3D EBSD: Tomographic orientation microscopy in a FIB SEM


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Motivation and Features

Methods

Experimental set-up
Data analysis (GND; interface textures;
simulation: CPFEM, phase field, MC, CA)

Examples

Indentation: GND
DP steels: GND
ECAP processing: interface textures

Conclusions
Special 3D features of FIB-EBSD tomography

- Increase phase space of microstructure analysis
  6D ($\varphi_1, \varphi_2, x, y, z$): Crystallography and texture with morphology
  8D ($\varphi_1, \varphi_2, h, k, x, y, z$): Interface crystallography (interface texture)

- Spatial texture and phase correlations (connectivity, percolation, correlations)

- Connection to 3D microstructure modeling

Recent reviews:
- Uchic et al., MRS Bulletin 32 (2007) 408
Advantages of FIB-EBSD tomography (3D EBSD)

• 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
  – high measurement speed
  – fully automatic
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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

Approaches to 8D interface analysis and 3D GND analysis

Plane boundaries defined by triple junctions in two adjacent layers

Triangulation the interfaces for defining the grain boundaries

GND (Kröner-Nye)
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Cu, 60° conical, tip radius 1µm, loading rate 1.82mN/s, loads: 4000µN, 6000µN, 8000µN, 10000µN

Hardness and GND* in one experiment

Higher GND density at smaller scales?

* GND: geometrically necessary dislocations (accommodate curvature)
Nanoindentation (smaller is stronger): 3D EBSD and CPFEM

Cu, 60° conical, tip radius 1μm, loading rate 1.82mN/s, loads: 4000μN, 6000μN, 8000μN, 10000μN

* GND: geometrically necessary dislocations (accommodate curvature)
Comparison, crystal rotations (absolute, about [11-2] axis)

Absolute rotations

experiment

CPFEM, dislocation-based

From local misorientations to GNDs

\[ \triangle \phi = \phi(2) \phi^{-1}(1) \]

misorientation

\[ |\triangle \phi| = \min \{ \cos^{-1} \{ \text{tr} \left[ (O_i^{\text{cry}} \phi(1)) \phi_T^{(2)} O_j^{\text{cry}} \right] \} \} \]

\[ i = 1 \ldots 24, \ j = 1 \ldots 24 \]

orientation difference

\[ \phi(2) - \phi(1) = (\triangle \phi - I) \phi(1) \]

orientation gradient

\[ g_{ij,k} = \frac{\phi(2)_{ij} - \phi(1)_{ij}}{d_k} \]

(spacing d from EBSD scan)
From local misorientations to GNDs

\[
\beta_{ij} = \frac{\delta u_i}{\delta x_j} = \beta_{ij}^{el} + \beta_{ij}^{pl}
\]

**distortion**

(sym, a-sym)

<table>
<thead>
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<th>(u=u(x,y,z))</th>
<th>((x_1,y,z))</th>
<th>(u_1(x,y,z))</th>
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From local misorientations to GNDs

\[ \beta_{ij} = \frac{\delta u_i}{\delta x_j} = \beta_{ij}^{el} + \beta_{ij}^{pl} \]

distortion (sym, a-sym)

\[ \alpha = \nabla \times \beta^{el} \]

\[ \alpha_{pi} = e_{pkj} \left( \epsilon_{ij,k}^{el} + g_{ij,k} \right) \]

\[ \alpha_{pi} = e_{pkj} g_{ij,k} \]

dislocation tensor (GND)

From local misorientations to GNDs

Slip and line directions of dislocations for GNDs in a FCC crystal

\[ \sqrt{2} \hat{b} : \begin{array}{ccccccccccc} 110 & 10\bar{1} & 0\bar{1}1 & 1\bar{1}0 & 101 & 01\bar{1} & 110 & 10\bar{1} & 0\bar{1}1 & 1\bar{1}0 & 101 & 011 & \bar{1}10 & 10\bar{1} & 0\bar{1}1 \end{array} \]

\[ \sqrt{6} \hat{t} : \begin{array}{ccccccccccc} \bar{1}12 & \bar{1}21 & 2\bar{1}1 & \bar{1}12 & 121 & 211 & 1\bar{1}2 & 121 & 2\bar{1}1 & 112 & \bar{1}21 & 21\bar{1} & 110 & 101 & 011 & \bar{1}10 & 10\bar{1} & 0\bar{1}1 \end{array} \]

\[ \mathbf{B} = \mathbf{b}(\hat{\mathbf{t}} \cdot \mathbf{r}) = (\mathbf{b} \otimes \hat{\mathbf{t}})\mathbf{r} \]

Frank loop through area \( \begin{array}{ccccccccccc} b,t \end{array} \) combinations

\[ \alpha_{ij} = \sum_{a=1}^{18} \rho_{gnd}^a b_i^a t_j^a \]

\[ \alpha_{ij} = \sum_{a=1}^{9} \rho_{gnd}^a b_i^a t_j^a \]

18 \( b,t \) combinations

9 \( b,t \) combinations
Extract geometrically necessary dislocations
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3D-EBSD - results

Image Quality

Kernel Average Misorientation (martensite highlighted in black)

Calcagnotto et al. ISIJ 48 (2008) 1096

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DP steel micromechanics

GND density vs. FM interface fraction

average GND density in ferrite [1/m²]

interface covered by martensite [%]

large grain

small grain

0-1 μm³
1-2 μm³
2-4 μm³
> 4 μm³

DP steel: effect of GNDs on local hardness?

Coarse grained DP (12.4 µm), Berkovich 50 nm, 500 µN

![Graph showing nanohardness vs. distance from phase boundary for coarse and fine grained DP steels.](image-url)
Overview

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ECAP processing: 3D microstructure and interface analysis

Cu-0.17wt%Zr – as deformed

4 ECAP passes

8 ECAP passes

color: crystal direction parallel to ECAP direction (ED)
ECAP processing: 3D microstructure and interface analysis

Cu-0.17wt%Zr – 8 ECAP passes and heat treated at 650°C, 10 min.

color: crystal direction parallel to ECAP direction (ED)

experiment: texture

experiment: interfaces

phase field simulation: Melzer, Nestler
Cu-0.17wt%Zr – 8 ECAP passes and heat treated at 650°C, 10 min.

Misorientation angles of grain boundaries

Highest fraction
Cu-0.17wt%Zr – 8 ECAP passes and heat treated at 650°C, 10 min.

60° [111]

Max. 818.70

Resolution: 8.18°

CLS method: Rollett
ECAP processing: 3D microstructure and interface analysis

Cu-0.17wt%Zr – as deformed after 8 passes

Area fraction of $\Sigma 3 \sim 3$

8 ECAP passes

Misorientation angles of grain boundaries

Number fraction [%]

Misorientation angle [degrees]
ECAP processing: 3D microstructure and interface analysis

Cu-0.17wt%Zr – as deformed after 8 passes

Resolution: 8.18°

Max. 5
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- **Conclusions**
• 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: $\approx$ 50 x 50 x 50 µm$^3$
• Angular resolution: 0.5° (precision of tilt)
• Time per cut: 15 … 60 min /cycle
• Examples: Size effect, interface crystallography, interface strengthening