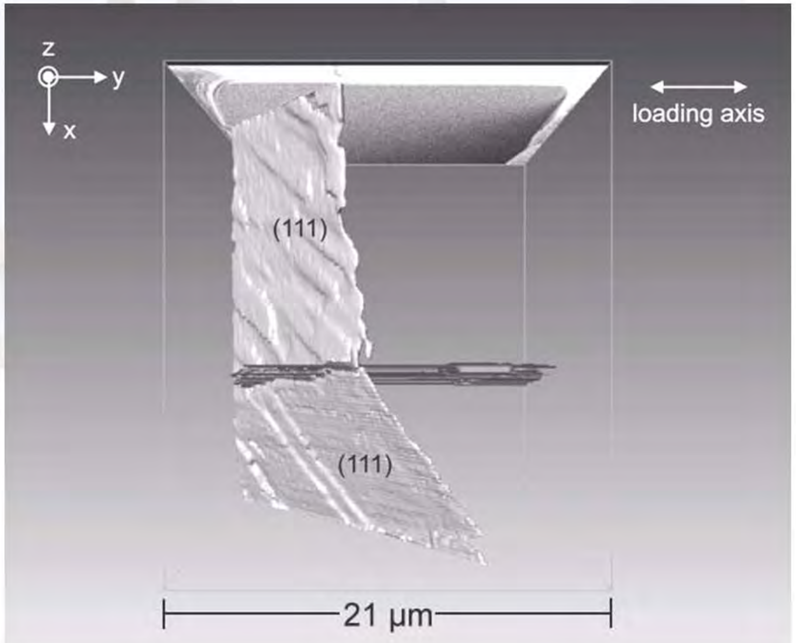
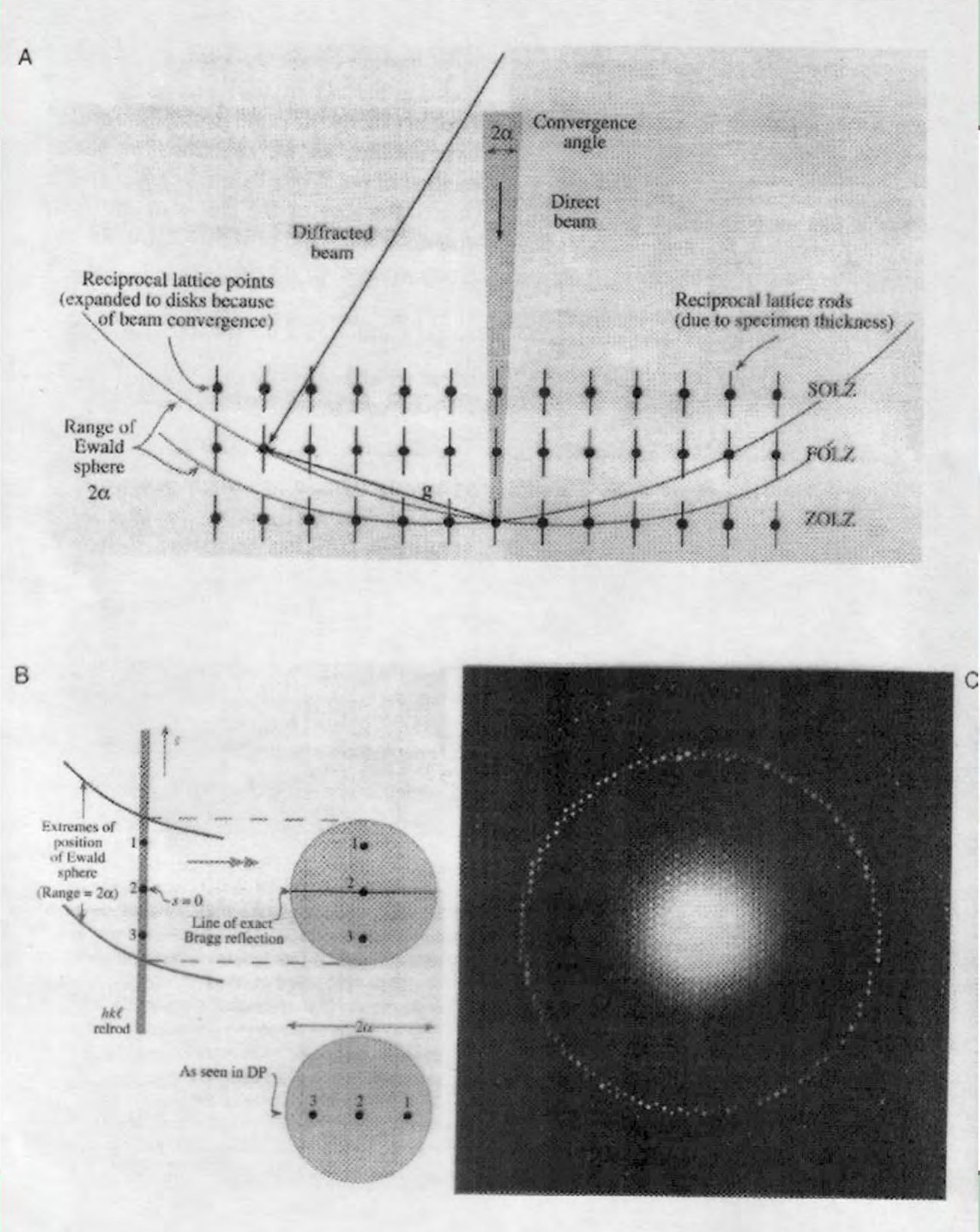
From 3D-Tomography to lattice rotation in nano sized bicrystals

What can be learned from local characterization methods

Horst Vehoff, Michael Marx, Afrooz Barnoush

- EBSD and Orientation gradient mapping
- FIB Tomography
- Nano indentation
- Interaction crack - grain boundary
- Nanopillar bicrystals
- Strength of interfaces
- Ultra fine grained materials

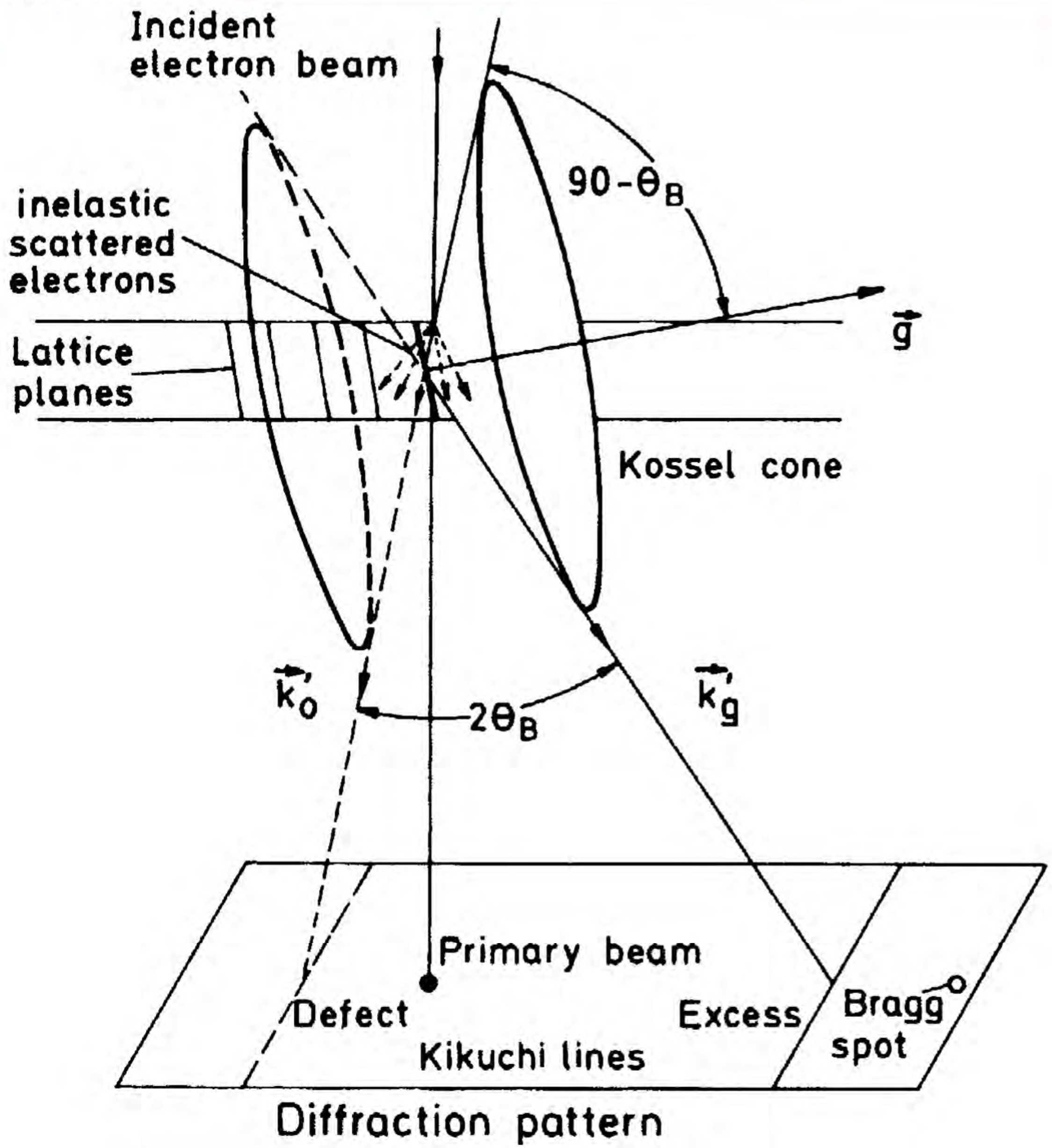




Beugungsbild im TEM

Mit Hilfe eines divergenten Strahls werden mehrere Lauezonen erfasst

EBSD



Streuung der Elektronen an den Netzebenen in alle Richtungen auf zwei Kegelmäntel (Kosselkegel) gestreut.

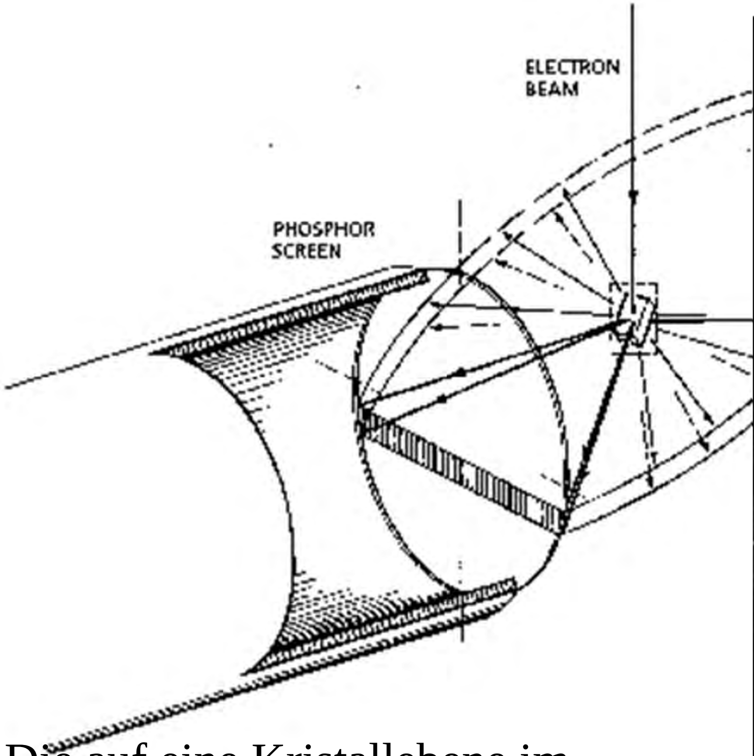
geringen Öffnungswinkels des Kegels (um 1°)
→ Schnitt der Kegelmäntel mit dem Schirm Geraden.

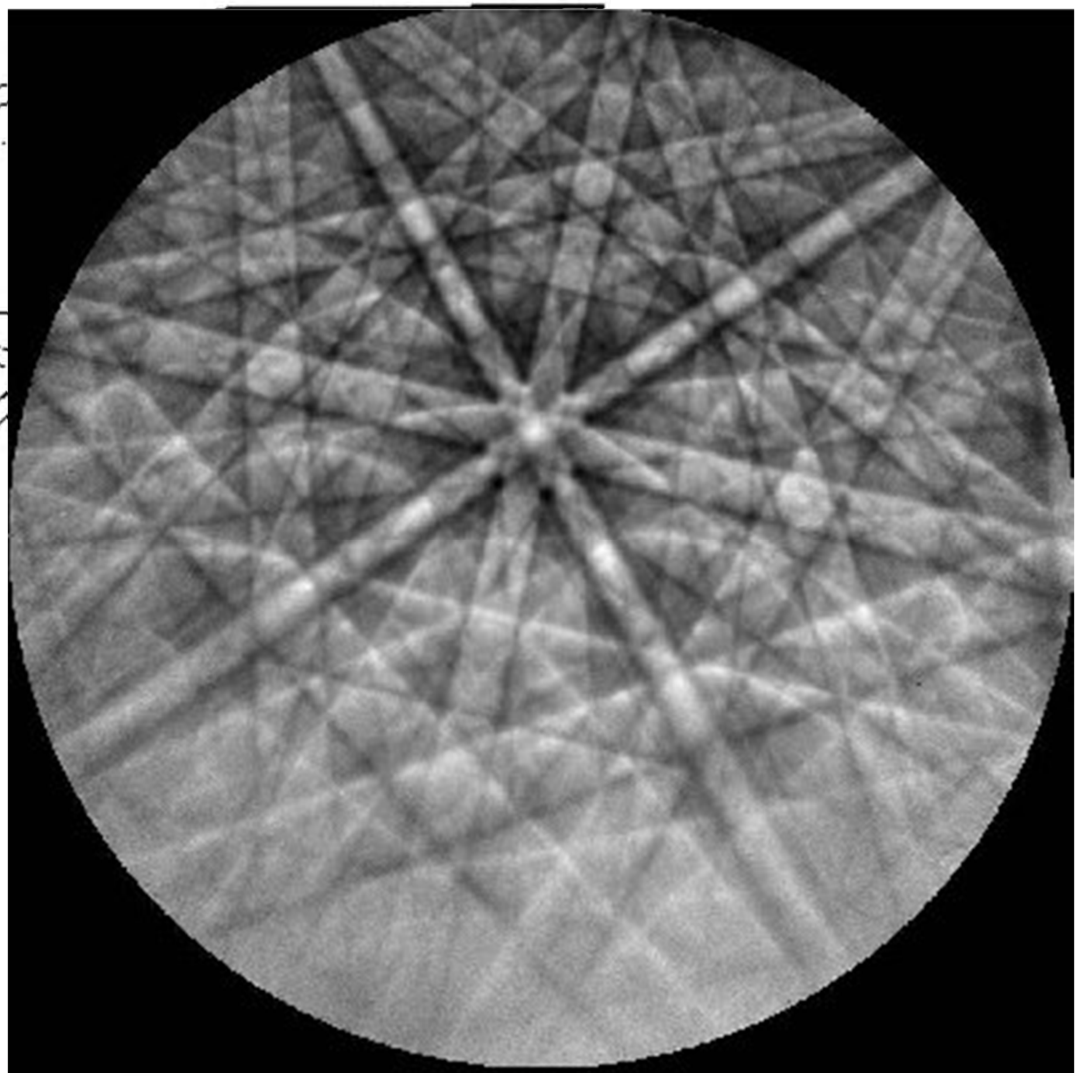
Zu jeder Netzebenenschar gehören eine helle und eine dunkle Kikuchilinie

Schema der Bildung der Kikuchilinien

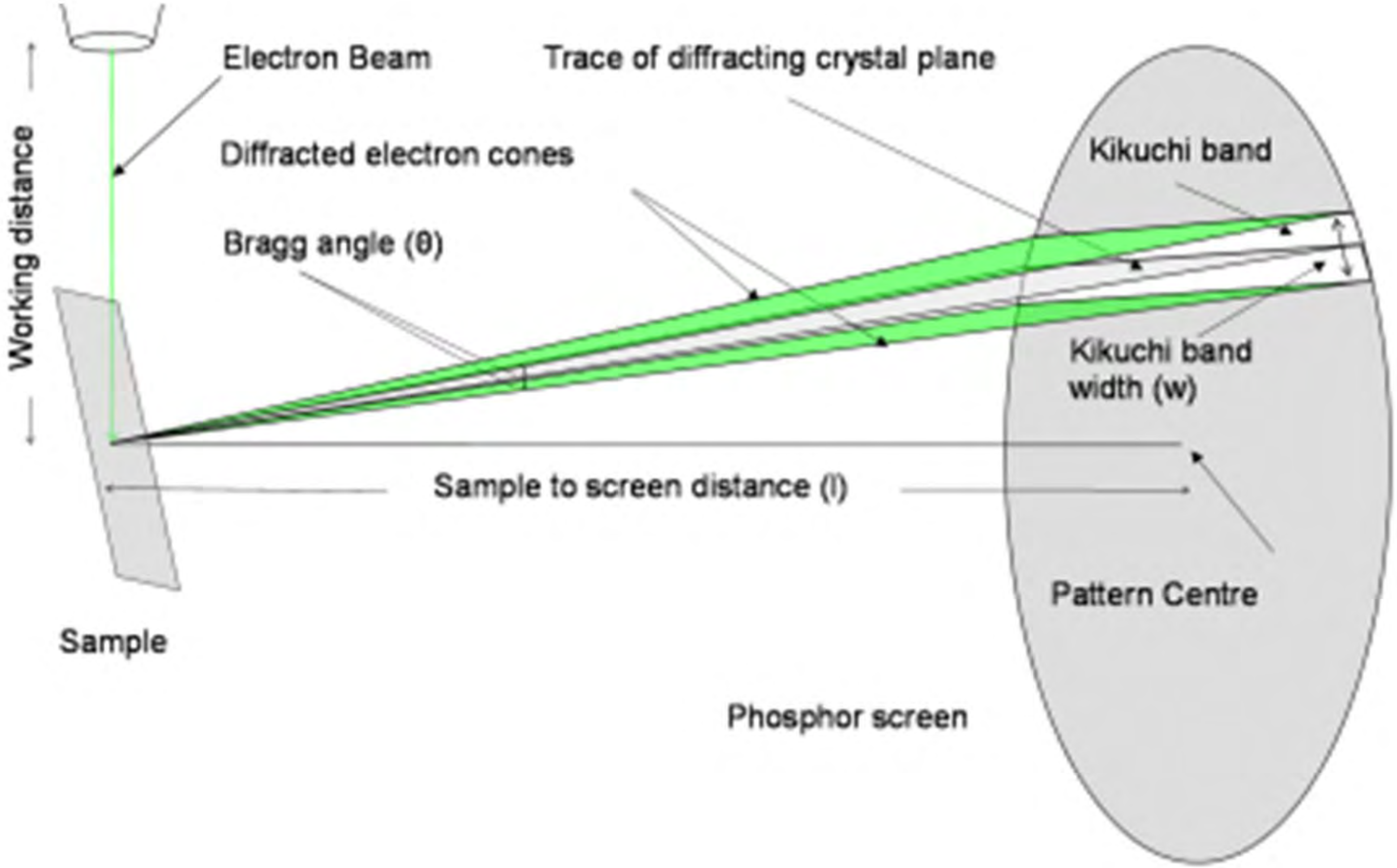
EBSD

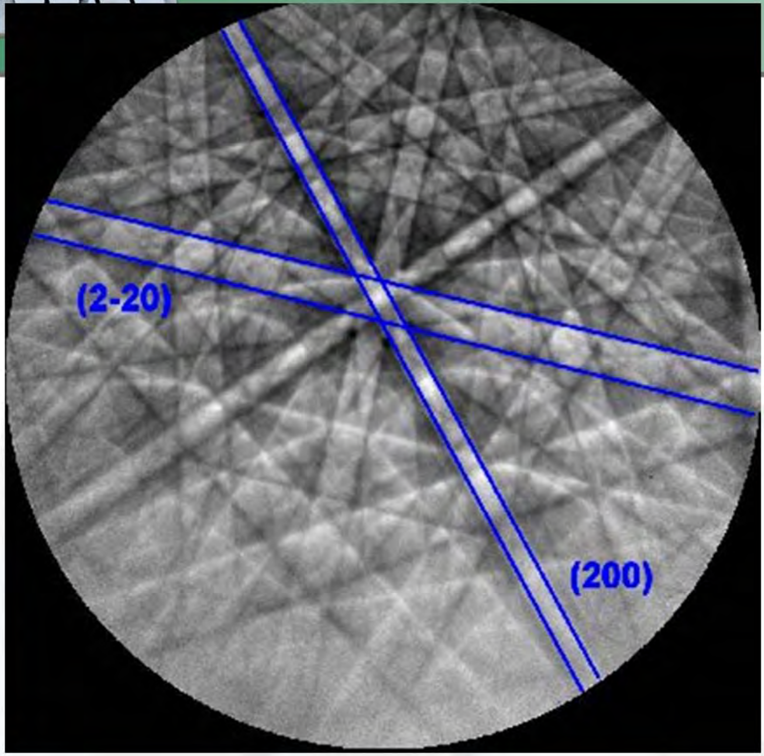
Bildung der ‚Electron Backscatter Diffraction Pattern‘ (EBSP)





Die auf eine Kristallebene im Braggwinkel einfallenden Elektronen werden in zwei Kegel gebeugt und Kikuchi Bänder im Beugungsmuster bilden sich


Electron Beam
Trace of diffracting crystal plane
Working distance
Diffracted electron cones
Kikuchi band
Bragg angle (θ)
Kikuchi band
width (w)
Sample to screen distance (l)
Pattern Centre
Sample
Phosphor screen



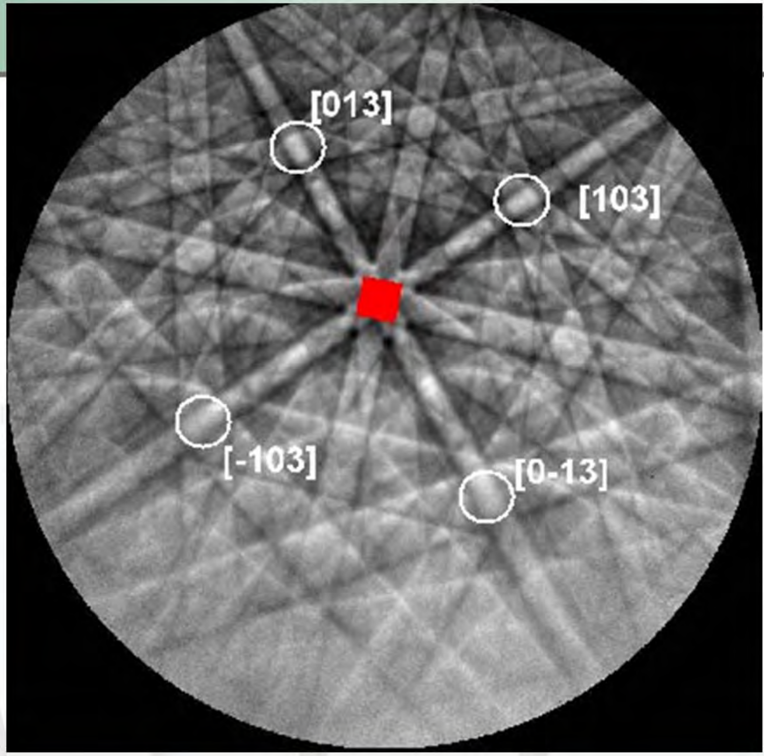
Kikuchi-Linienbreite

Ebenenabstand d:

d(200) > d(2-20)

→ Kikuchi-Bänder-Breite b

b(200) < b(2-20)

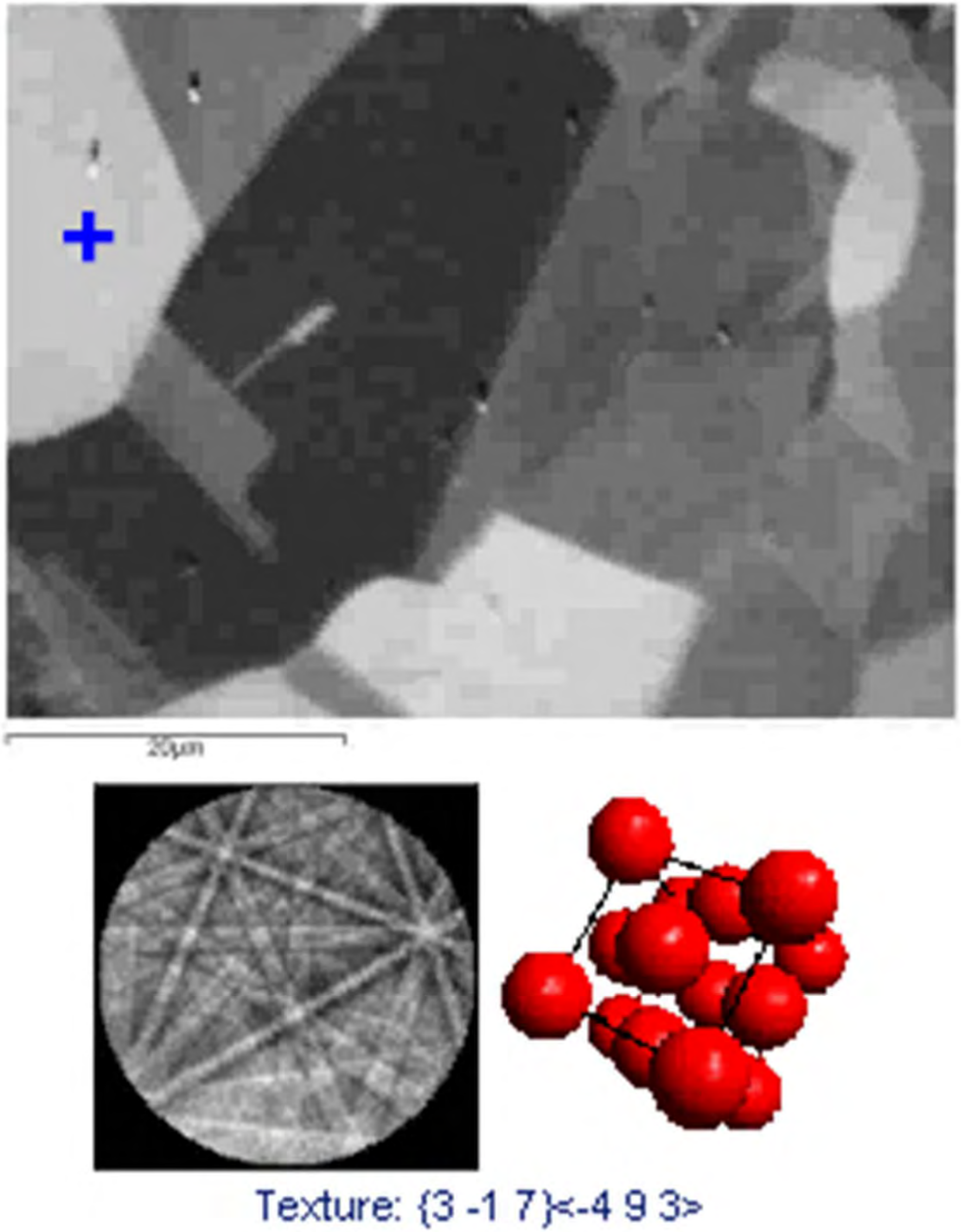


Kristallsymmetrie

z. B. vierfältige Symmetrie um die [001]-Richtung durch symmetrisch äquivalente <013> Zonenachsen

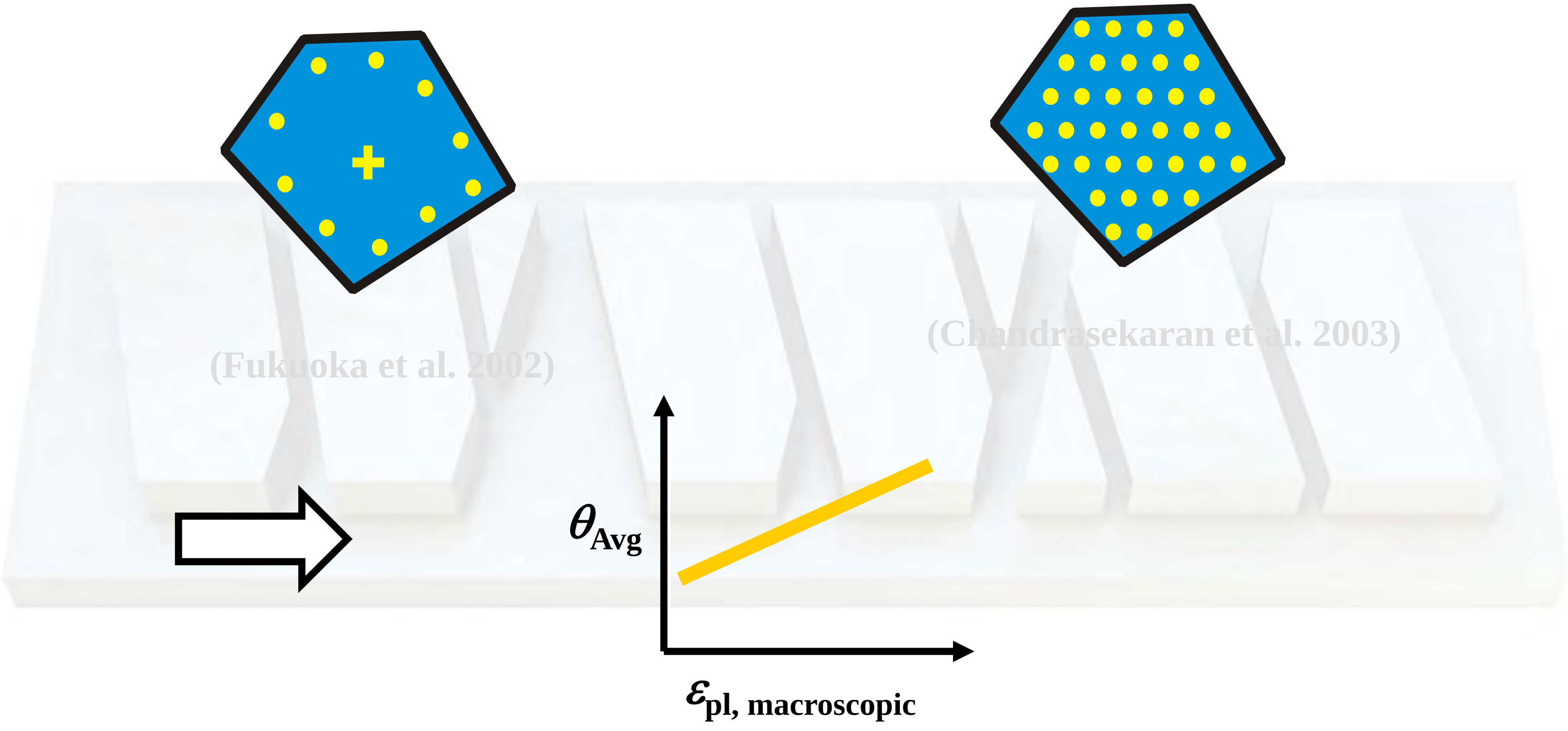
$$b \approx 2l\theta \approx \frac{nl\lambda}{d}$$

Automatischer Scan



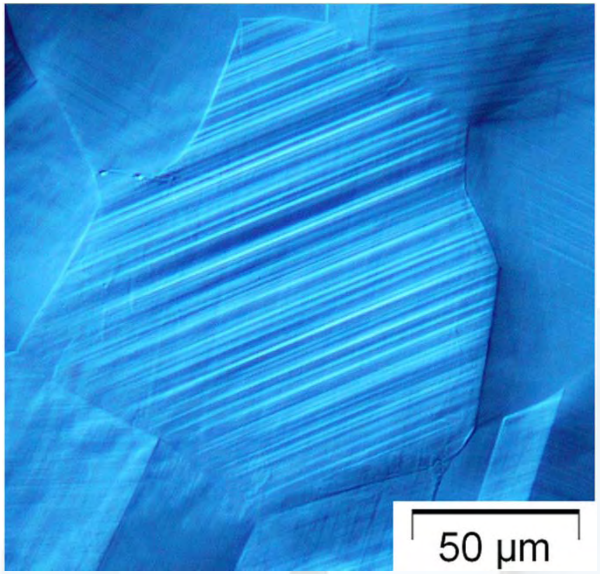
Methods – Orientation Gradient Mapping (OGM)

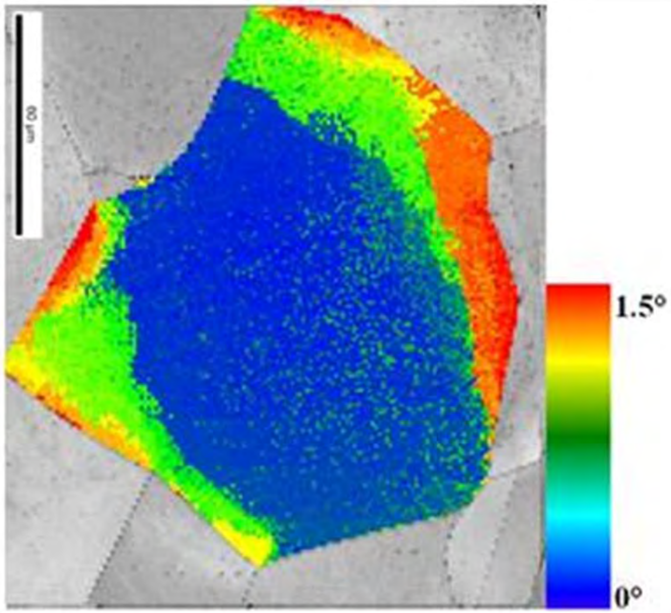
Why: lattice rotations can be easier compared with simulations than dislocation densities



Results for uniform and cellular dislocation structure are consistent

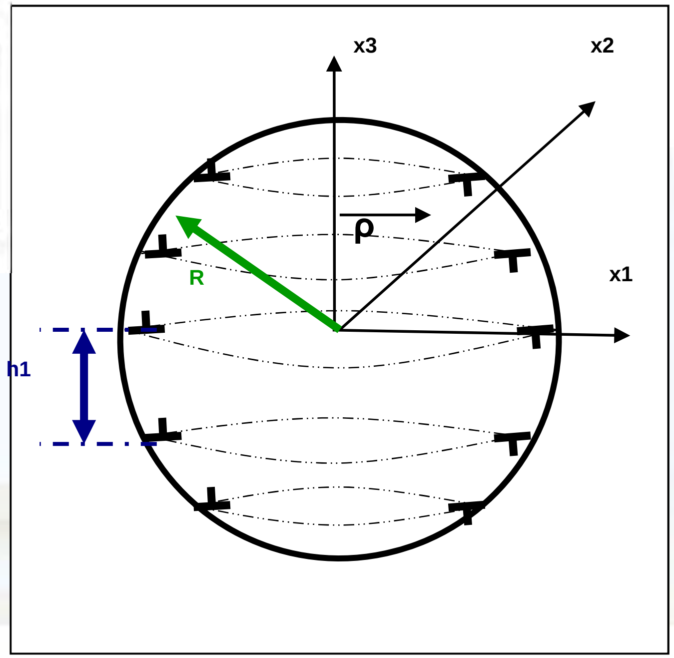
Dislocations and boundaries





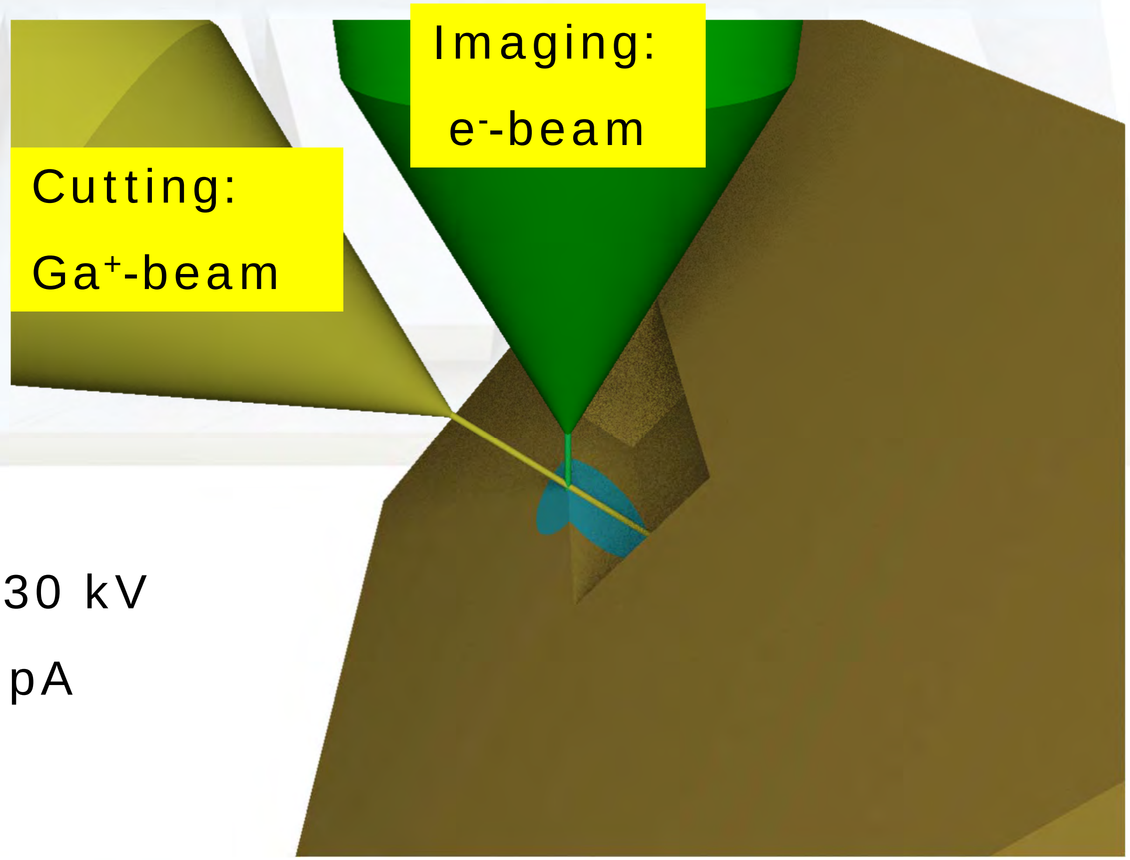
Analytical model of the problem

$$\mathbf{b} = \begin{pmatrix} b \\ 0 \\ 0 \end{pmatrix}$$



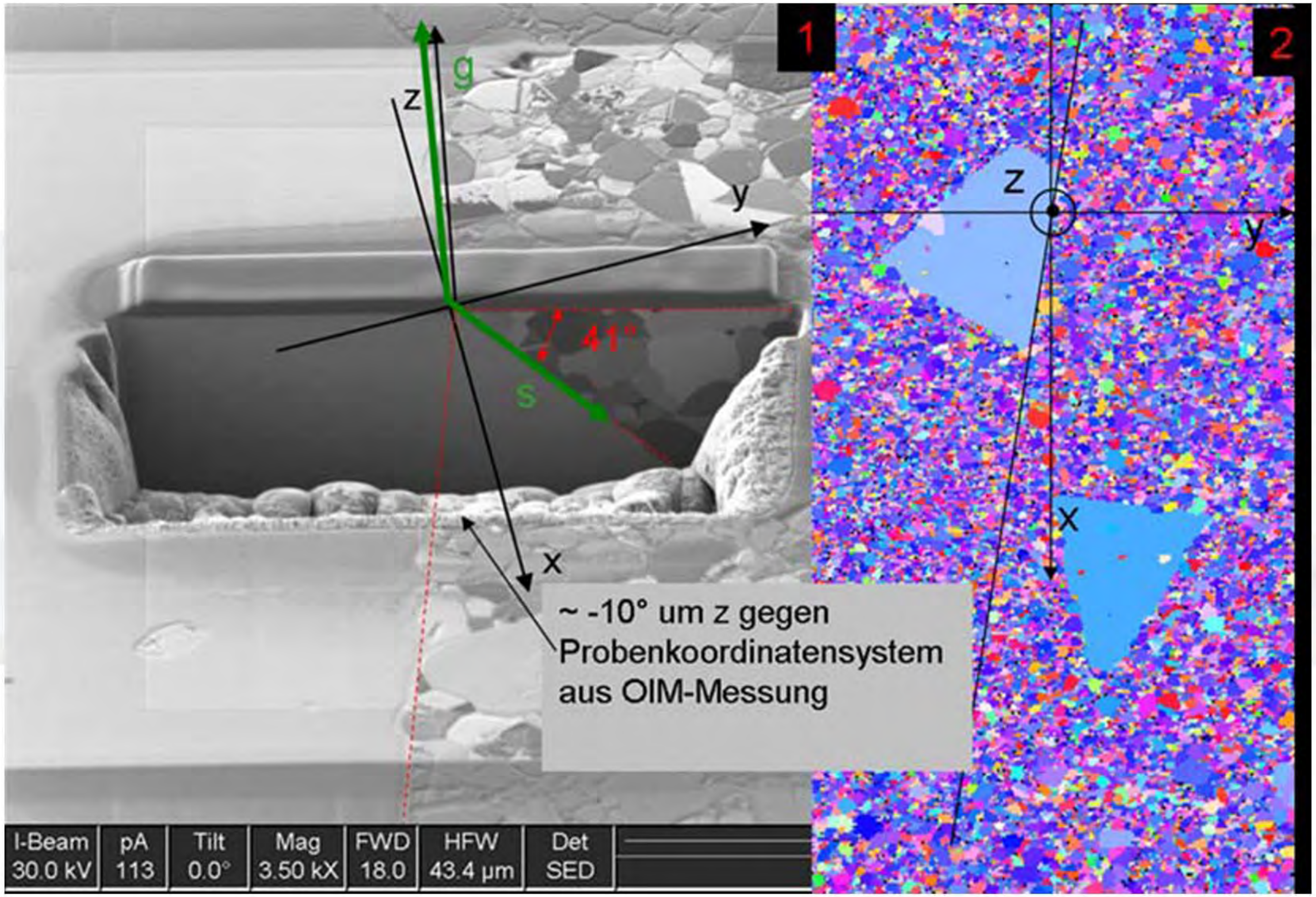
Orientation gradient mapping
Lattice rotations due to GND's

Cutting micro cracks near grain boundaries with the correct geometry



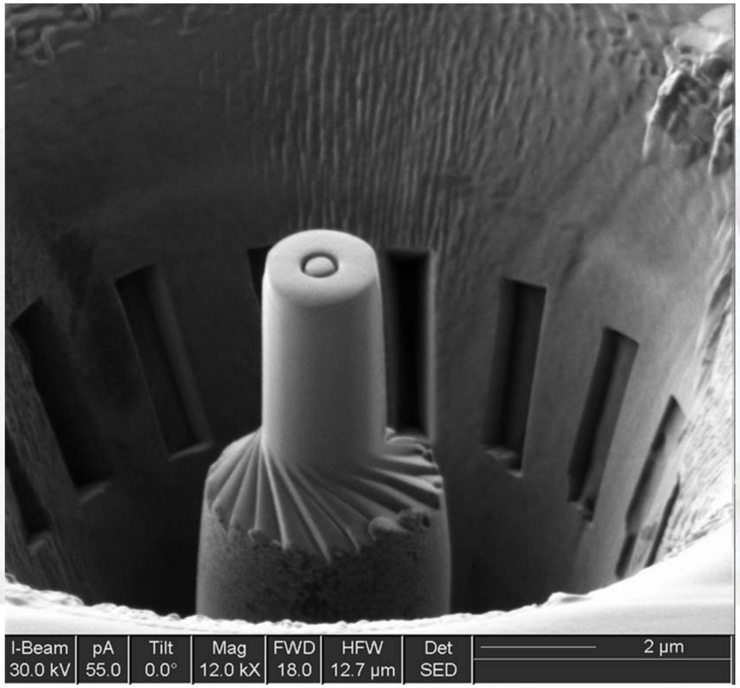
Acceleration voltage: 30 kV

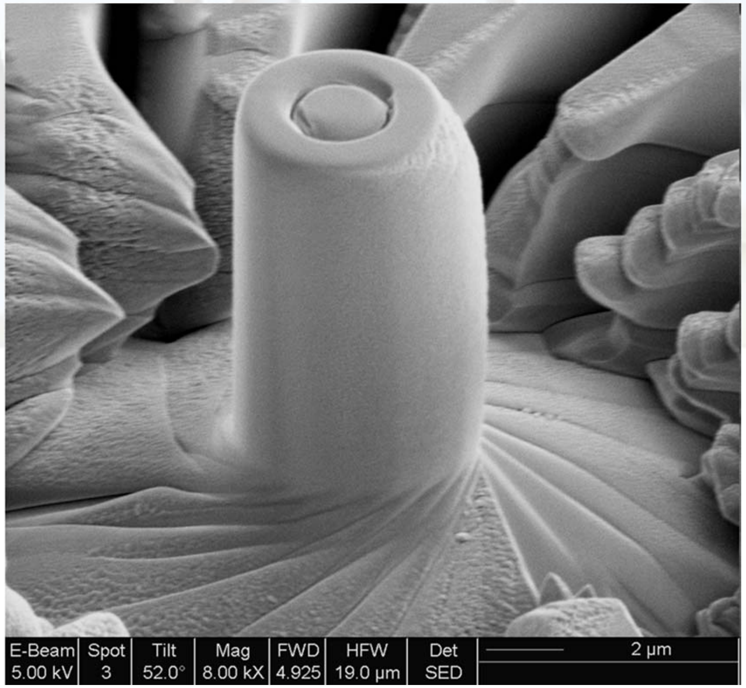
Beam current: 20.000 pA



Techniques used to characterize grains

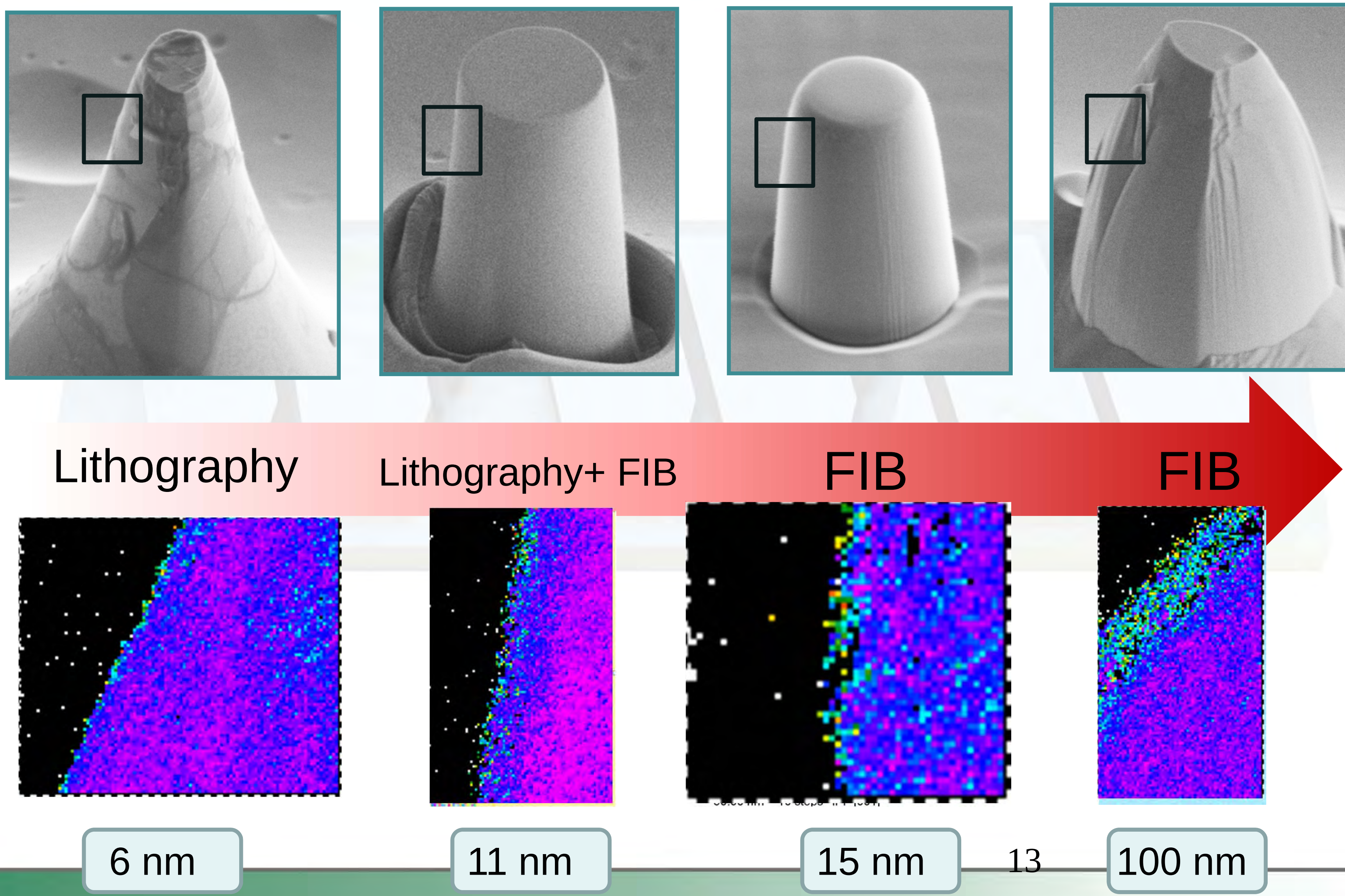
Sputtering process by FIB



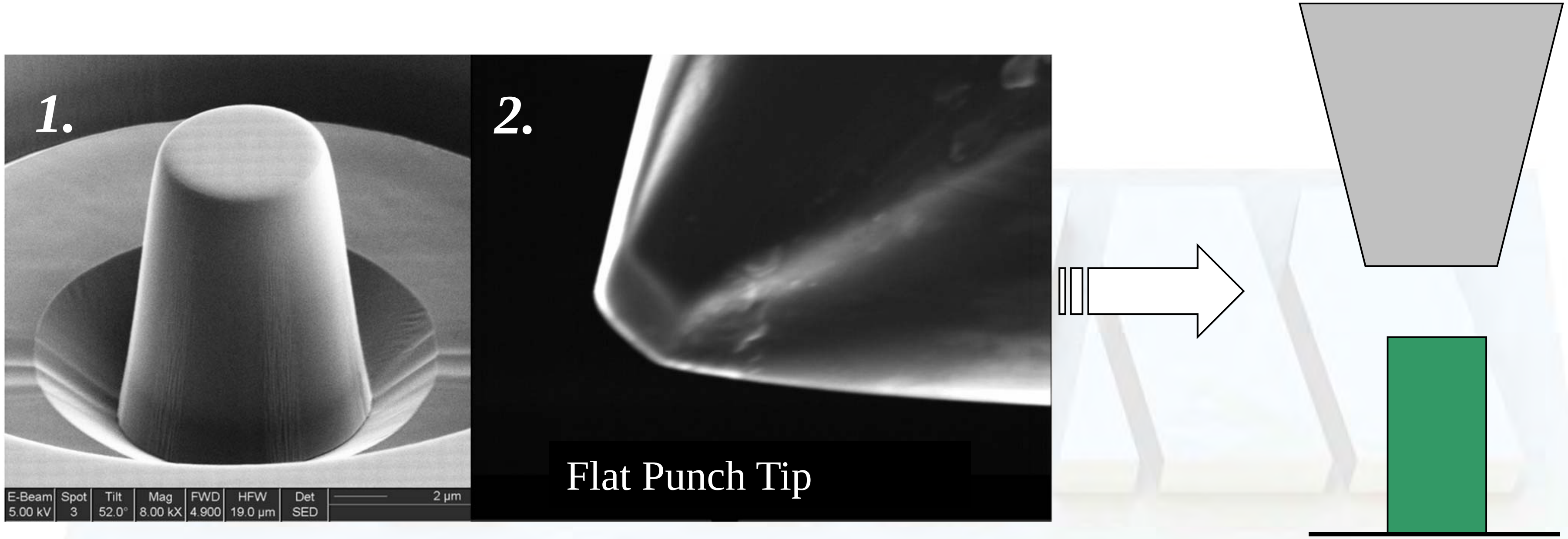


1. M. D. Uchic, D. M. Dimiduk, Materials Science and Engineering A 400-401, 268-278 (2005)

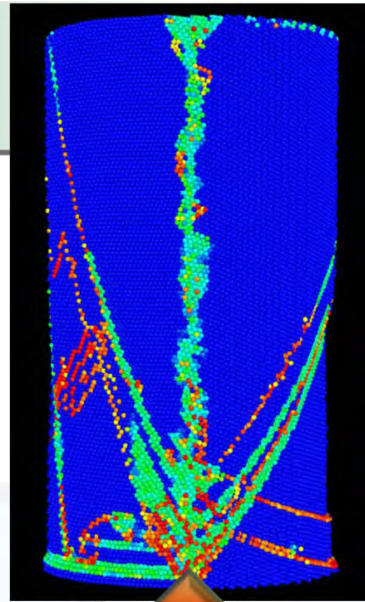
FIB Damage



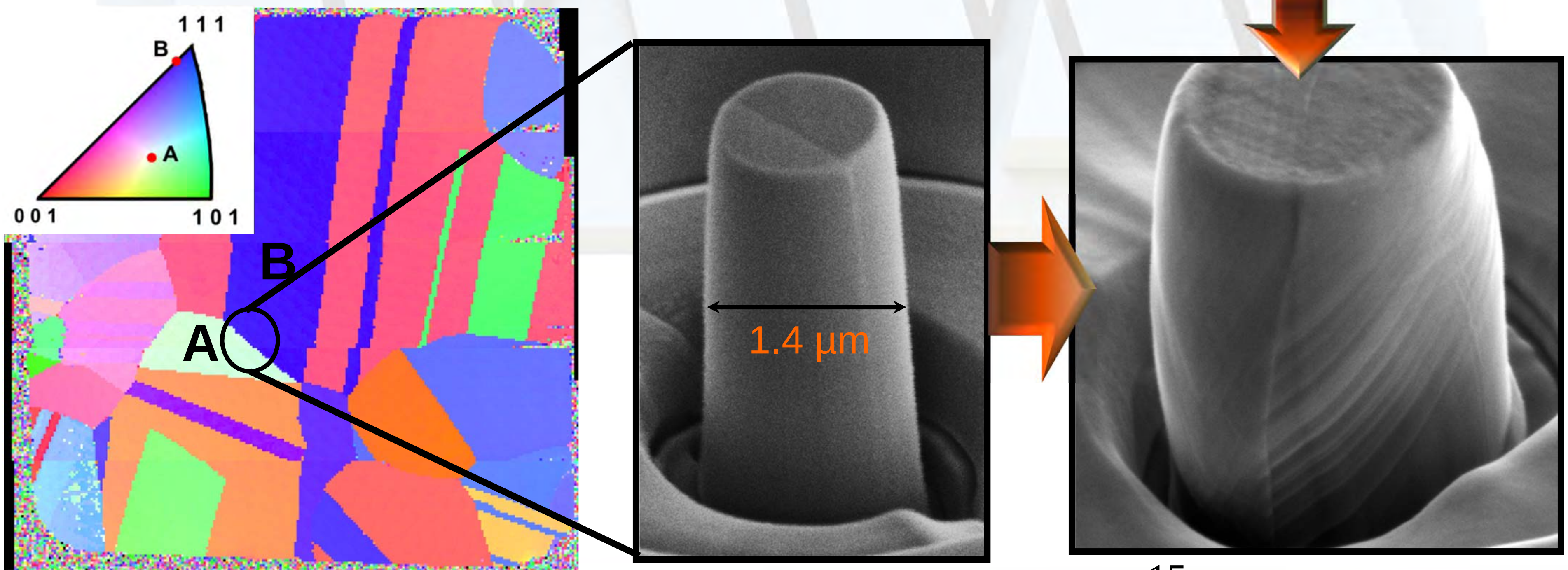
Mikroproben



Experimental



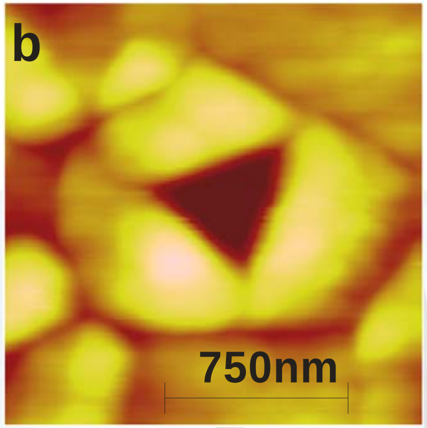
- ✓ Mesoscopic length scale
- ✓ Molecular Dynamics (MD) simulation
- ✓ 1 to 4 Mio. atoms
- ✓ Embedded atom potential

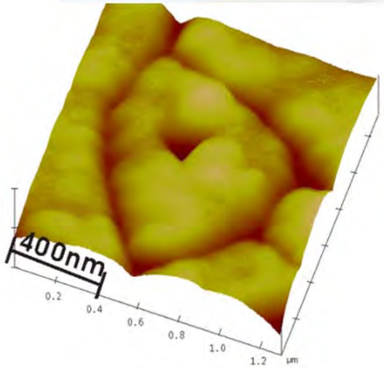


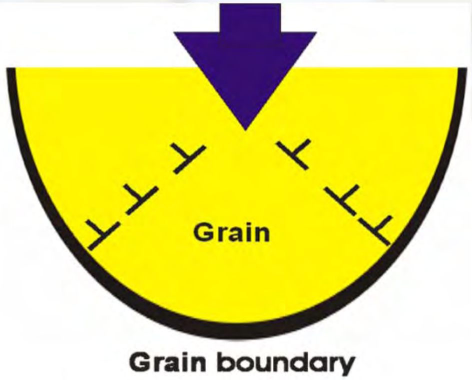
In cooperation with Dr. Ing. Afrooz Barnoush (WWM) and Dr. Mao Wen (Shanghai Jiao Tong University, China)

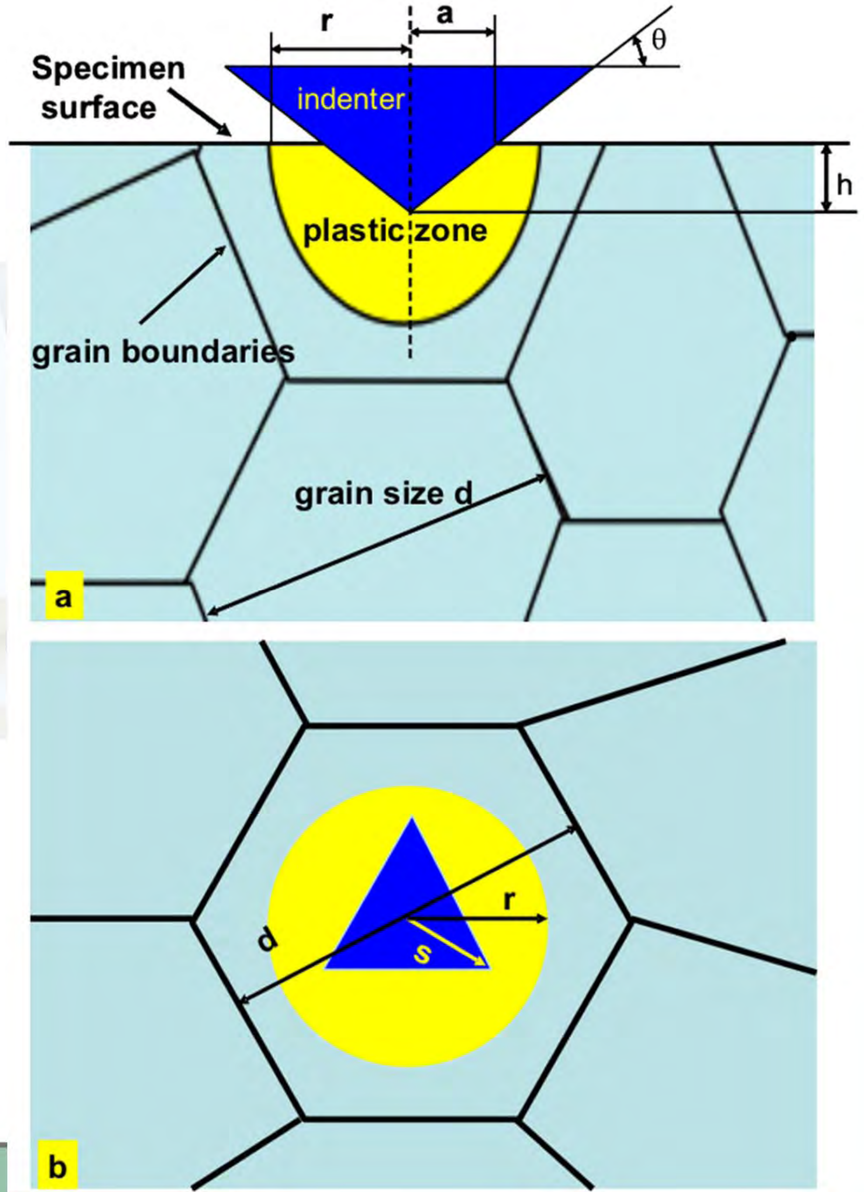
The indentation size effect and grain boundaries

- Nanoindentation

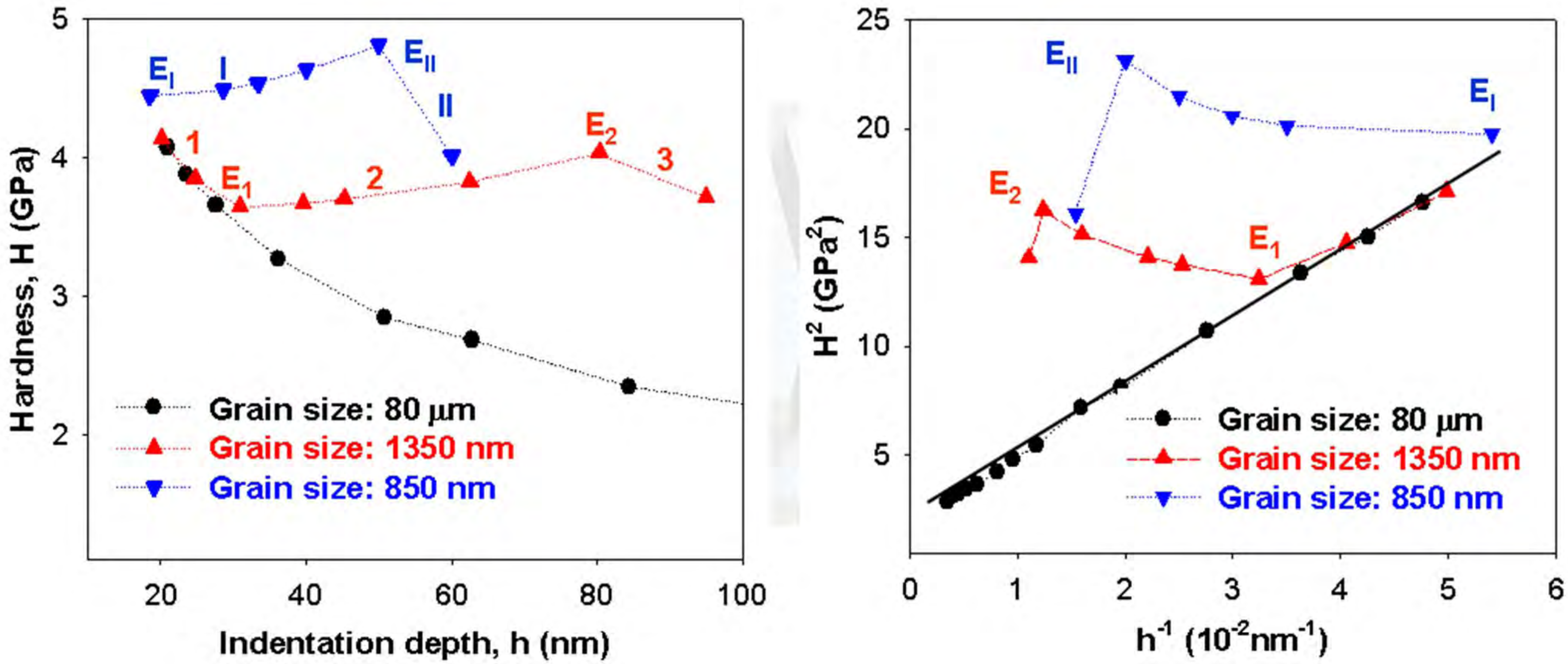








Indentation size effect and lateral boundaries



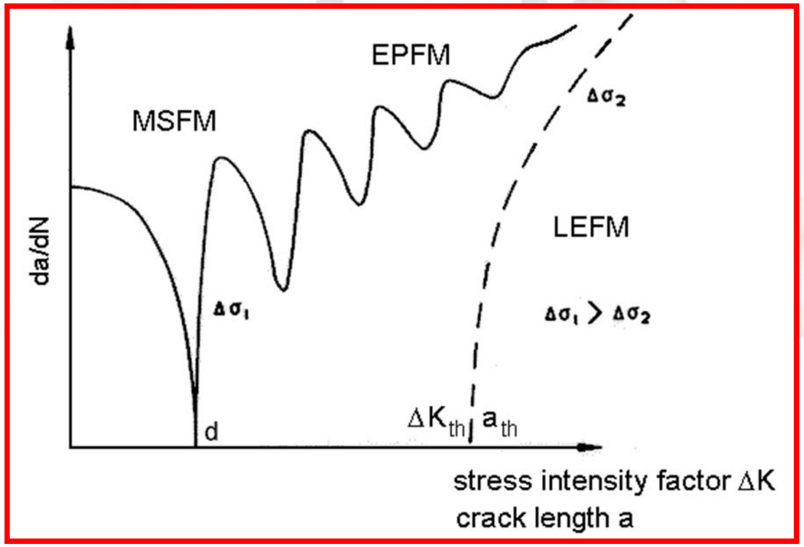
$$H^2 = H_0^2\left(1 + \frac{h_0}{h}\right)$$

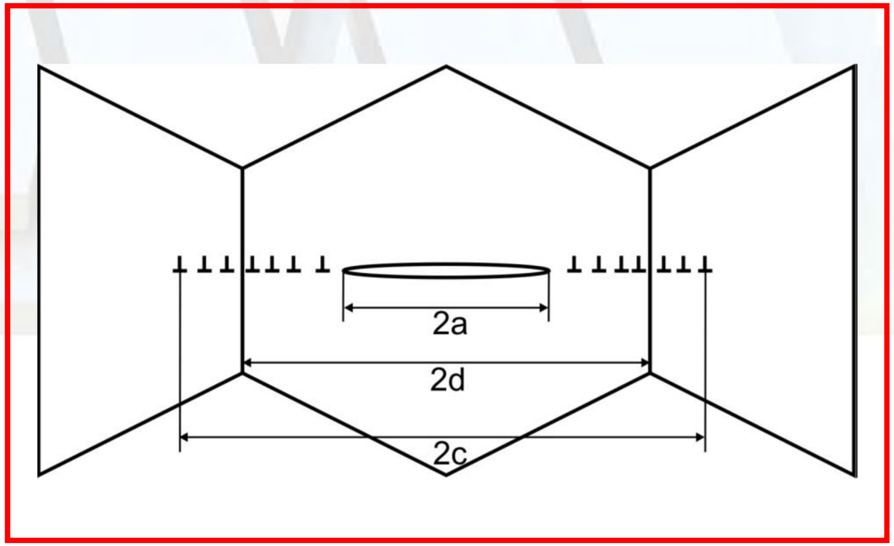
Grain boundary in front of the crack tip:

fluctuating crack propagation rate

crack stop possible

which parameters describe the resistance of a grain boundary against crack propagation?

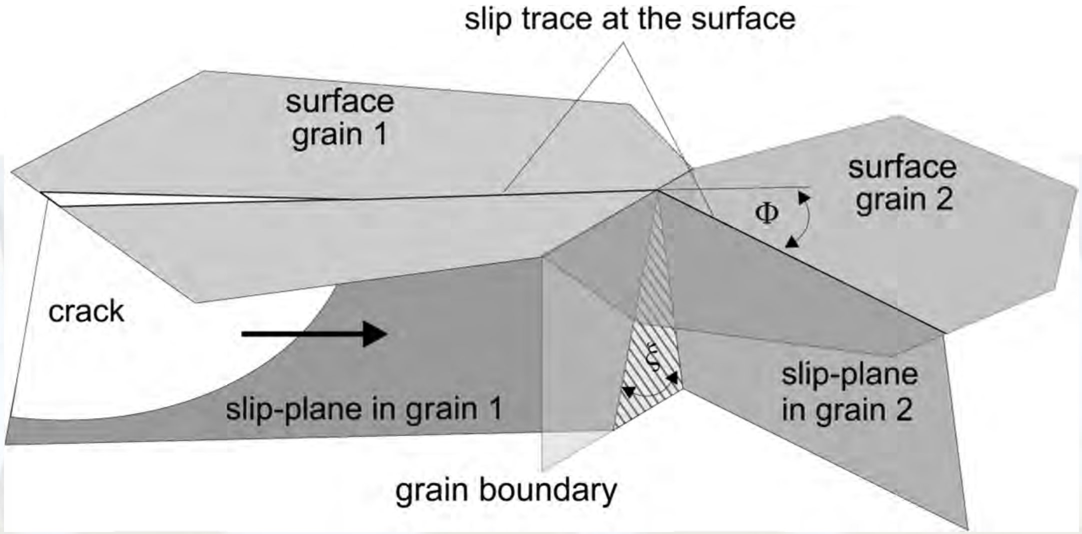




Experiments:

- Same Boundary, distance between crack and boundary is varied
- Same Crack, but different types of boundaries and grains

3-D-problem: model of Zhai as example:



Mechanisms of interaction:

- inclination angle of the grain boundary?

- involved slip planes?

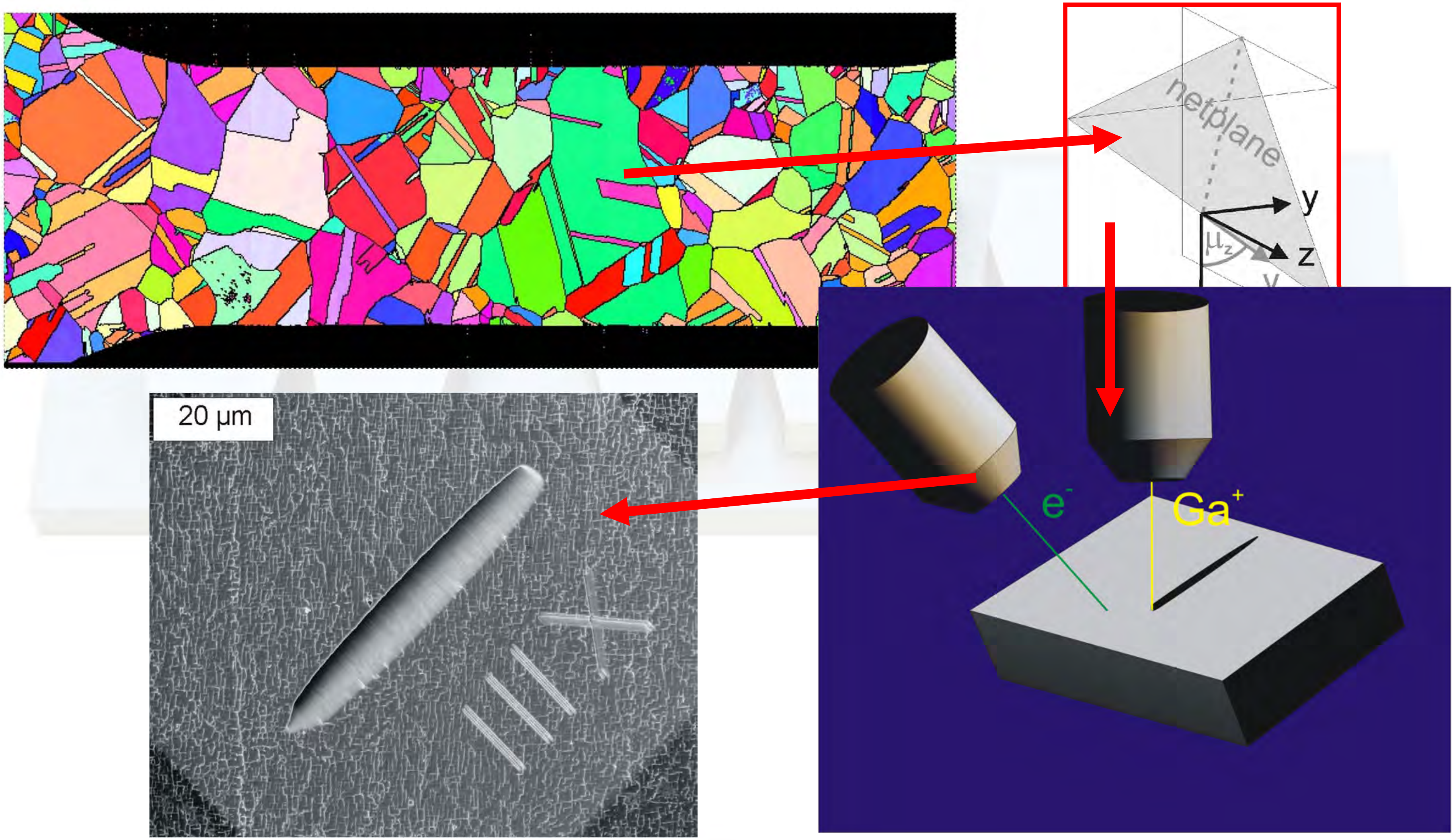
=> 3 dimensional information needed!

=> quantifying the model by 3D FIB tomography

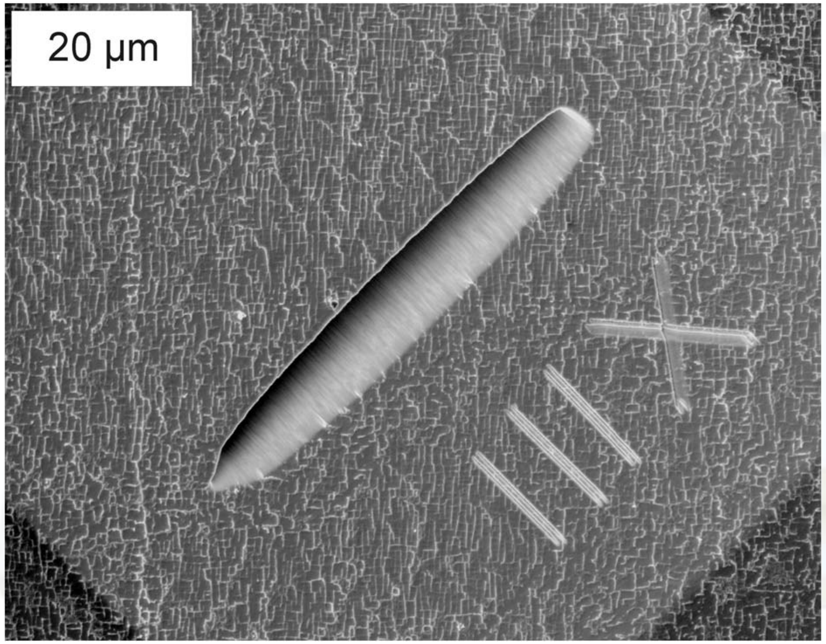
T. Zhai et al, International Journal of Fatigue 27 (2005) 1202-1209

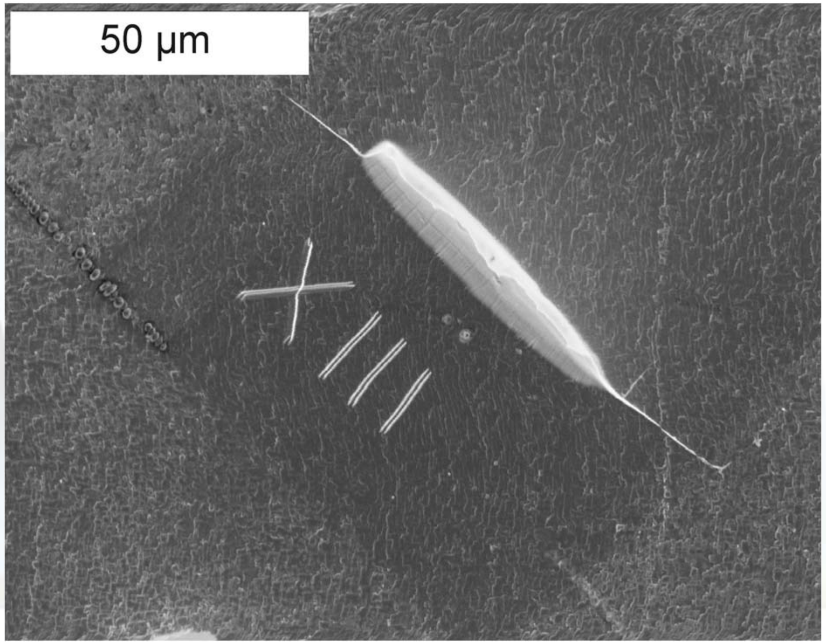
Artificial crack initiation

Initiating a crack with a FIB cut notch:



Stage I crack parallel to a slip plane

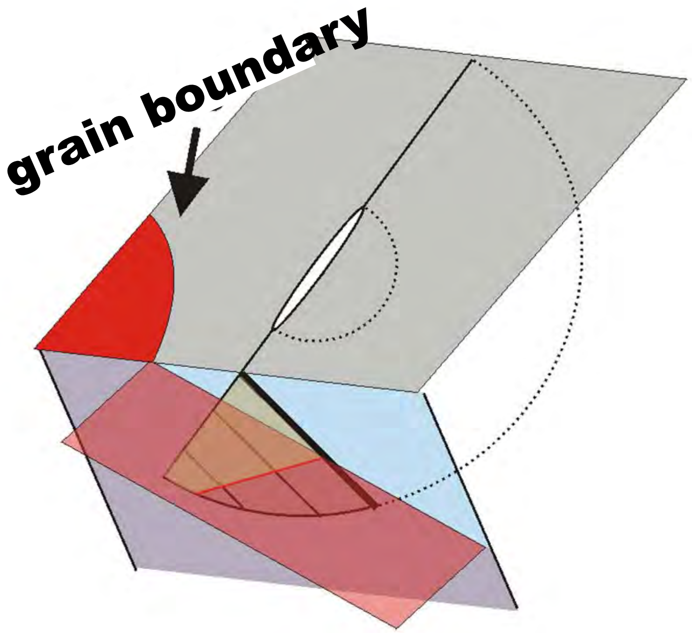


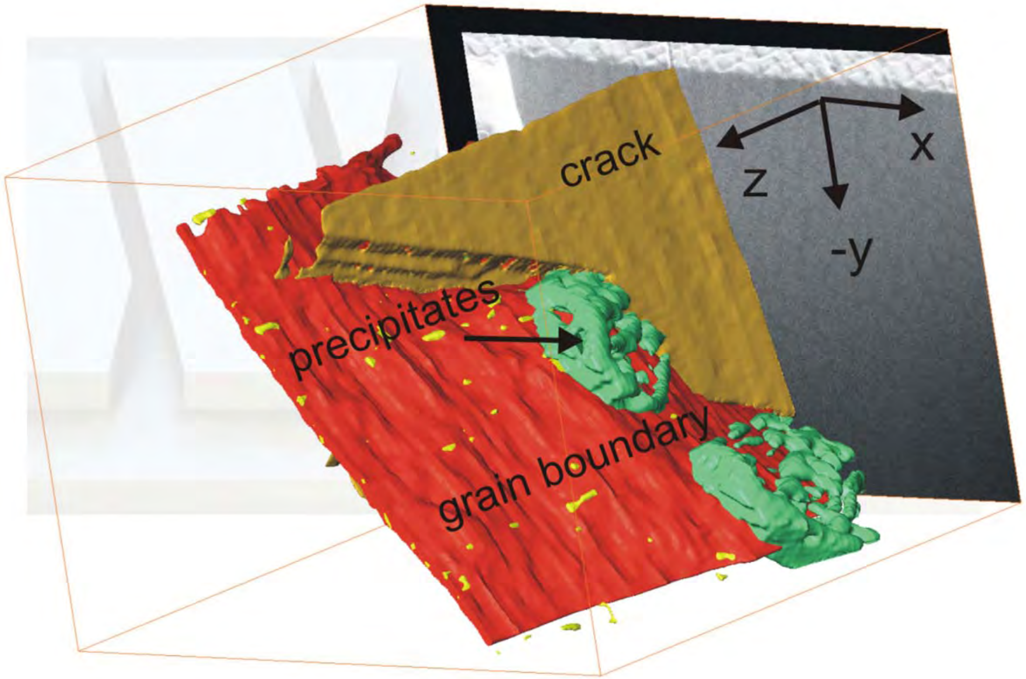


Combining OIM with FIB

Schäf, Marx, Holzapfel, Vehoff, Mat. Sci. 2006

Analysis of the images enables **a 3D reconstruction of the crack**





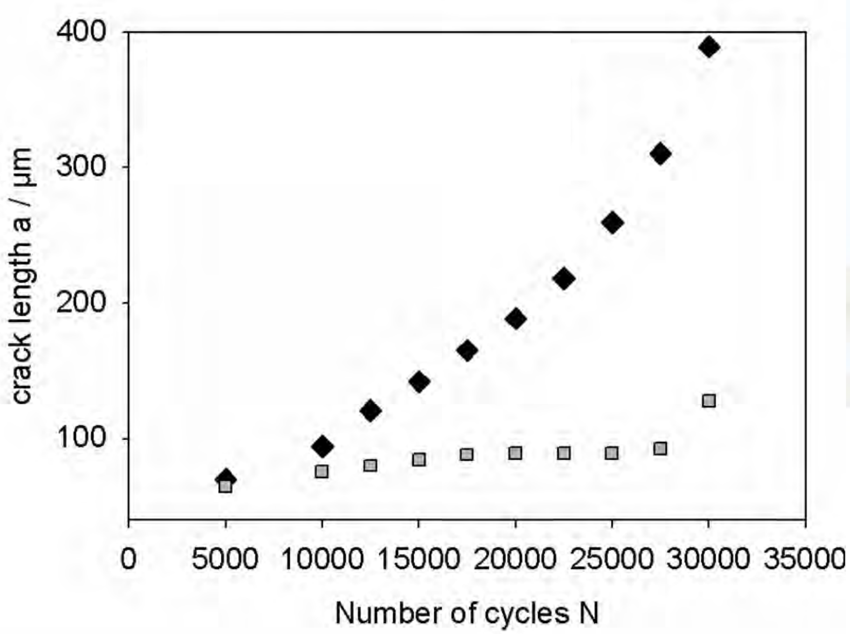
Growth of artificial cracks

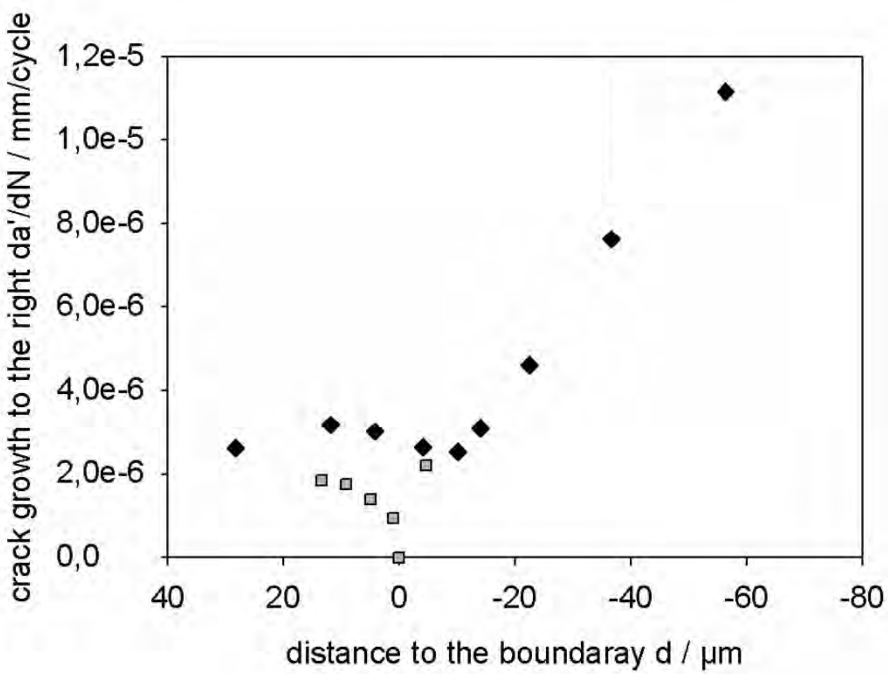
Fatigue tests interrupted for replicas

- stress amplitude: σ_A = 300 MPa

- load ratio R = -0.1

Two cracks with different lengths interact with the same grain boundary





different crack propagation rates!

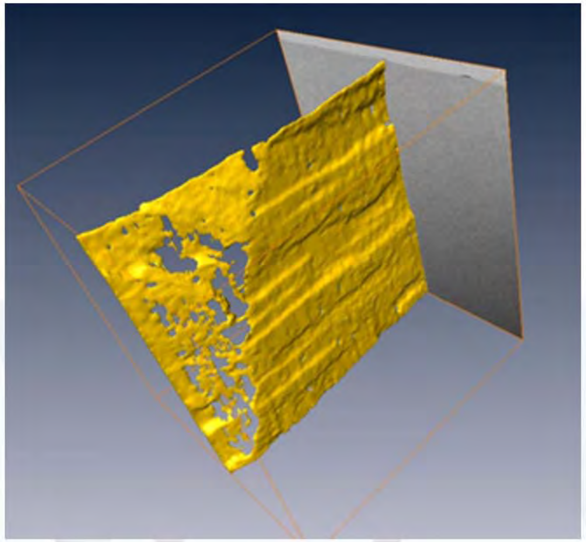
Some cracks stop or only move slower, why?

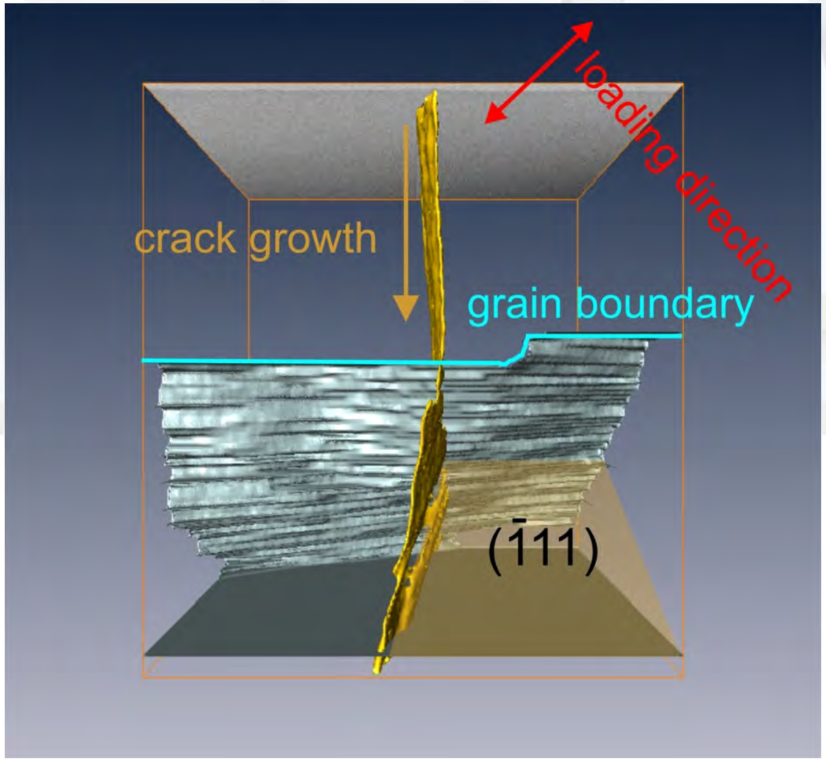
Influence of the microstructure

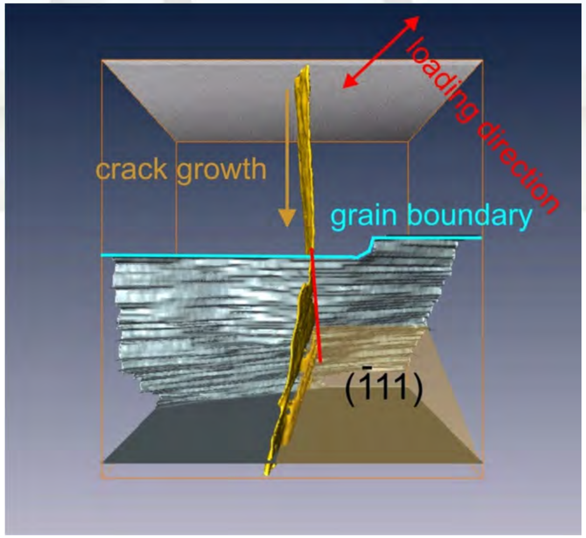
Mechanisms of interaction:

- large misorientation angle

- but only deceleration







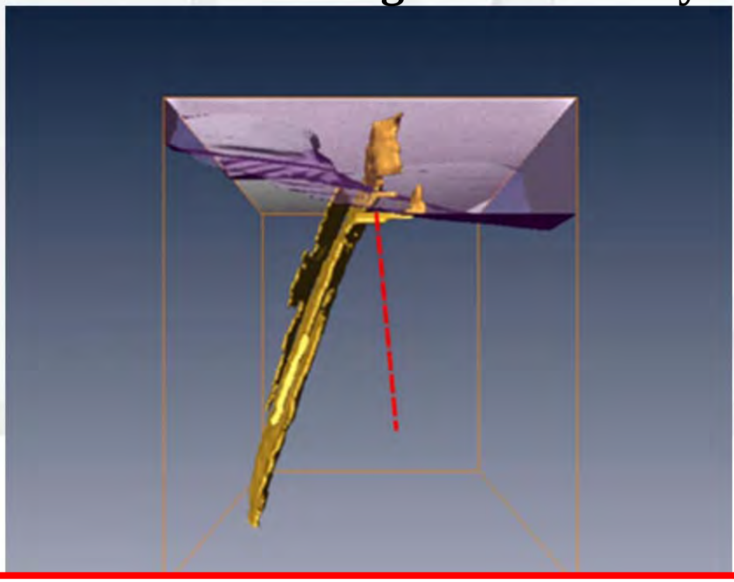
Influence of the microstructure

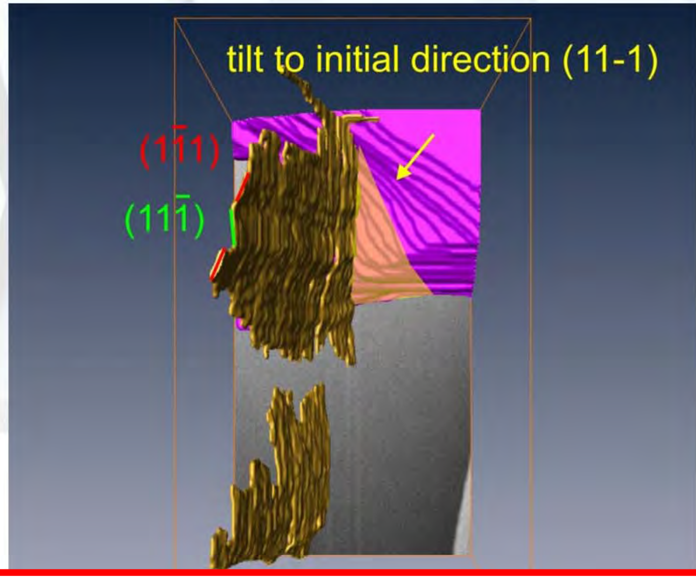
Mechanisms of interaction:

- same grain boundary

- second crack with identical crack parameters

- other inclination of the grain boundary





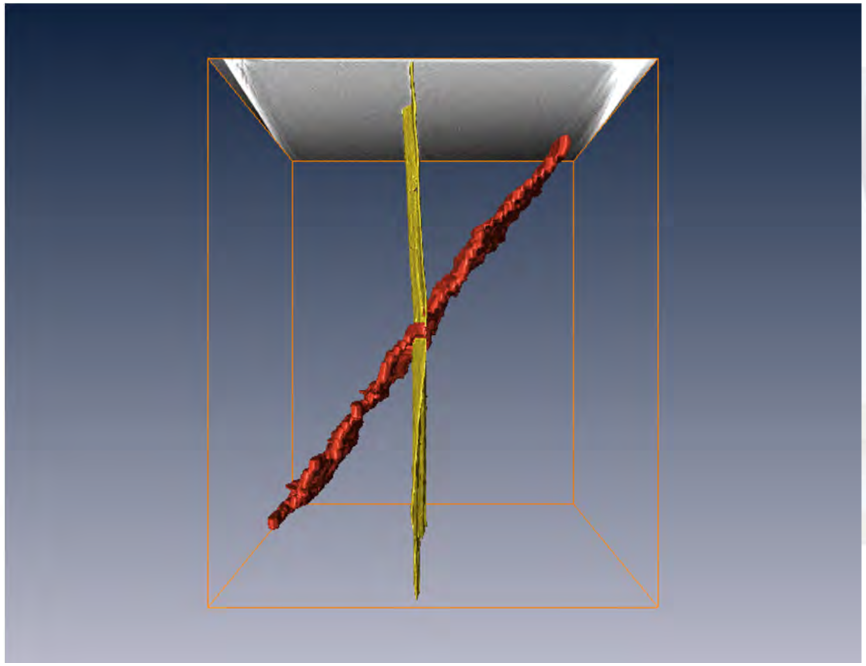
resistance of a grain boundary against crack propagation determined by:

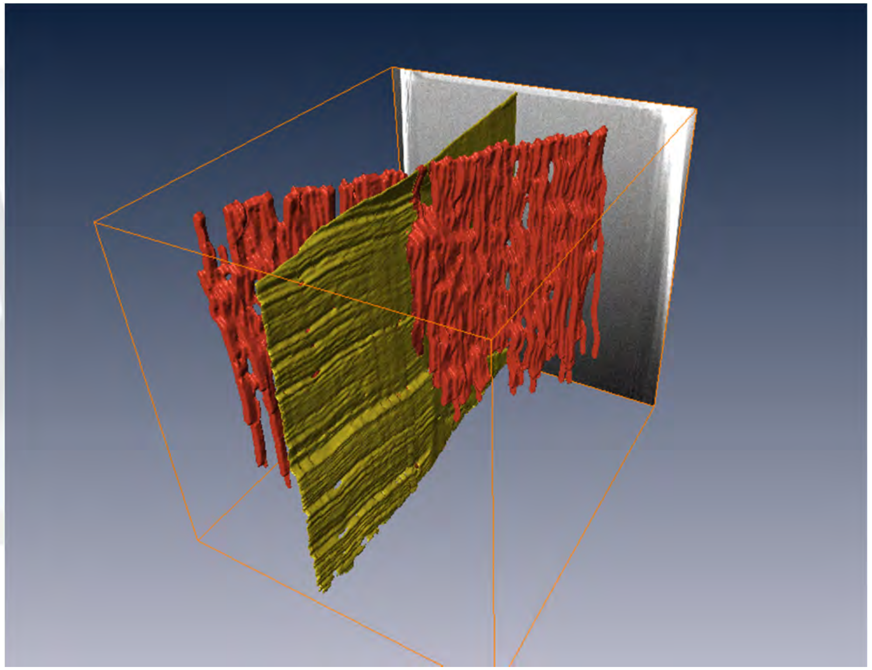
- possibility for the crack to propagate continuously through the grain boundary

- not determined by the misorientation angle of the active slip planes?

Mechanisms of interaction:

- grain boundary with special orientation





crack passes the grain boundary by alternating activation of (111) slip planes for a continuous propagation through the grain boundary

Quantifying the crack propagation:

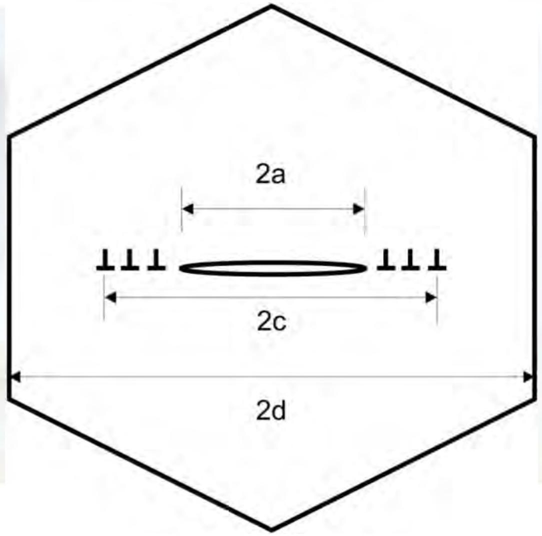
1. for a slip band without grain boundary (Equilibrium Slip Bands, ESP):

$$\frac{da}{dN} = C \cdot \Delta CTSD^n$$

BCS-model used by Tanaka[1]:

crack tip sliding displacement

$$\Delta CTSD = \left(\frac{2\tau^* a}{\pi^2 A}\right) \ln\left(\frac{c}{a}\right), \qquad A = \frac{G}{2\pi(1-\nu)}$$



τ* = shear-stress necessary to move a dislocation

$$\frac{a}{c} = \cos\left(\frac{\pi \Delta\tau}{2\tau^*}\right)$$

parameters needed:

- constants C and n

- shear-stress τ*

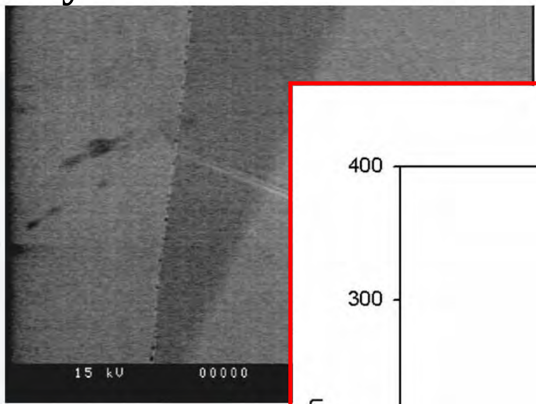
- Schmid-factor (to calculate Δτ from applied load σ)

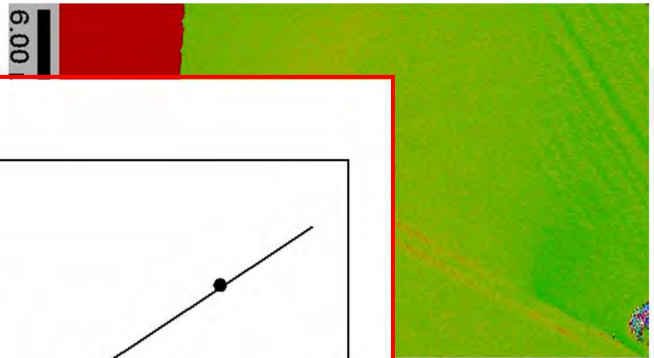
[1]Tanaka K., Akiniwa Y., Nakai y., Wei R.P.: Engineering Fracture Mechanics, Vol.24, 803-819, 1986

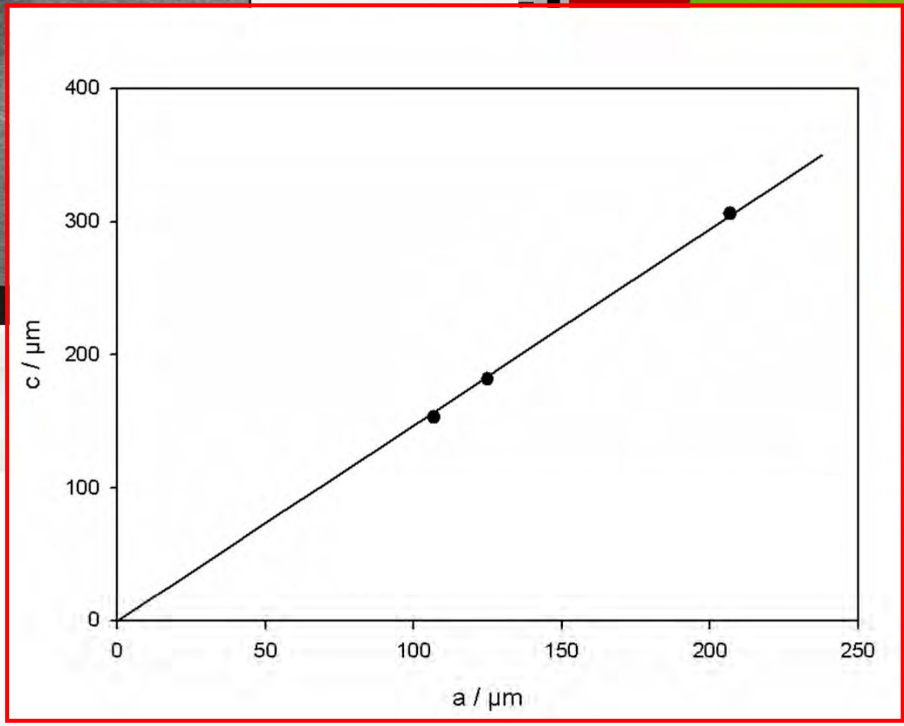
Quantifying the crack propagation:

measuring the plastic zone size:

by ECCI **and by EBSD**







$$\frac{\mathbf{a}}{\mathbf{c}} = \cos\left(\frac{\pi\Delta\tau}{2\tau^*}\right)$$ **=> shear-stress τ* = 502 MPa**

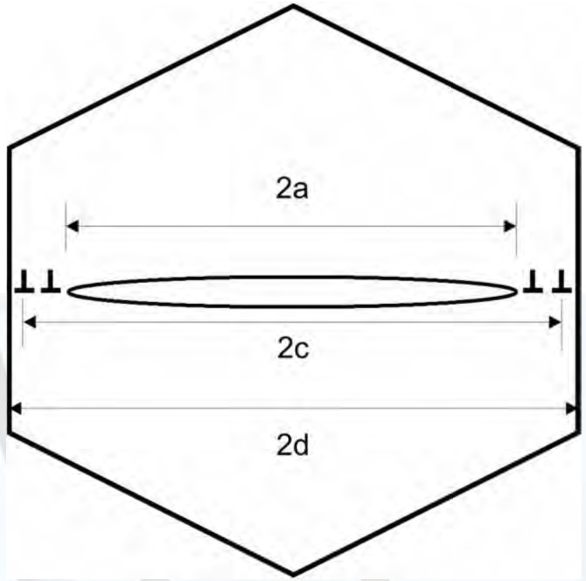
Quantifying the crack propagation:

2. for a slip band with grain boundary

2 a) plastic zone blocked by the grain boundary (Blocked Slip Band, BSB):

$$\Delta CTSD = \left(\frac{\beta \Delta \tau}{\pi A}\right)\left(c^2 - a^2\right)^{1/2} + \left(\frac{2\tau^* a}{\pi^2 A}\right)\ln\left(\frac{c}{a}\right)$$

$$\beta = 1 - \left(\frac{2\tau^*}{\pi \Delta \tau}\right)\arccos\left(\frac{a}{c}\right)$$



2 b) plastic zone spread in the neighboring grain (Propagating Slip Band, PSB):

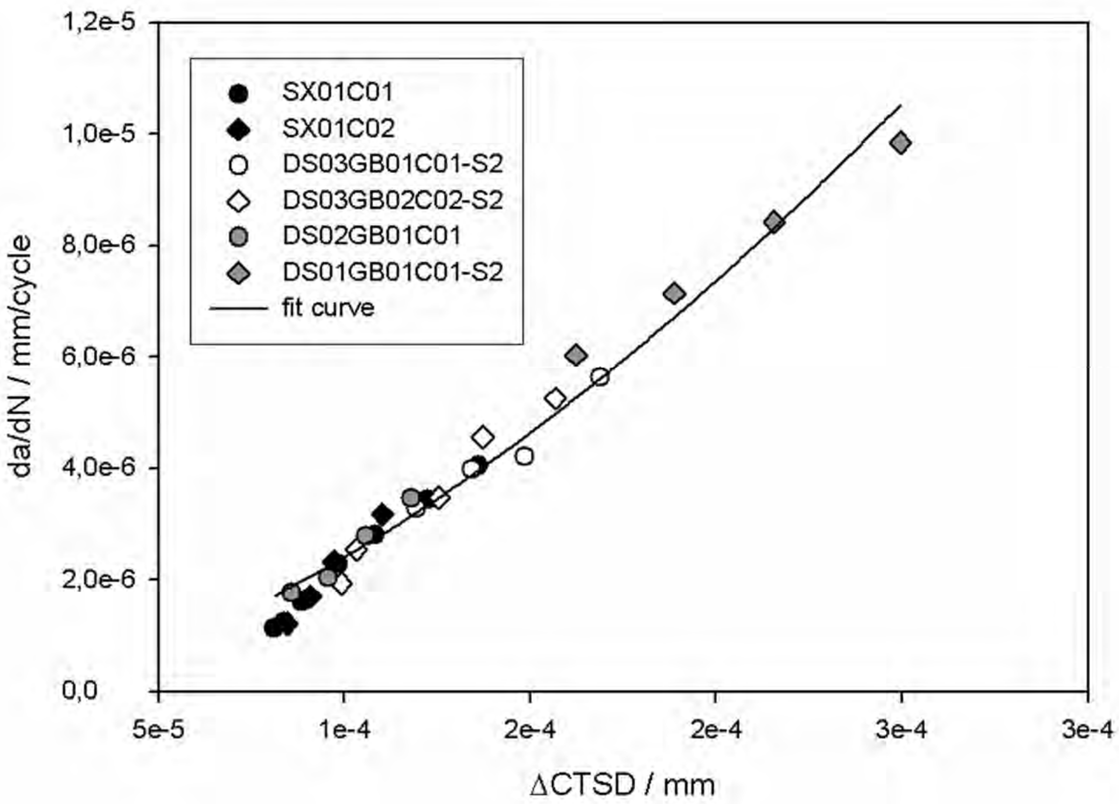
resulting shear-stress τ^* determined by τ_1 and τ_2 of both grains:

$$\Delta CTSD = \left(\frac{2\tau^* a}{\pi^2 A}\right)\ln\left(\frac{c}{a}\right) + \left(\frac{\tau^*_2 - \tau^*}{\pi^2 A}\right) g(a;c,d)$$

$$g(a;c,d) = d \cdot \ln\left|\frac{\sqrt{c^2 - d^2} + \sqrt{c^2 - a^2}}{\sqrt{c^2 - d^2} - \sqrt{c^2 - a^2}}\right| - a \cdot \ln\left|\frac{a\sqrt{c^2 - d^2} + d\sqrt{c^2 - a^2}}{a\sqrt{c^2 - d^2} - d\sqrt{c^2 - a^2}}\right|$$

Quantifying the crack propagation:

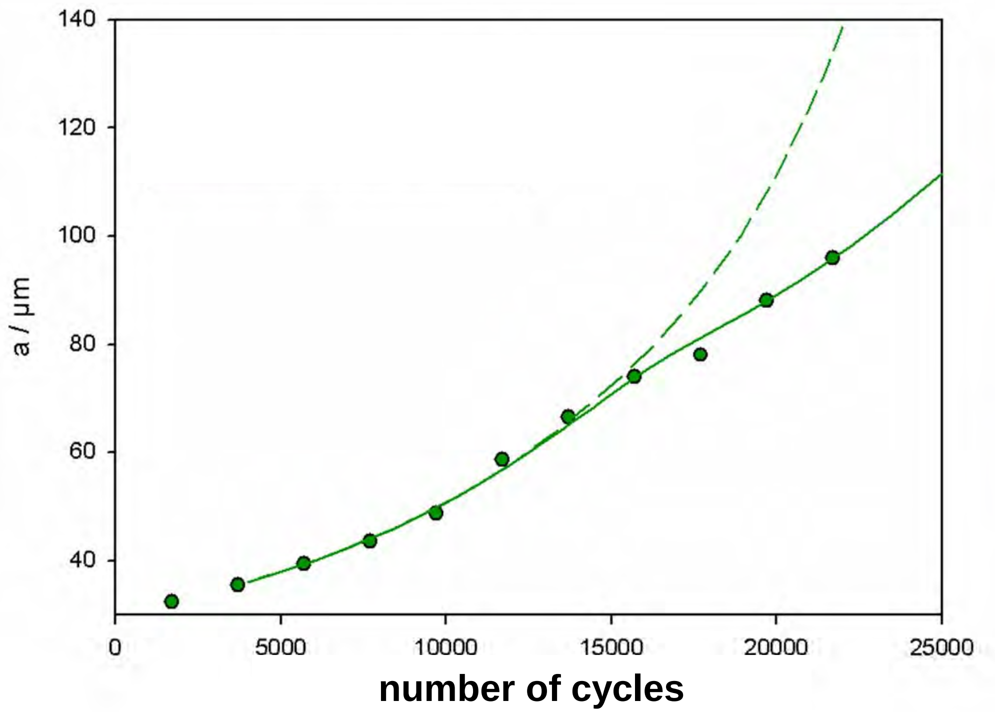
measuring the crack propagation rate by replica technique



Quantifying the crack propagation:

2. for a slip band with grain boundary

2 b) plastic zone spread in the neighboring grain (Propagating Slip Band, PSB):



calculation done with the parameters from the single crystal measurements

no further fit-parameter!

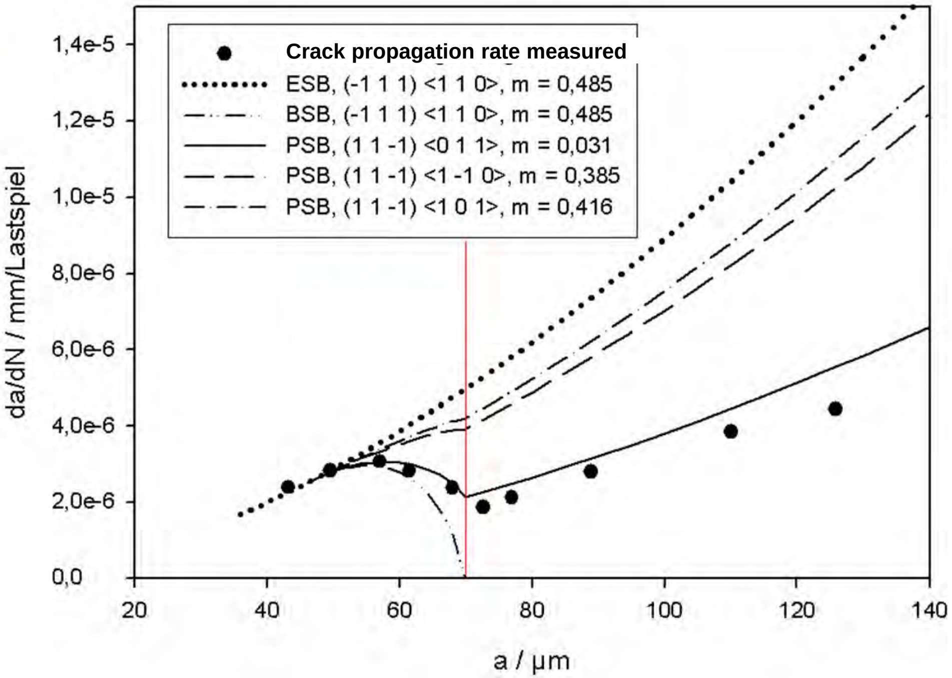
Numerical model

Quantifying the crack propagation:

2. for a slip band with grain boundary

2 b) plastic zone spread in the neighboring grain (Propagating Slip Band PSB):

shear-stress τ^* determined by τ_1 and τ_2 of both grains:

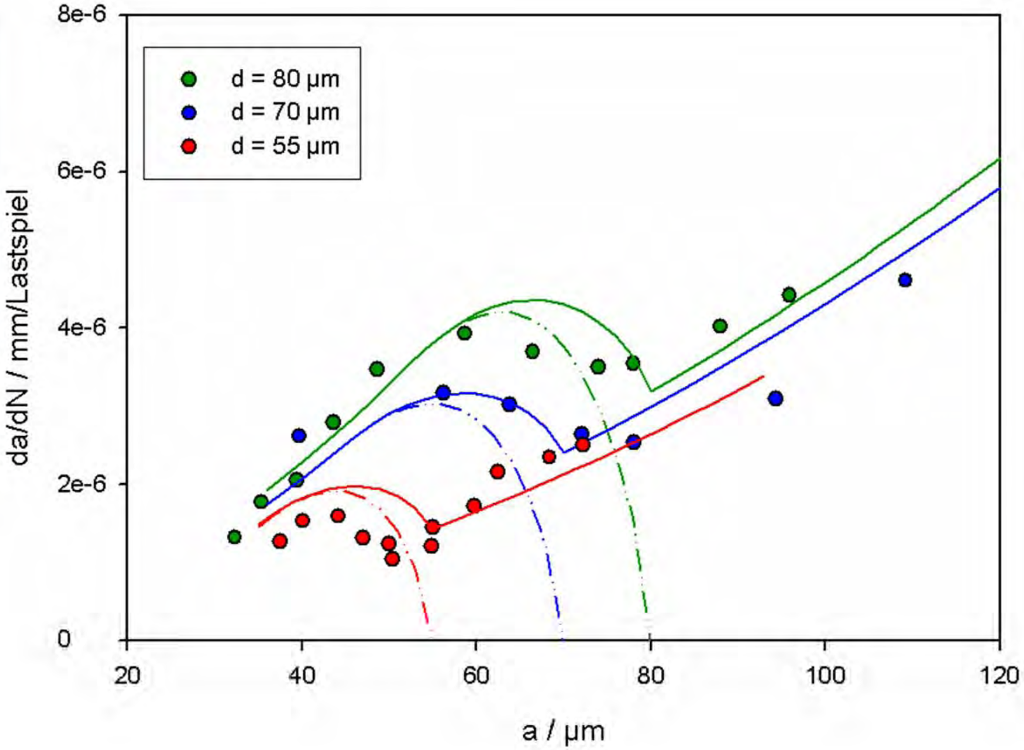


τ_2 determined for the adjacent grain by the slip system with the lowest Schmid-factor

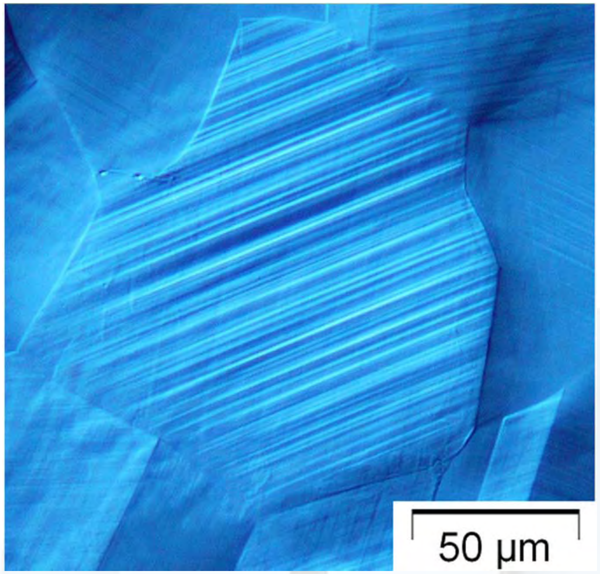
Quantifying the crack propagation:

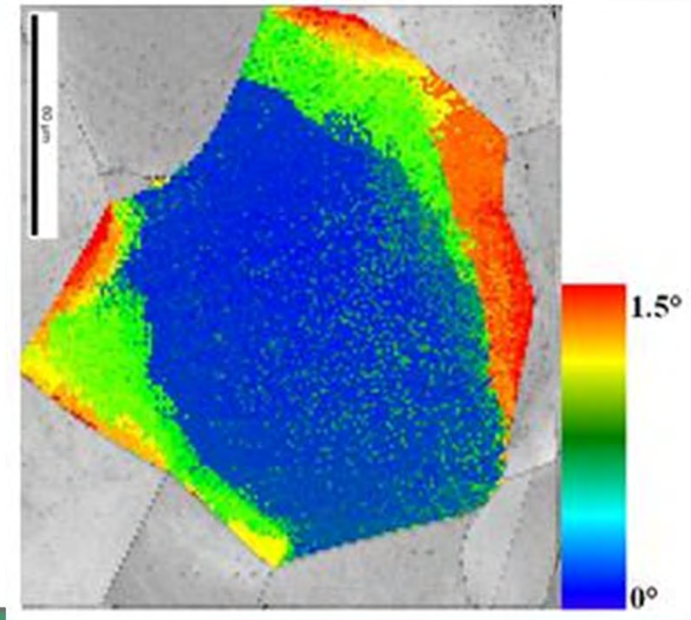
2. for a slip band with grain boundary and

different distances between notch tip and grain boundary

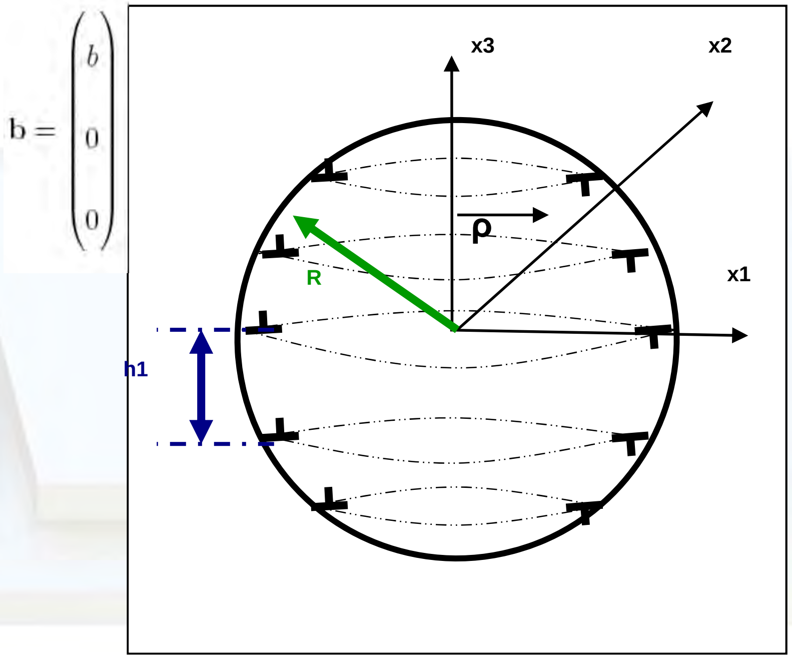


Dislocations and boundaries



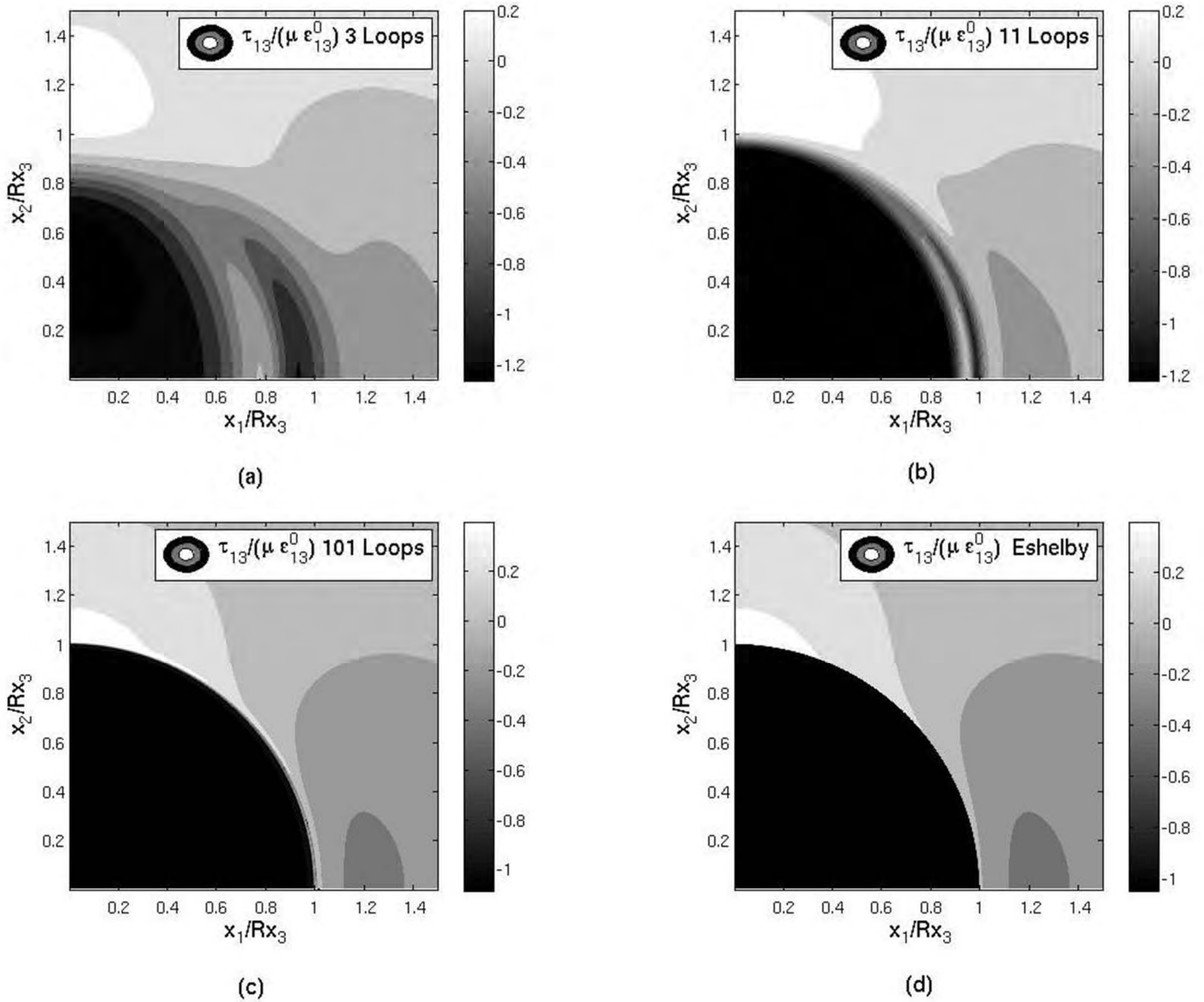


Analytical model of the problem



Orientation gradient mapping
Lattice rotations due to GND's

Stress concentration at grain boundaries as a function of the number of discrete slipbands



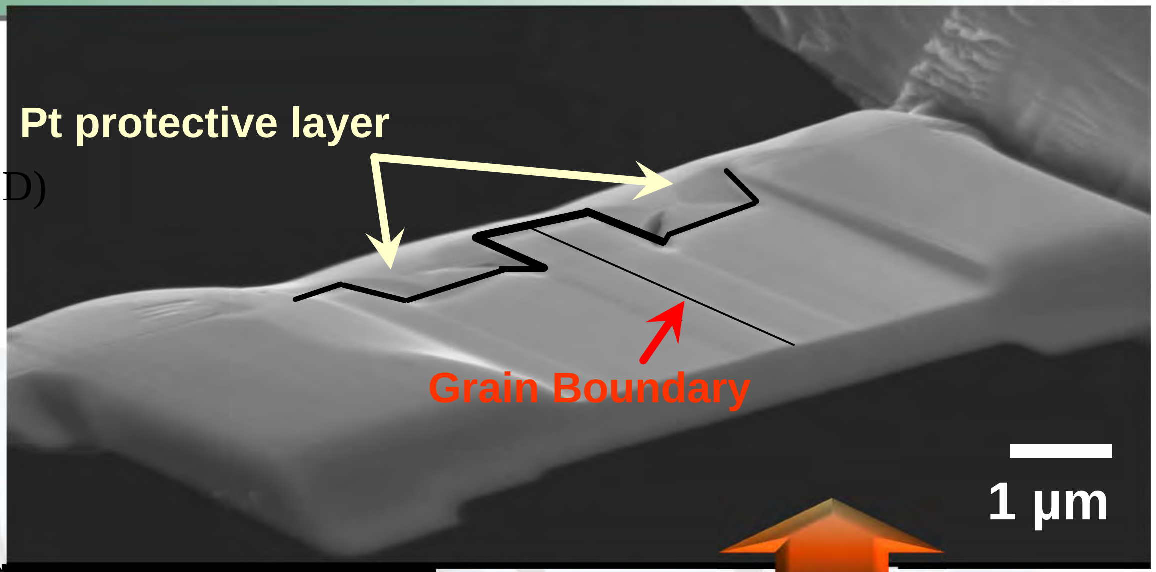
3 and 11 Slip bands

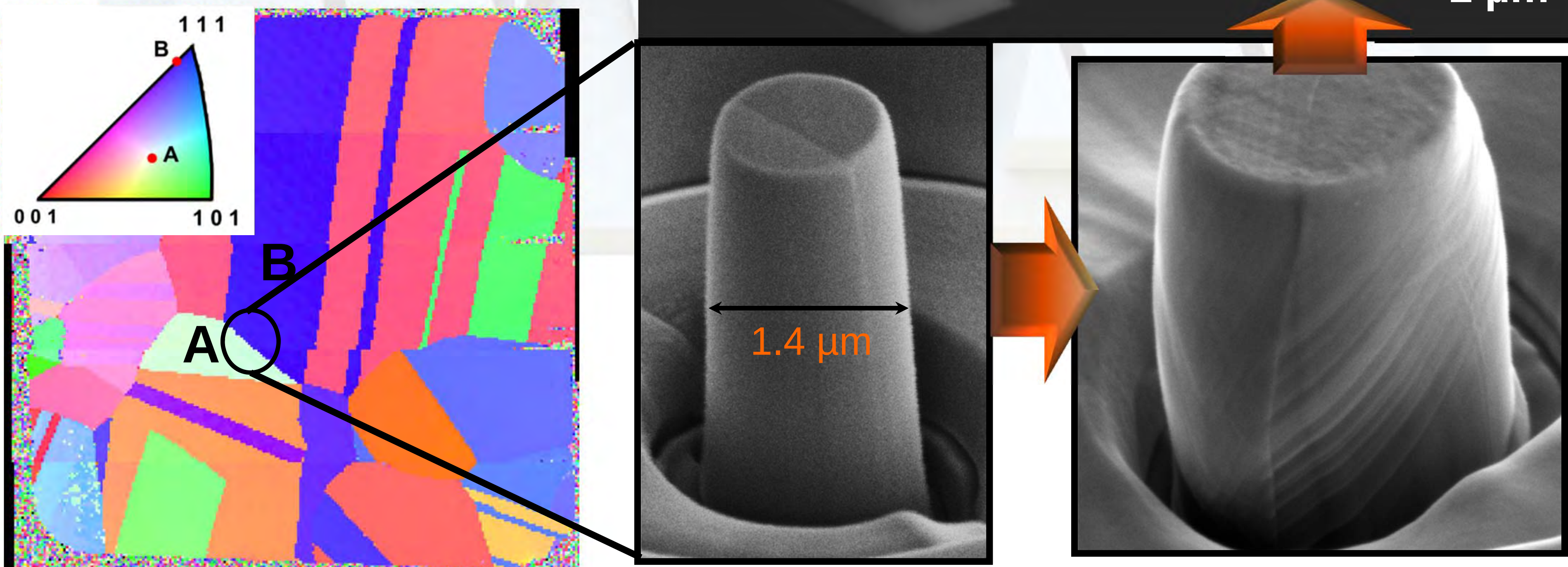
101 and continuum

Camille Perrin, Stephane Berbenni, Horst Vehoff, Marcel Berveiller: Acta Mat. 58, 4639-4649, 2010

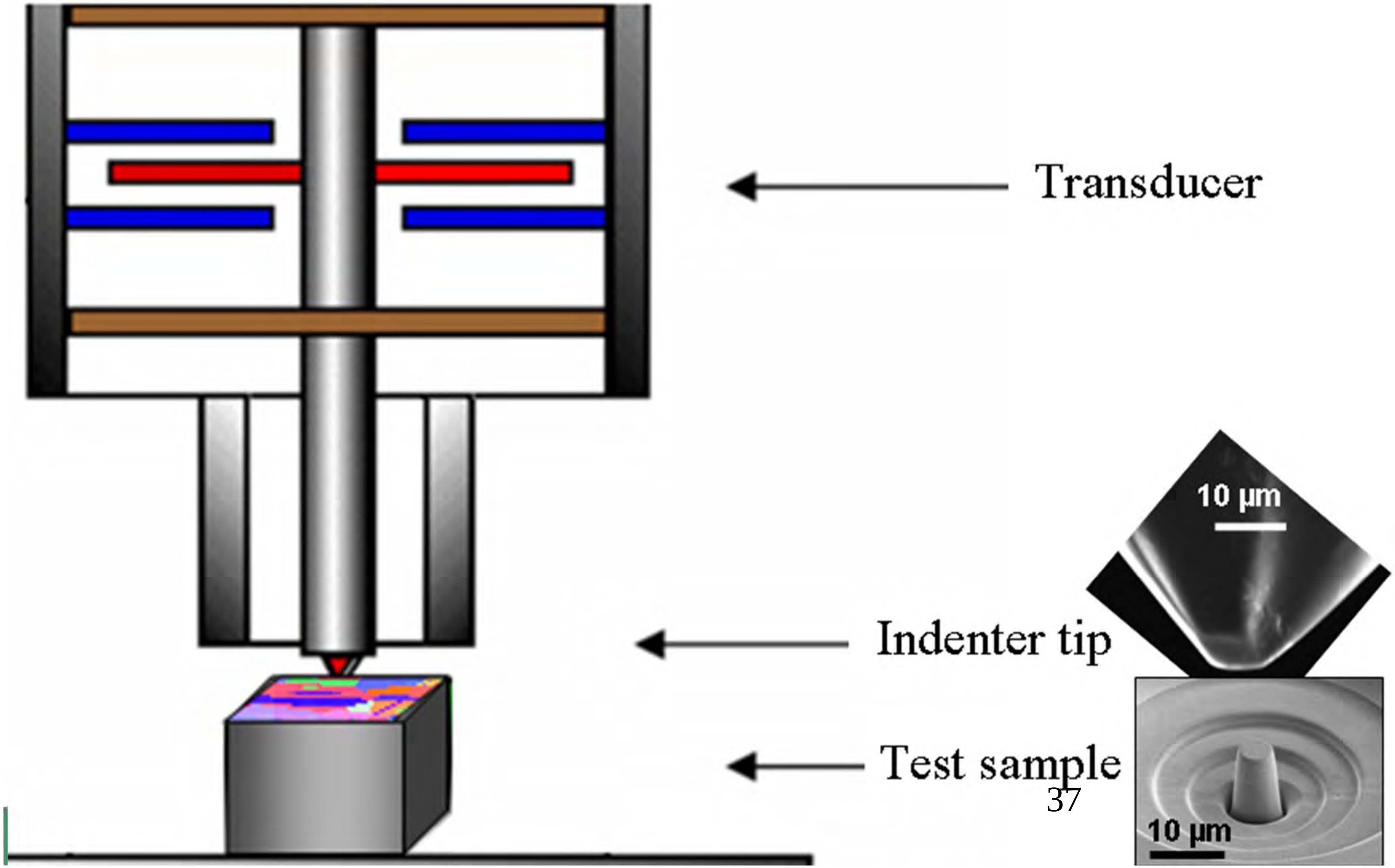
Nanopillar bicrystals, the effect of boundary strength

- ✓ Mesoscopic length scale
- ✓ Electron Backscattered Diffraction (EBSD) measurements

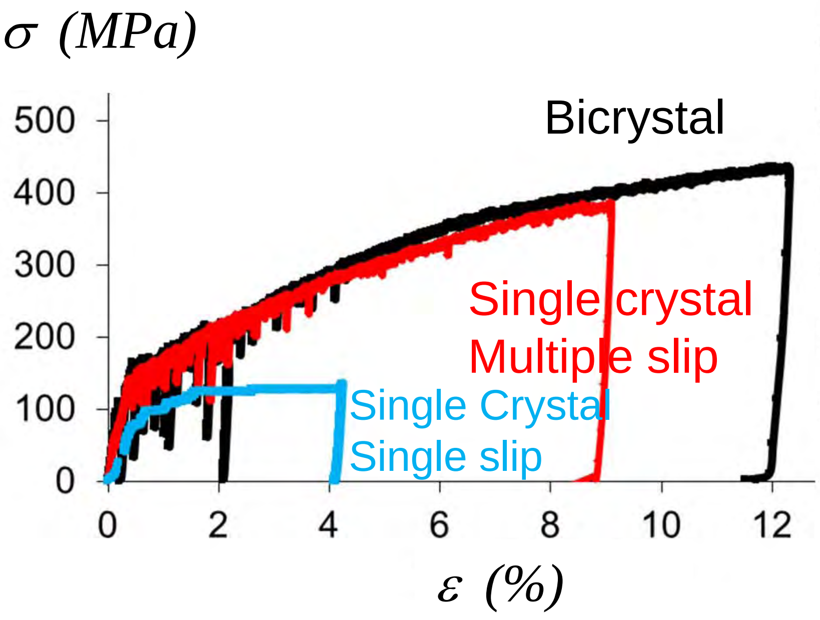




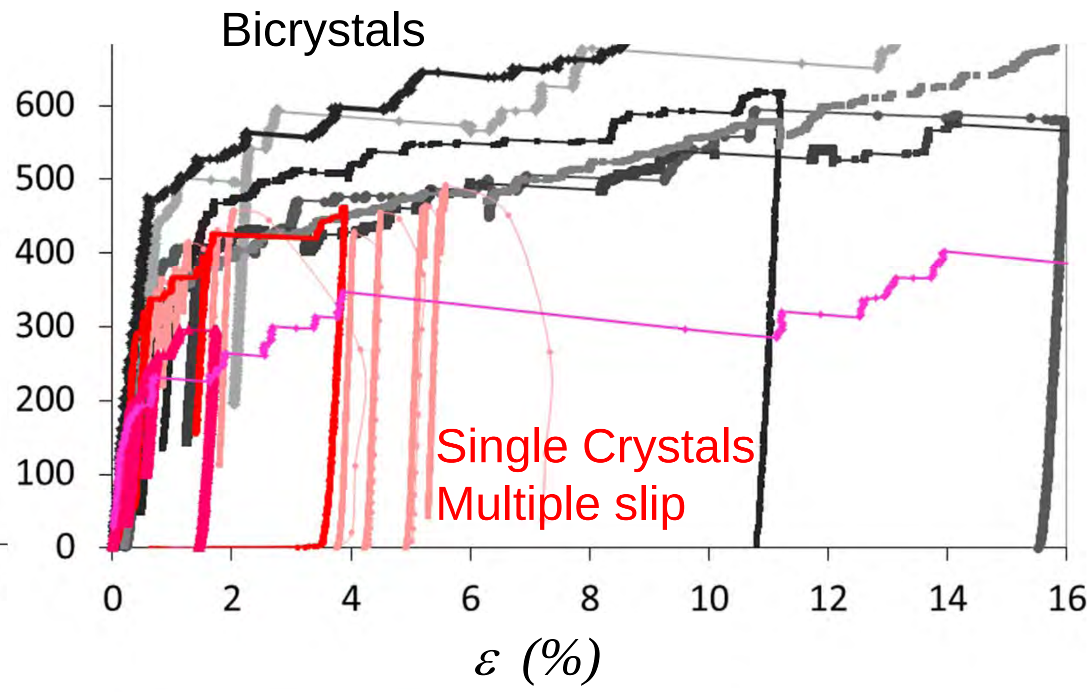
Micromechanical testing



Compression test results



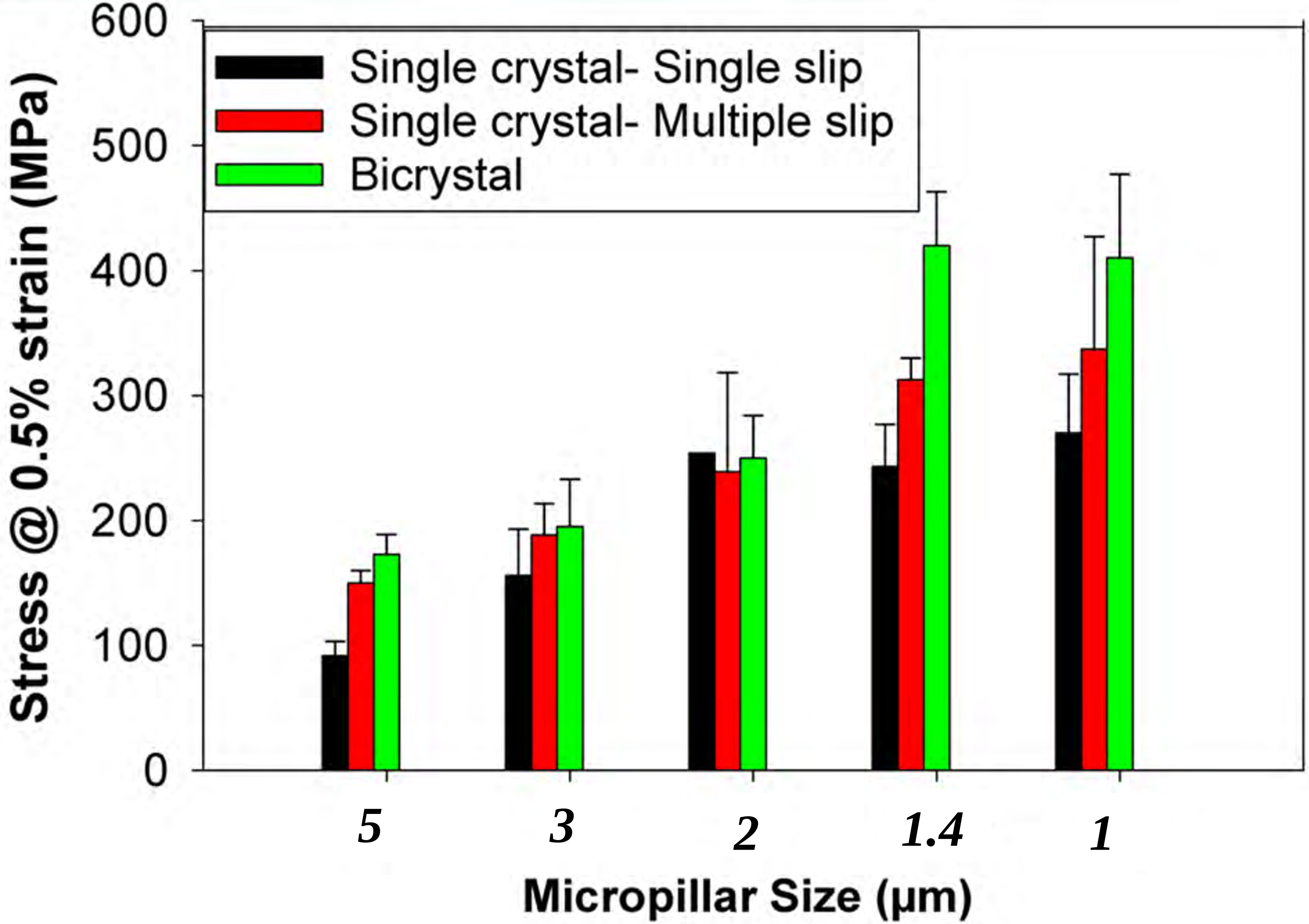
$$\phi = 5\,\mu\text{m}$$



$$\phi = 1\,\mu\text{m}$$

Kheradmand Nousha; Vehoff Horst: ADVANCED ENGINEERING MATERIALS 14, 153-161, 2012

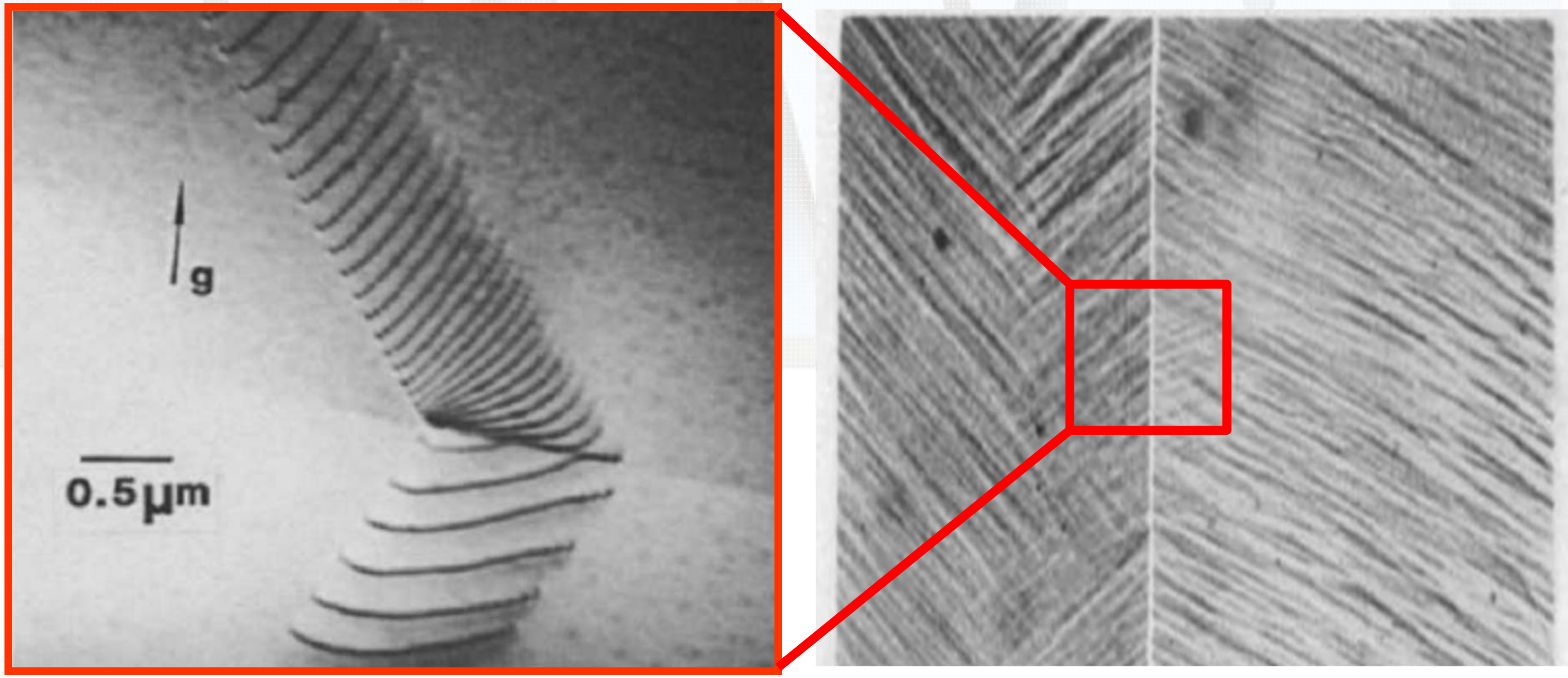
The effect of grain size on strength



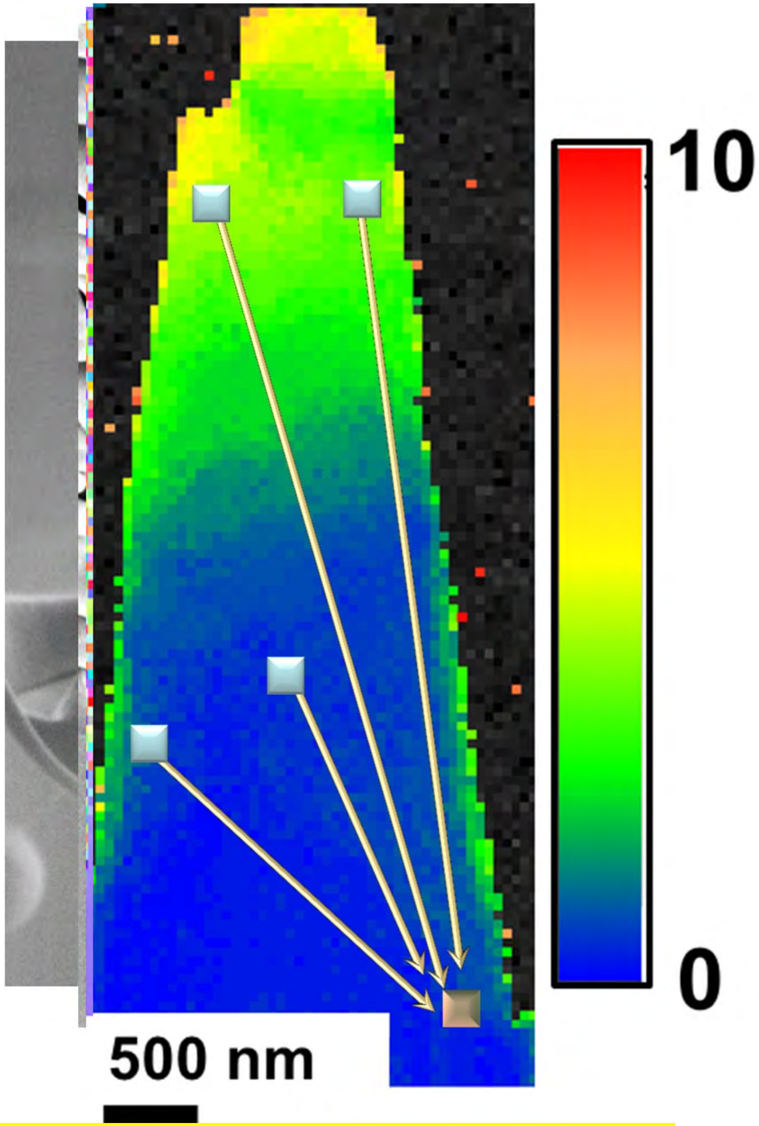
The gb effects the strength

$\phi < 2\ \mu m$

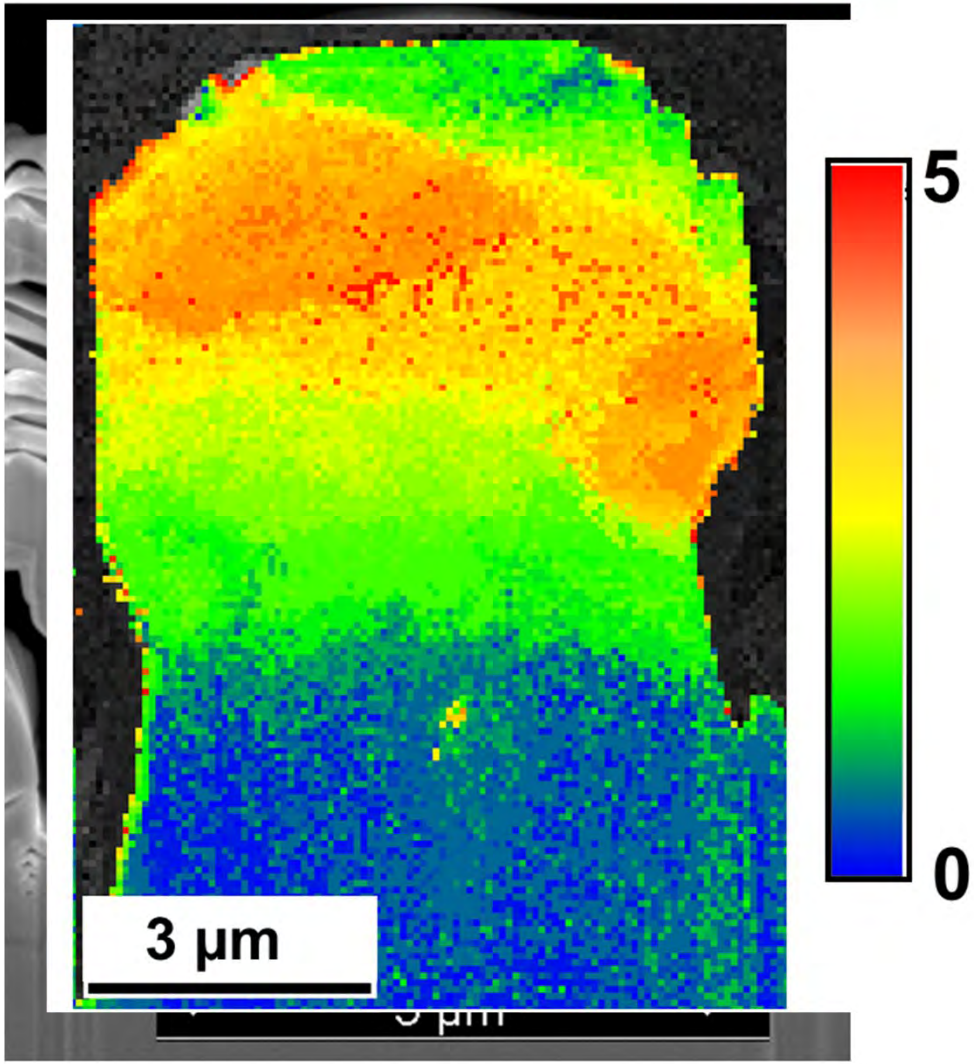
- Local
- Macroscopic bicrystals (Chalmers et al. from1937)
- Microscopic bicrystals (TEM)



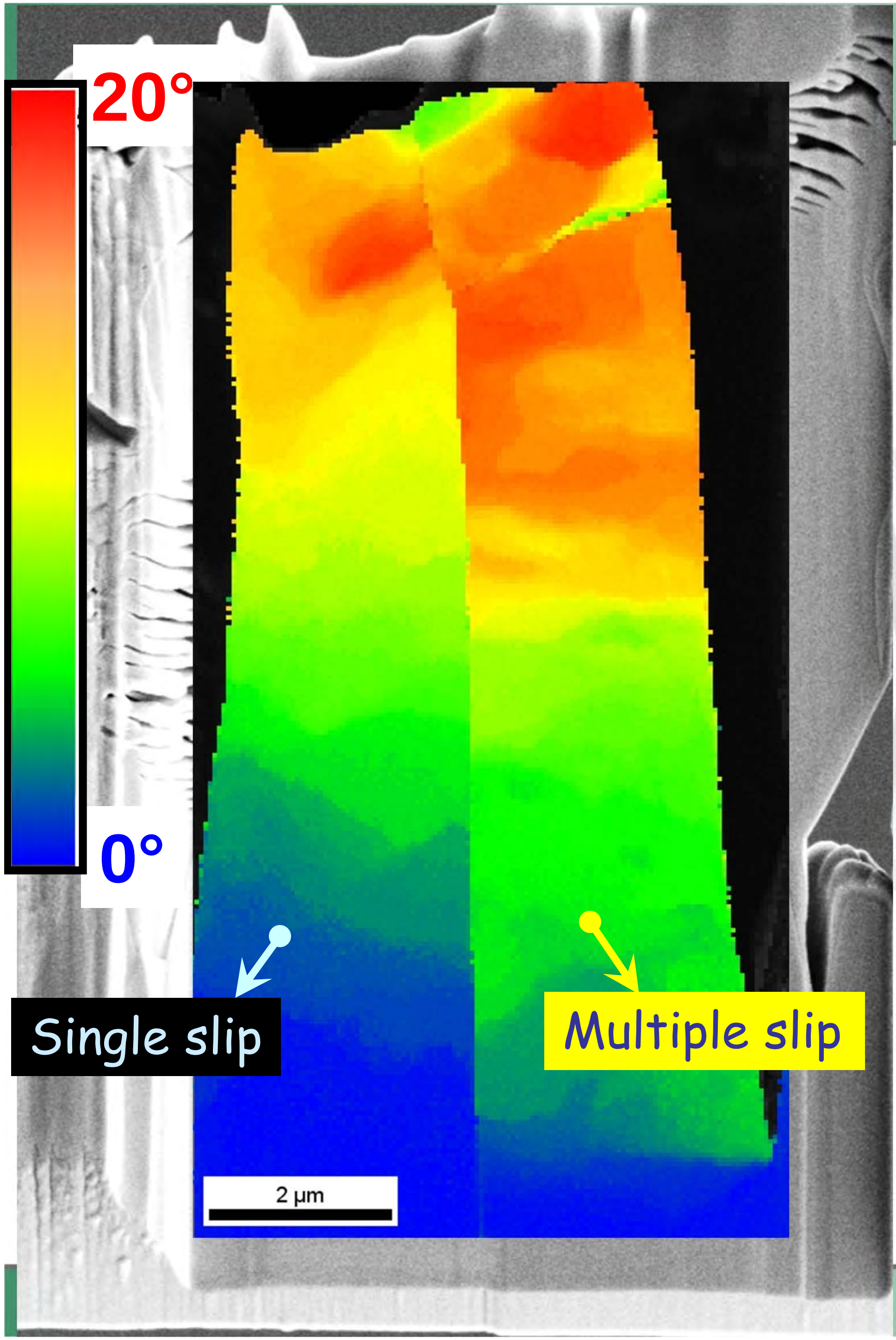
Z.Shen et al., Acta Metallurgica 36,1988

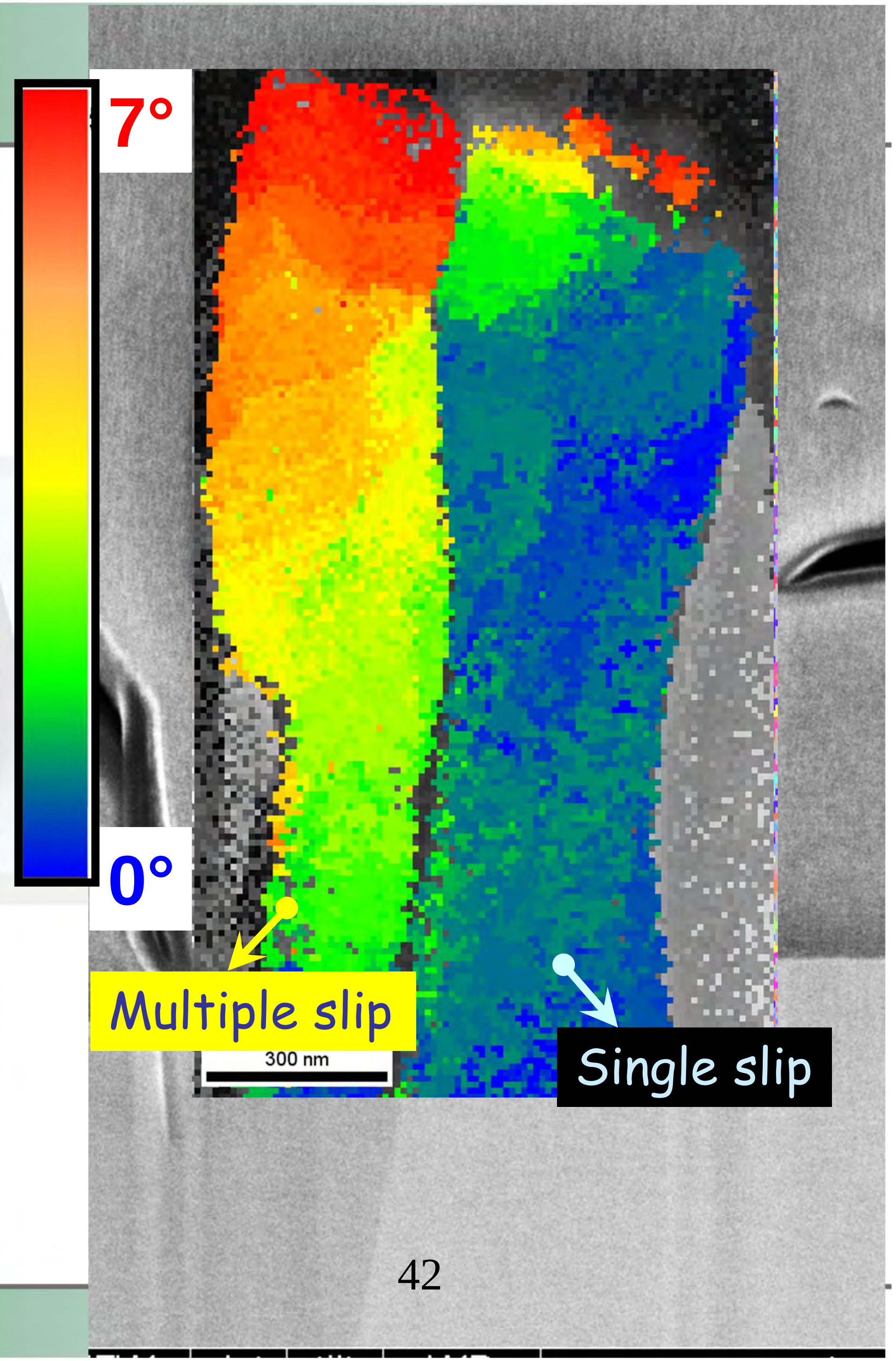


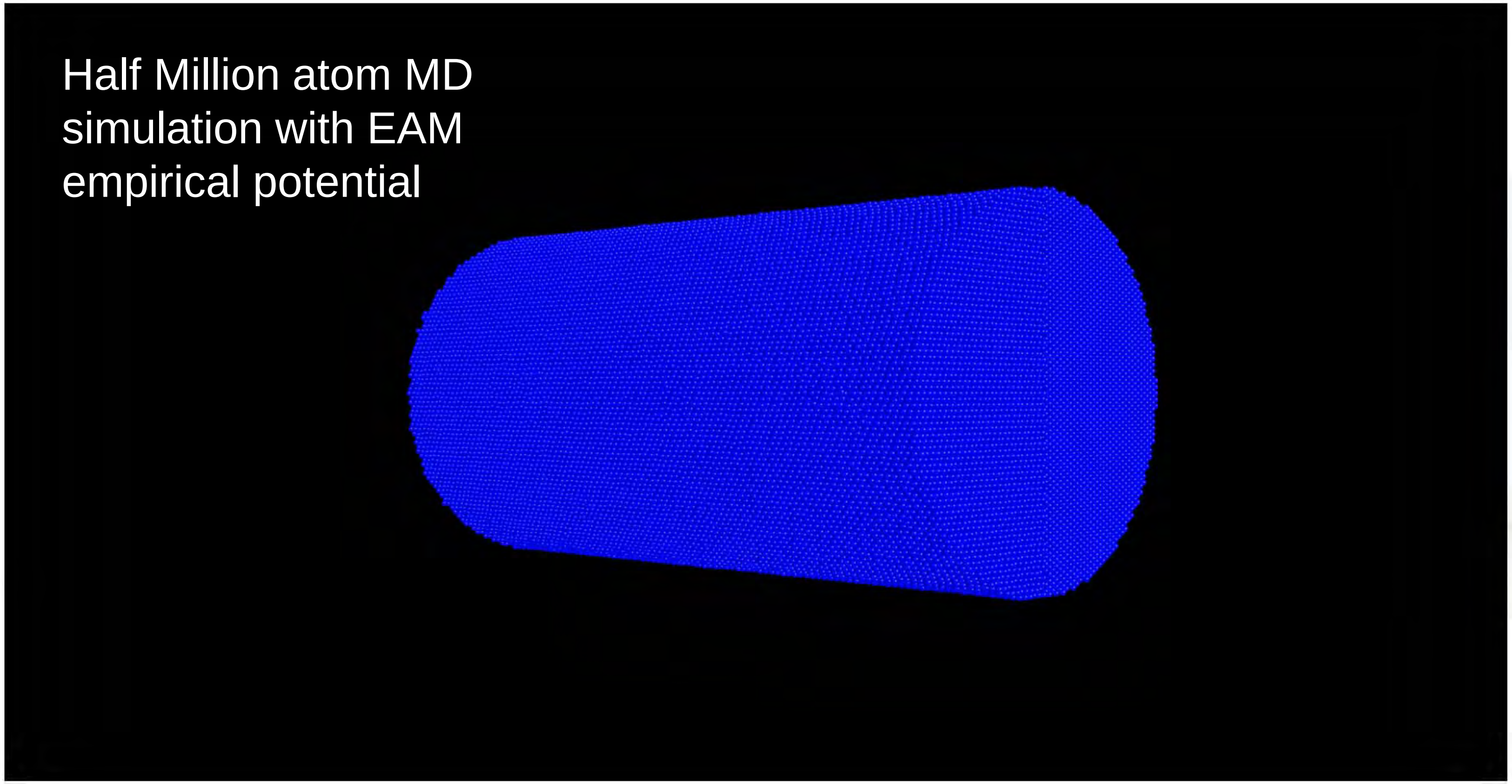
Single slip orientation
(-12 25 -9)

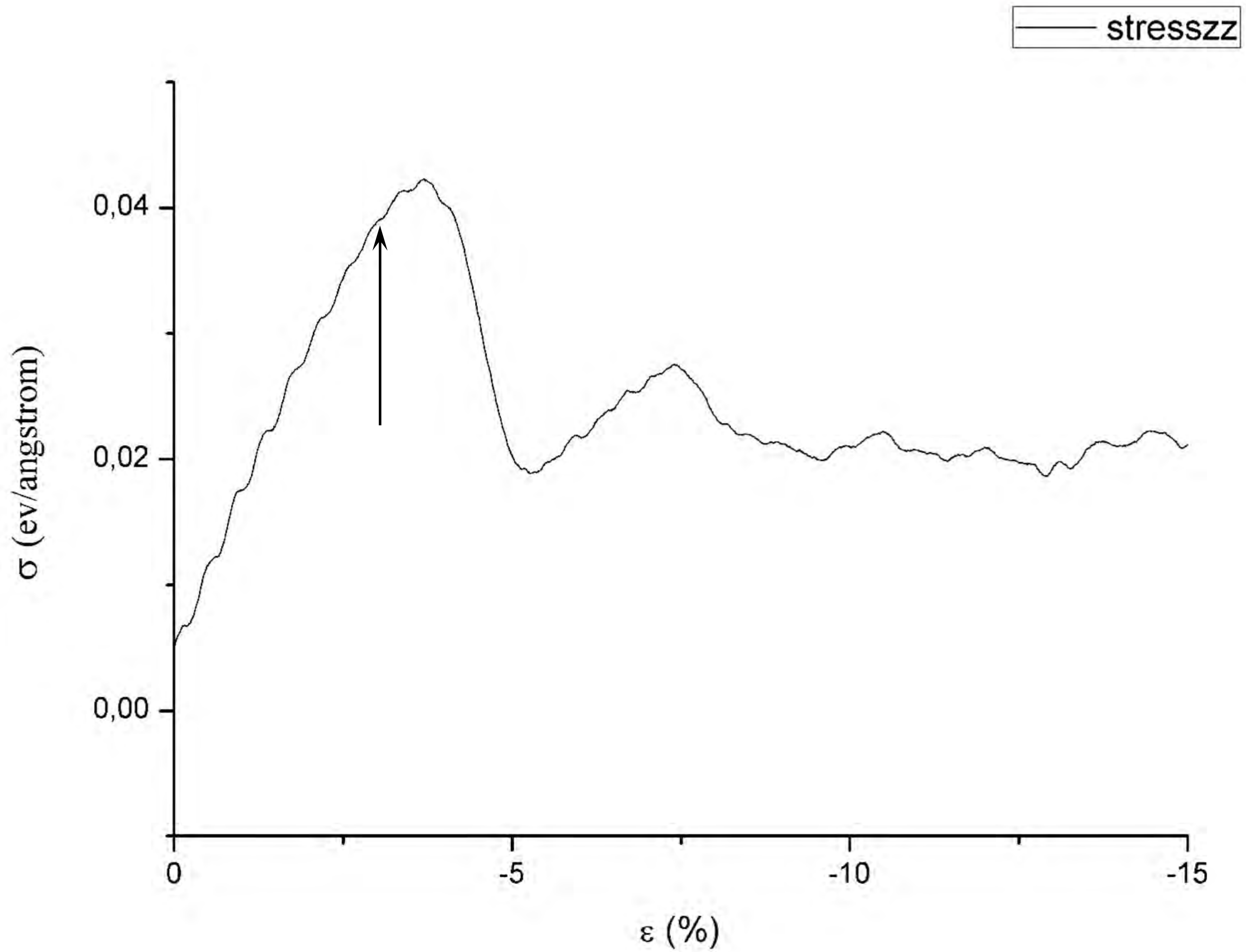


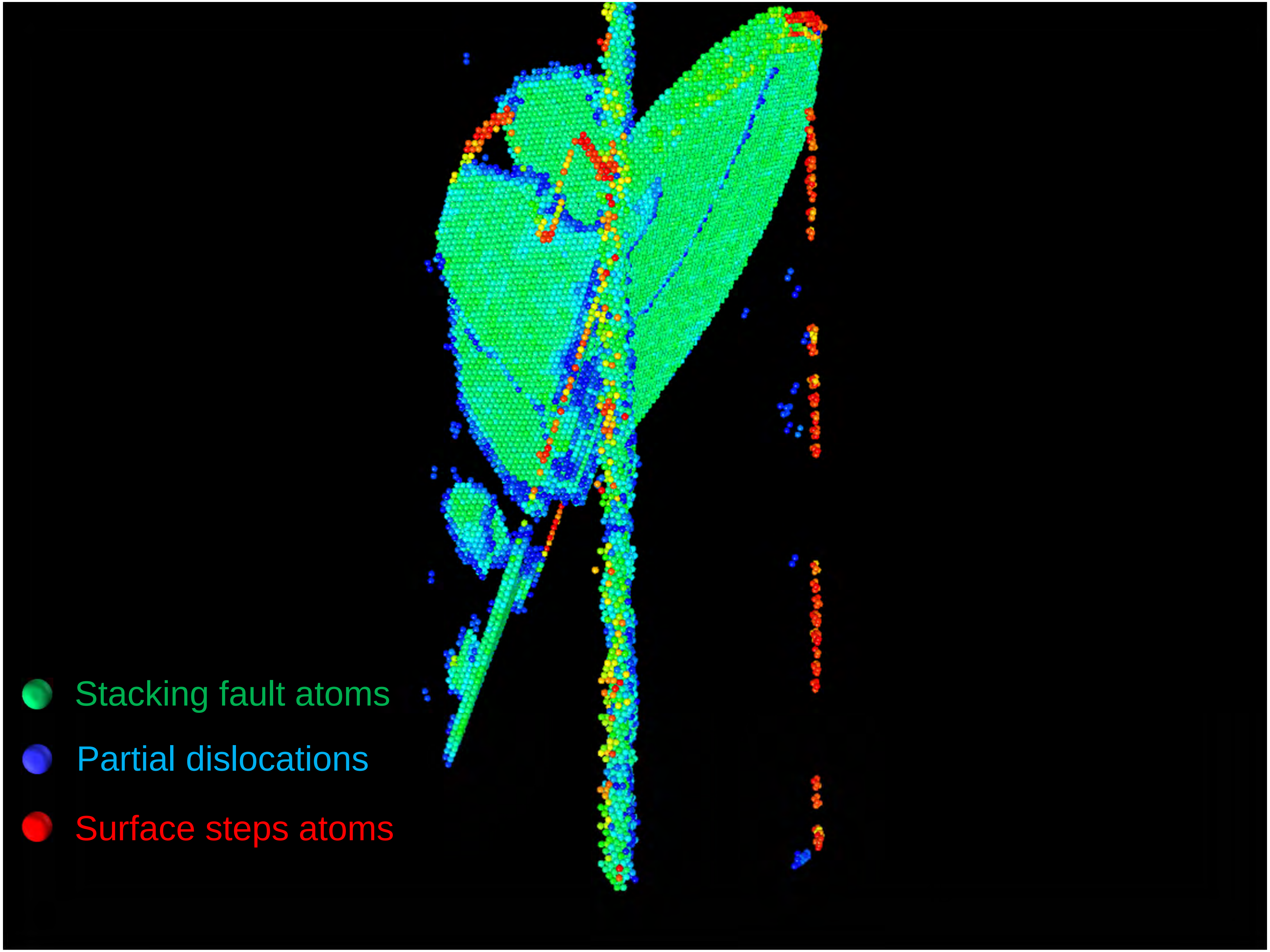
Double slip orientation
(11 1 18)

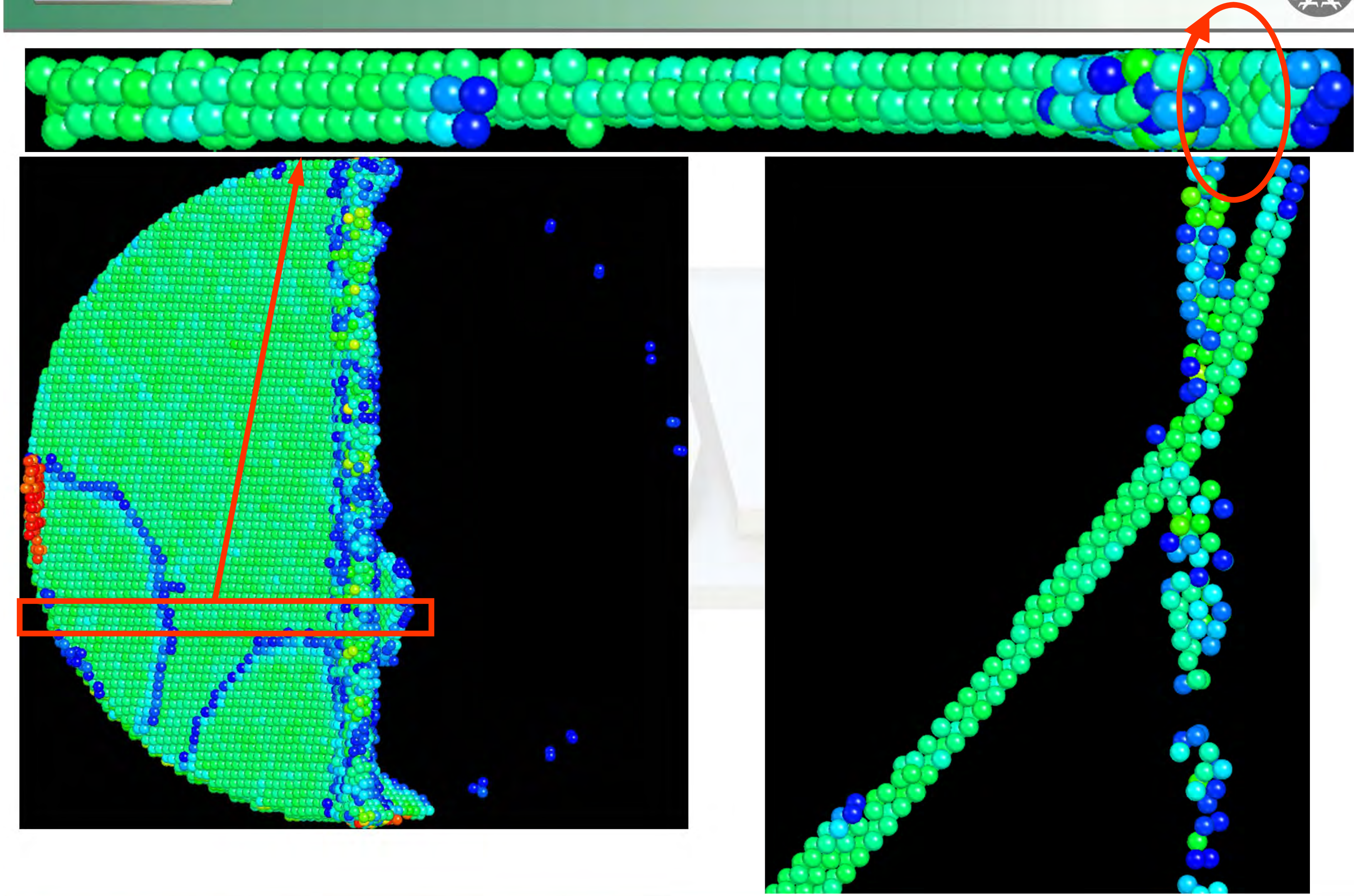

20°
0°
Single slip
Multiple slip
2 µm


7°
0°
Multiple slip
300 nm
Single slip

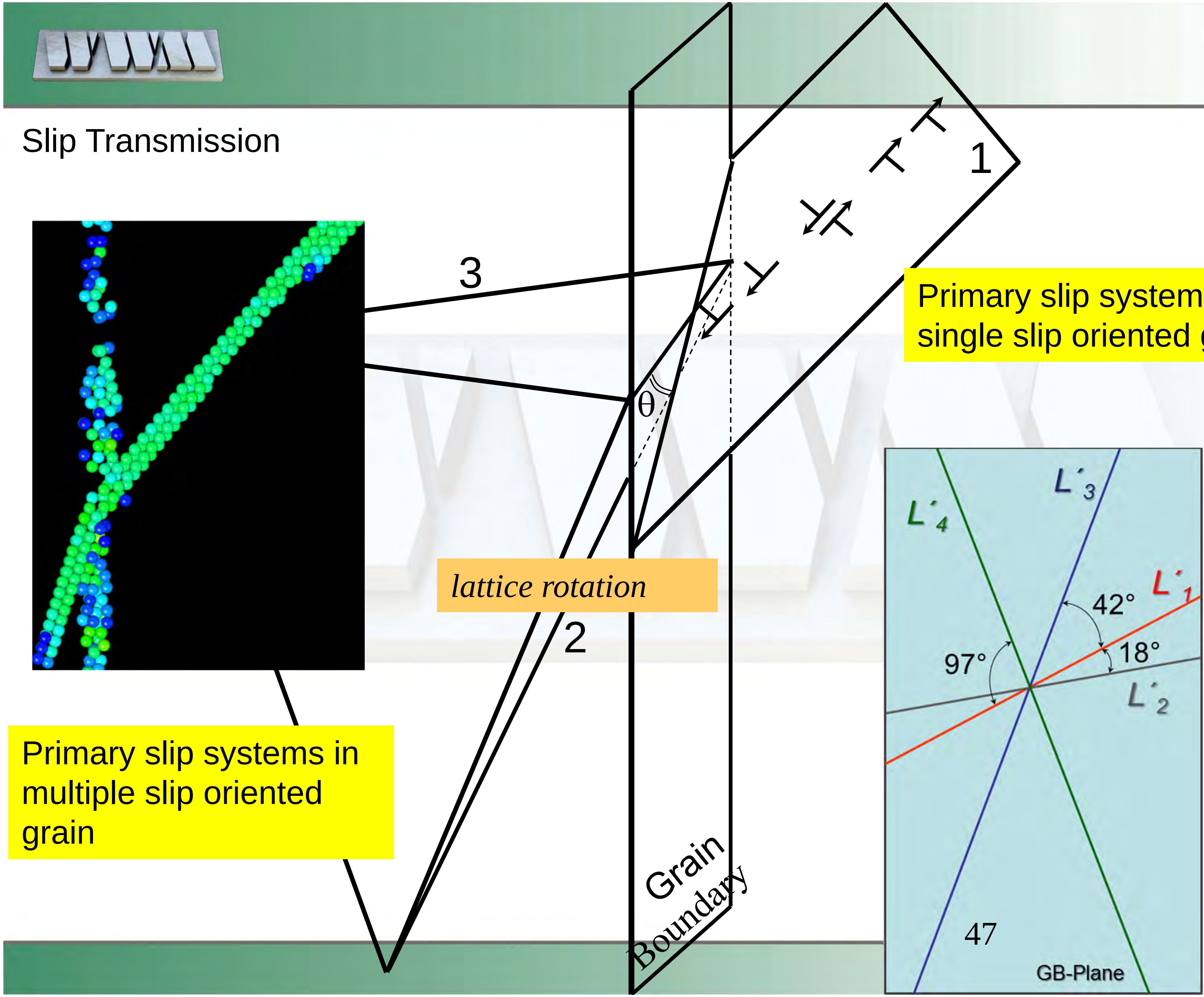

Half Million atom MD
simulation with EAM
empirical potential


stresszz
0,04
0,02
0,00
σ (ev/angstrom)
0
-5
-10
-15
ε (%)

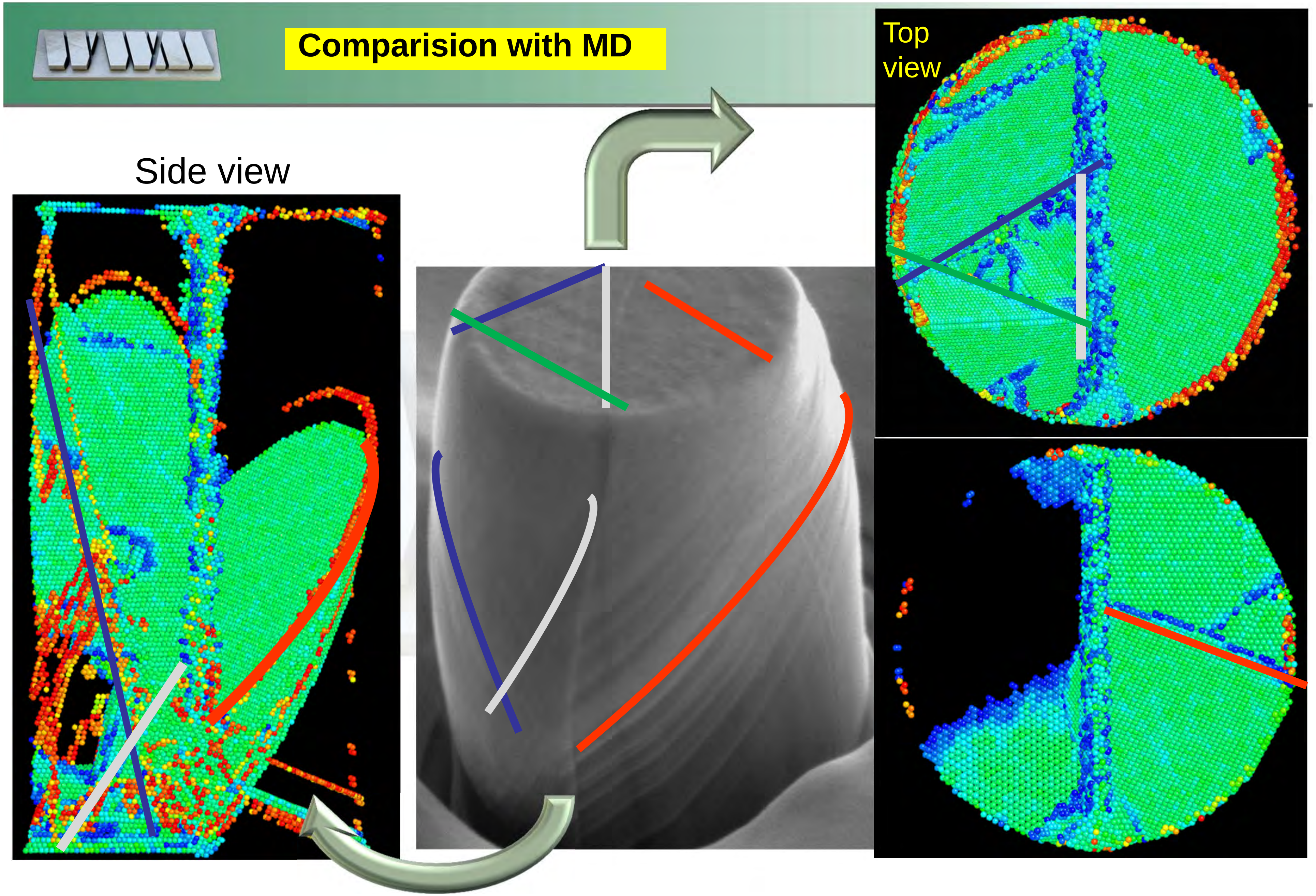

Stacking fault atoms
Partial dislocations
Surface steps atoms


90°

Slip Transmission

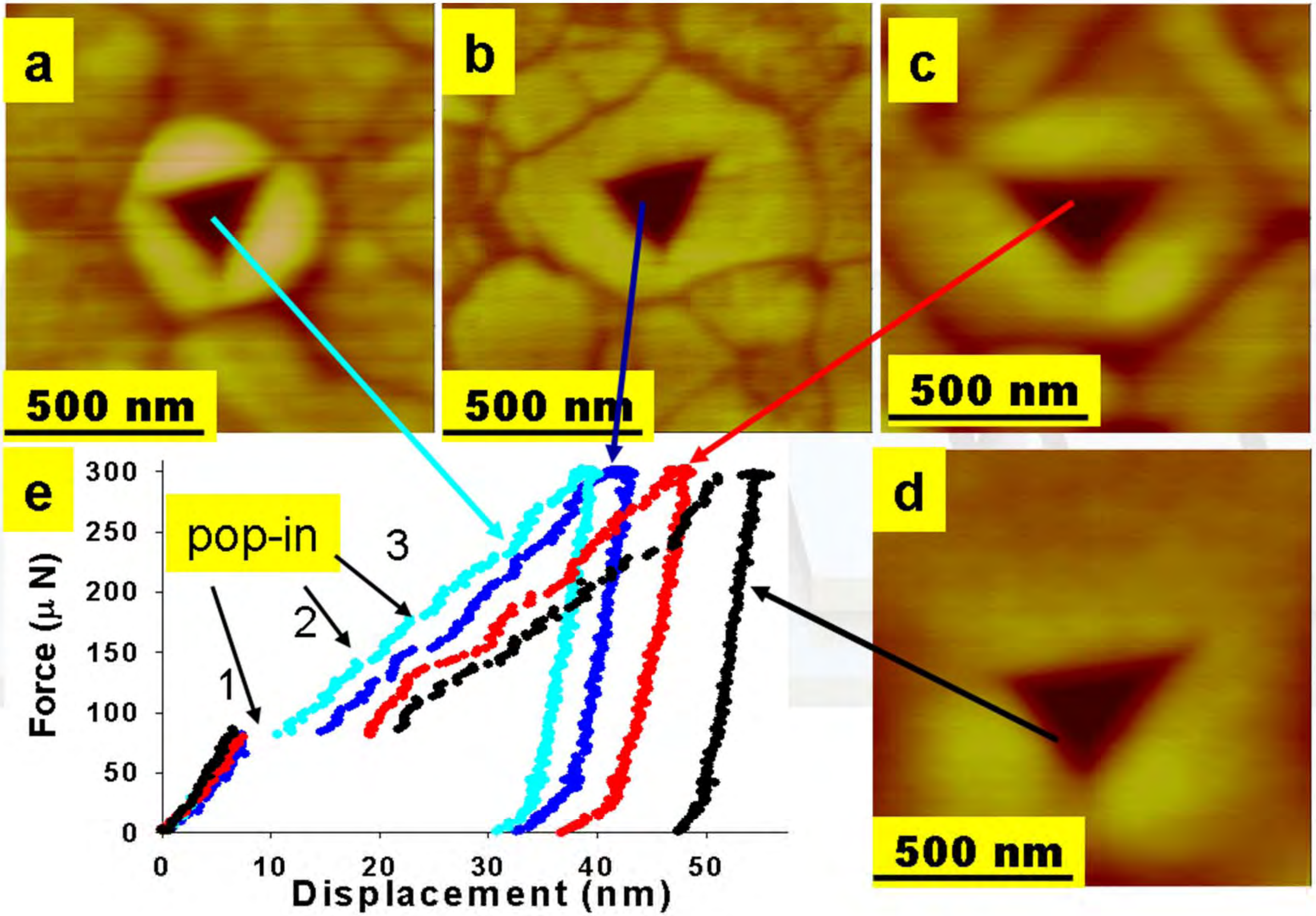


Comparision with MD



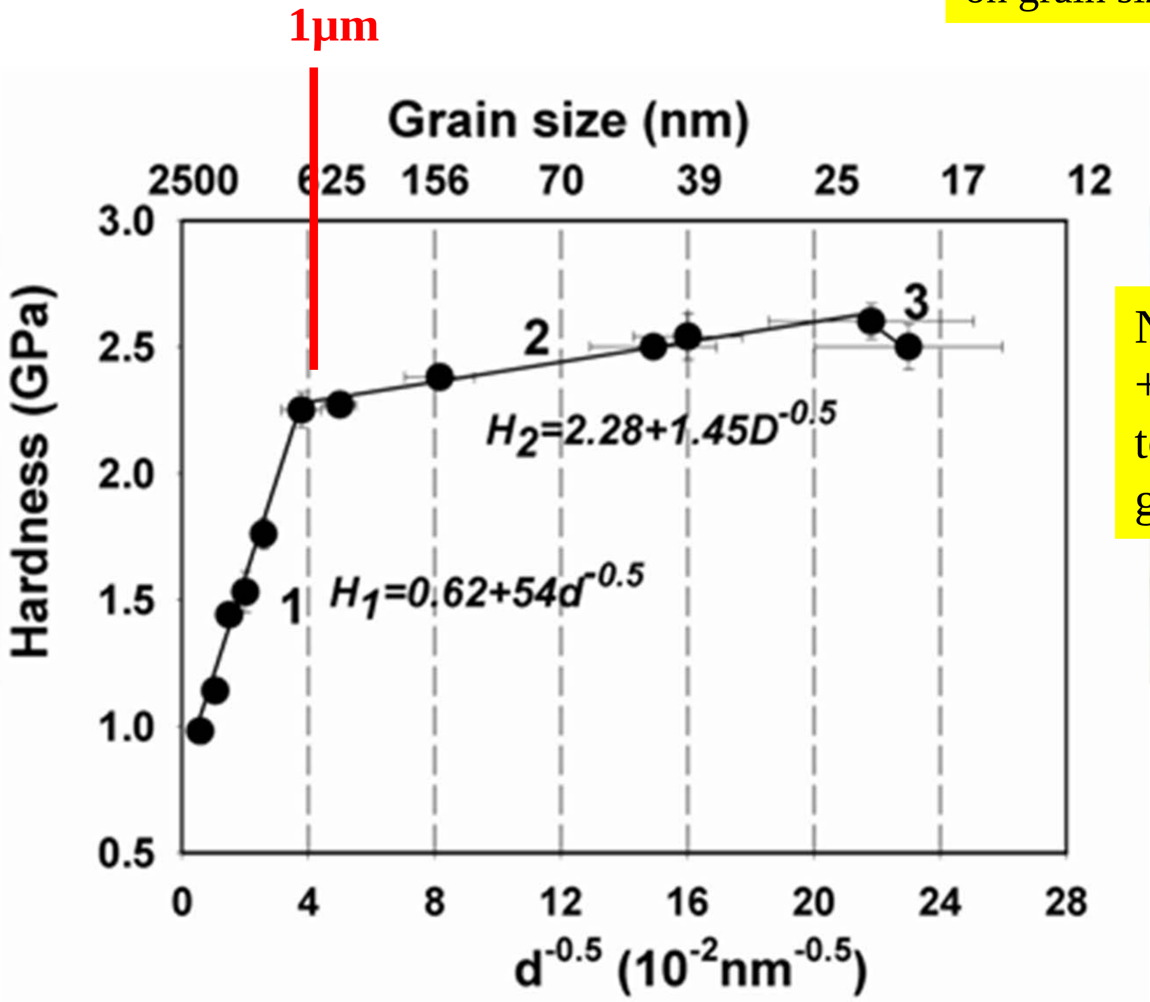
A. Barnoush and coworkers, in preparation

Nanoindentation in einzelnen Körnern



Deformation and size

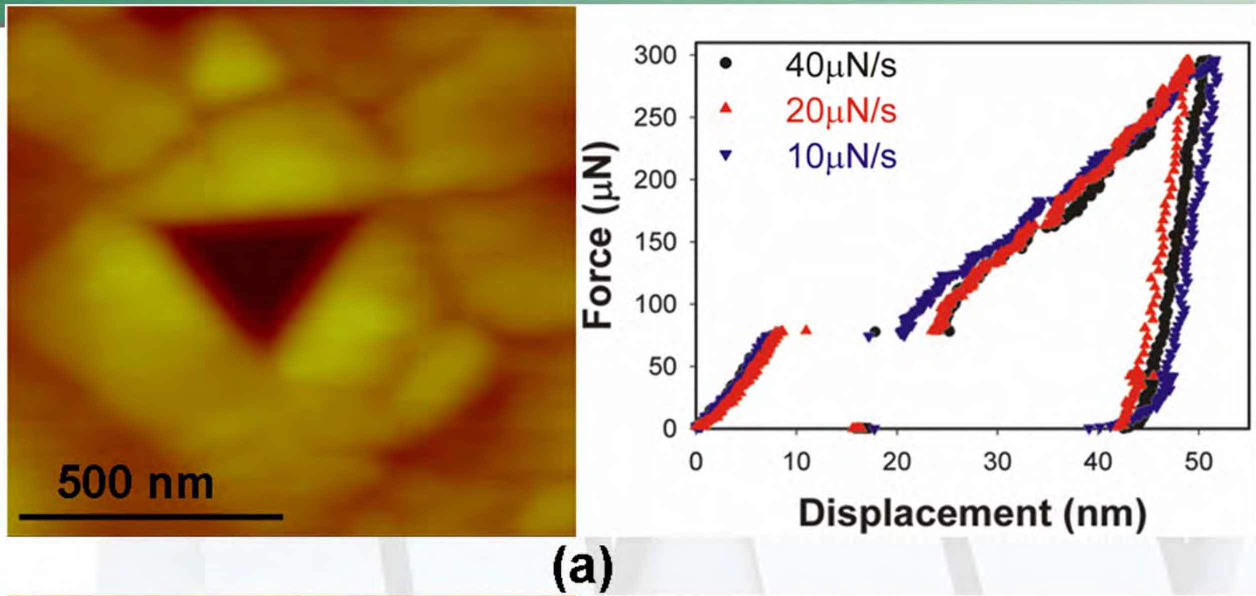
Constitutive equations depend on grain size



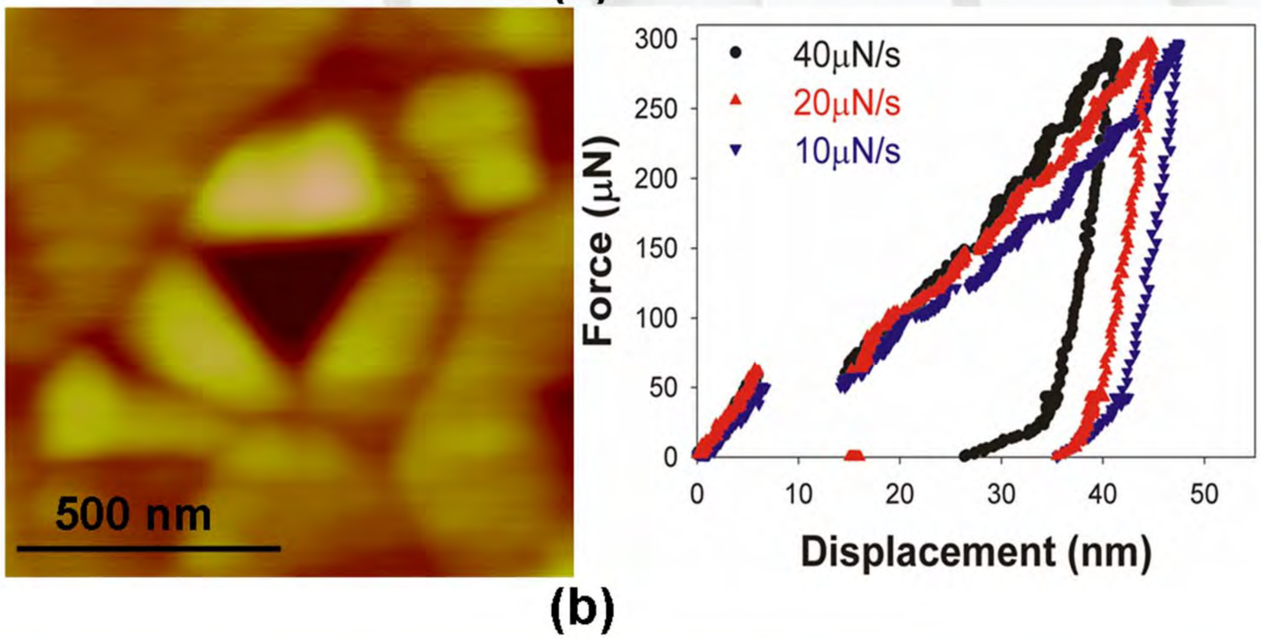
Ni, PED
+ heat treatment
to produce the different grain sizes

Bo Yang, Horst Vehoff: Acta Mater., 55(3):849–856, February 2007

Strain rate sensitivity



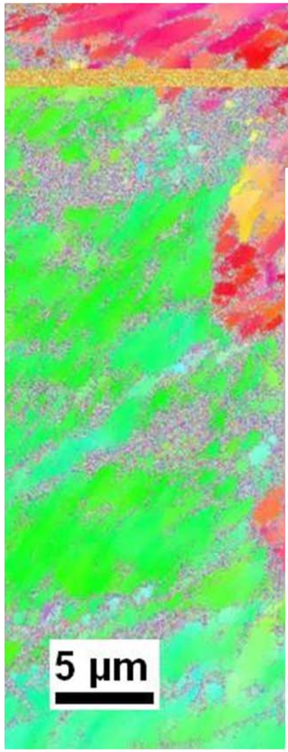
Konstante
Pop-in-Weite

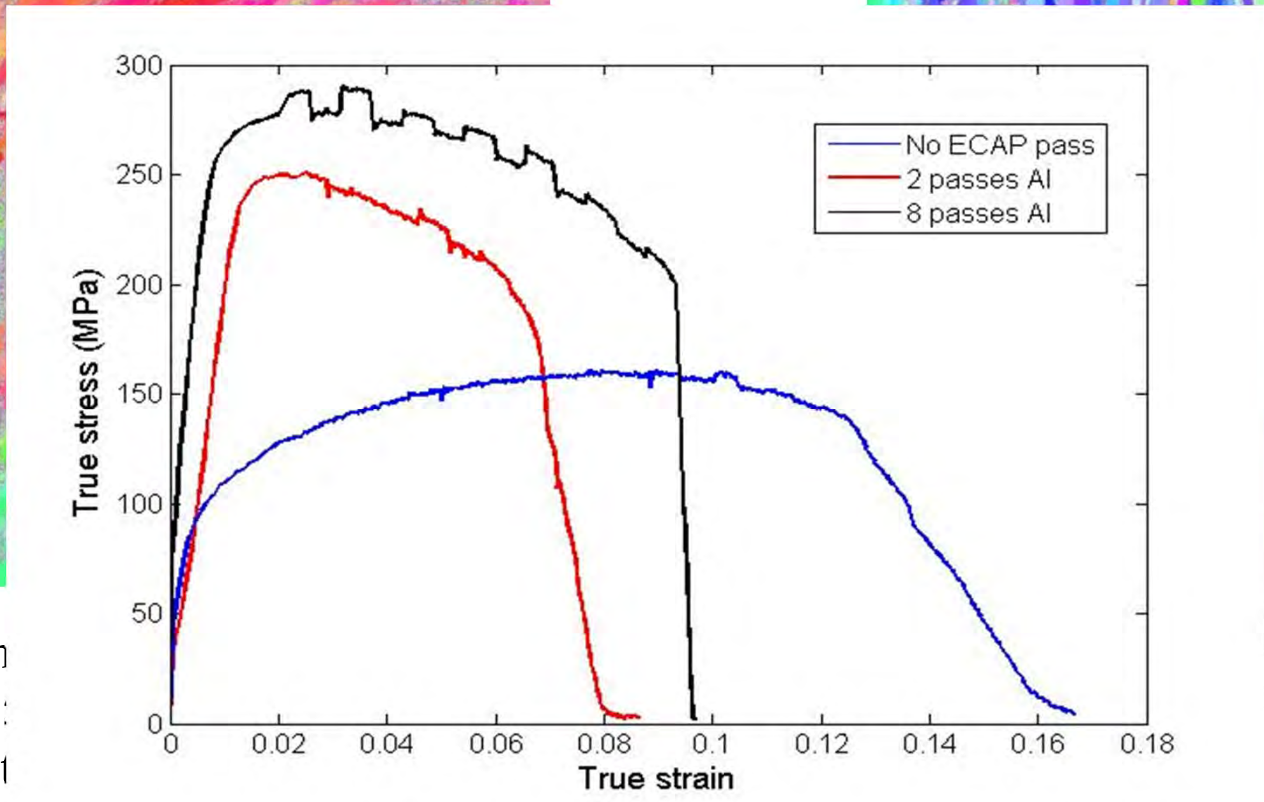


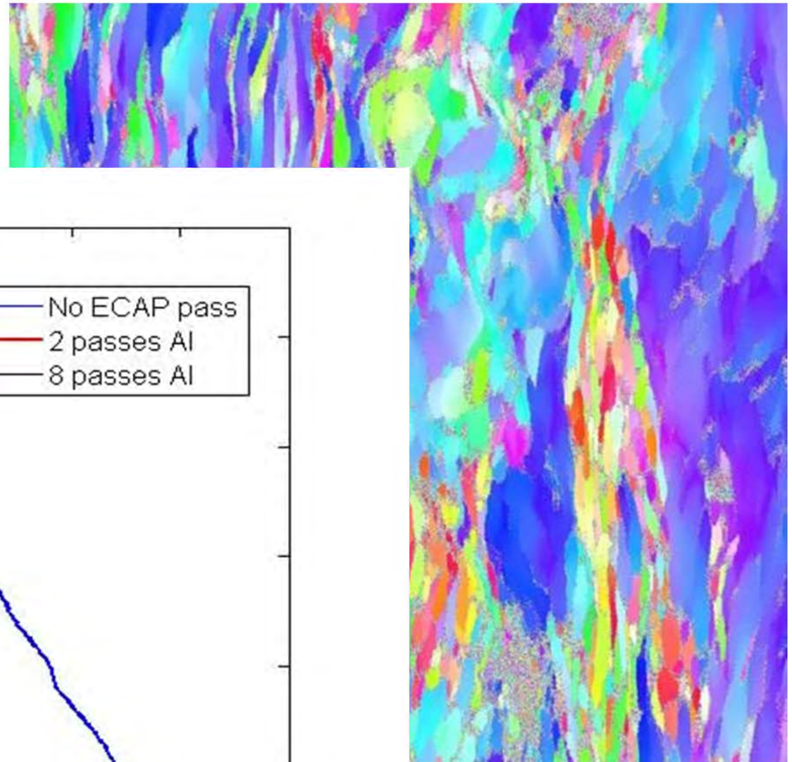
Pop-in-Weite begrenzt
durch Korngrenzen

-> dehnratenempfindlich

Global results: Al (AlMg0.5Si0.4) route C





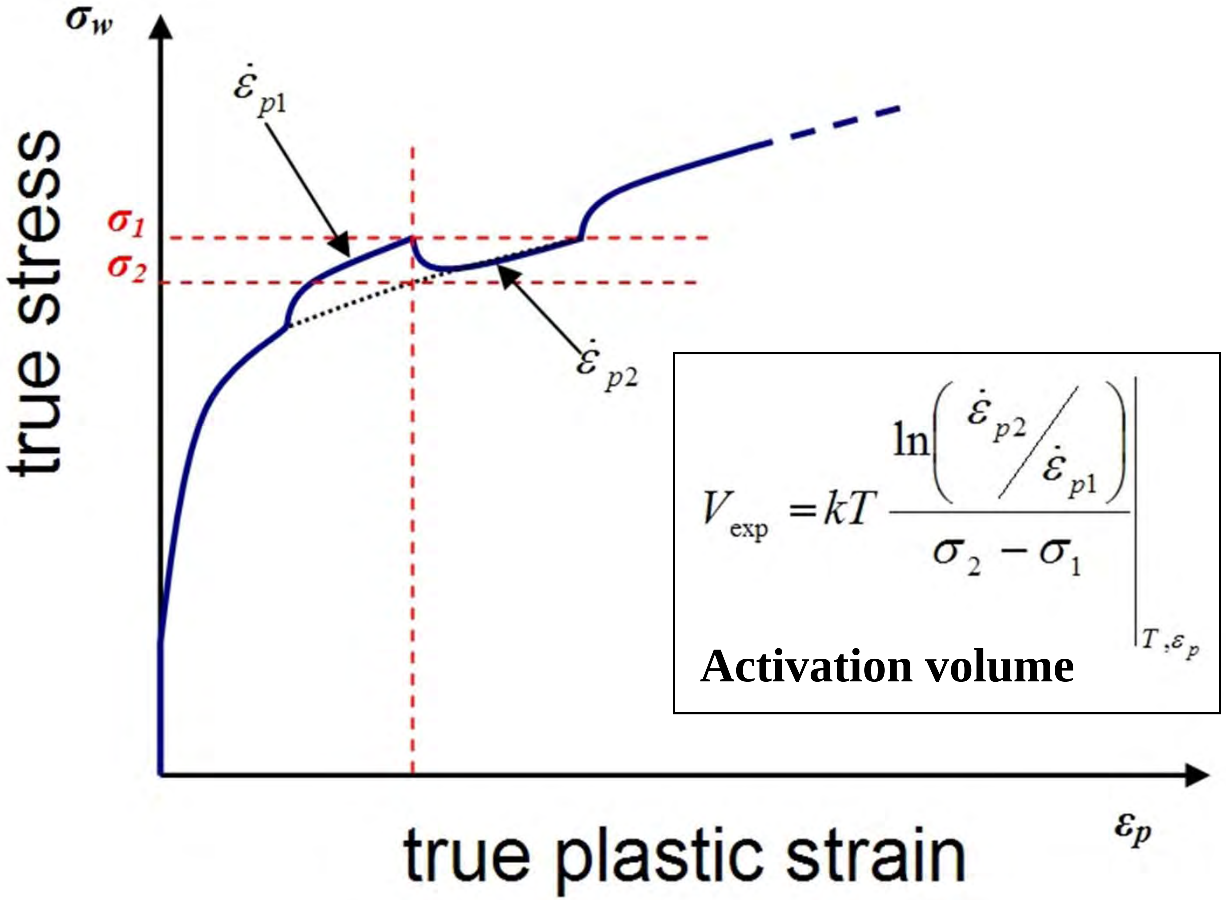


- 2 mean orien
- Grain sizes: :
- High amount

listribution
round 350 nm
HAGB (+20 %)

Strain rate sensitivity depends on grain size

Global Method: strain rate jump test



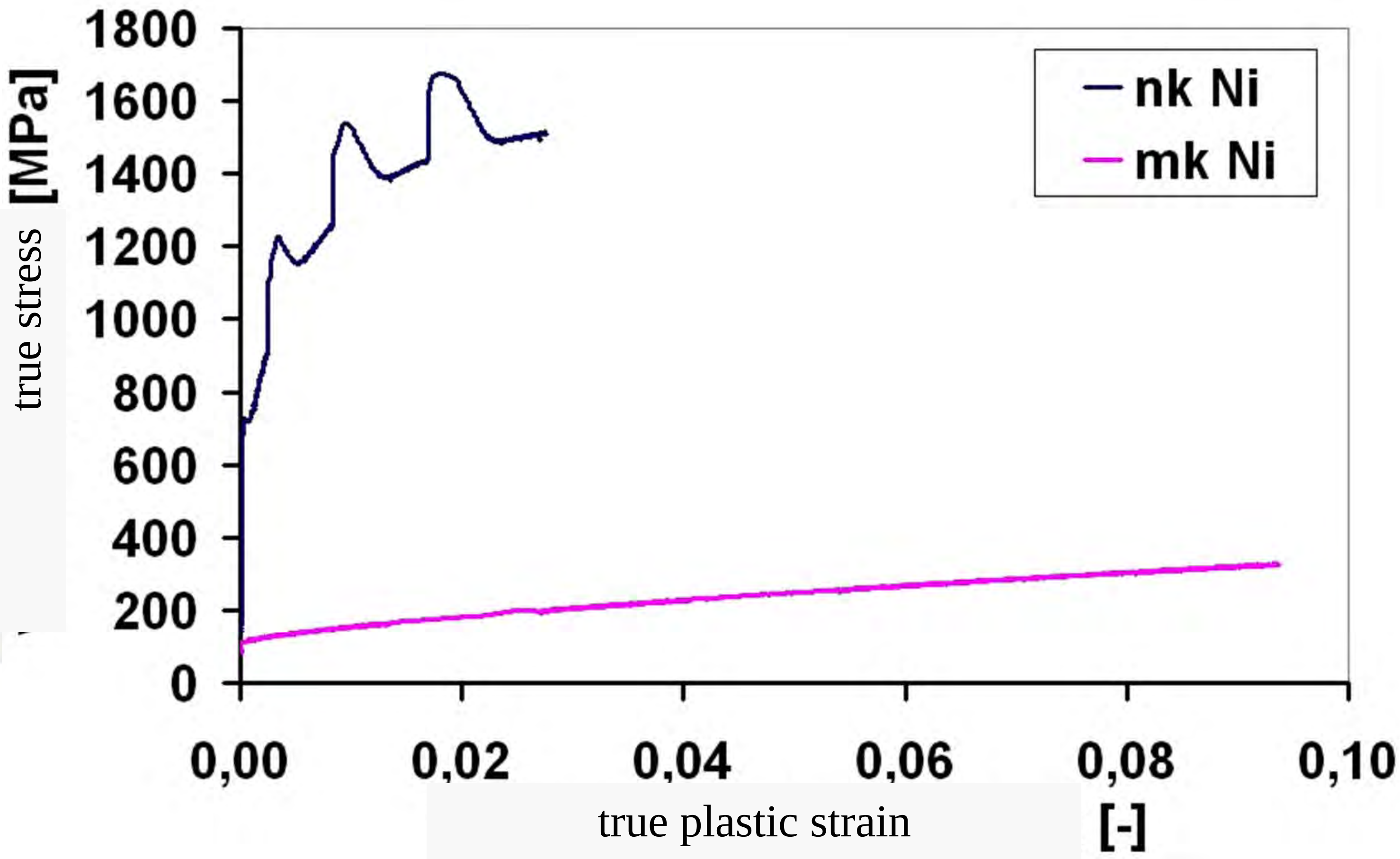
$$V_{\exp} = kT \left. \frac{\ln\left(\dot{\varepsilon}_{p2} / \dot{\varepsilon}_{p1} \right)}{\sigma_2 - \sigma_1} \right|_{T,\varepsilon_p}$$

Activation volume

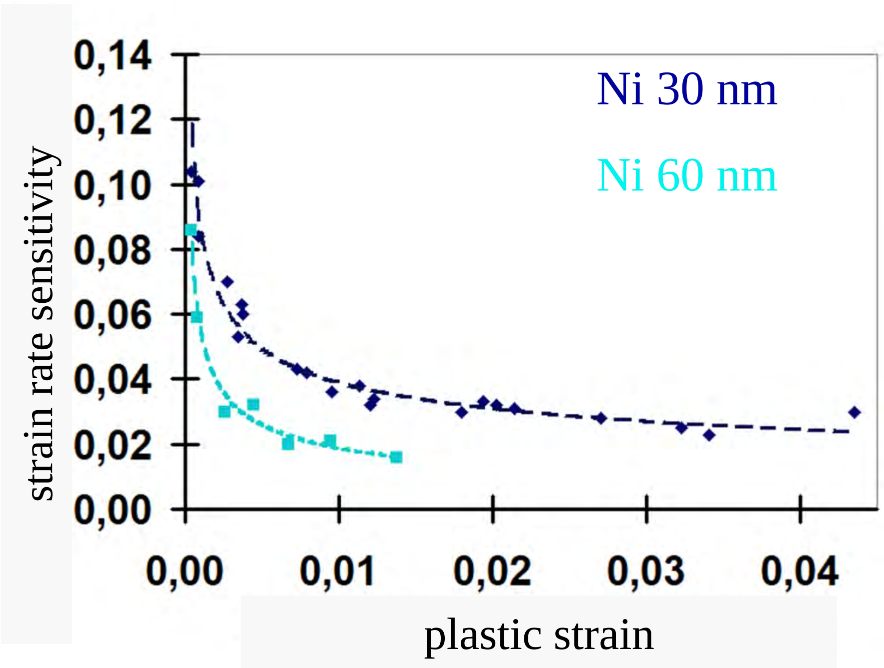
$$m = \left. \frac{\partial \ln \sigma}{\partial \ln \dot{\varepsilon}_P} \right|_{T,\varepsilon_P}$$

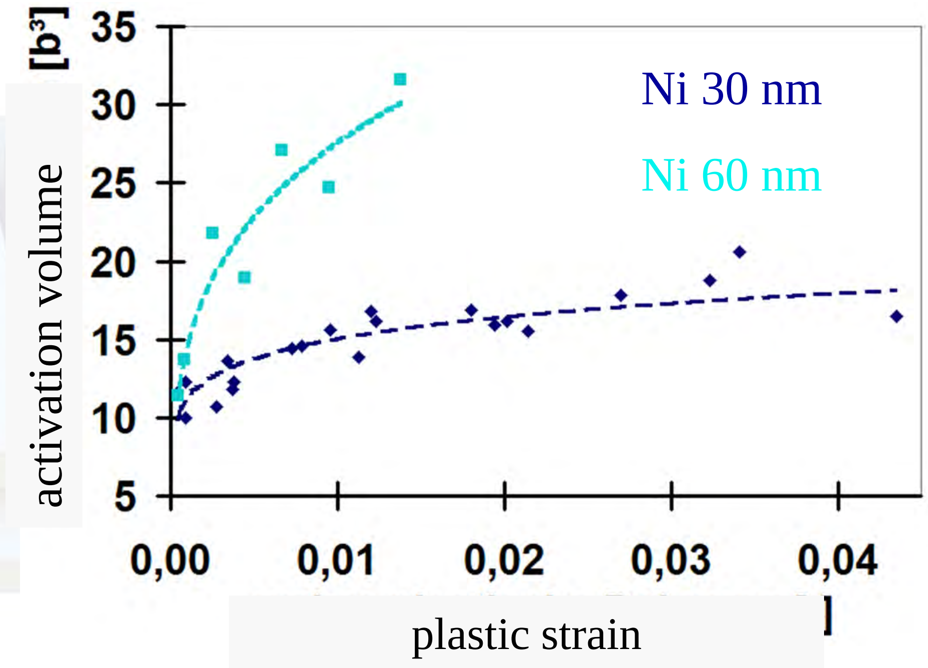
Strain rate sensitivity

Strain rate sensitivity of Nk and mk Ni



Influence of grain size

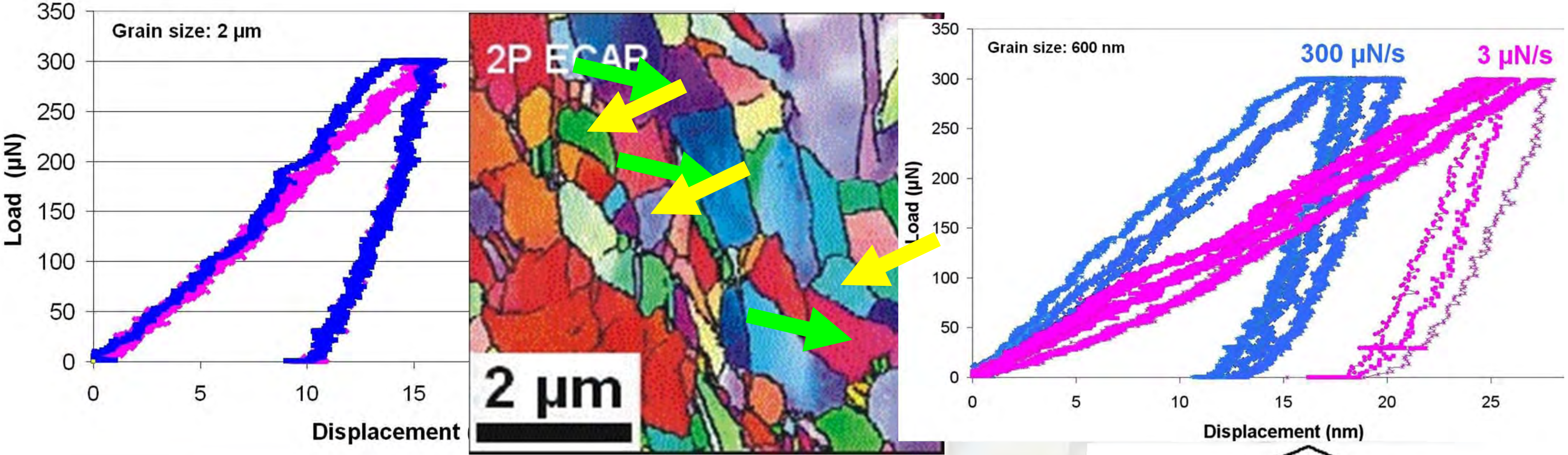




Activation volume decreases with grain size

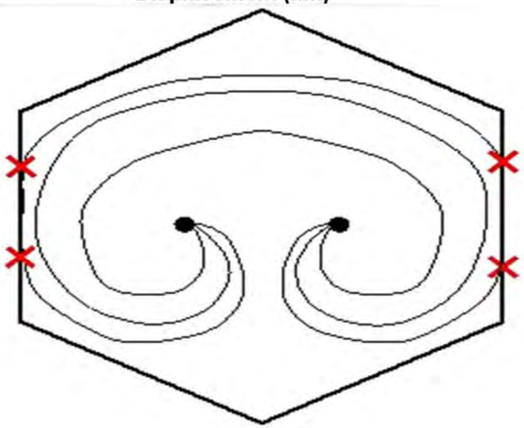
Indentation of single grains

ECAP-Ni: complex microstructure

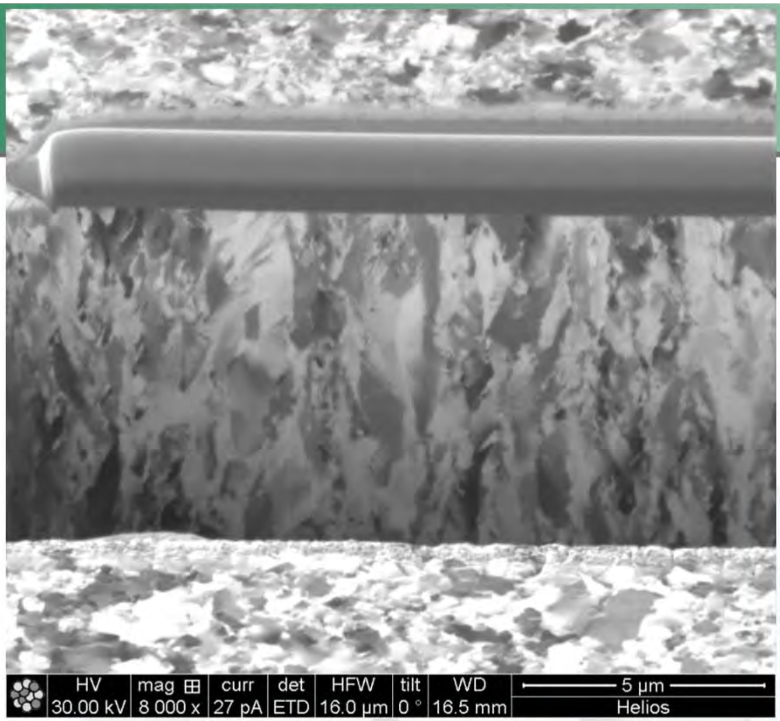


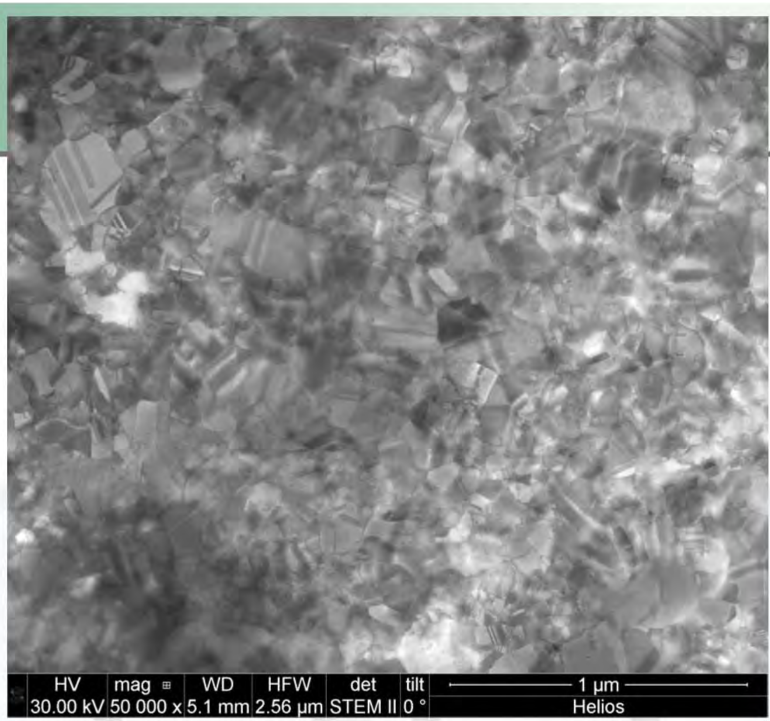
Direct dislocation grain boundary-interaction

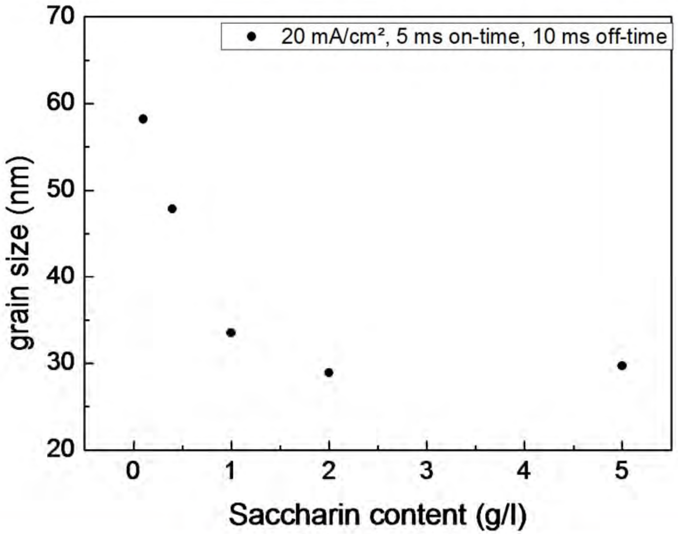
Strain rate sensitivity

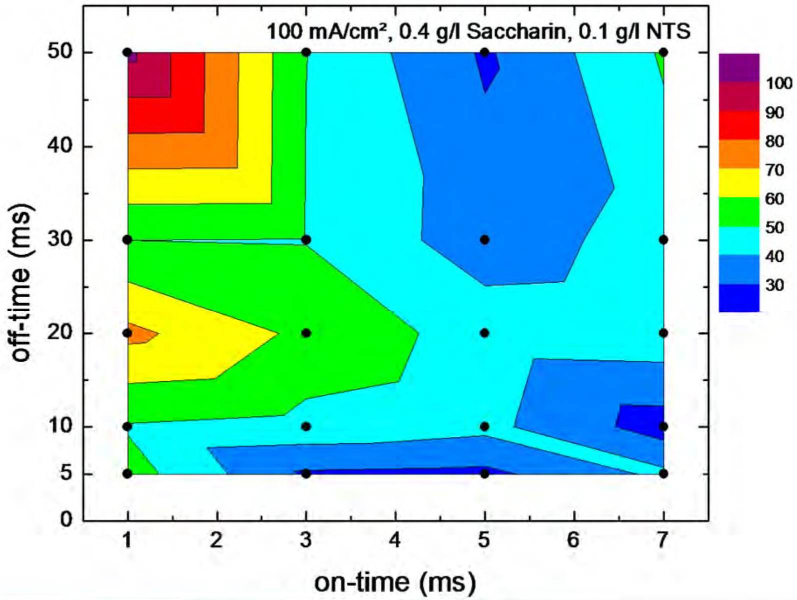


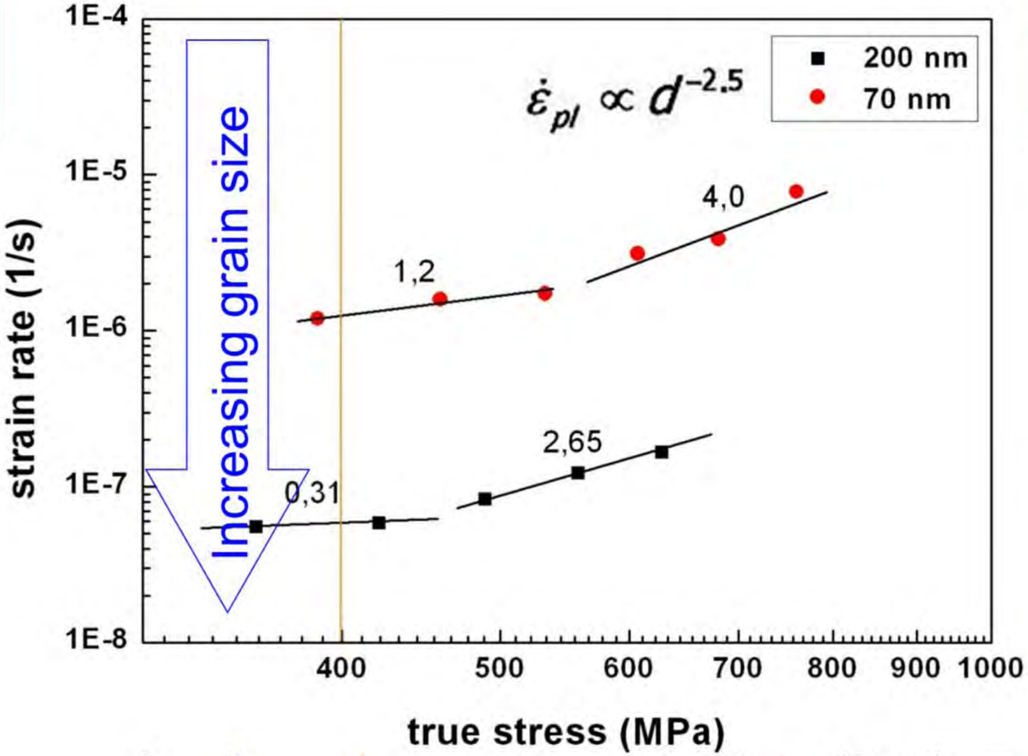
Horst Vehoff, Delphine Lemaire, Kerstin Schüler, Thomas Waschkies, and Bo Yang. International Journal of Materials Research, 98(4):259–268, April 2007.


HV 30.00 kV
mag 8 000 x
curr 27 pA
det ETD
HFW 16.0 µm
tilt 0 °
WD 16.5 mm
5 µm
Helios

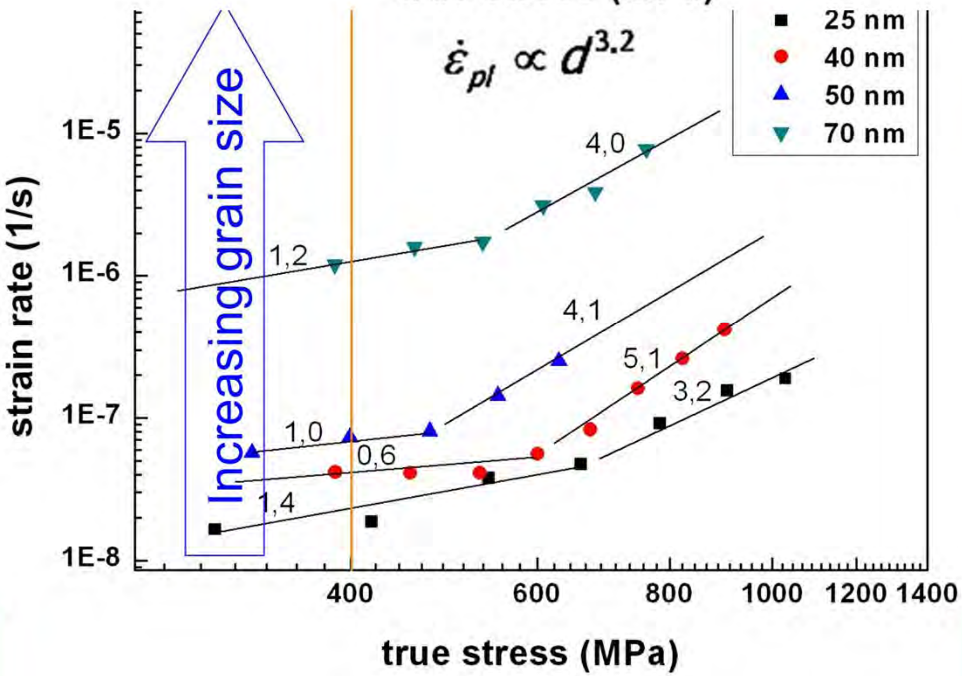

HV 30.00 kV
mag 50 000 x
WD 5.1 mm
HFW 2.56 µm
det STEM II
tilt 0 °
1 µm
Helios


20 mA/cm², 5 ms on-time, 10 ms off-time
grain size (nm)
Saccharin content (g/l)


100 mA/cm², 0.4 g/l Saccharin, 0.1 g/l NTS
off-time (ms)
on-time (ms)
100
90
80
70
60
50
40
30



Clean boundaries



Segregated boundaries