

3D EBSD analysis of mechanical size effects during indentation: EBSD tomography and crystal plasticity FEM

E. Demir, N. Zaafarani, F. Weber, D. Ma, S. Zaefferer, F. Roters, D. Raabe



**Max-Planck-Institut
für Eisenforschung GmbH**

Department of Microstructure Physics and Metal Forming
Düsseldorf, Germany

d.raabe@mpie.de

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- Motivation and approach
- Sample preparation
- Deformation experiments
- Characterization
- Orientation gradients
- Dislocation analysis
- Results
- Other examples: Wire bending, pillar compression
- Conclusions



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Challenges in size dependent mechanics

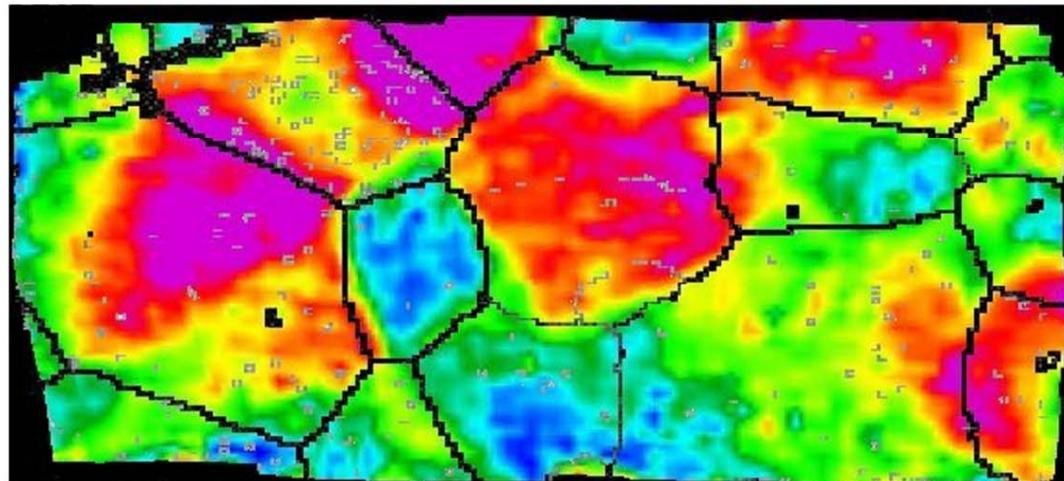
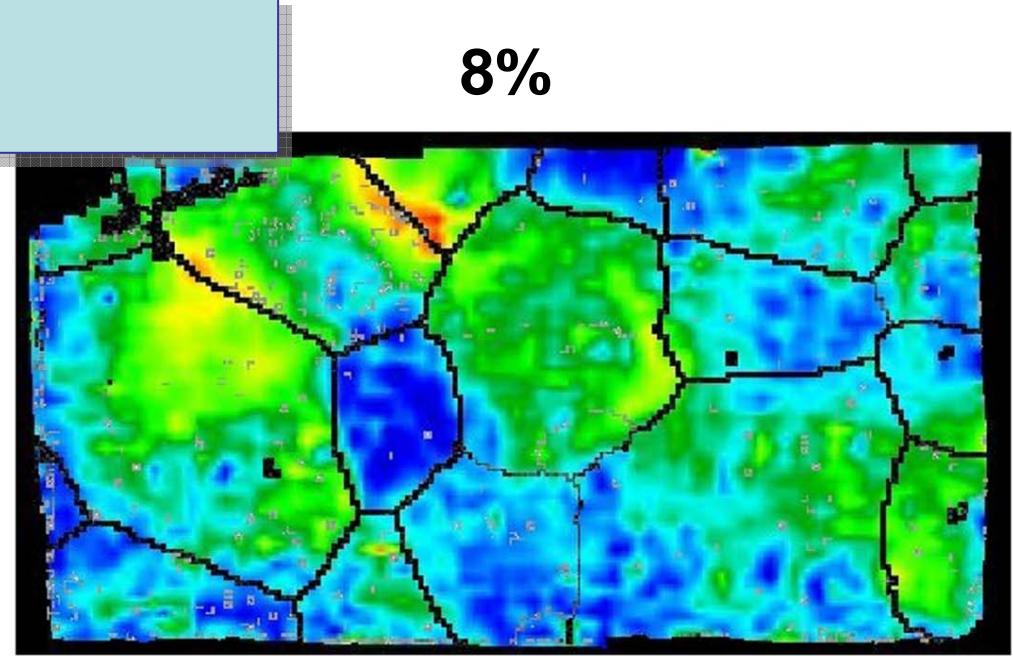
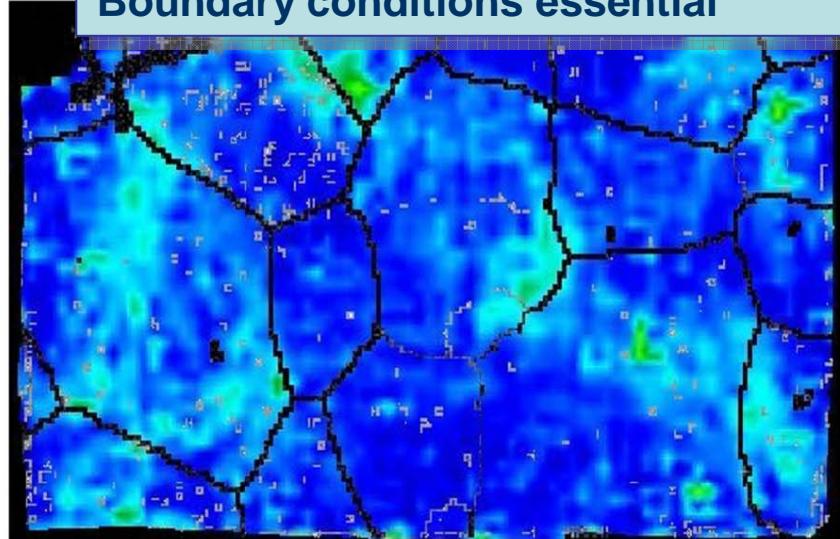
- **Theoretical concept (GND, gradients, sources, hardening, localization, ...)**
- **Experiments asking a specific question**
- **Homogeneity of the microstructure**
- **Boundary conditions of the experiment**

Homogeneity and boundary conditions – large scale

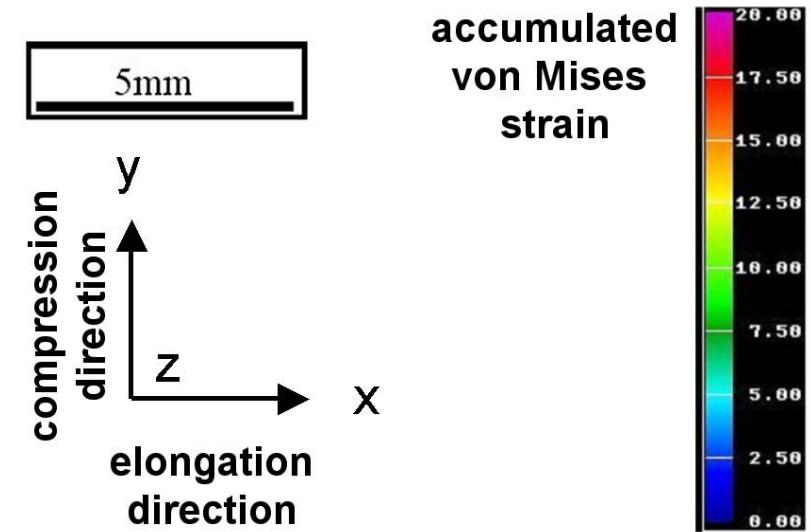


Microstructure not homogeneous

Boundary conditions essential



15%

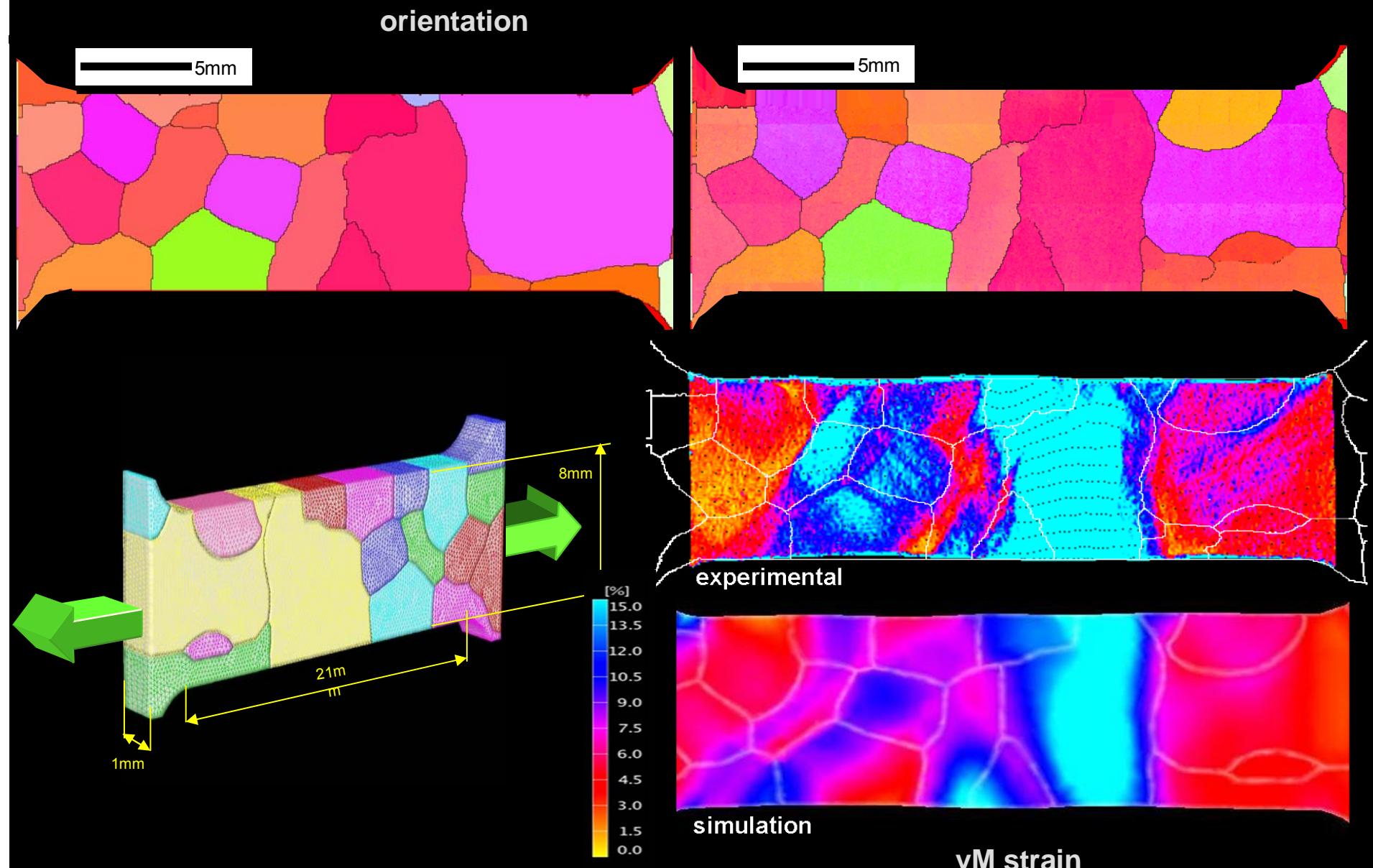


M. Sachtleber, Z. Zhao, D. Raabe: Mater. Sc. Engin. A 336 (2002) 81

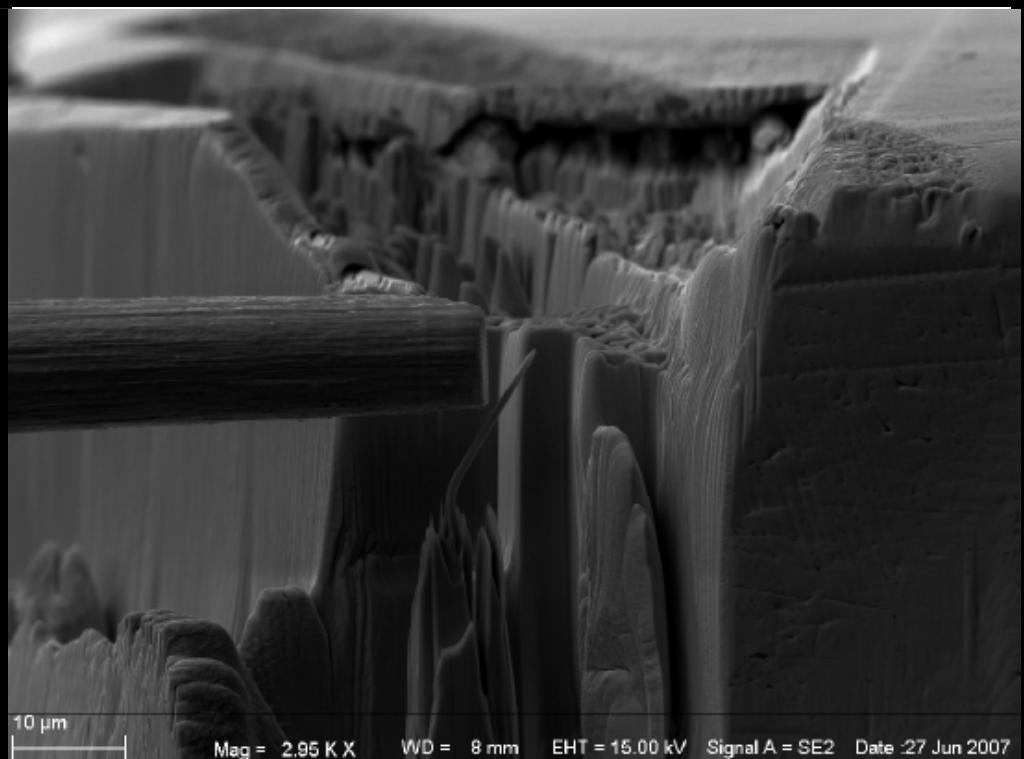
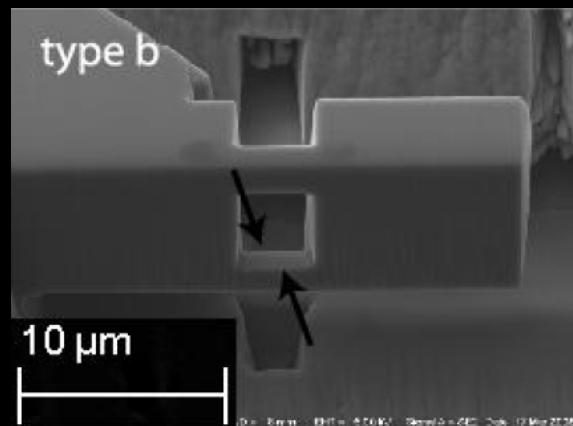
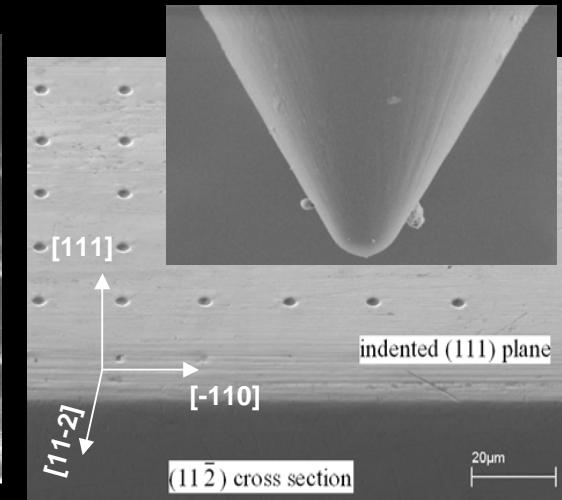
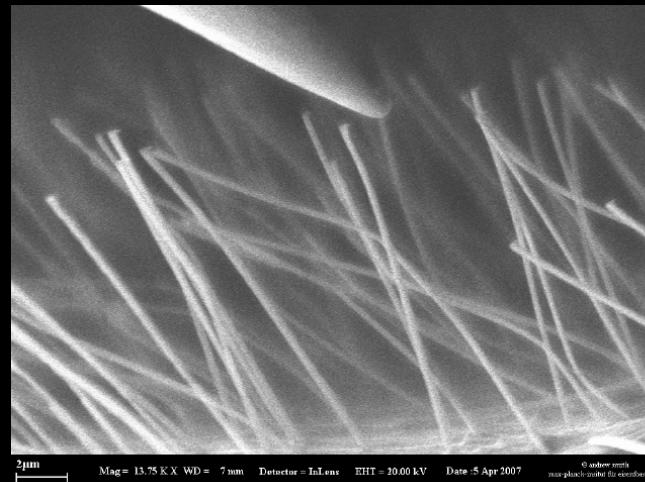
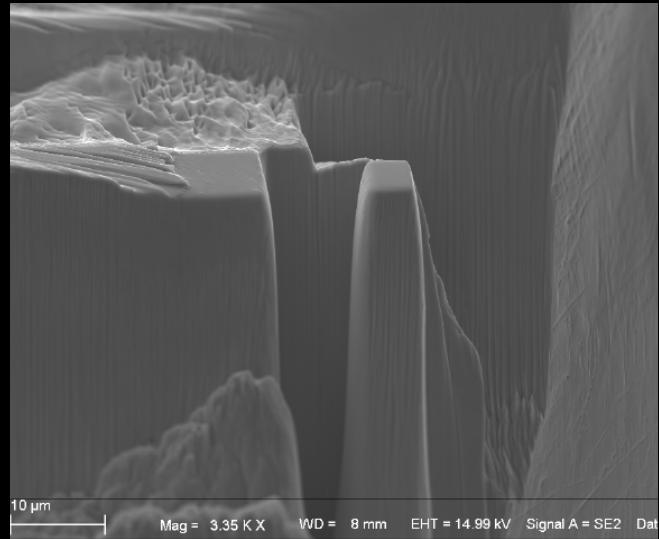
D. Raabe, M. Sachtleber, Z. Zhao, F. Roters, S. Zaefferer: Acta Mater. 49 (2001) 3433–3441

4

Crystal plasticity FEM, grain scale mechanics (3D)



Homogeneity and boundary conditions – small scale





- Relationship between ISE and GND: hardness and GND in the same experiment
- Indents of different depths, Cu single crystal
- High resolution 3D EBSD
- Determine plastic volume beneath indents
- Calculate GNDs around indents from 3D orientation gradients and plastic volume
- Also: Wire bending; pillar compression

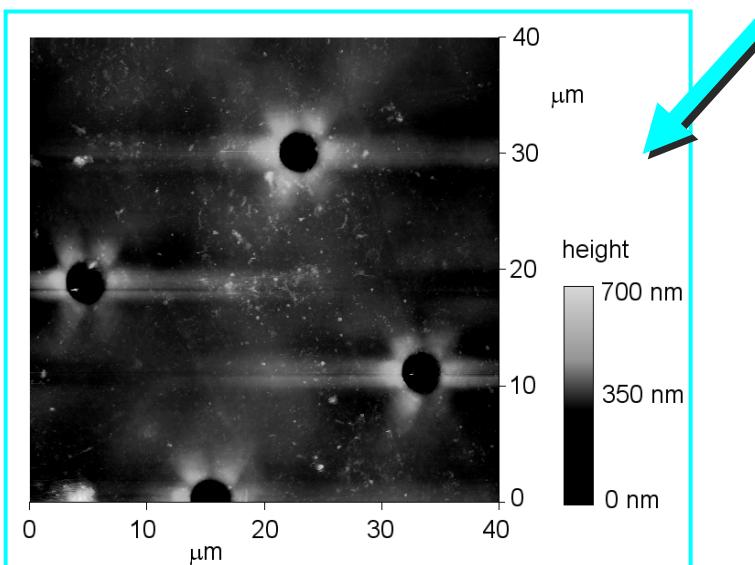
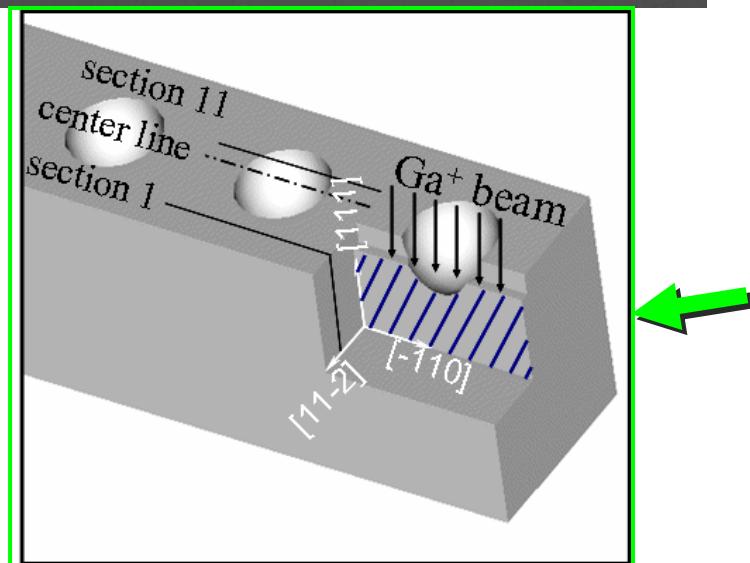
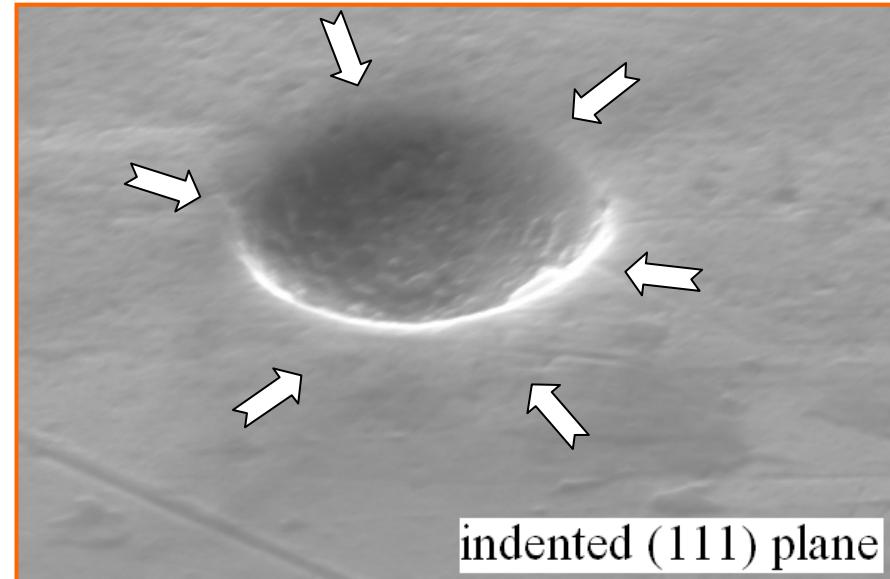
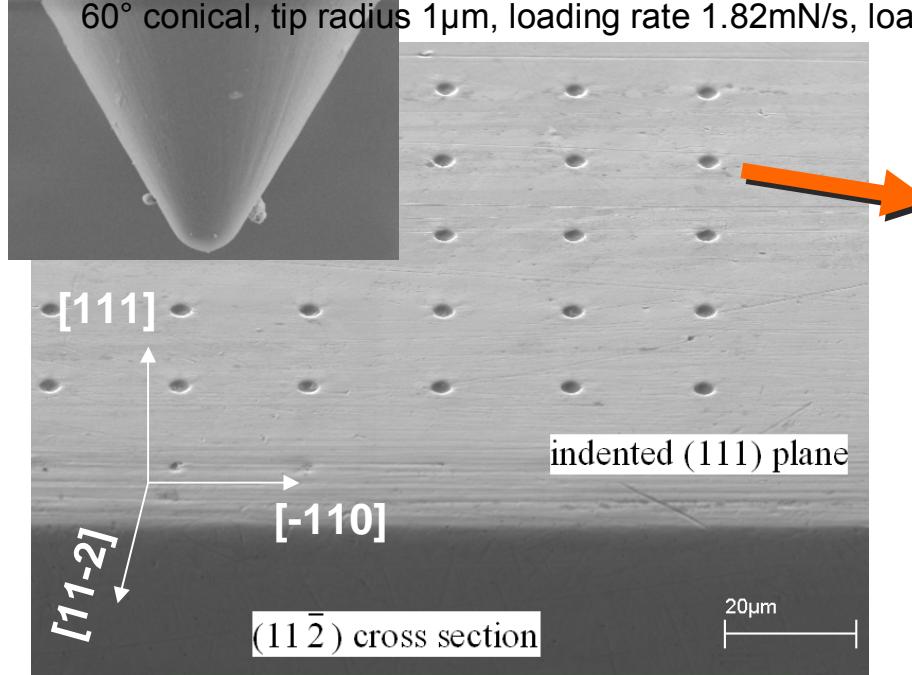


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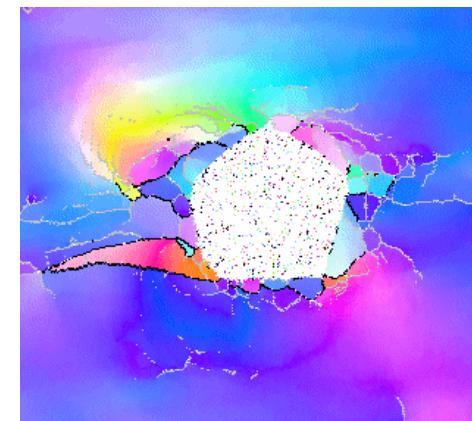
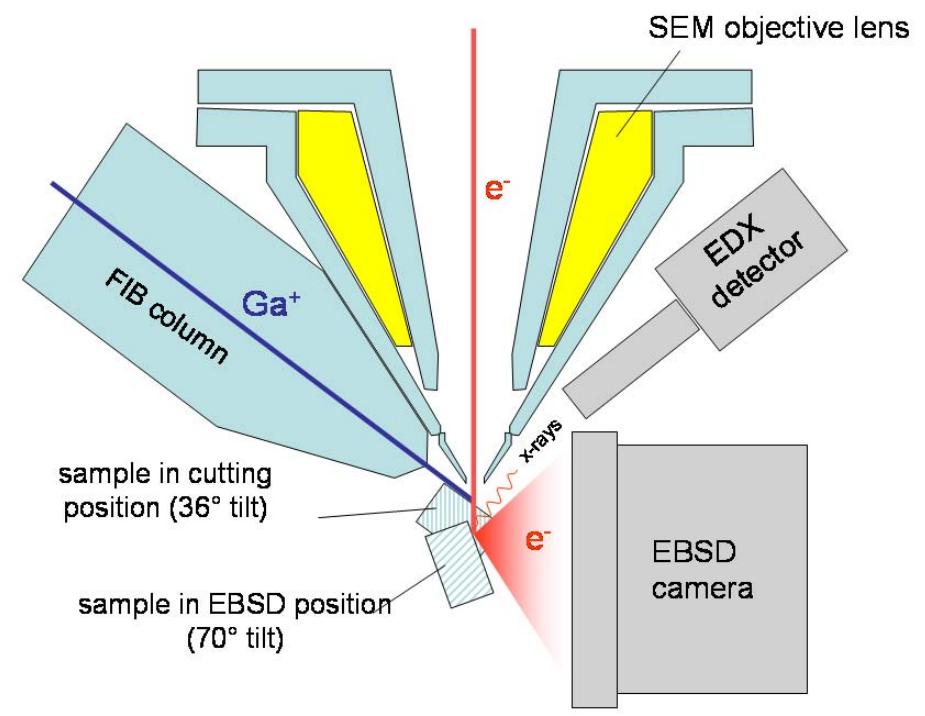
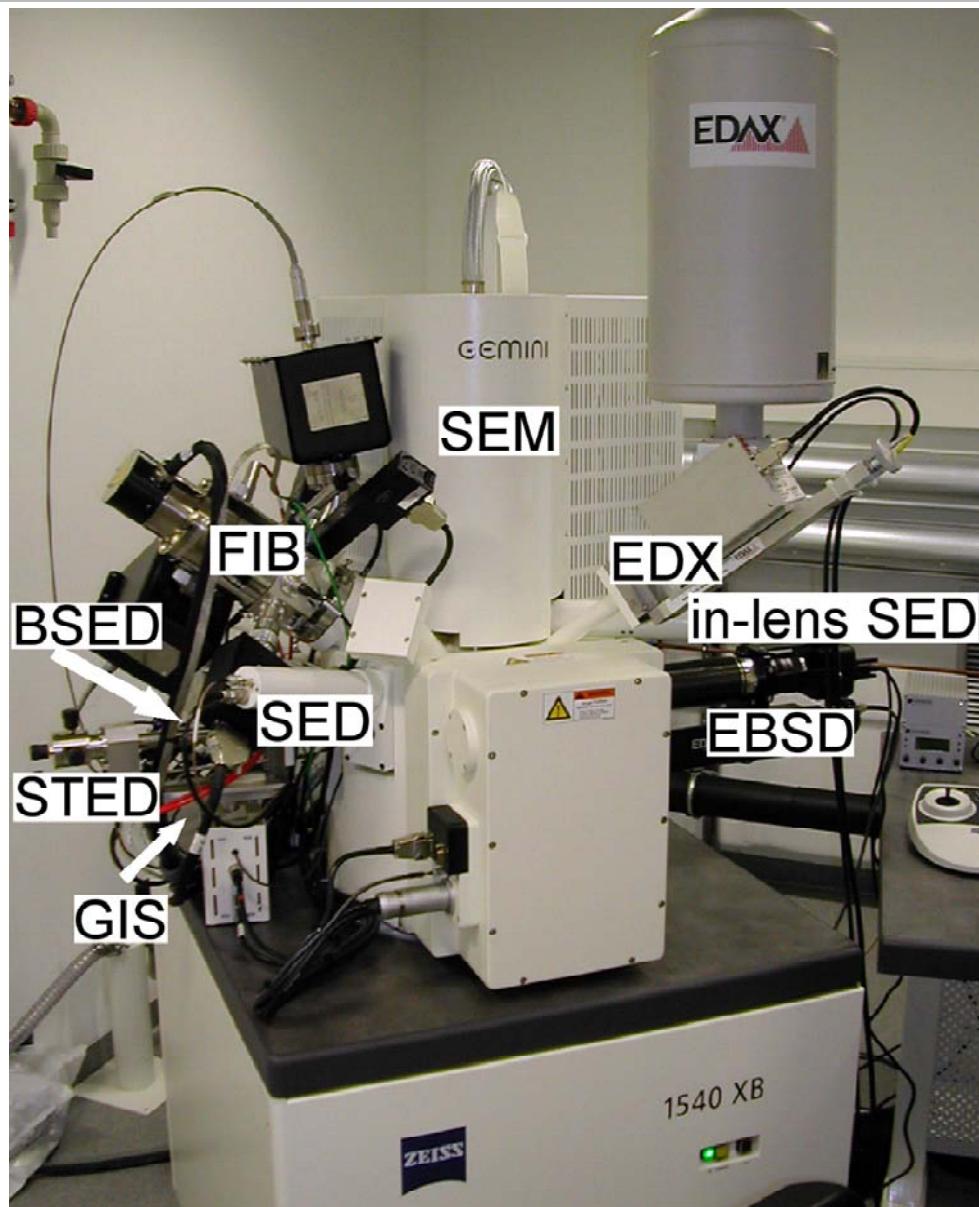
Nanoindentation



60° conical, tip radius 1 μ m, loading rate 1.82mN/s, loads of 4000 μ N, 6000 μ N, 8000 μ N and 10000 μ N



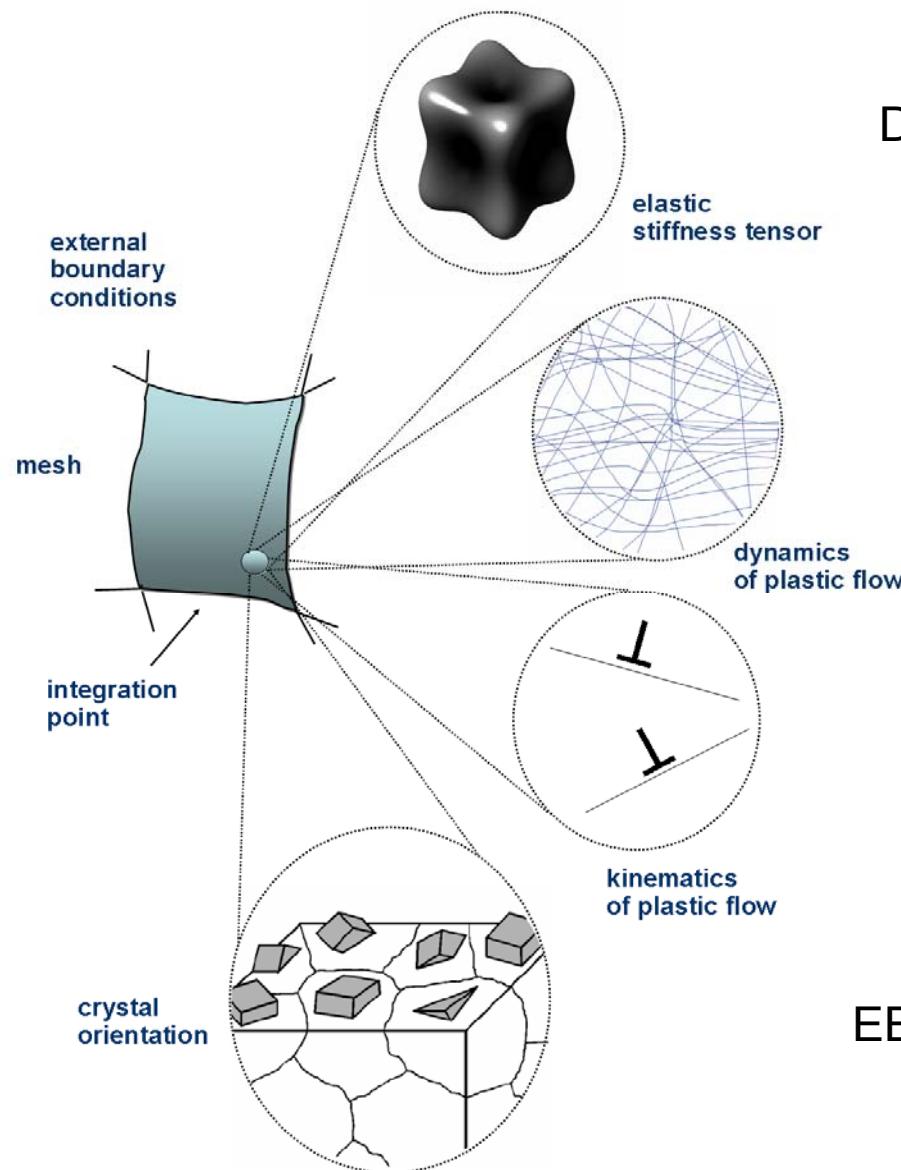
3D electron microscopy, 3D EBSD, tomography



J. Konrad, S. Zaefferer, D. Raabe, Acta Mater. 54 (2006) 1369
S. Zaefferer, S. I. Wright, D. Raabe, Metall. Mater. Trans. A 39A (2008) 374

Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany

Dierk Raabe, PLASTICITY 2009, Jan. 2009, d.raabe@mpie.de



DFT, ultrasonics

Statistical dislocation-based hardening law in CPFEM

DDD, TEM, single crystal tests

Crystallography, MD, Peierls analysis

Kinematics in CPFEM

EBSD, RVE,....

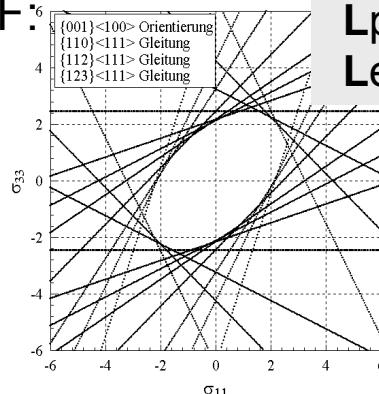


Multiplicative Decomposition of the Deformation Gradient

constitutive reduction of DOF:

$$\tilde{\mathbf{L}}_p = \sum_{\alpha=1}^{12} \dot{\gamma}_\alpha \tilde{\mathbf{d}}_\alpha \otimes \tilde{\mathbf{n}}_\alpha$$

flow law



\mathbf{F}^* : elastic and rotation deformation gradient

\mathbf{F} : total deformation gradient

\mathbf{F}_p : plastic deformation gradient

\mathbf{L}_p : plastic velocity gradient

\mathbf{L}_e : elastic velocity gradient

Definitions:

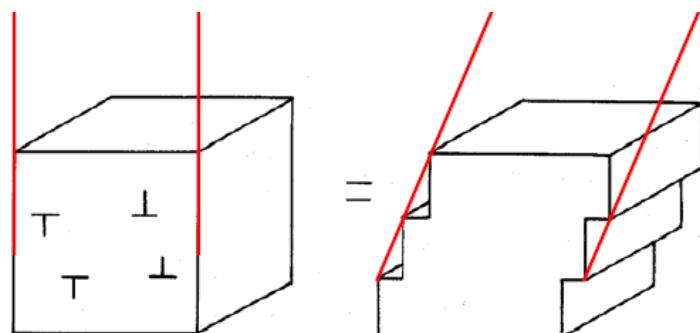
$$\mathbf{L} := \dot{\mathbf{F}} \mathbf{F}^{-1}$$

$$\mathbf{L}_e := \dot{\mathbf{F}}_e \mathbf{F}_e^{-1}$$

$$\tilde{\mathbf{L}}_p := \dot{\mathbf{F}}_p \mathbf{F}_p^{-1}$$

$$\mathbf{L}_p := \mathbf{F}_e \tilde{\mathbf{L}}_p \mathbf{F}_e^{-1}$$

$\dot{\gamma}_\alpha(\tau_\alpha, \tau_{c\alpha}, \theta) \longrightarrow \text{Phenomenological}$
 $\dot{\gamma}_\alpha(\tau_\alpha, \rho, \theta) \longrightarrow \text{Dislocation density laws}$



$$\dot{\gamma} = \frac{d\gamma}{dt} = n \frac{dx}{X} \frac{b}{Z} \frac{1}{dt} = \rho_m b v$$

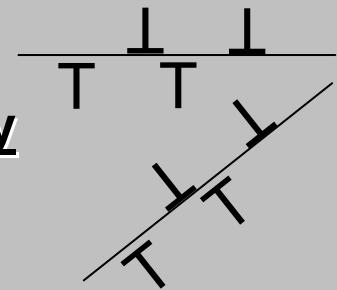


1

1. set
internal
variables

dyadic flow law based on dislocation rate theory

Taylor, Kocks, Mecking, Estrin, Kubin,...

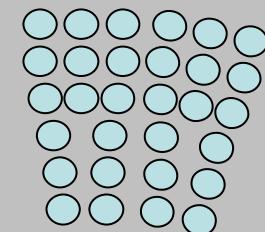


2

2. set
internal
variables

plastic gradients,
size scale and orientation gradients (implicit)

Nye, Ashby, Kröner,....

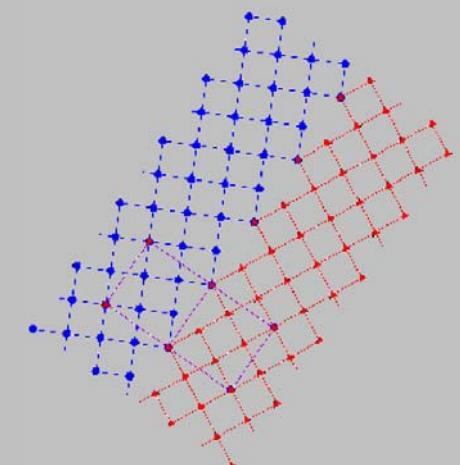


3

3. set
internal
variables

grain boundaries

activation concept:
energy of formation upon slip penetration: conservation law





➤ ***Mobile dislocations***

accommodate the external plastic deformation

➤ ***Immobile dislocations (cell interior, cell walls)***

work hardening, including locks and dipoles

➤ ***Geometrically necessary dislocations***

preserve the lattice continuity

Rate equations are formulated for the immobile dislocation densities:

$$\dot{\rho}_{\text{SSD}} = (\dot{\rho}_I^+)_{\text{SSD}} + (\dot{\rho}_{II}^+)_{\text{SSD}} + (\dot{\rho}_I^-)_{\text{SSD}} + (\dot{\rho}_{II}^-)_{\text{SSD}}$$

Four mechanisms:

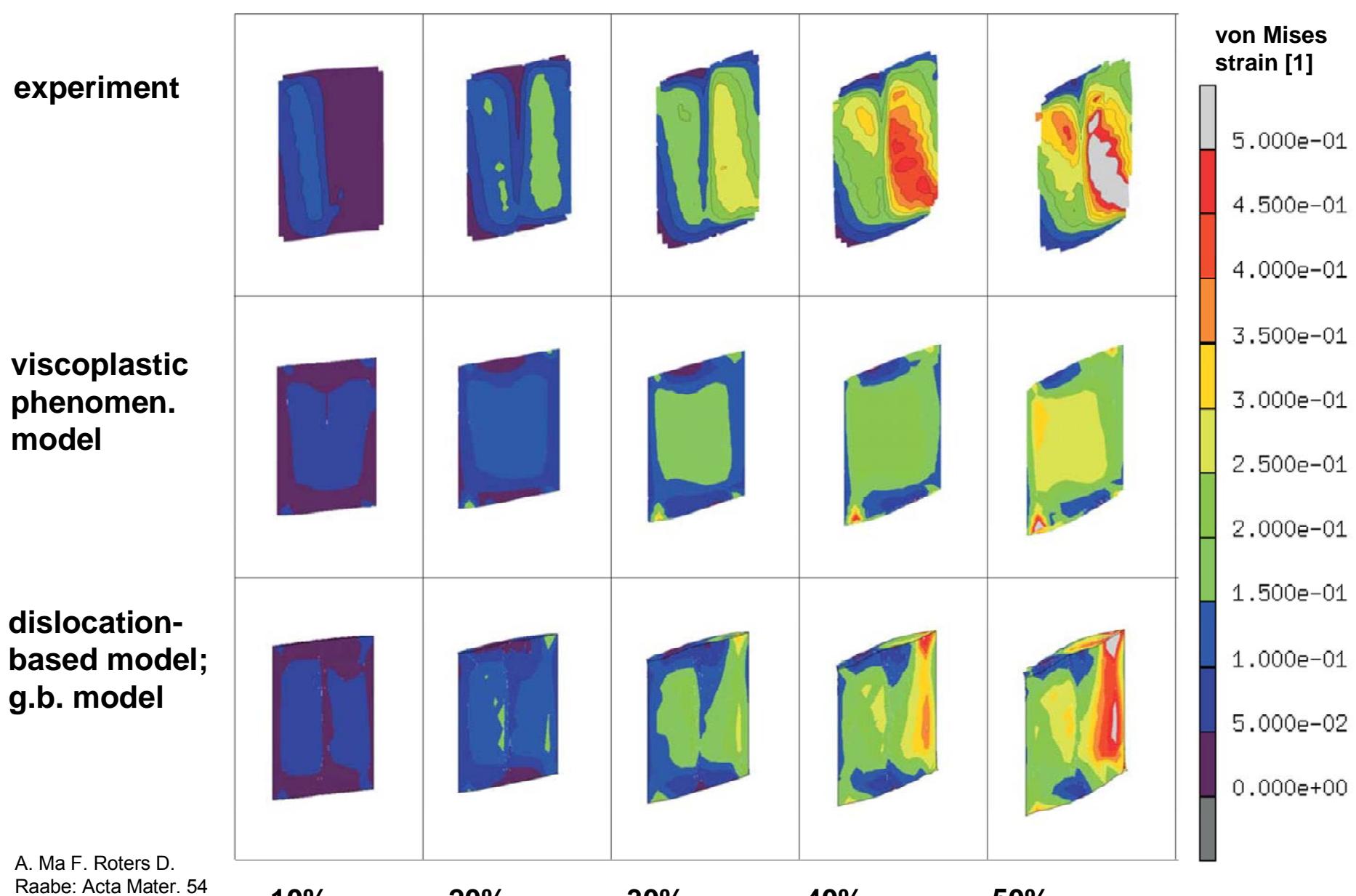
mechanism 1 : lock formation

mechanism 2 : dipole formation

mechanism 3 : athermal annihilation

mechanism 4 : thermally activated annihilation by climb

Al Bicrystals, low angle g.b. [112] 7.4°, v Mises strain



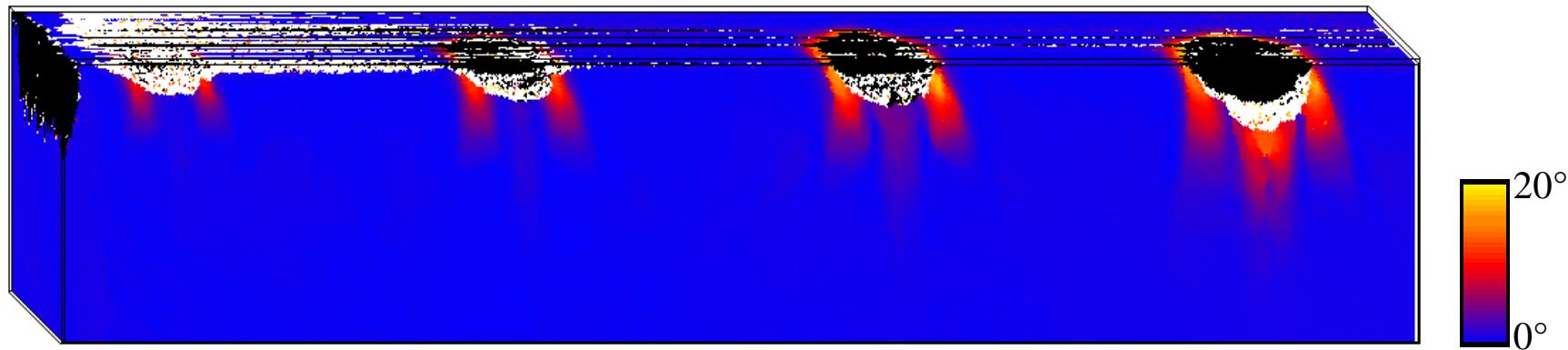
A. Ma F. Roters D.
Raabe: Acta Mater. 54
(2006) 2181

16

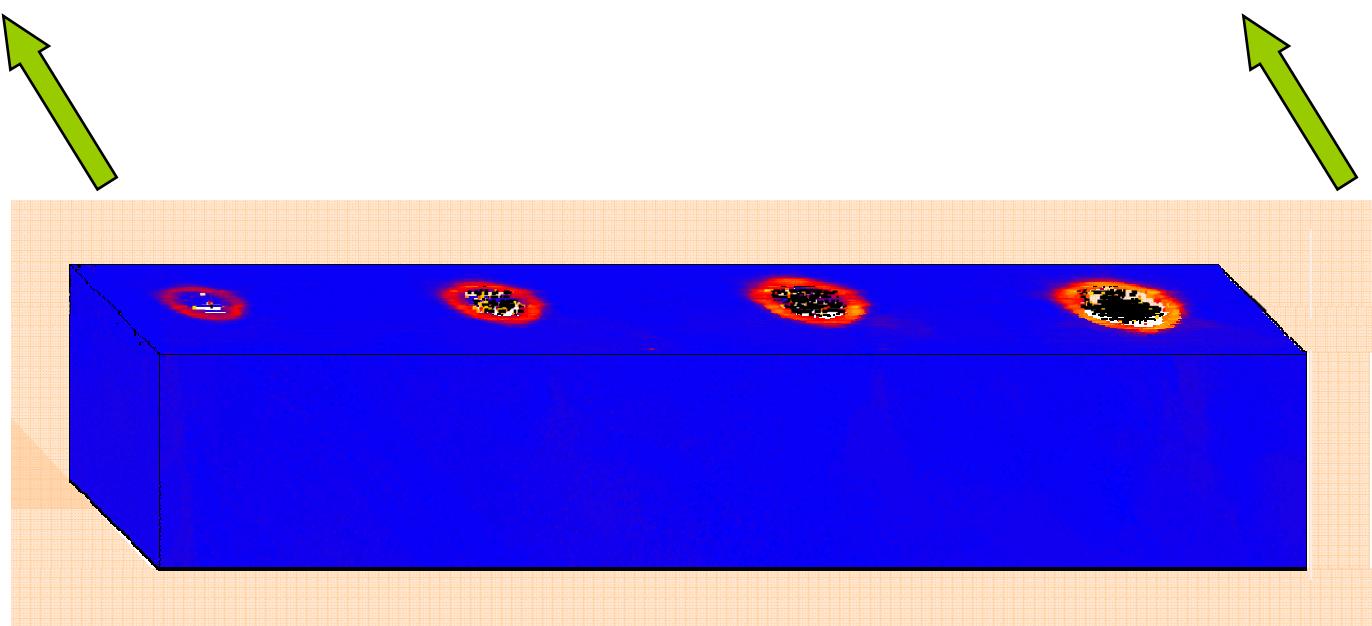
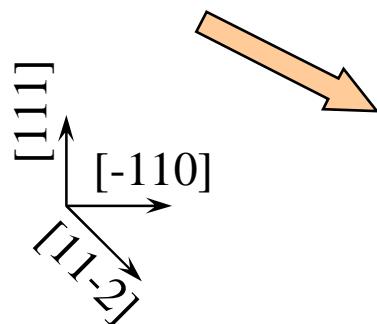
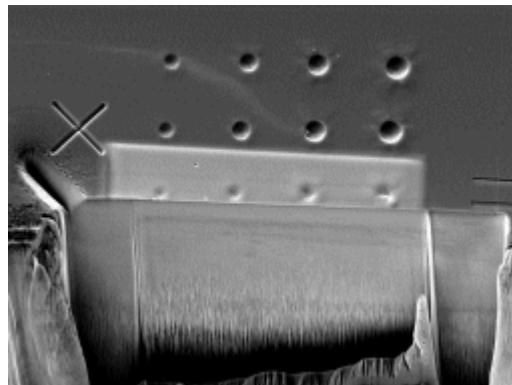


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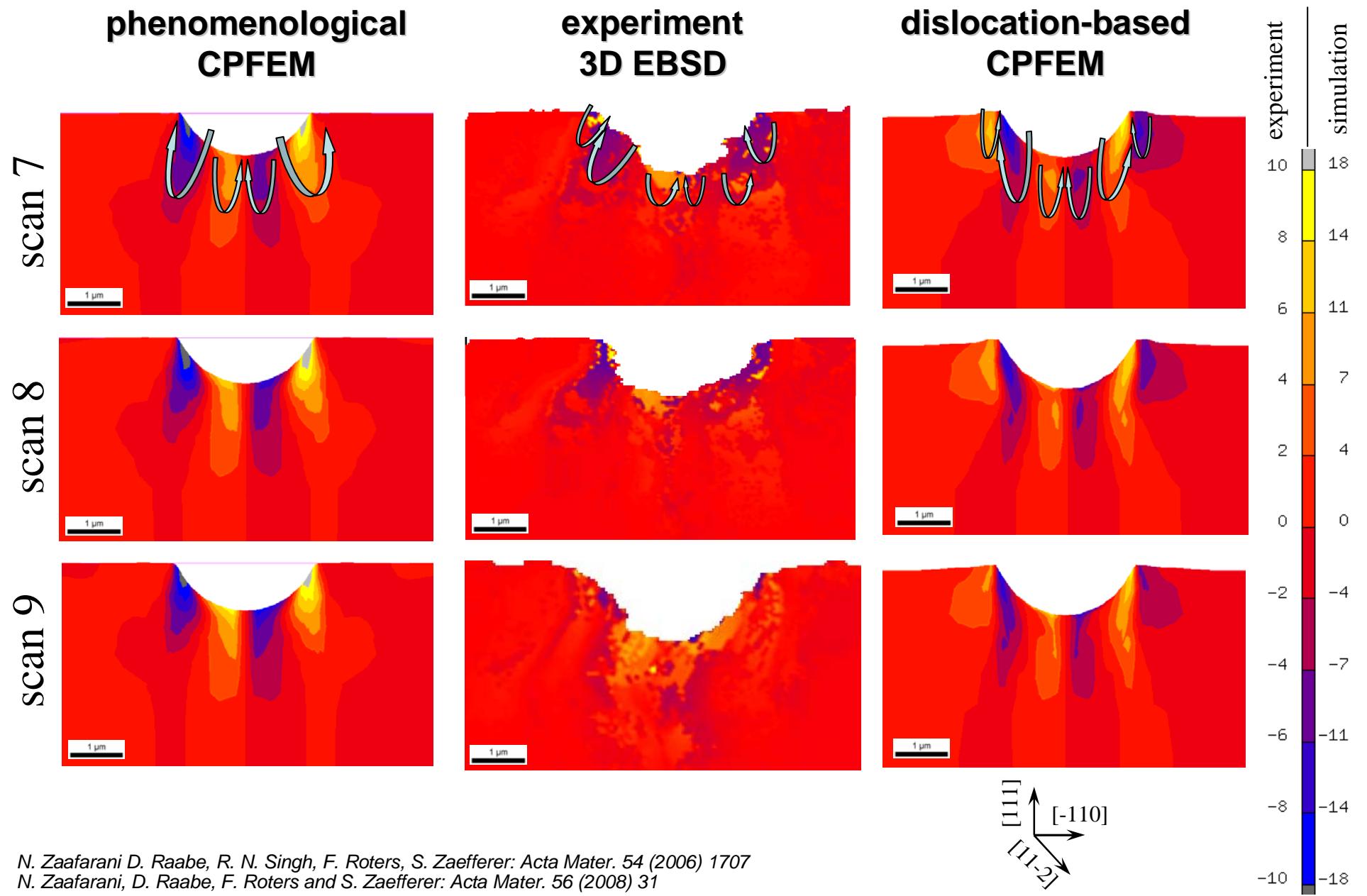
Crystal orientation distribution around nanoindents



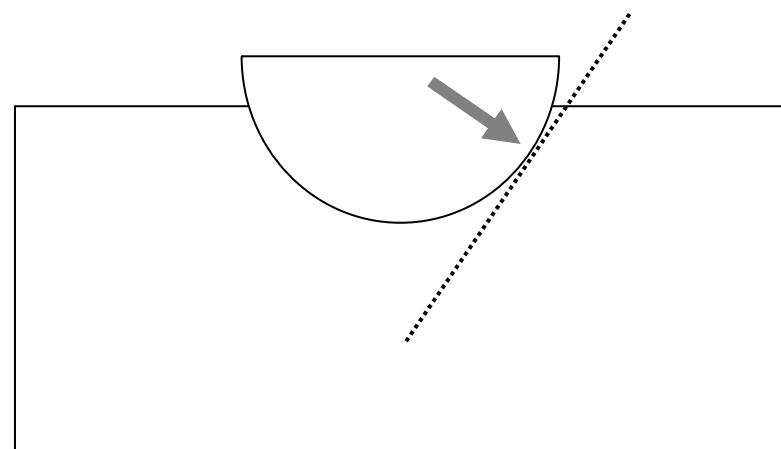
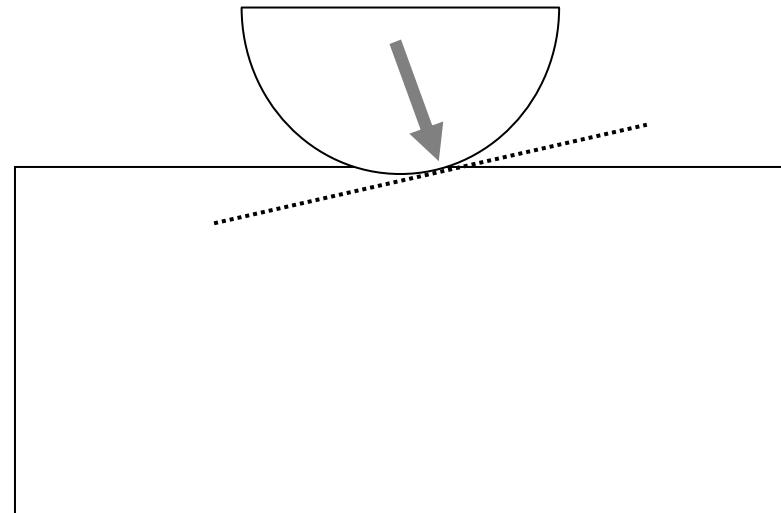
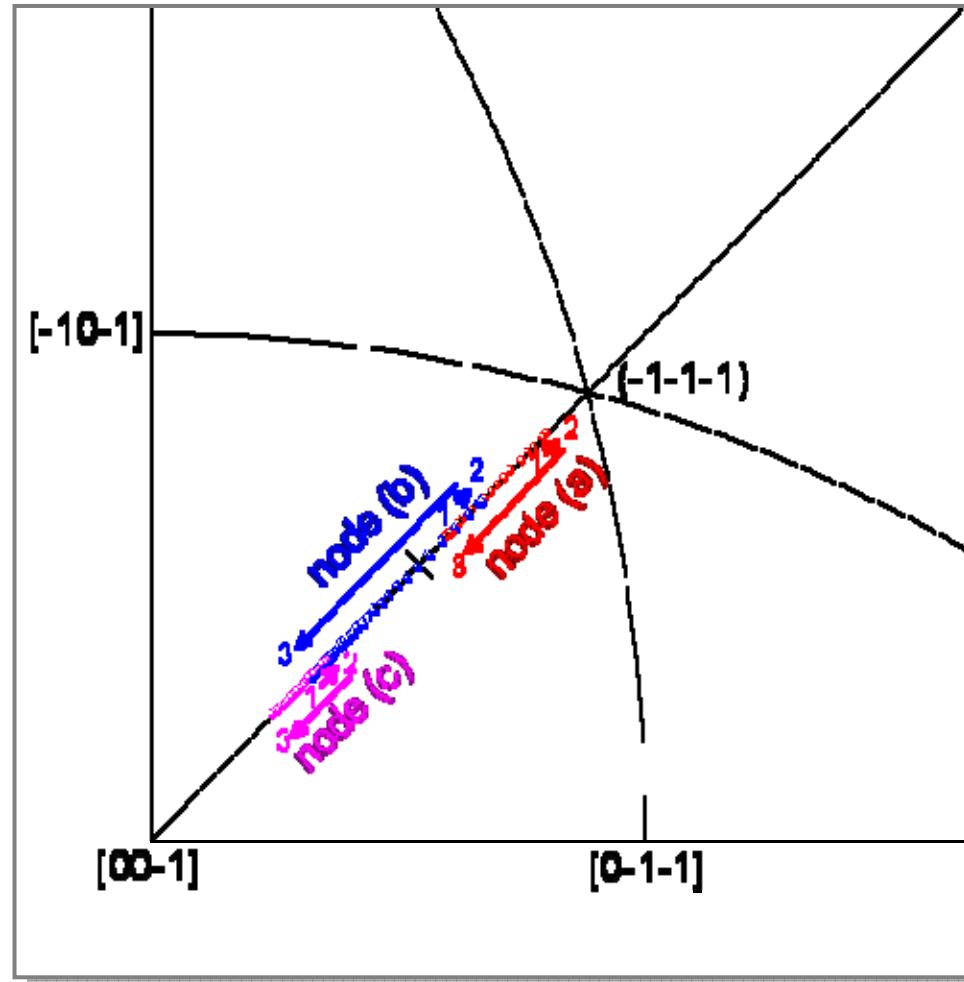
Misorientation angle



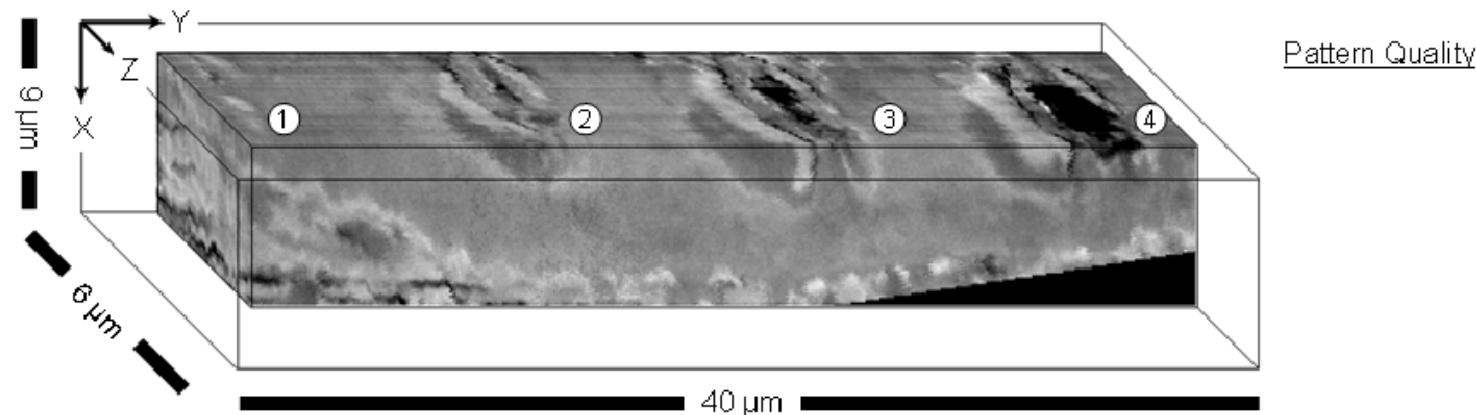
Comparison, crystal rotations about [11-2] axis



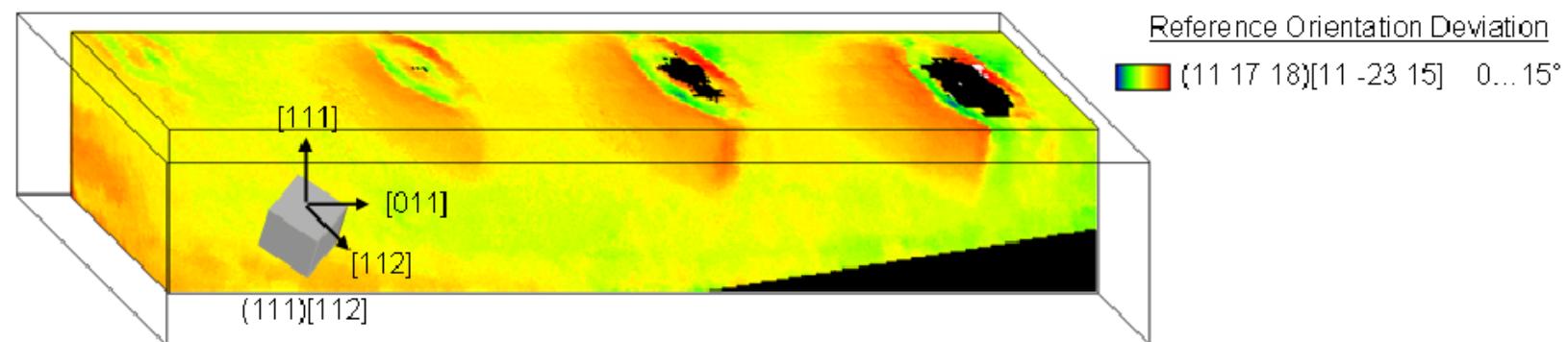
N. Zaafarani, D. Raabe, R. N. Singh, F. Roters, S. Zaefferer: Acta Mater. 54 (2006) 1707
N. Zaafarani, D. Raabe, F. Roters and S. Zaefferer: Acta Mater. 56 (2008) 31



Pattern quality, rotation field, indents of different size



Pattern Quality



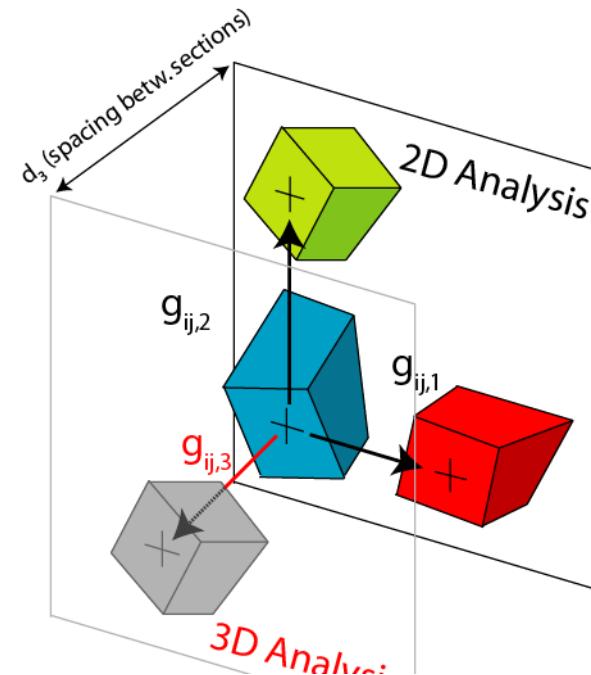
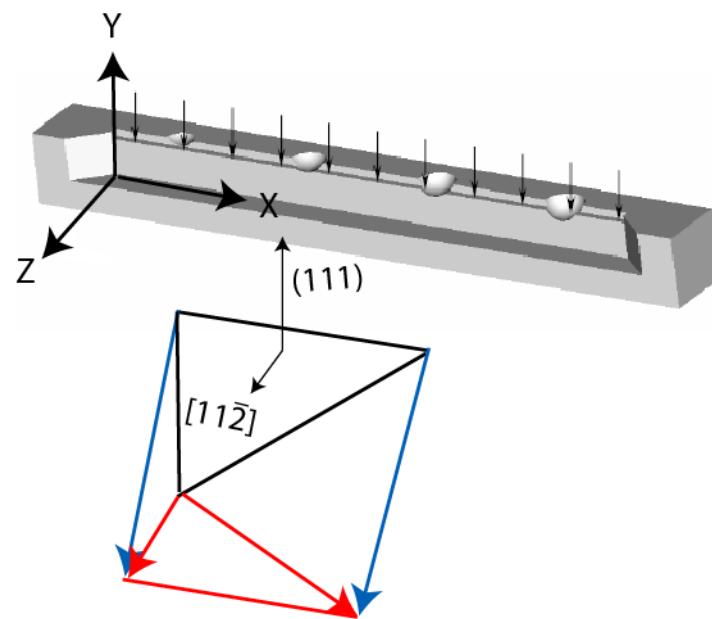
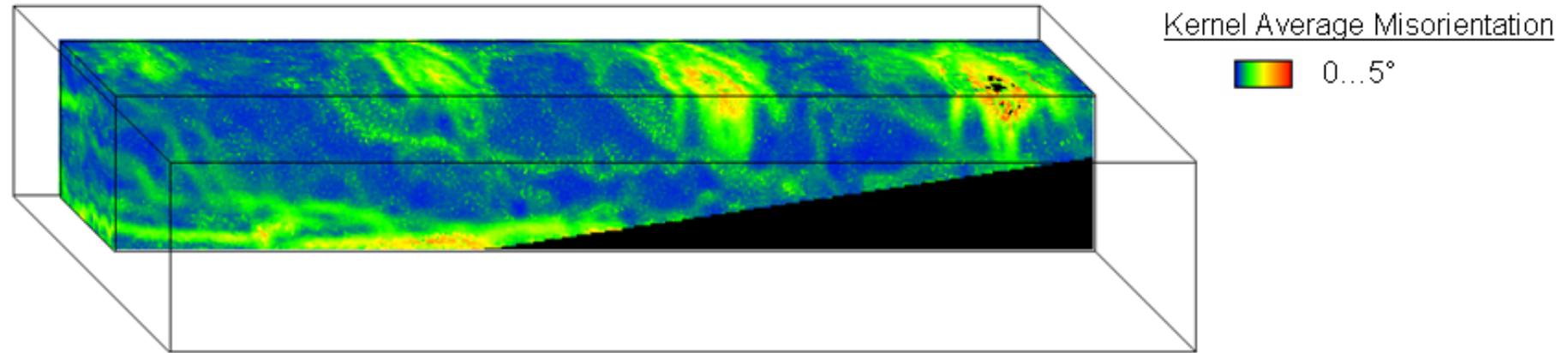
Reference Orientation Deviation

(11 17 18)[11 -23 15] 0...15°



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Local misorientation analysis



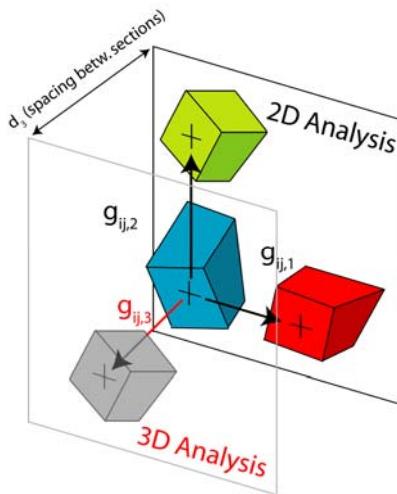
From local misorientations to GNDs



$$\Delta\phi = \phi_{(2)} \phi_{(1)}^{-1} \quad \text{misorientation}$$

$$|\Delta\phi| = \min\{\cos^{-1}\{tr[(O_i^{cry}\phi_{(1)})\phi_{(2)}^T O_j^{cry}]\}\} \quad i = 1 \dots 24, j = 1 \dots 24$$

$$\phi_{(2)} - \phi_{(1)} = (\Delta\phi - I)\phi_{(1)} \quad \text{orientation difference}$$



$$g_{ij,k} = \frac{\phi_{(2)ij} - \phi_{(1)ij}}{d_k} \quad \text{orientation gradient (spacing } d \text{ from EBSD scan)}$$



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

distortion
(sym, a-sym)

$$\alpha = \nabla \times \beta^{el}$$

$$\alpha_{pi} = e_{pkj} (\epsilon_{ij,k}^{el} + g_{ij,k})$$

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

dislocation tensor (GND)

J. F. Nye. Some geometrical relations in dislocated crystals. Acta Metall. 1:153, 1953.

E. Demir, D. Raabe, N. Zaafarani, S. Zaefferer: Acta Mater. 57 (2009) 559–569

E. Kröner. Kontinuumstheorie der Versetzungen und Eigenspannungen (in German). Springer, Berlin, 1958.

E. Kröner. Physics of defects, chapter Continuum theory of defects, p.217. North-Holland Publishing, Amsterdam, Netherlands, 1981.



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

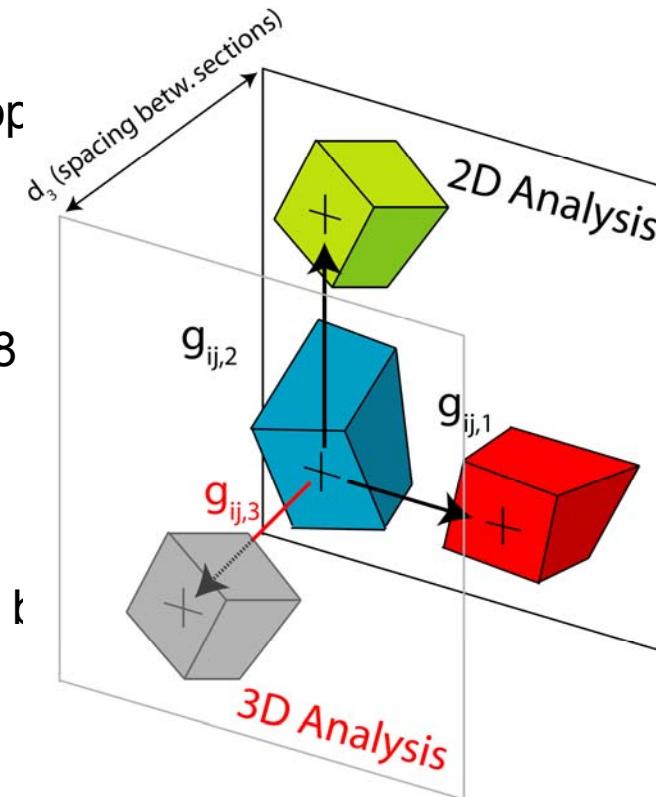
$\sqrt{2} \hat{\mathbf{b}}$: $\bar{1}10 \ 10\bar{1} \ 0\bar{1}1 \ \bar{1}\bar{1}0 \ 101 \ 01\bar{1} \ 110 \ \bar{1}01 \ 0\bar{1}\bar{1} \ 1\bar{1}0 \ \bar{1}0\bar{1} \ 011 \ 110 \ 101 \ 011 \ \bar{1}10 \ 10\bar{1} \ 0\bar{1}1$

$\sqrt{6} \hat{\mathbf{t}}$: $\bar{1}\bar{1}2 \ \bar{1}2\bar{1} \ 2\bar{1}\bar{1} \ \bar{1}1\bar{2} \ \bar{1}\bar{2}1 \ 211 \ 1\bar{1}\bar{2} \ 121 \ \bar{2}\bar{1}1 \ 112 \ 1\bar{2}\bar{1} \ \bar{2}1\bar{1} \ 110 \ 101 \ 011 \ \bar{1}10 \ 10\bar{1} \ 0\bar{1}1$

$$\mathbf{B} = \mathbf{b}(\hat{\mathbf{t}} \cdot \mathbf{r}) = (\mathbf{b} \otimes \hat{\mathbf{t}})\mathbf{r} \quad \text{Frank loop}$$

$$\alpha_{ij} = \sum_{a=1}^{18} \rho_{gnd}^a b_i^a t_j^a \quad \text{DDT in terms of 18}$$

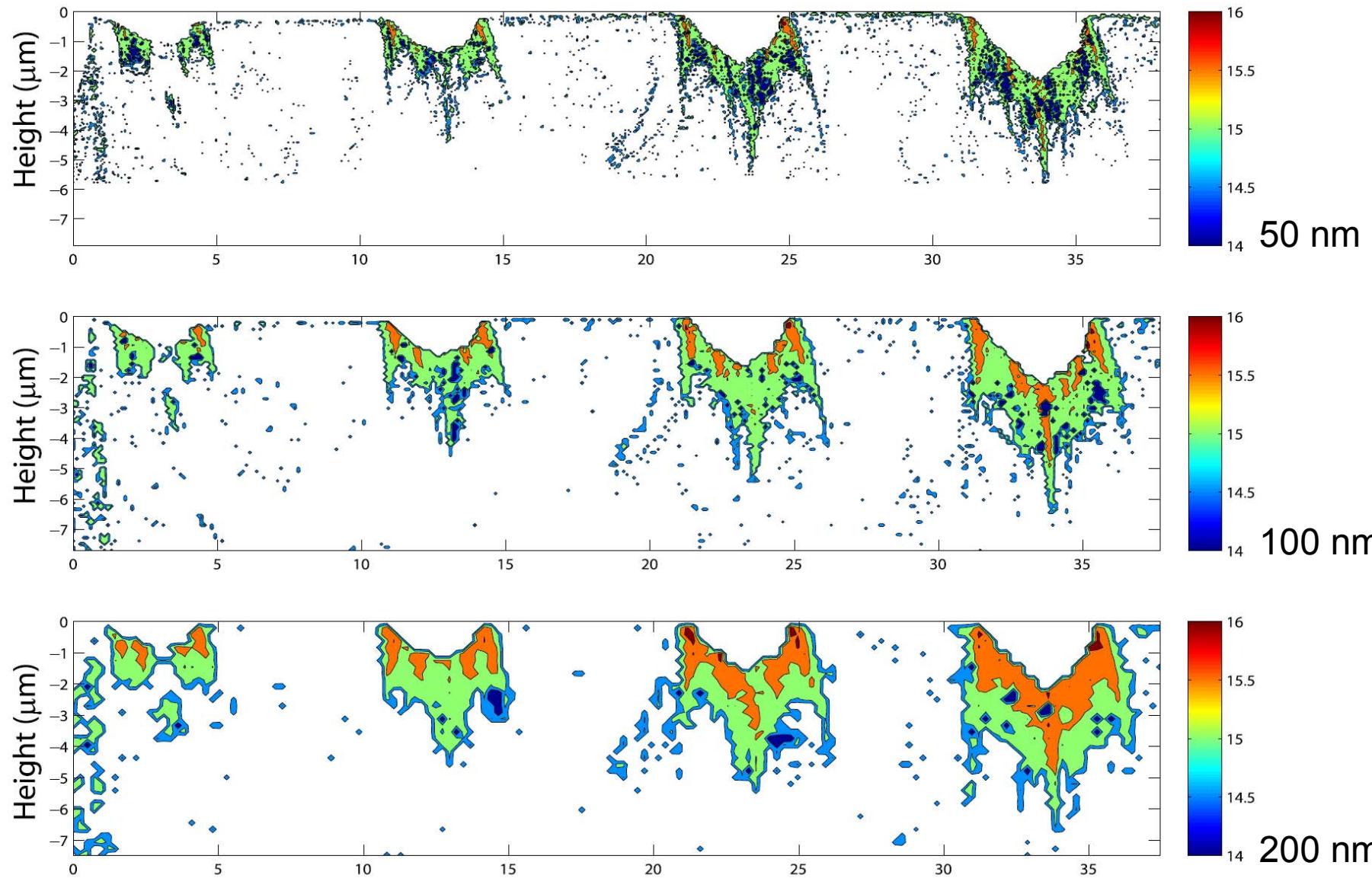
$$\alpha_{ij} = \sum_{a=1}^9 \rho_{gnd}^a b_i^a t_j^a \quad \text{DDT in terms of 9 } \mathbf{t}$$





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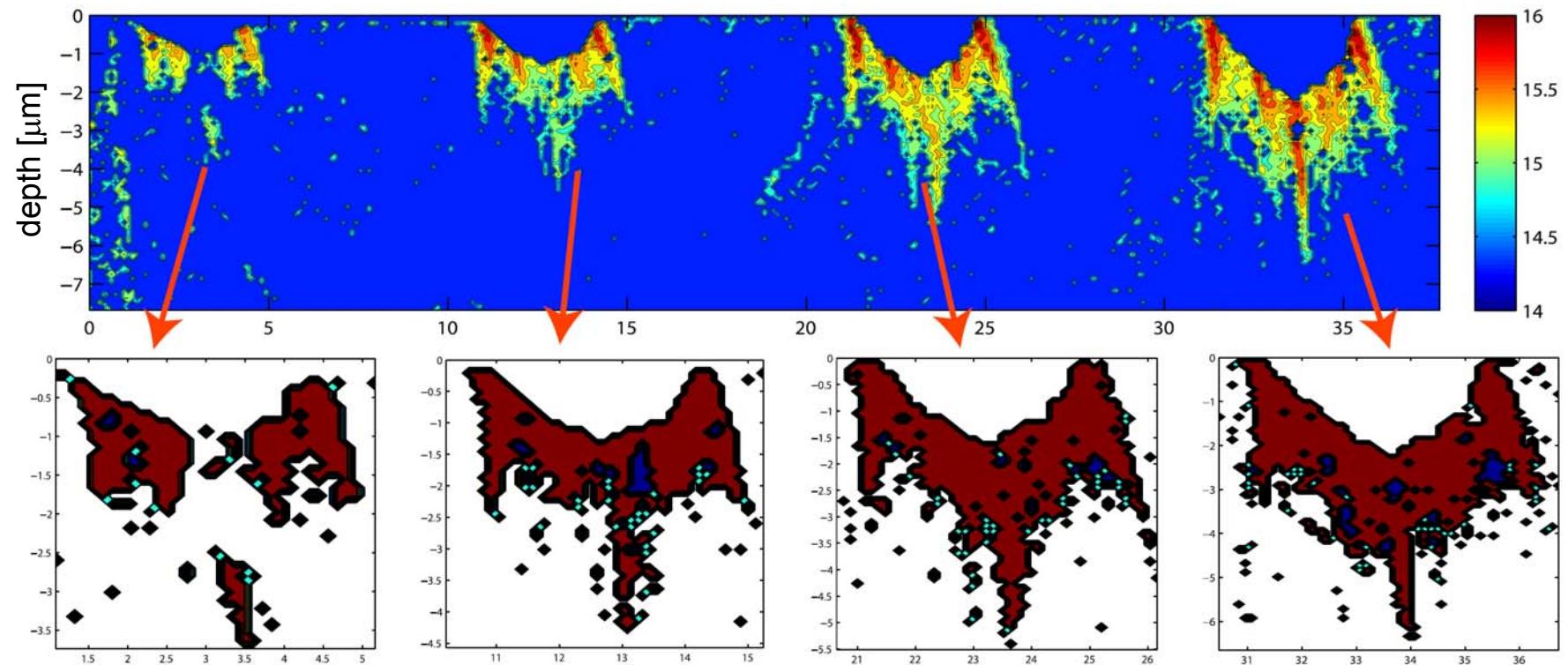
Distribution of GNDs for different gradient resolutions



center section, 2D analysis, color code: GND density (decadic log. ($1/m^2$))

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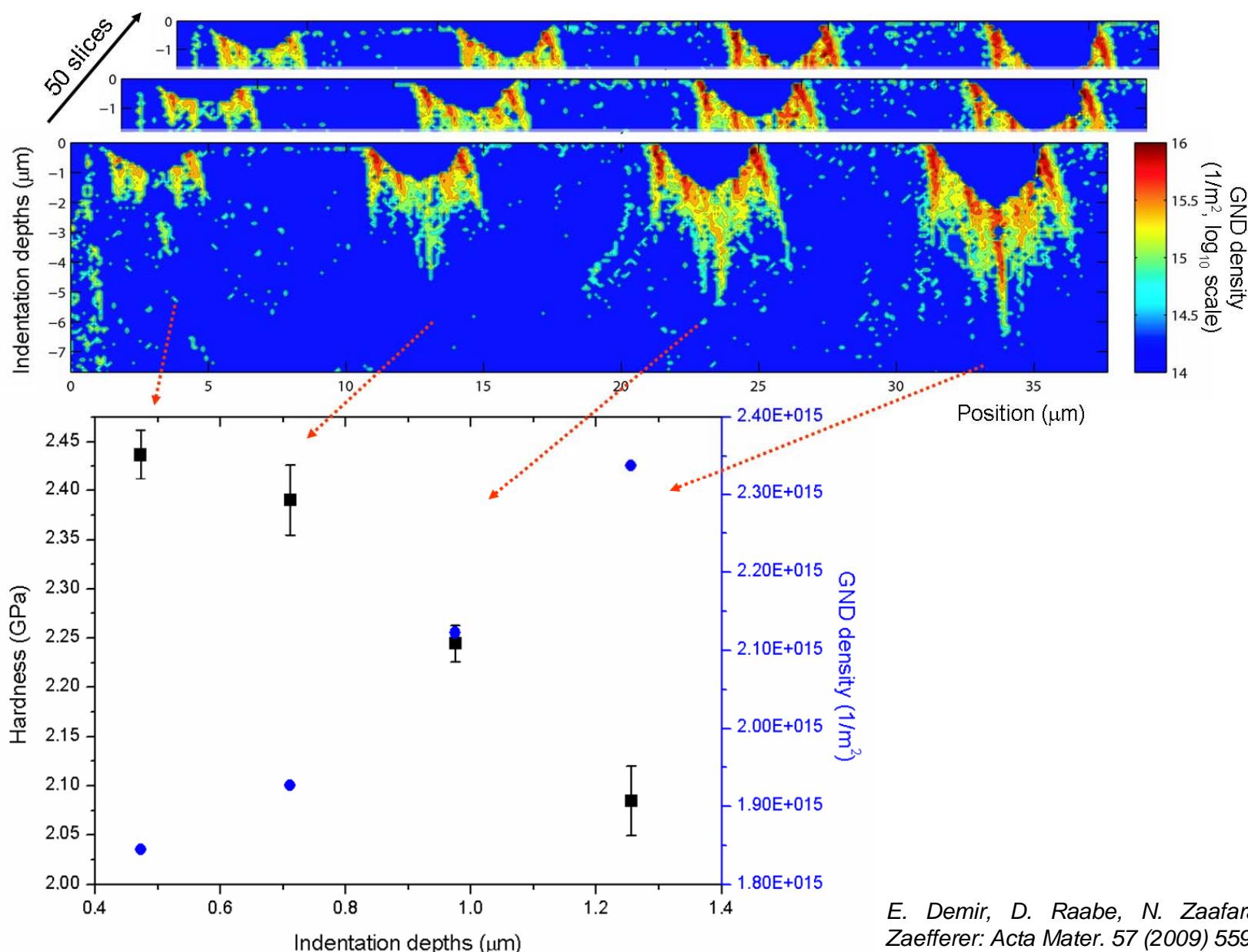
Identification of the plastic volume



Reference volumes (red color) indicates the plastic volume using lower threshold of GNDs of $10^{14}/\text{m}^2$

center section, 2D analysis, color code: GND density (decadic log. ($1/\text{m}^2$))

Extract geometrically necessary dislocations

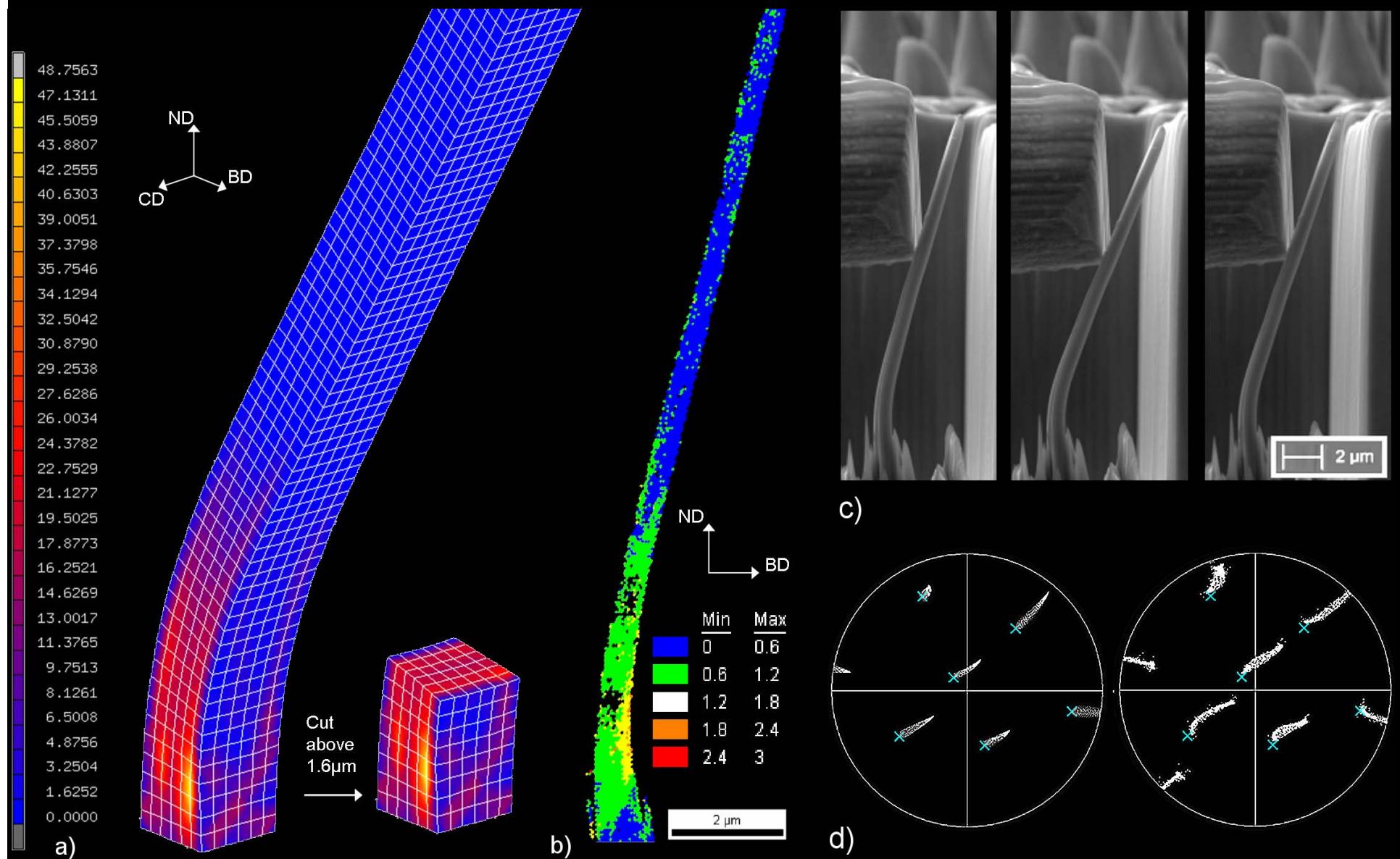


E. Demir, D. Raabe, N. Zaafarani, S. Zaefferer: Acta Mater. 57 (2009) 559–569



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Similar experiment for bending

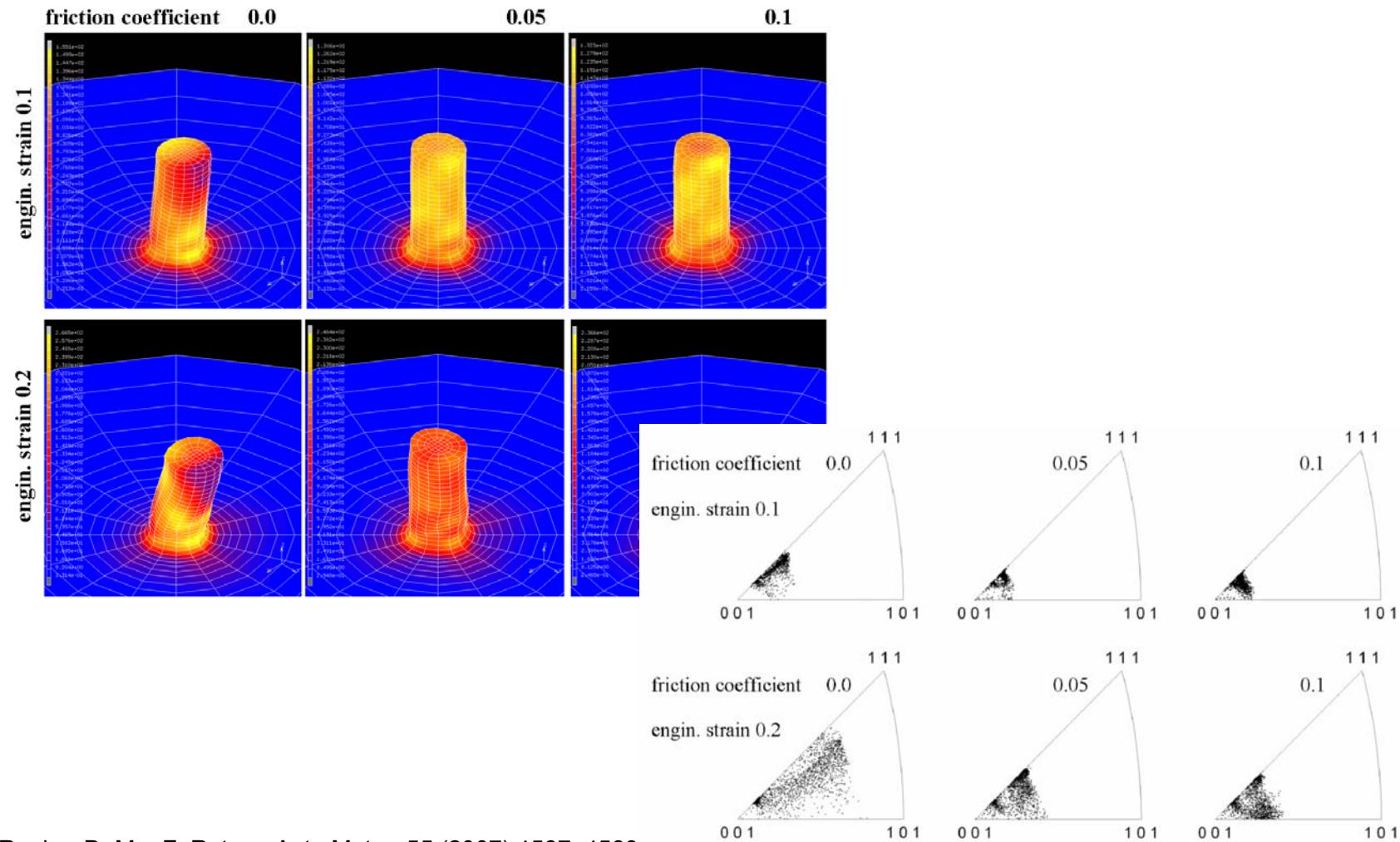


F. Weber, I. Schestakow, D. Raabe, F. Roters: Adv. Engin. Mater. 10 (2008) 737-741

Metallic micropillar deformation



Parameter study: friction



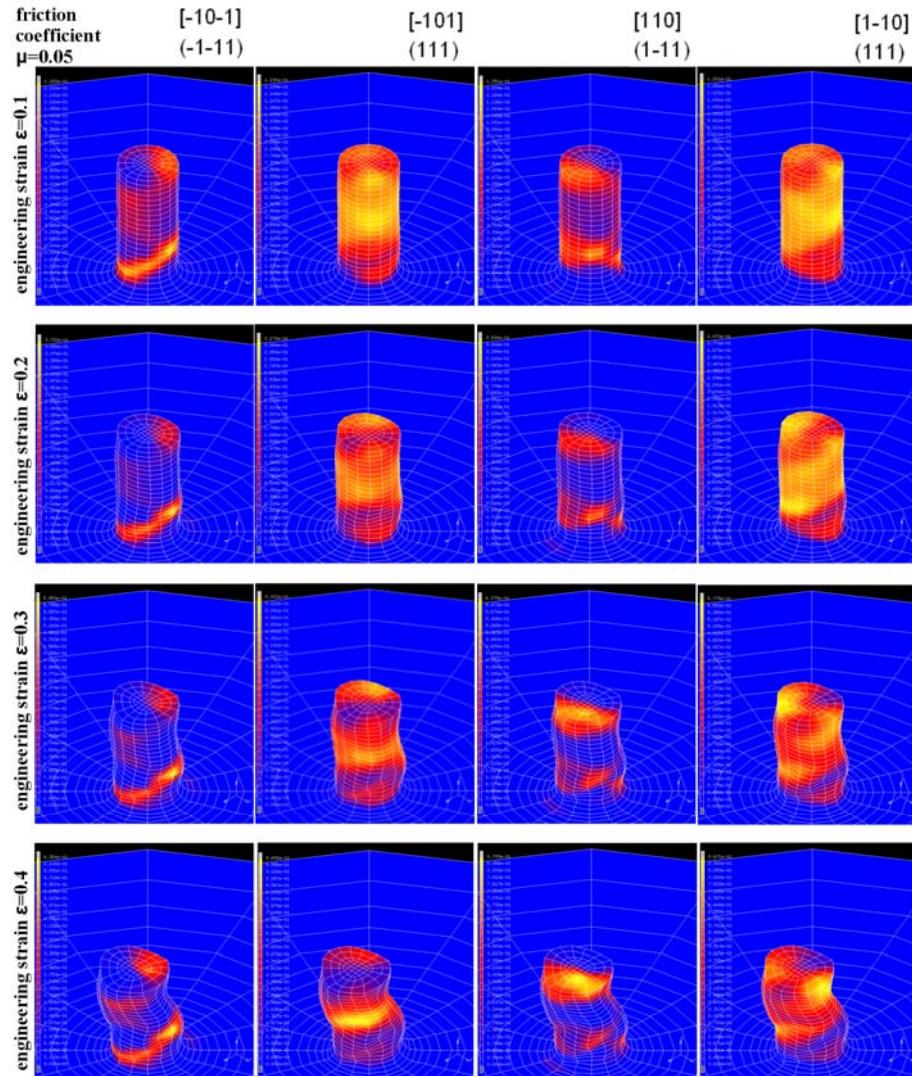
D. Raabe, D. Ma, F. Roters: Acta Mater. 55 (2007) 4567–4583

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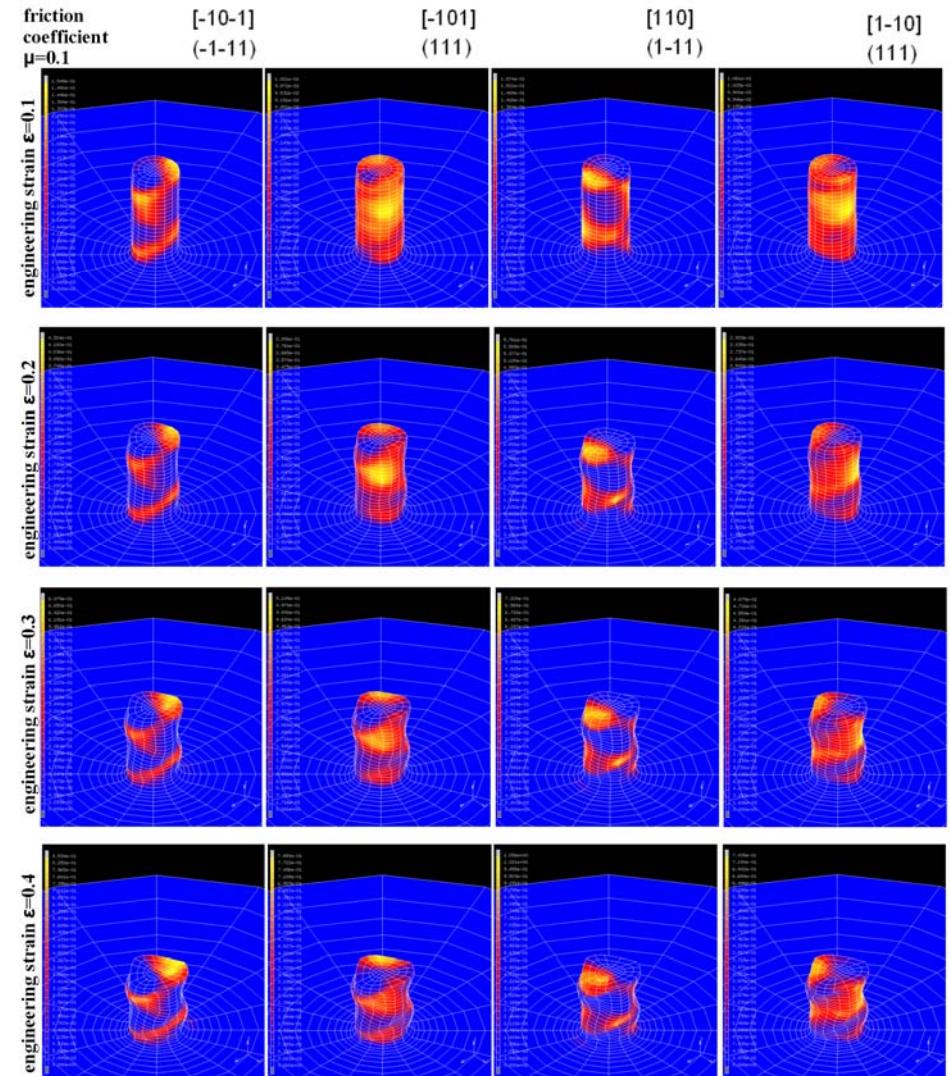
Metallic micropillar deformation



Parameter study: individual slip activity and heterogeneity

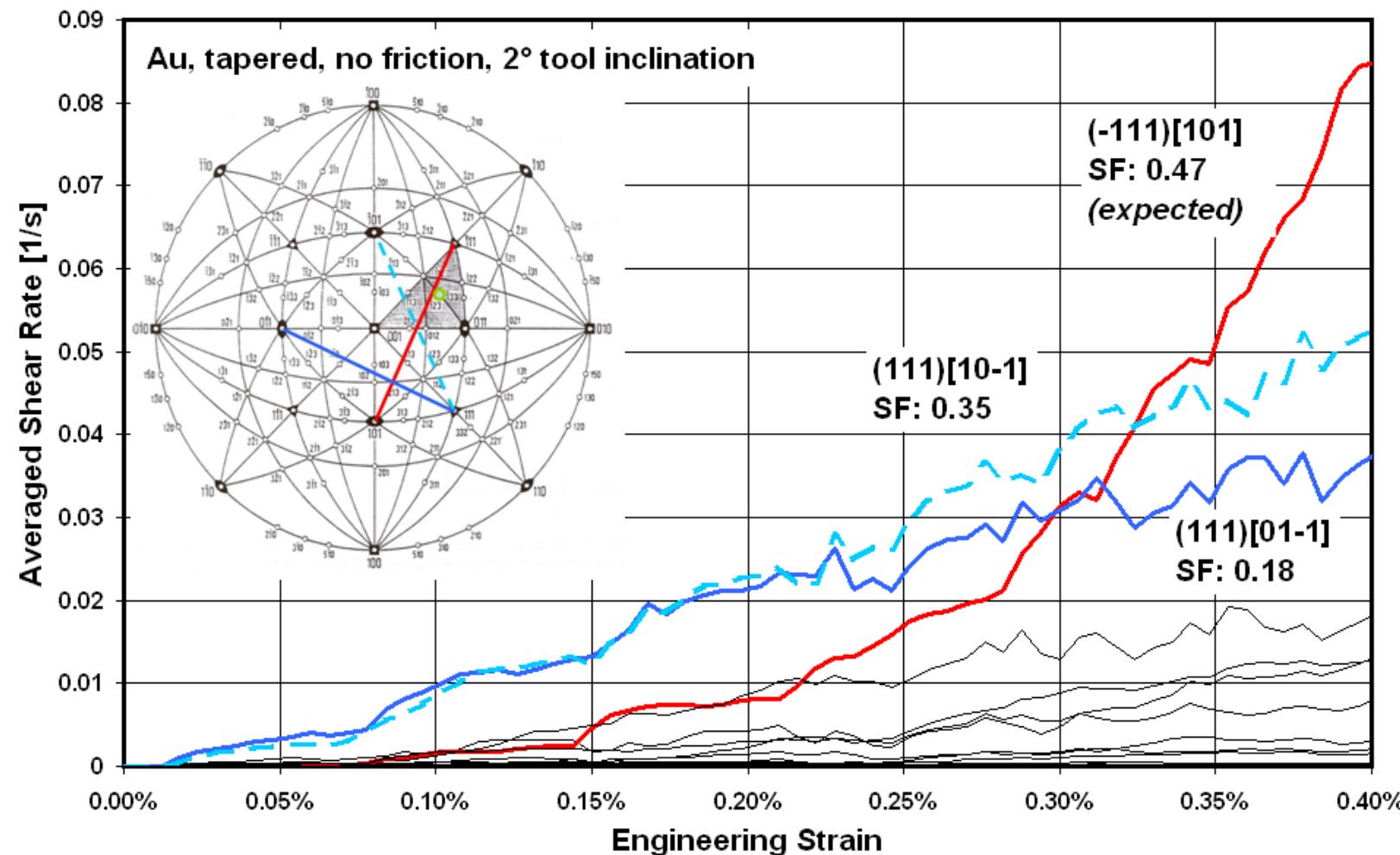


D. Raabe, D. Ma, F. Roters: Acta Mater. 55 (2007) 4567–4583



corresponding experiments by : Robert Maaß, Steven Van Petegem, Julien Zimmermann, Daniel Grolimund, Helena Van Swygenhoven

Simulation results





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- The GNDs have an inhomogeneous distribution underneath the indents with very high local density values. No homogeneous distribution.
- The total GND density below the indents decreases with decreasing indentation depths. This observation does not conform to strain gradient theories attributing size-dependent material properties to GNDs.
- The amount of deformation imposed reduces with decreasing indentation depths. Therefore, SSDs that evolve through strain do not account for the increasing hardness values with decreasing indentation depth.
- Inhomogeneous distribution of GNDs plays a main role.
- Explaining size dependent material strengthening effects by using average density measures for both GNDs and SSDs is not sufficient to understand the indentation size effect.
- More critical experiments on physics of size effects
- Control and numerical analysis of boundary conditions