

## Techniques and application of 3D orientation microscopy based on EBSD tomography



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*with*

*P. Konijnenberg, (3D analysis software)*

*G. Stechmann (CdTe solar cells)*

*A. Khorashadizadeh (SPD copper)*

### Outline

- Measurement techniques
  - 3D orientation microscopy by FIB serial sectioning
- Alignment of data sets
- Voxel-based analysis
  - Calculation of geometrically necessary dislocation densities
- Interface-based analysis
  - Marching tetraeder for interface detection
  - Smoothing of interfaces
  - grain boundary characterization
- Conclusions

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## The need for 3D orientation microscopy

<u>2D Stereology</u>	<u>3D Destructive</u>	<u>3D Non-destructive</u>
statistical observations:	static observations:	process observations:
<ul style="list-style-type: none"> <li>• grain size distribution</li> <li>• grain shape (from 2 sample sections)</li> <li>• volume fraction and distribution of 2nd phase constituents</li> <li>• texture-microstructure relations</li> </ul>	<ul style="list-style-type: none"> <li>• comprehensive morphology information</li> <li>• 3D connectivity of features</li> <li>• grain boundaries</li> <li>• input data for modelling</li> <li>• 3D deformation structures</li> </ul>	<ul style="list-style-type: none"> <li>• recrystallization (e.g. nucleation, grain growth)</li> <li>• deformation (e.g. texture formation)</li> <li>• phase transformation (e.g. variant selection)</li> </ul>

Note: Many problems can be solved by 2D statistical observations; only for some 3D observations are essential

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## Serial sectioning methods

### Sectioning:

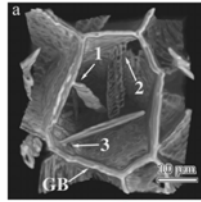
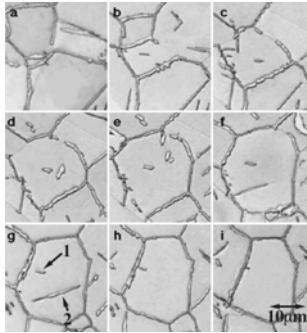
- mechanical or chemical polishing
- LASER ablation, FIB milling....

### 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 allotropic cementite by mechanical polishing

## Advantages of FIB-EBSD tomography

- Sectioning by FIB
  - accurate depth definition
  - flat and parallel sections ( $< 1^\circ$  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 ( $\sim 50$  nm)
- Combination of FIB and EBSD
  - table-top instrument
  - “high” measurement speed
  - fully automatic

### Reviews:

- *Uchic et al., MRS Bulletin 32 (2007) 408-416*
- *Zaefferer et al., Met. Mater. Trans. 39A, (2008) 374-389*
- *Zaefferer et al. in Schwarz et al: EBSD in Materials Science, Springer 2010*

## Alternative sectioning methods

- Automated mechanical or chemical-mechanical polishing (Uchic in: S. Ghosh and D. Dimiduk (eds.), Computational Methods for Microstructure-Property Relationships, c Springer Science+Business Media, 2011)

- large area/volume, no beam damage,
  - difficult to automate, depth accuracy
  
- Combination of laser ablation and FIB milling (Echlin et al., A new TriBeam system for three-dimensional multimodal materials analysis, Rev. Sci. Instrum. 83, 023701 (2012))

- large area/volume,
  - depth accuracy, beam damage
  
- Milling with a Plasma-FIB (Tescan Fera) (Hrnčir et al., Novel plasma FIB/SEM for high speed failure analysis, 3D tomography and other applications, Proc. EMC 2012, Manchester)

- large area/volume, less beam damage than Ga-FIB?
  - surface quality

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## Length scale of 3D orientation microscopy

The diagram illustrates the length scale of 3D orientation microscopy techniques, ranging from 1 nm to 1 mm. The scale is divided into several regions:

- 1 nm to 100 nm:** nano crystals
- 100 nm to 1000 nm:** submicron crystals
- 1000 nm to 10000 nm:** deformation structures
- 10000 nm to 100000 nm:** recrystallization structures "conventional" grains
- 100000 nm to 1000000 nm:** grain size & texture
- 1000000 nm to 10000000 nm:** macroscale texture fields

Key microscopy techniques and their characteristics are listed:

- TEM tomography:** 1 nm resolution, 50<sup>3</sup> nm<sup>3</sup> volume. H.H. Liu et al., Science 332 (2011)
- FIB-EBSD tomography:** 50 nm resolution, 50<sup>3</sup> μm<sup>3</sup> volume. Zaeferrer, Wright & Raabe Mat. Trans. A (2008) and Mulders & Day Mat Sci Forum 495-497 (2005)
- DAXM tomography:** 500 nm resolution, 100<sup>3</sup> μm<sup>3</sup> volume. B.C. Larson et al., Nature 415 (2002) 887
- MP-EBSD tomography:** 1 μm resolution, 1<sup>3</sup> mm<sup>3</sup> volume. M.V. Kral, G. Spanos, Acta Mater. 47 (1999) 711
- 3DXRD tomography:** 1...5 μm resolution, 1<sup>3</sup> mm<sup>3</sup> volume. H.F. Poulsen et al., J. Appl. Cryst. 34 (2001) 751

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### Principle of serial sectioning & orientation microscopy

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

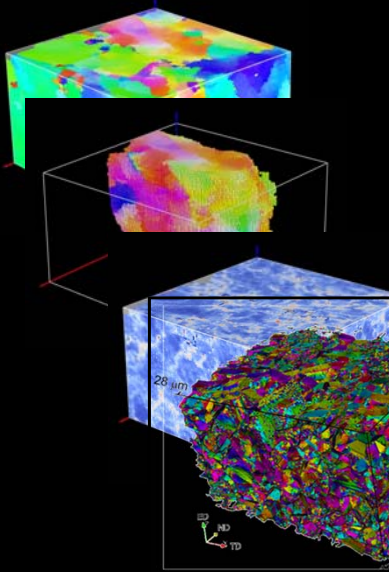
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### Geometrical set-up alternatives for FIB-EBSD

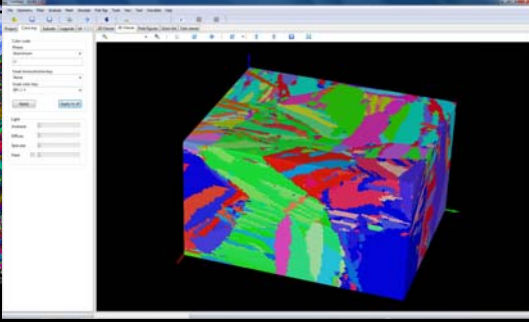
<p><b>tilt set-up</b></p> <ul style="list-style-type: none"> <li>+/- medium tilt positioning accuracy</li> <li>+ tilt inaccuracies create linear distortions</li> <li>+ simple software correction possible</li> <li>+ freely selectable milling position</li> </ul> <p><small>Zaefferer et al., <i>Met. Mater. Trans.</i> 39A, 374-389 (2008)</small></p>	<p><b>static set-up</b></p> <ul style="list-style-type: none"> <li>+ no stage movement required</li> <li>+ highest possible positioning accuracy</li> <li>+ unconventional but non-problematic EBSD set-up</li> <li>+ high measurement speed</li> </ul> <p><small>Proposed in: Zaefferer et al. in Schwarz et al.: <i>EBSD in Materials Science</i>, Springer 2010 Realized by N. Gey, Metz</small></p>	<p><b>rotation set-up</b></p> <ul style="list-style-type: none"> <li>+ high stage positioning accuracy</li> <li>+/- rotation inaccuracies create shear distortions</li> <li>+/- software correction more complex</li> <li>+/- every milling position requires a different holder</li> </ul> <p><small>Mulders, Day, <i>Mat. Sci. Forum</i> 495-497, 237-242 (2005)</small></p>
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## QUBE software package



- 3D Data in/output (all Laue sym.)
- Quaternion based crystallography
- Structure wizard
- Geometry editor
- Orientation filtering
- Orientation data analysis
- Boundary meshing / analysis
- Simulation tools / links

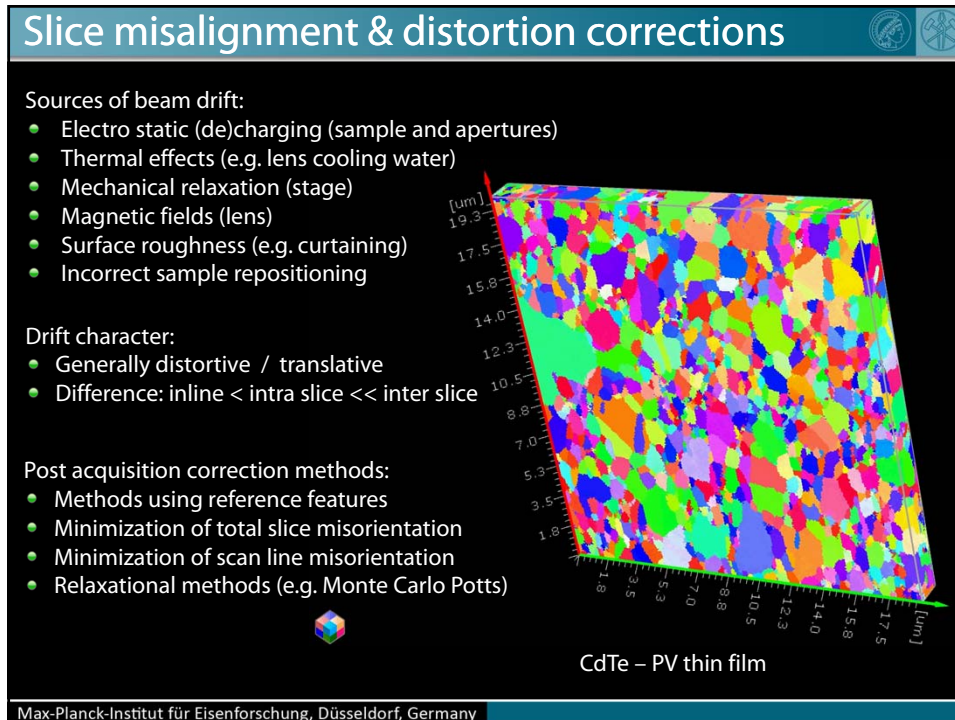
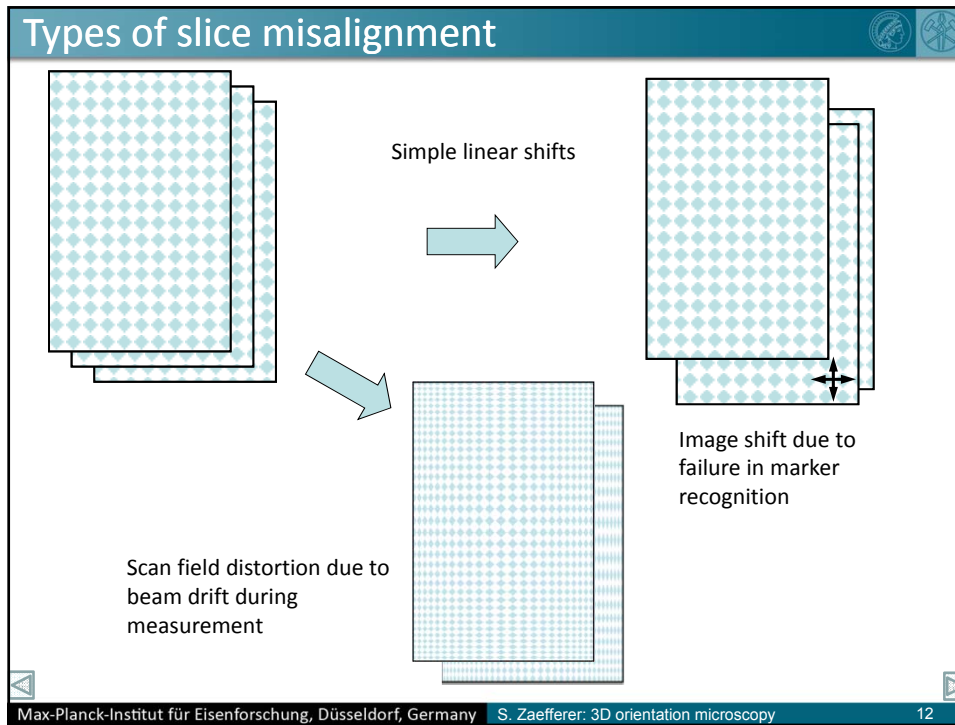


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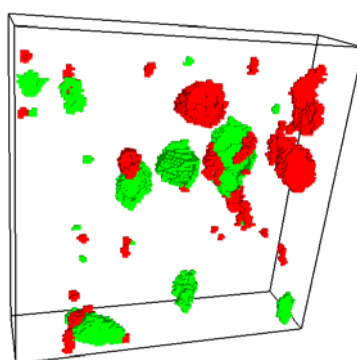
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## Combination of crystallographic and compositional data



Phase  
 NdO  
 pseudo hex Nd<sub>2</sub>O<sub>3</sub>

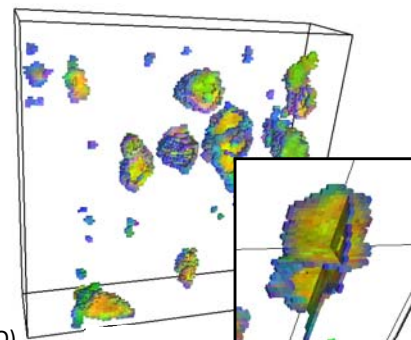
Fe-Nd-B Magnet material

150 x 150 x 40 voxels;

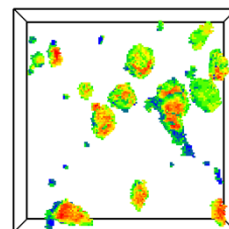
Simultaneous EBSD & EDX mapping

Courtesy T. Woodcock, IFW, Dresden

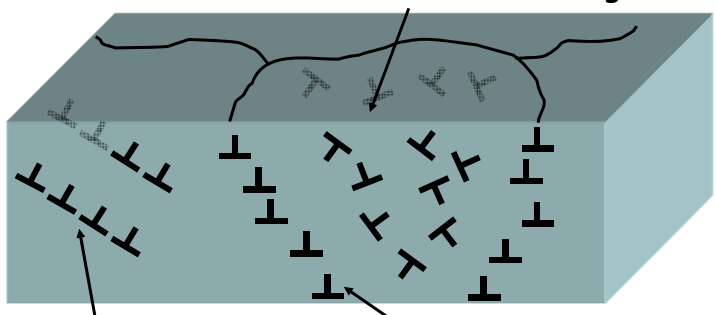
Red: EDS (O)  
 Green: EDS (Nd)  
 Blue: EDS (Fe)



3D Oxygen map



## Classification of dislocations for density calculations



SSDs inside of subgrains

GNDs in pile-ups      GNDs in subgrain boundaries

Geometrically necessary dislocations: GND  
 ⇒ Create measurable lattice rotations

Statistically stored dislocations: SSD  
 ⇒ Only lead to pattern blurring

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## Determination of GND densities from Nye tensor

Fundamental equation of continuum dislocation theory:

$\alpha$ : dislocation tensor       $\alpha = \text{curl} \beta_{el.}$        $\beta$ : el. strain gradient

The dislocation tensor is the rotation (curl) of the orientation field

e: permutation symbol       $\alpha_{ij} = e_{ikl} g_{jl,k}$       g: orientation tensor

The dislocation tensor can also be described as the local density sum of geometrically necessary dislocations

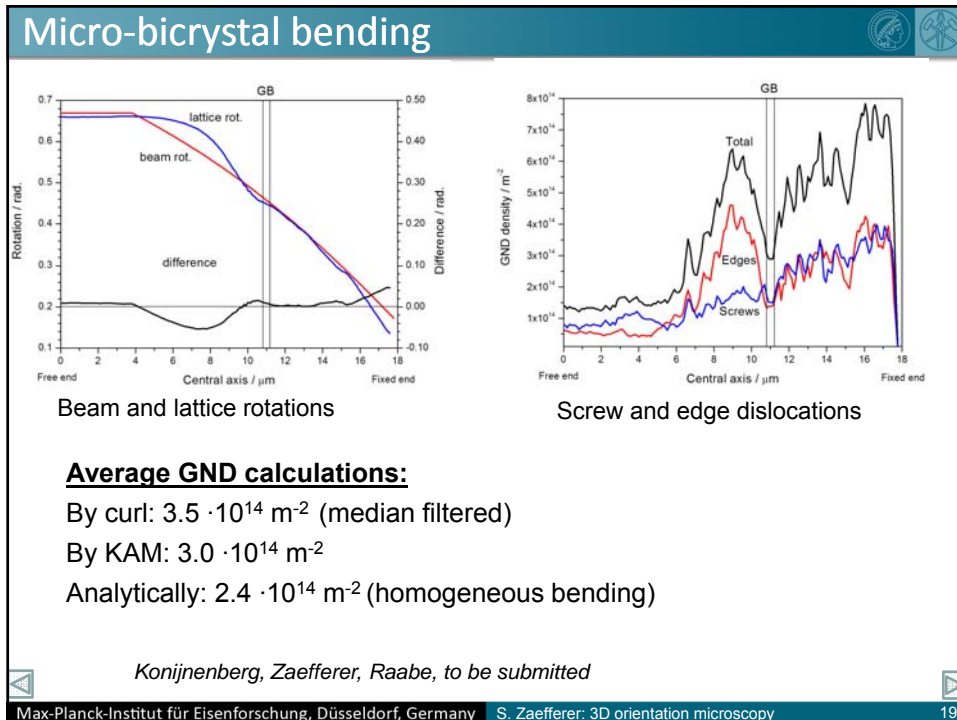
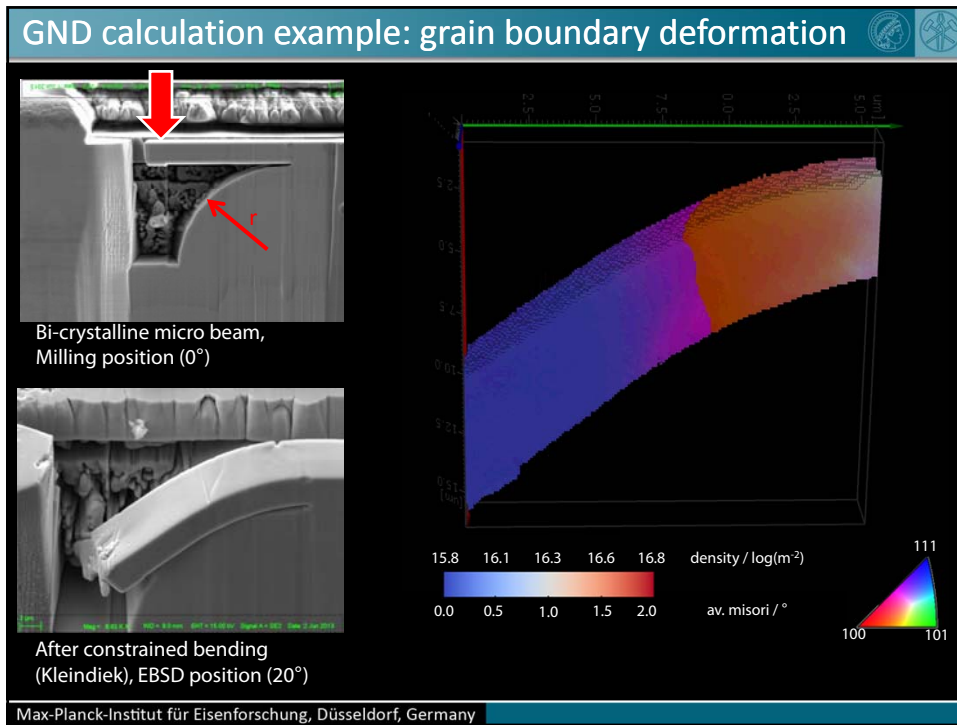
$\phi$ : disl. density of type k       $\alpha_{ij} = \sum_{k=1}^K \phi^k b_i^k \otimes t_j^k$       b: Burgers vector, t: line element  
 Dyadic product

Problem:  $\alpha$  has 9 components but, e.g. fcc crystals have 18 slip systems

Possible permutations:       $p = \binom{18}{9} = 48620$

Additional solution finding criterium: e.g. minimization of line energy.

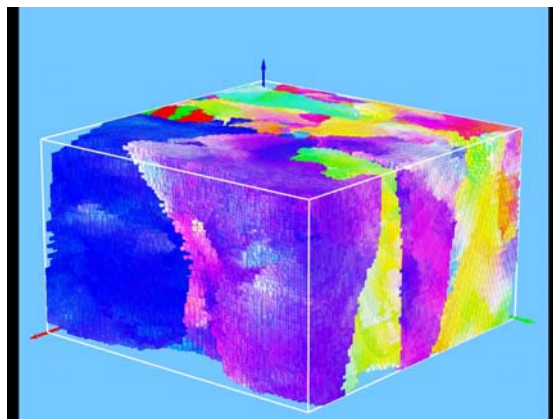
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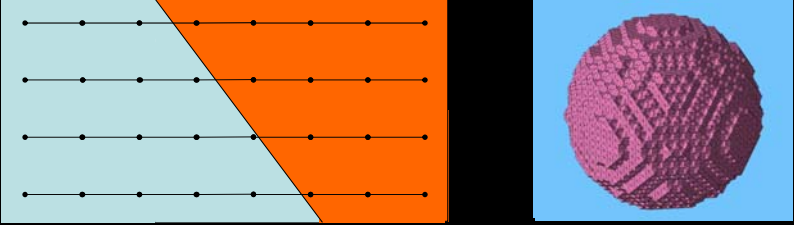
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## Transition from voxel to mesh description



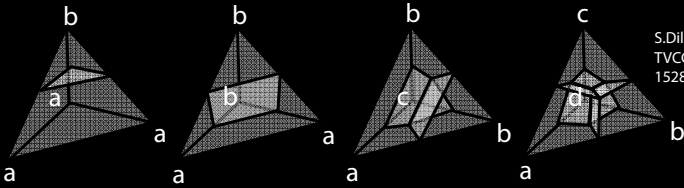
- Segmentation
- Meshing: marching tetraeder
- Smoothing: constrained vertex model
- Rendering

### Mesh generation: Marching Tetraeder



Spherical grain, radius = 14 voxels

S.Dillard et al. IEEE TVCG. 13(6) (2007), 1528-1535



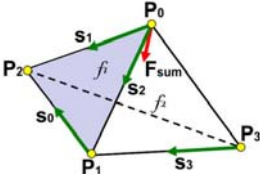
- Identifies 3 basic constituents (higher order junctions are decomposed)
- No topological ambiguities (compared to Marching Cube)
- High triangle density though

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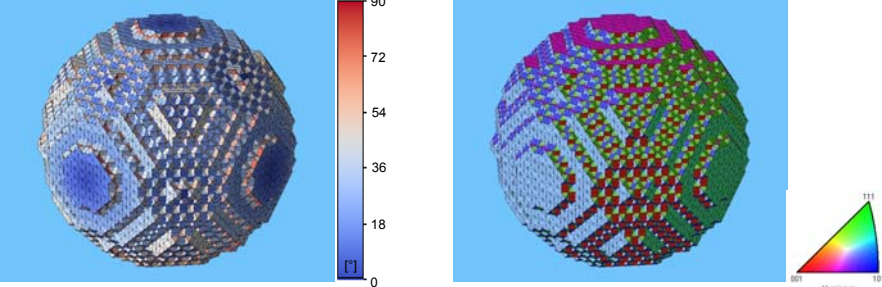
### Boundary smoothing by Constrained Vertex Model (CVM)

- Vertices are free to move under the action of a line/surface tension, mobility is fixed.
- All quadruple junctions remain fixed, no topological changes are allowed.
- Iterative smoothing of triple lines and boundaries to a desired degree.

$$v = m_{eff} F_{sum}$$

$$F_{sum} = \frac{\gamma_{GB}}{2} \sum_{i=1}^n \frac{s_{0i} \times (s_{1i} \times s_{0i})}{\|s_{1i} \times s_{0i}\|}$$


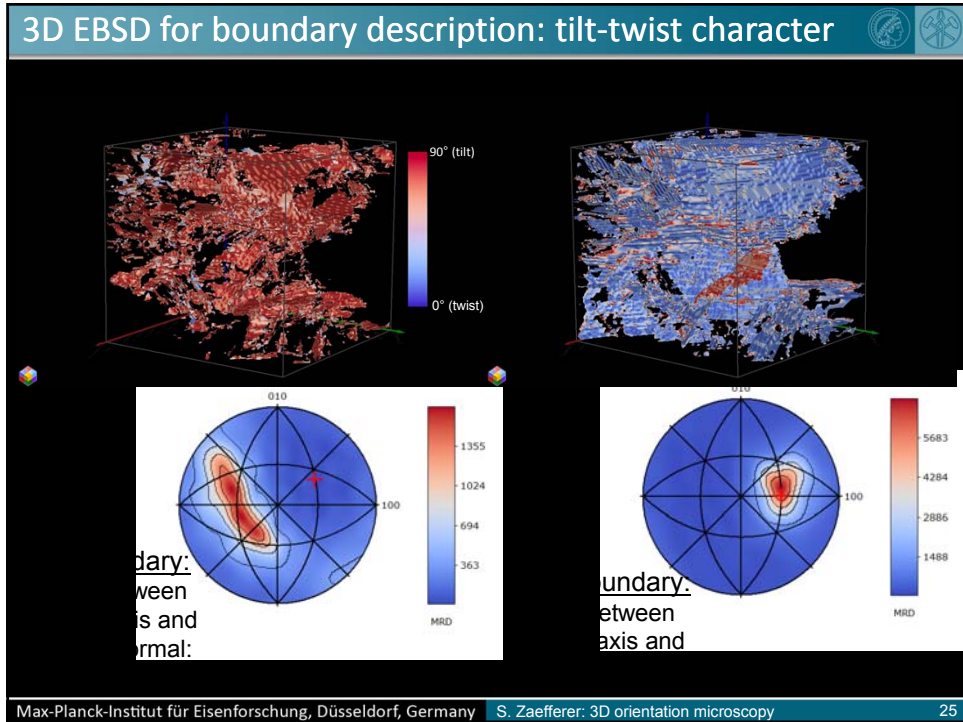
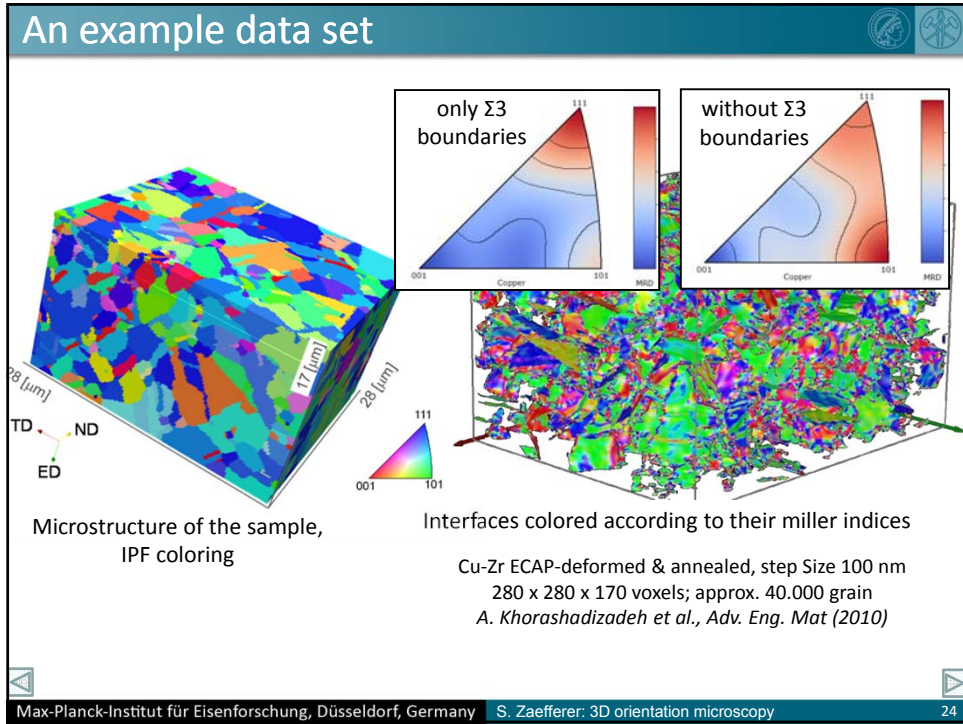
[1] L.A. Barrales Mora et al. Acta Materialia 56 (2008)

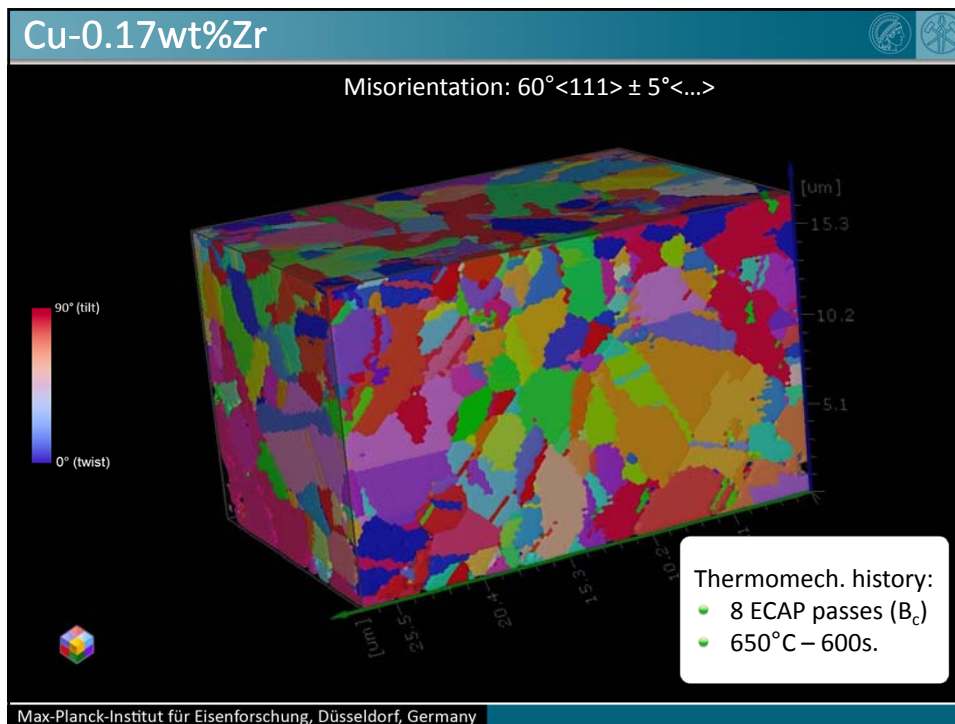


Surface normal deviation

Surface normal in cubic (m3m) IPF colors

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**CdTe thin film solar cells**

- CdTe solar cell provided by Laboratory for Thin Films and Photovoltaics, EMPA, Dübendorf.  
(collaboration with Prof. Tiwari and Dr. Buecheler)
- The CdTe layer was deposited by **High Vacuum Evaporation (HVE)** and has undergone a **CdCl<sub>2</sub> treatment**.

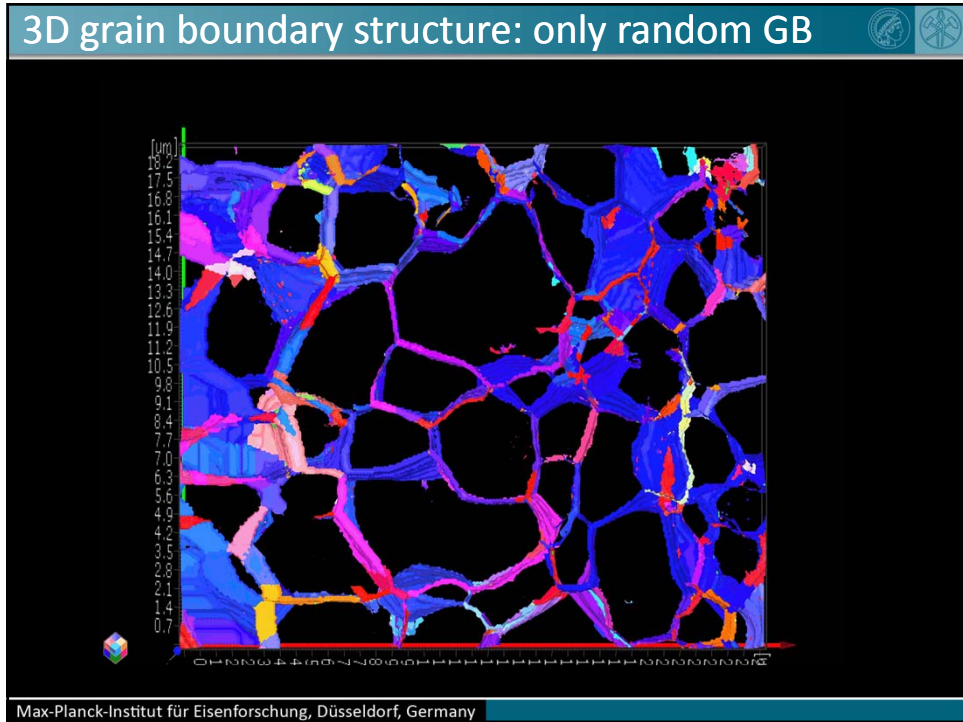
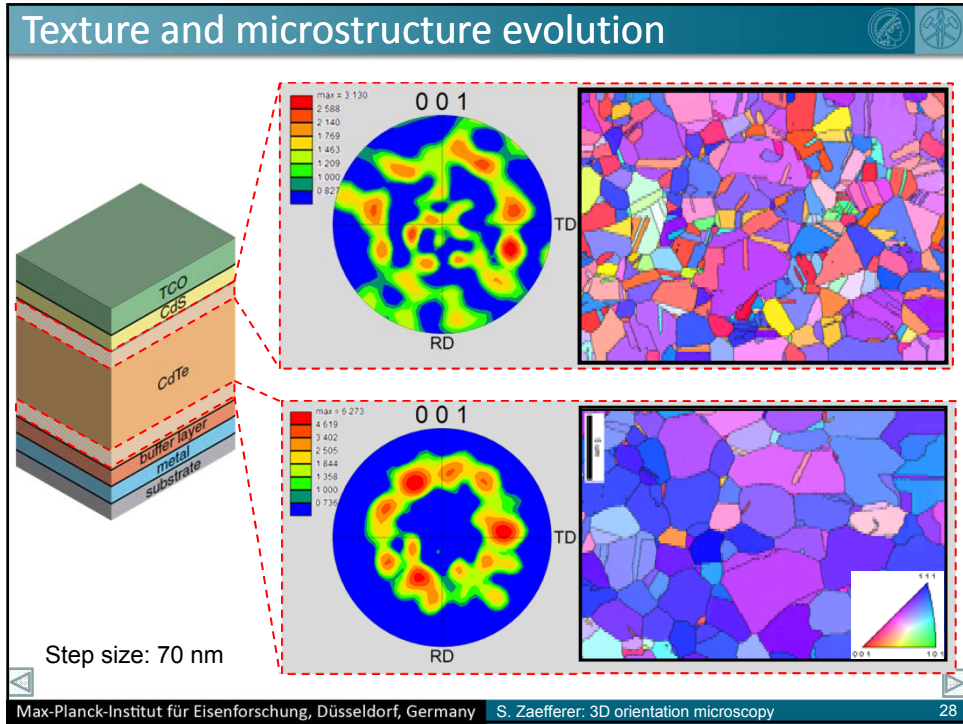
incidence of light

TCO  
CdS  
CdTe  
buffer layer  
metal  
substrate

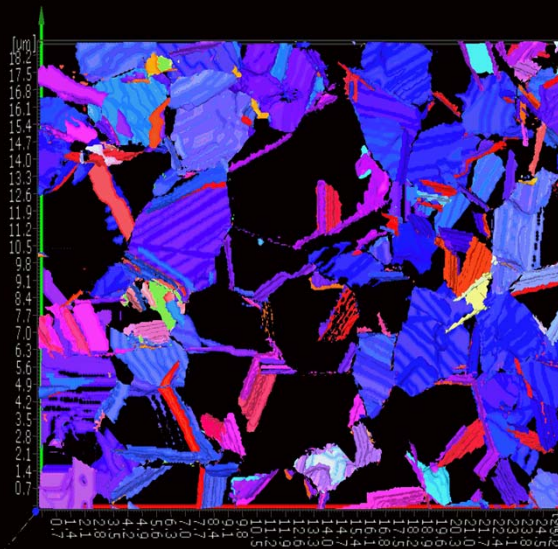
deposition order

*Gretener et al., Prog. Photovolt: Res. Appl., 2012*

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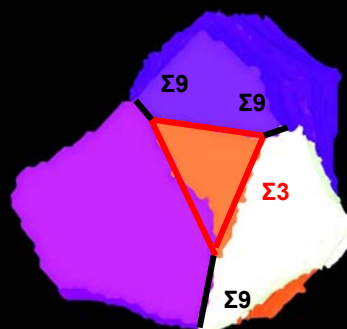


## 3D grain boundary structure: only twin GB



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## 3D twin structure



- Twin boundaries are only horizontal close to the substrate
- During the growth process they become vertical and form  $\Sigma 9$  boundaries
- What are their properties?

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### Chathodoluminescence (CL)

- electron beam creates electron-hole pairs
- electron hole pairs may diffuse in sample
- at non-defected areas: radiative recombination
  - light emission
- at defects: non-radiative recombination
  - no light emission

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### CL and EBSD to study electronic gb properties

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- Different boundaries show different charge carrier recombination intensity

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## Conclusions: 3D EBSD by FIB tomography

- Features:
  - Spatial resolution: min.  $50 \times 50 \times 50 \text{ nm}^3$
  - Observable volume: max.  $50 \times 50 \times 50 \text{ }\mu\text{m}^3$
- Problems (can be solved):
  - proper alignment of scan field distortions
  - orientation noise in the EBSD data
- Limits (cannot be overcome with this technique):
  - limited volume  $\Rightarrow$  statistically questionable data
  - destructive technique  $\Rightarrow$  no in-situ experiments
  - not applicable to FIB-sensitive materials
- Future:
  - new sectioning technique (mechanical, LASER)
  - analytical description of microstructure morphology

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