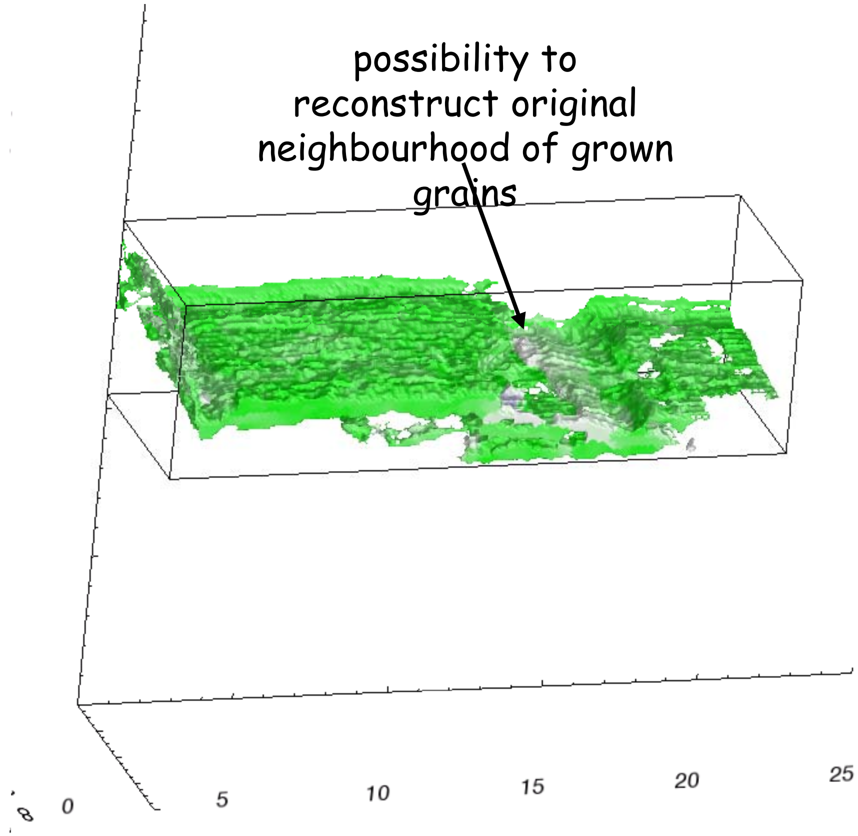
- Origin of the cube texture: oriented nucleation
- Possible reasons for texture selection:
  - stored energy differences *(Etter et al. Scripta Mat. 46 (2002) 311)*
  - grain boundary properties (e.g. 40° <111>) ("micro-oriented growth", *(Duggan et al., Acta metall. mater. 41 (1993) 1921)*)
  - differences in mobility of dislocations in different orientations (differences in recovery rate) *(Rhida & Hutchinson, Acta metall 30 (1982) 1929)*

3D-orientation microscopy on cold rolled material



Cube grains have low internal orientation fluctuations.
Some other orientations do have that as well.

Microstructure after 1 min annealing

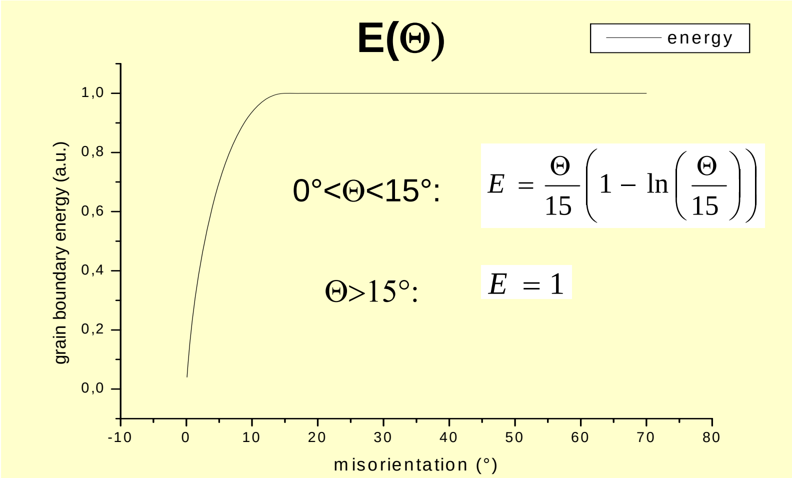


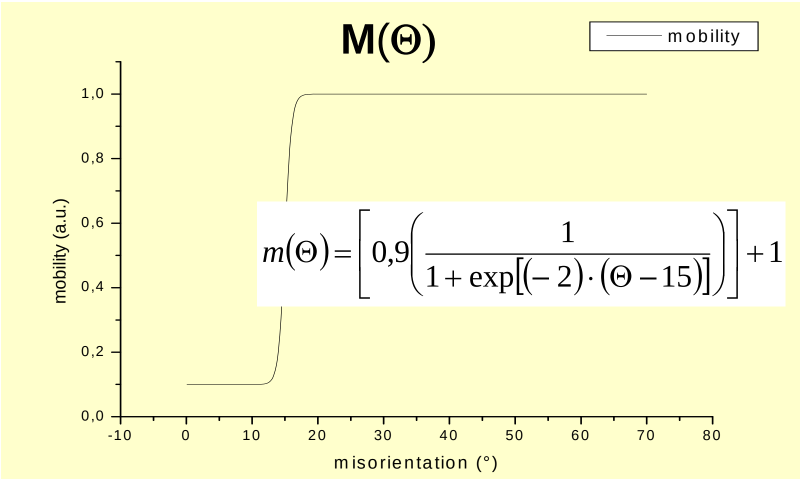
- only small cube-grain areas with 40°<111> orientation relation
- original neighbourhood of grown grain does not show any special boundaries with cube band

Direct observation of the nucleation process

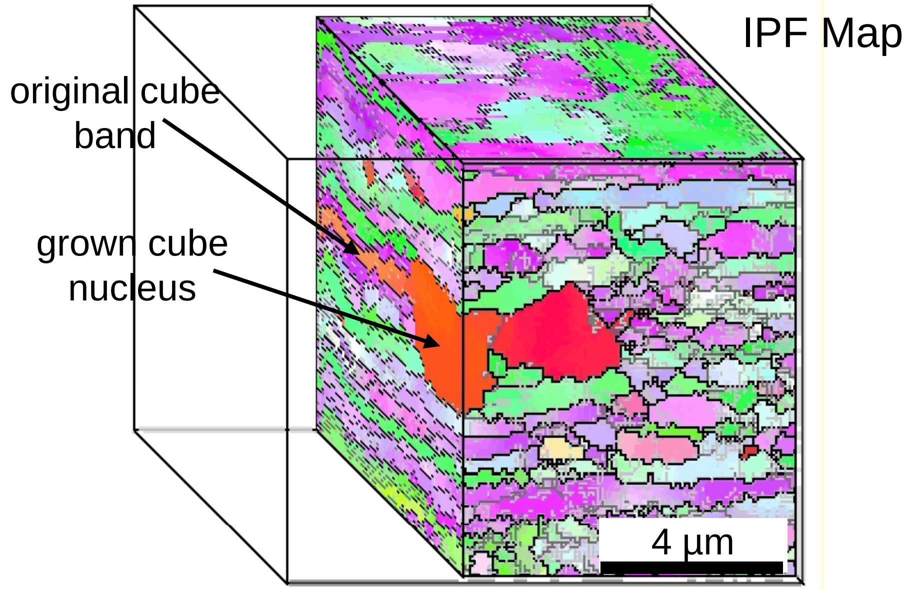
- In-situ observations are difficult
  (but see: *Nowell et al. ReX& GG2 (2004)*)
- Modelling on the basis of orientation microscopy data
  - Hypothesis: abnormal subgrain growth as nucleation mechanism of recrystallisation
    (*Humphreys, Acta Mater. (1997)*)
- 3D-Monte-Carlo Potts model for the simulation of subgrain growth
  - Freely selectable energy and mobility functions
  - Experimental microstructures as input data
  - Stored energy determined from local orientation gradients

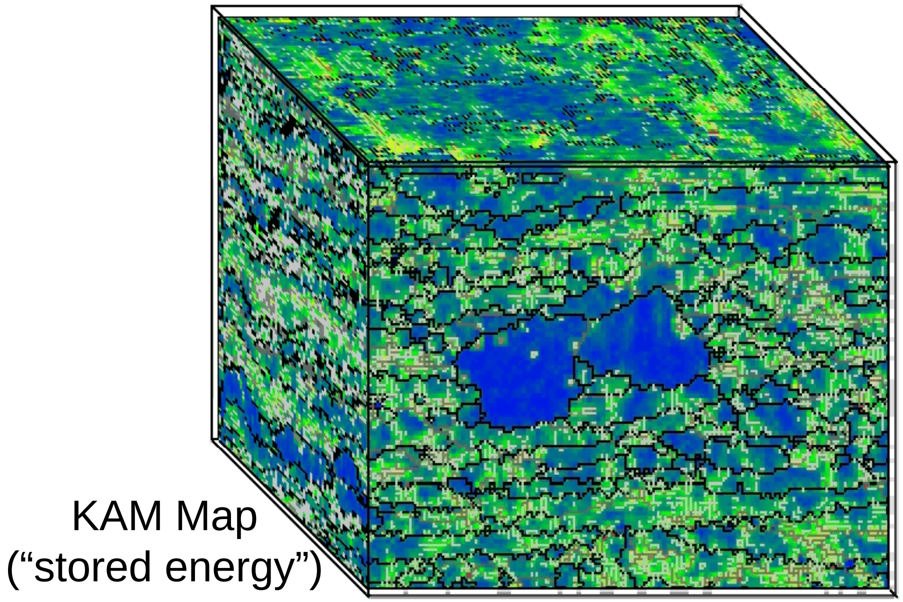
Nucleation simulation by a 3D-Monte Carlo Potts model



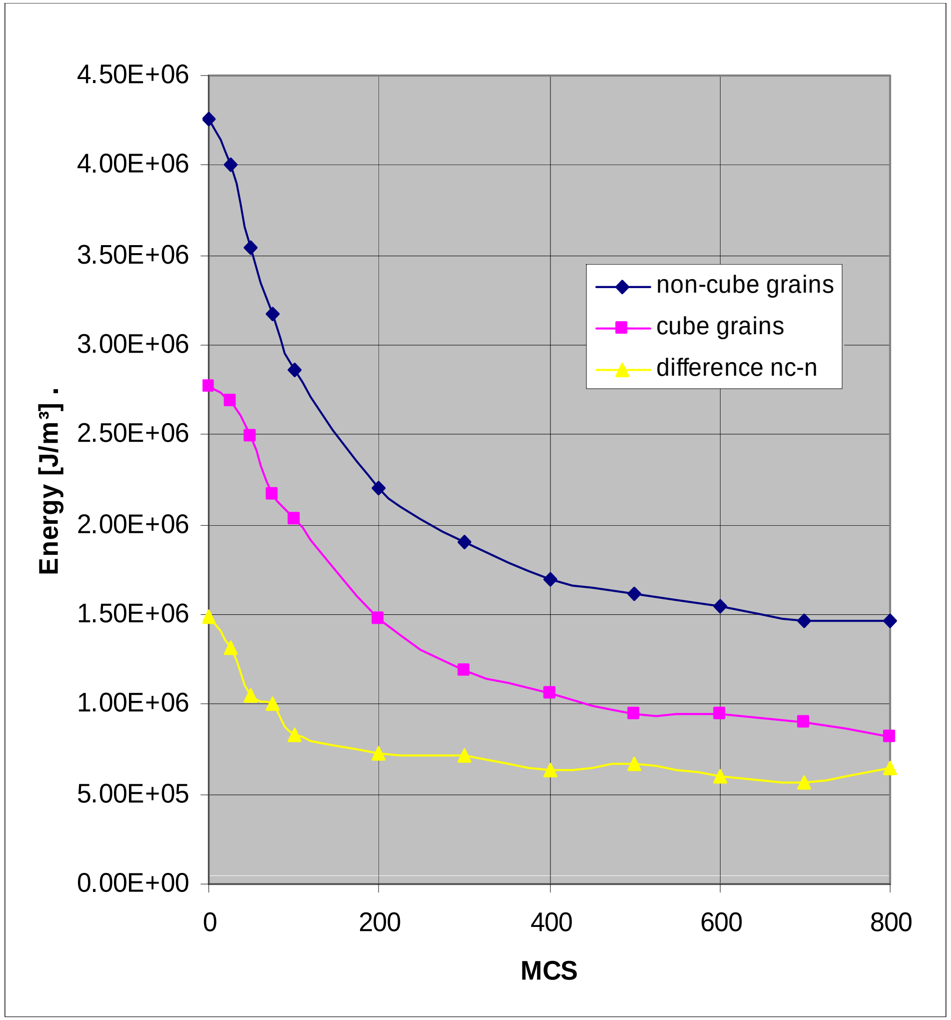


Stored energy according to Read-Shockley approach





Evolution of stored energy from MC simulations



- ⇨ Stored energy difference between cube and non-cube grains persists even after longer annealing periods
- ⇨ Growth advantage of the cube grains

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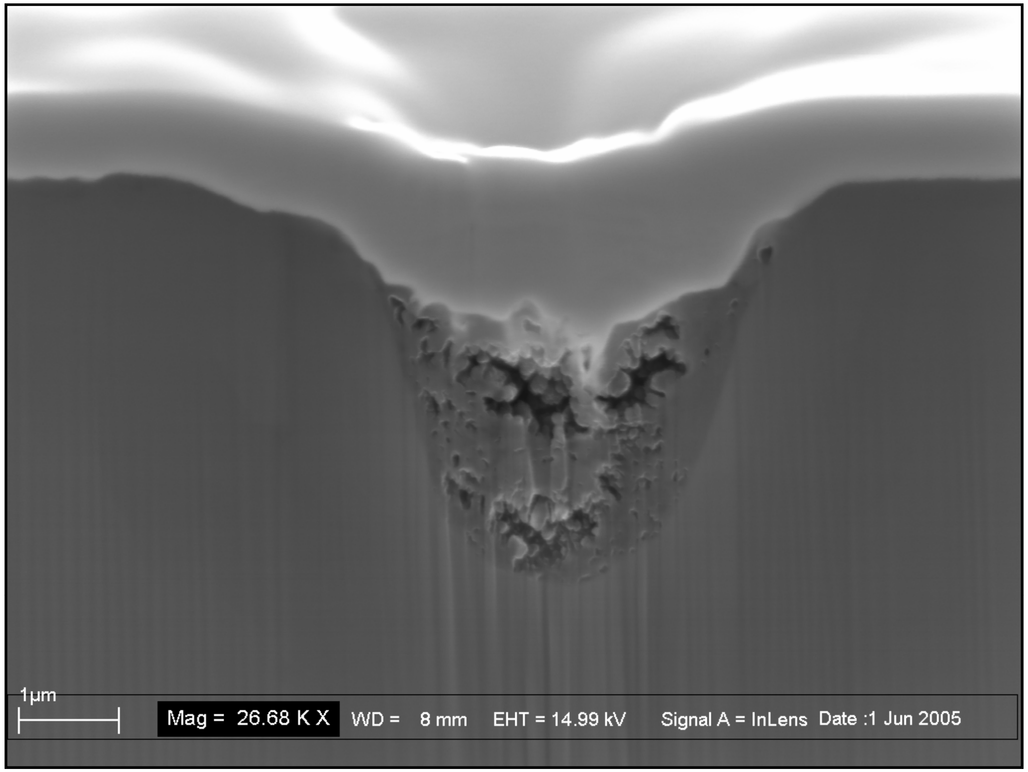
Materials restrictions of FIB-milling

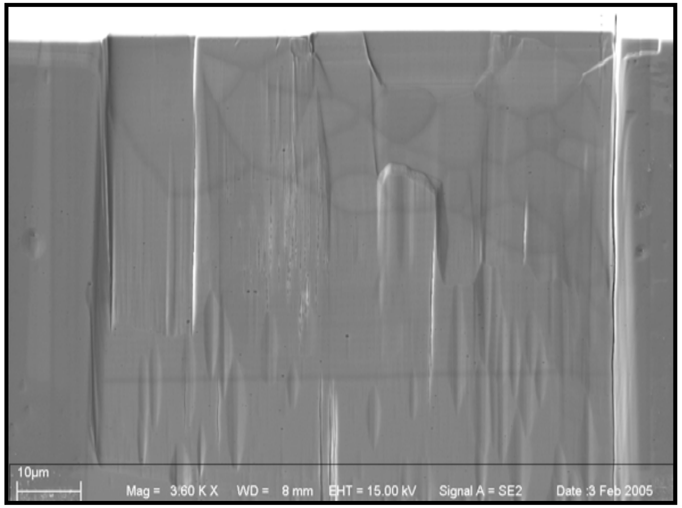
- Anisotropic sputtering and curtaining
- Amorphisation (beam damage)
- FIB-induced phase transformation
- Reaction between gallium and aluminium

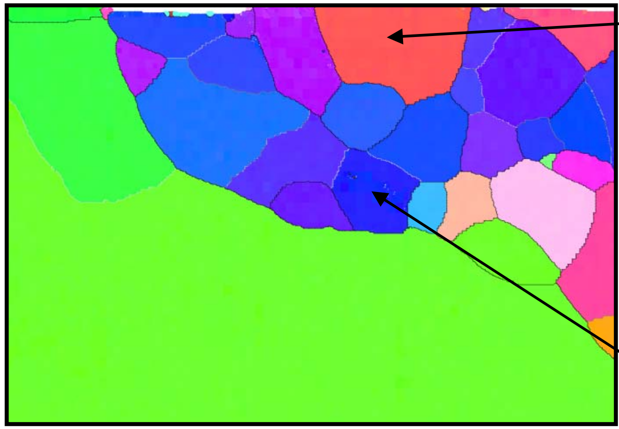


damage due to Ga-Al interaction at grain boundaries under a nano-indentation in Al

Anisotropic sputtering & curtaining

Fe 3% Si alloy: crystallographic origin of anisotropic sputtering





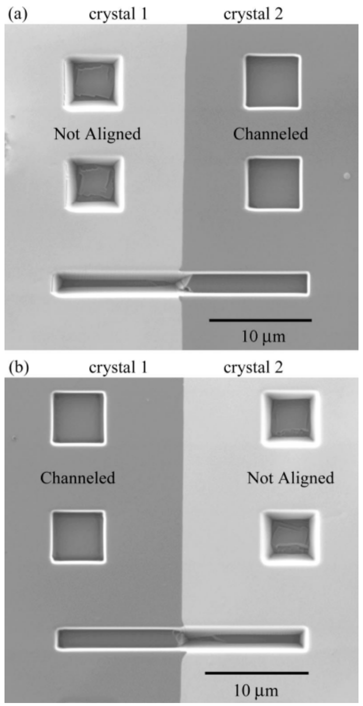
low resistance against sputtering

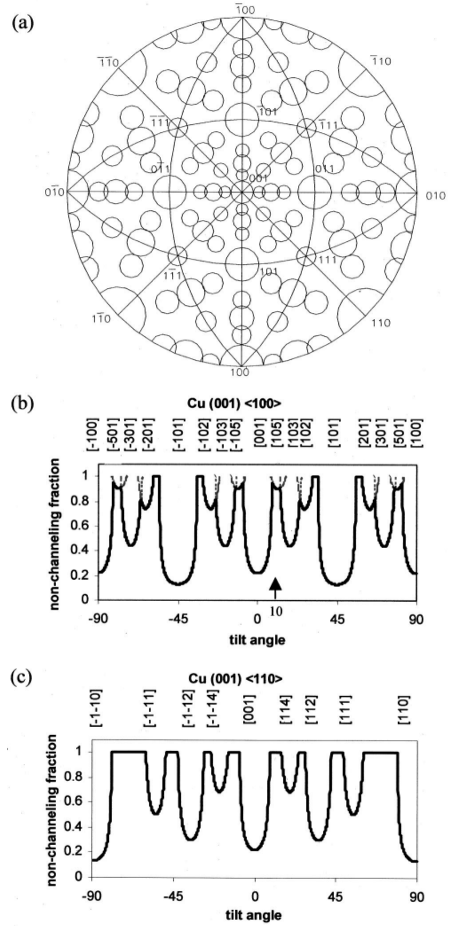
higher resistance against sputtering

⇨ easy sputtering: {100} crystal planes
hard sputtering: {111} crystal planes

Experiments and calculations on anisotropic sputtering of Cu

B.W. Kempshall et al., J.Vac.Sci.Tech. B19 (2001), 749

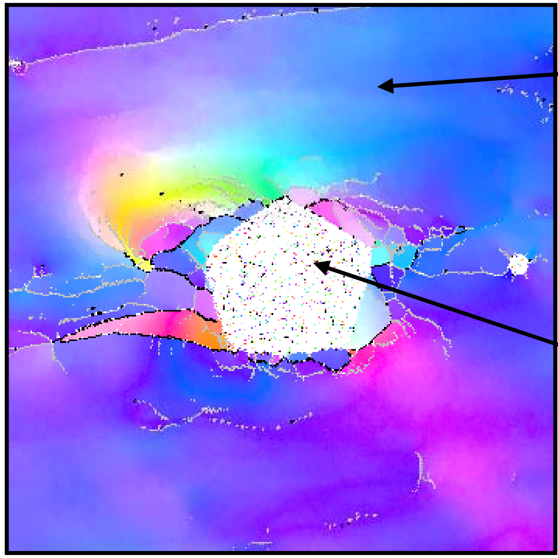




Amorphisation of lattice structure (beam damage)

- Investigation of *Kato et al., J.Vac.Sci.Tech. A17(1999), 1201*:
  - 20 nm side-wall amorphisation after 30 keV milling on silicon
  - 8 nm after 10 keV milling
    - ⇨ amorphisation depth is proportional to ion energy
    - ⇨ amorphisation depends on Z of target material

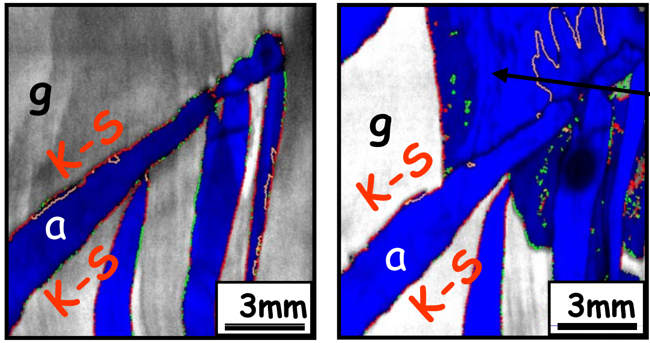
milling:
30 keV, 500 pA



FIB-beam-induced material changes

Beam-induced a-γ phase transformation in Fe-Ni

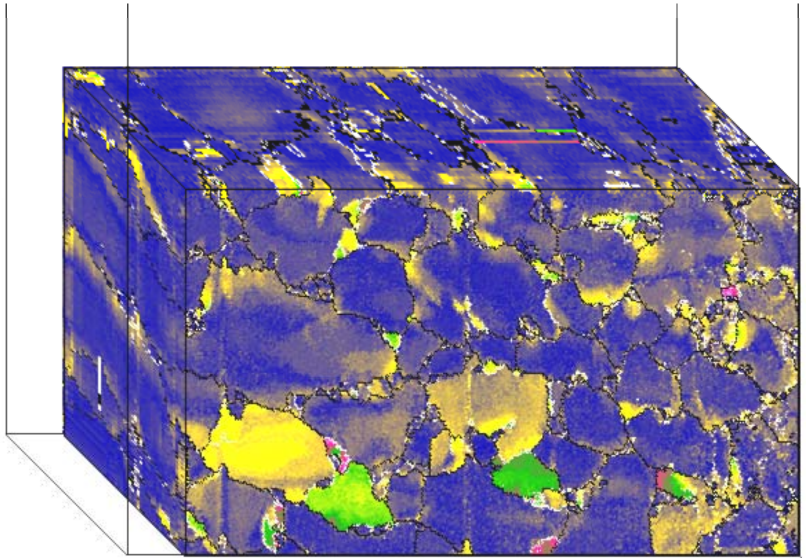
formation of a thin a-layer



transformation of metastable austenite into martensite during milling

3D orientation microscopy on a TRIP steel:

- no residual austenite left
- "bainitic" orientation gradients preserved



orientation deviation 0°...20°

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- **Measurement accuracy**
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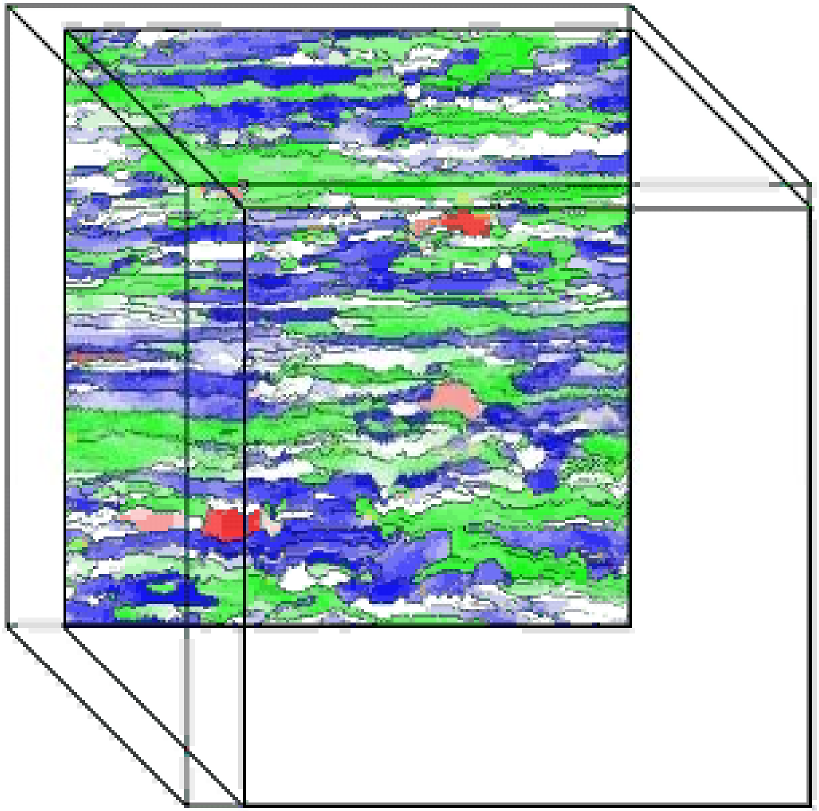
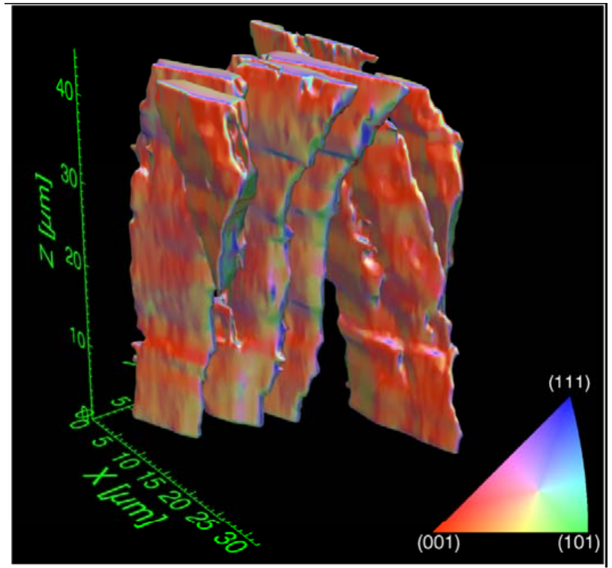
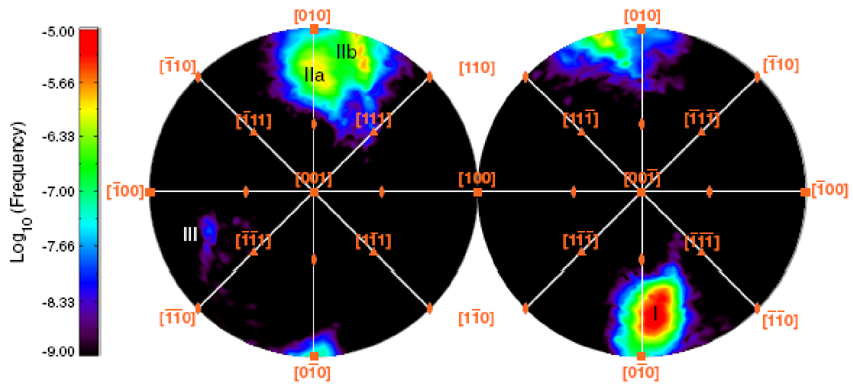
Problems of section alignment



Cube nucleus in Fe Ni





Crystallographic interface analysis of martensite plates
Rowenhorst et al. Scripta Mater. 55 (2006) 11–16

Bad section alignment leads to significant errors in plane determination

Software-based improvement of resolution

- Two sources of inaccuracy for the tilt set-up:
  - Tilt inaccuracies: linear expansion of measurement field $\Delta\alpha \leq 1°$ ⇨ $\Delta l/l \approx 1$ % (⇨ on a 10 µm field: 100 nm)
  - Shift inaccuracies: translations of measurement field usually in the order of 1 image pixel ≈ 50 nm
- Correction of inaccuracies:
  - Tilt: measurement of $\Delta\alpha$ by measurement of average misorientation between slices

    ⇨ correction by linear image distortion
  - Shift: minimization of Euler angle correlation coefficient between successive slices

$$C_{ij} = \sum_{i,j} \left| (\varphi_1, \phi, \varphi_2)_{t,x,y} - (\varphi_1, \phi, \varphi_2)_{w,x+i,y+j} \right| \overset{!}{=} \min$$

Advanced section alignment

- Non-systematic voxel shifts due to inaccurate beam movement
  - leads to locally changing misalignment of slices
- Approach:
  - short Monte-Carlo grain growth process
  - MC termination condition:

constant grain volume $$\frac{dV_g}{ds_{mc}} = 0$$

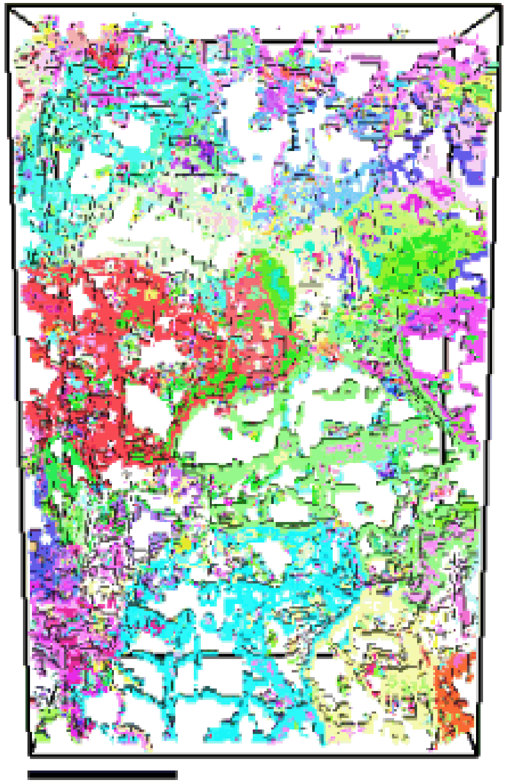
shrinking grain surface $$\frac{dA_g}{ds_{mc}} < 0$$

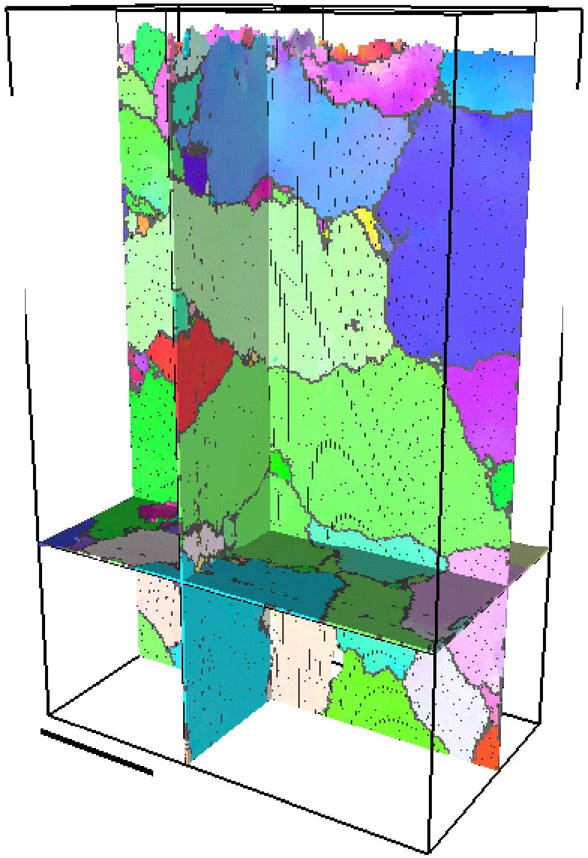
- conserving main grain shape and size, reducing grain boundary roughness
- conserving the internal structure of grains

Effect of MC clean-up

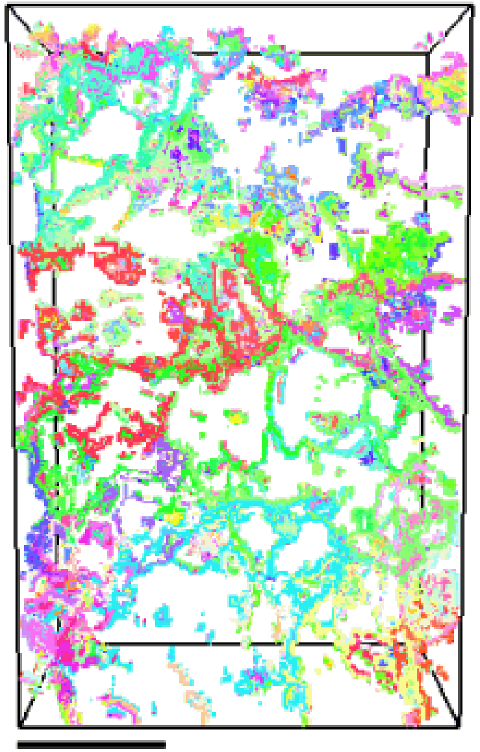
Sample: deformed TRIP steel

Raw data





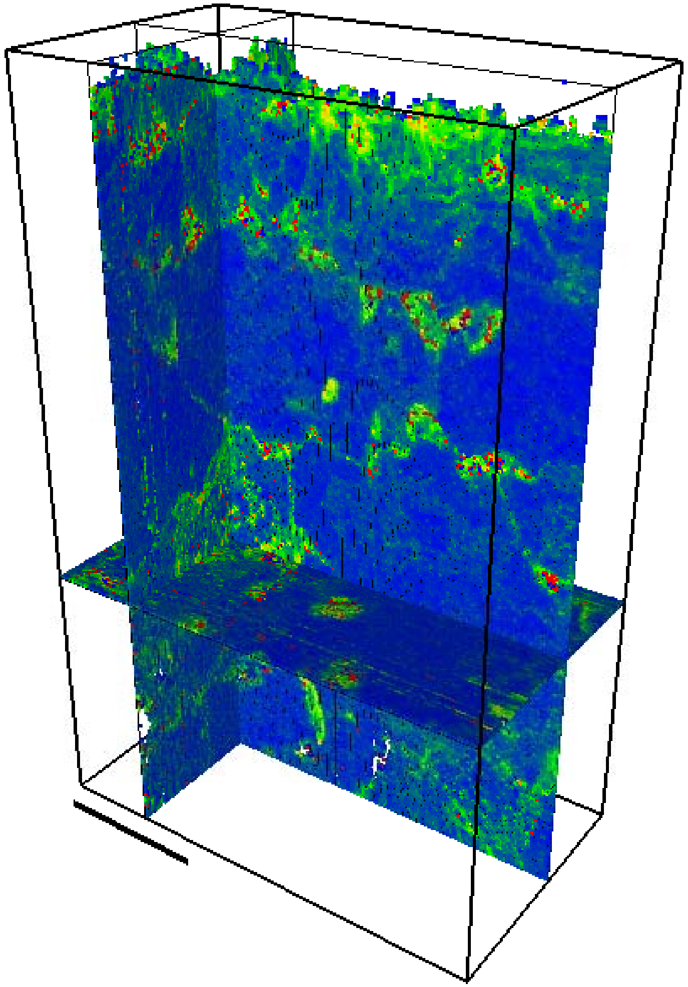
MCS = 5



effective clean-up: triple line reduction

MC clean-up: sub-structure conservation

Raw data



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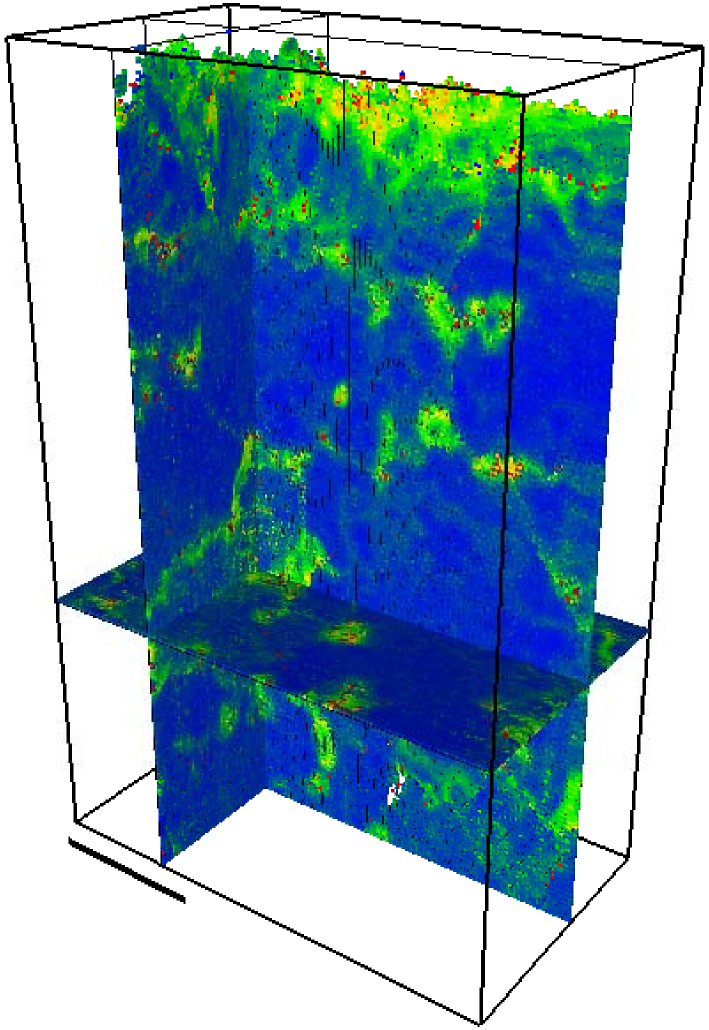

KAM values showing the local orientation gradients

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Conclusions I: general features

- A multi-dimensional microstructure vector is obtained at each 3D spatial position
  - phase, orientation, defect density, elemental composition
- Spatial resolution: 50 x 50 x 50 nm^3
- Observable volume: ≈ 50 x 50 x 50 μm^3
- Angular resolution: 0.5° (precision of tilt)
- Time consumation per cut: 15 … 60 min /cycle