Anwendungspotential neuer Methoden der Mikroanalyse zur Bestimmung lokaler mechanischer Eigenschaften, der chemischen Zusammensetzung und der Mikrostruktur im Rasterelektronenmikroskop

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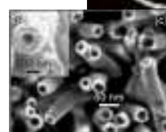
Hanau, April 2012

Materials Science & Technology

Laboratory for Mechanics of Materials and Nanostructures

materials for the
small world

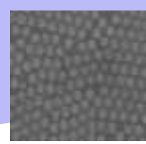




synthesis

of metals and semiconductors

Ebeam & UV-, FIB Litho, natural litho
Electroplating (LIGA),
ALD, PVD
Ion & ebeam deposition

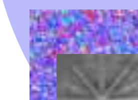
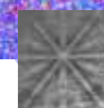
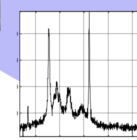
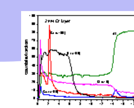



microstructure

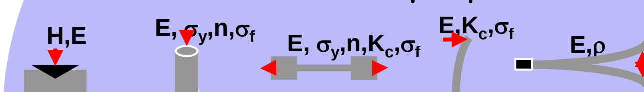
amorphous, nanocrystalline
monocrystalline

SEM, TEM, AFM, EBSD, EDX
Raman, GDOES, FIBSIMS

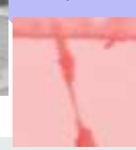
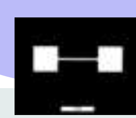
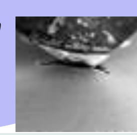
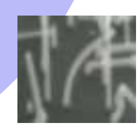
properties

mechanical material properties



and system properties like
residual stress/adhesion,
load bearing capacity

micro- & nanomechanical
testing

outline

Introduction to scanning electron microscopy

FIBSIMS

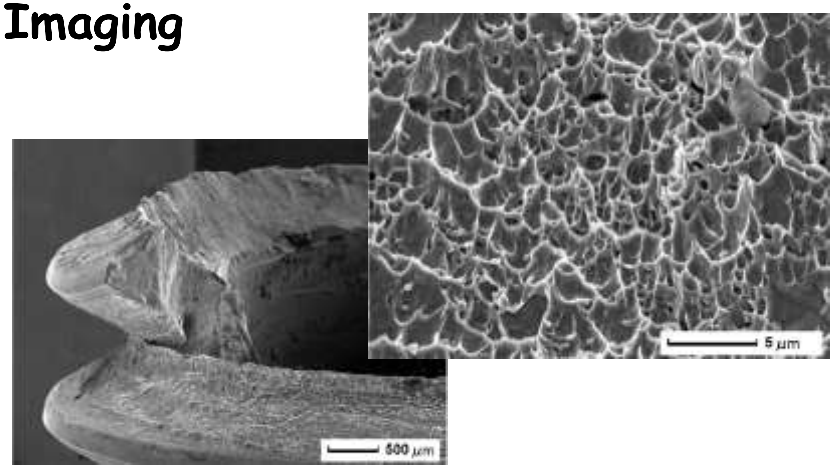
AFM in SEM

stress & strain from EBSD

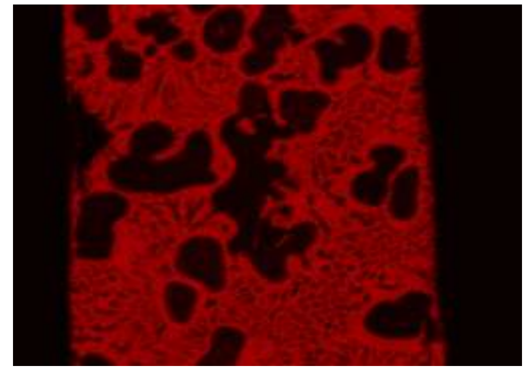
in-situ micromechanical testing

Applications of Scanning Electron Microscopy

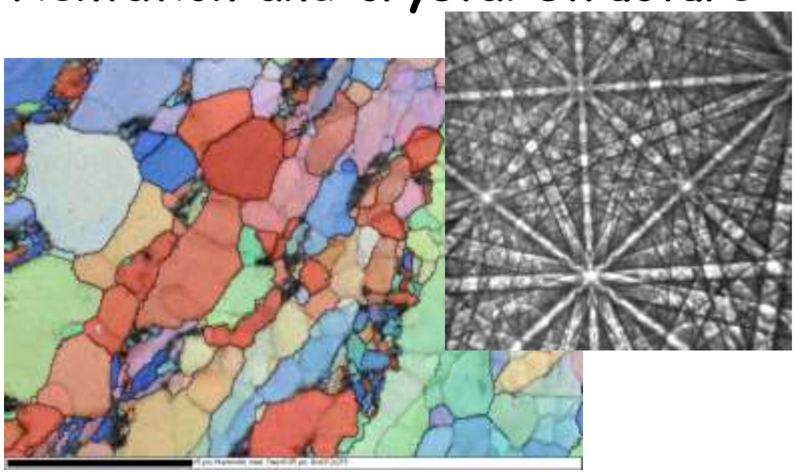
Imaging



Chemical composition



Orientation and crystal structure



Structuring

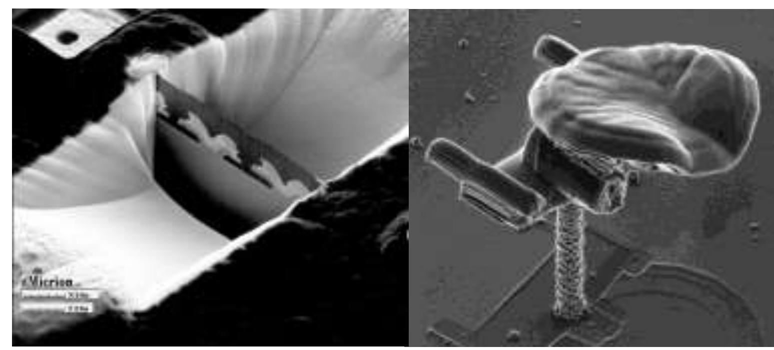
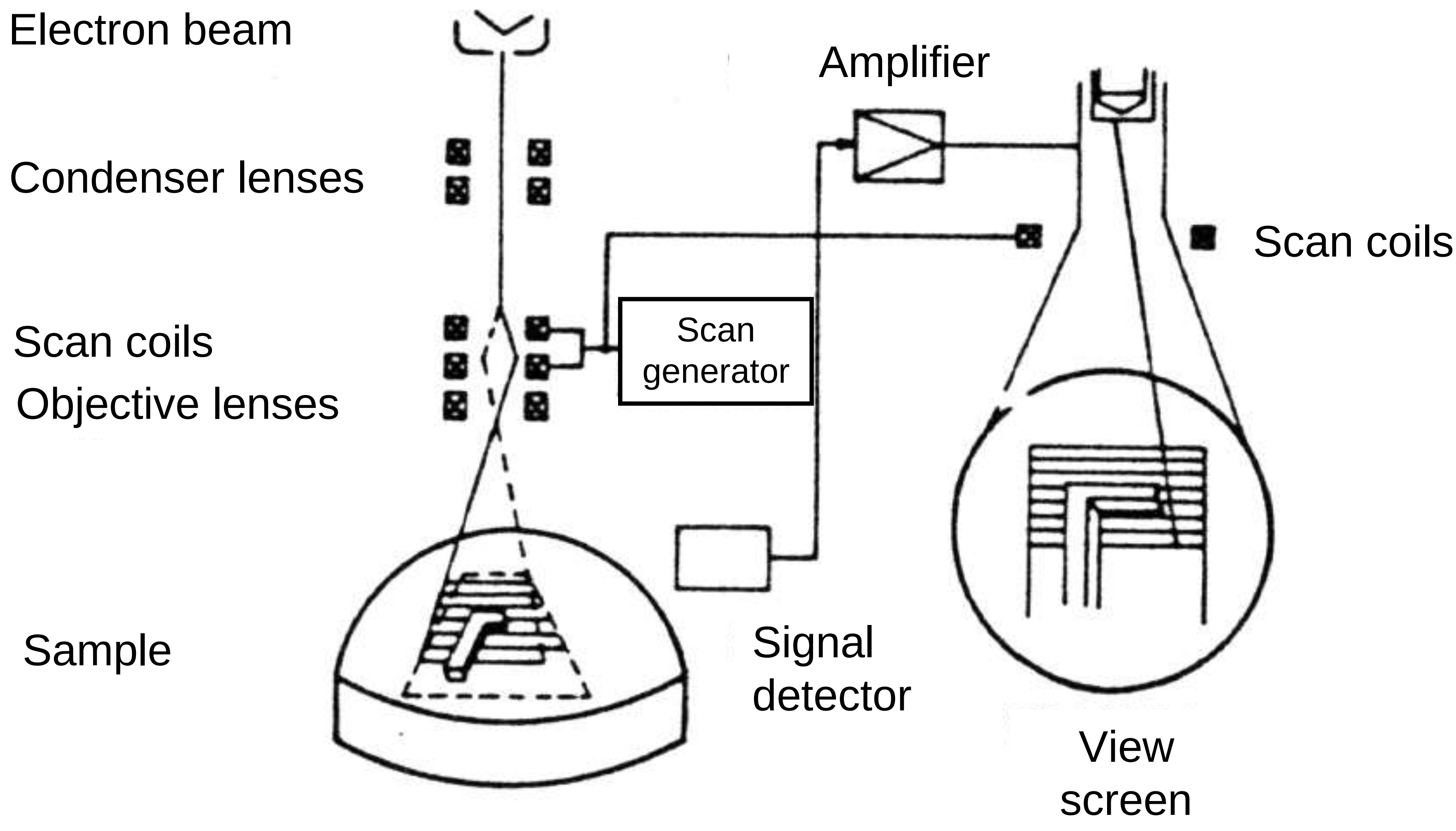
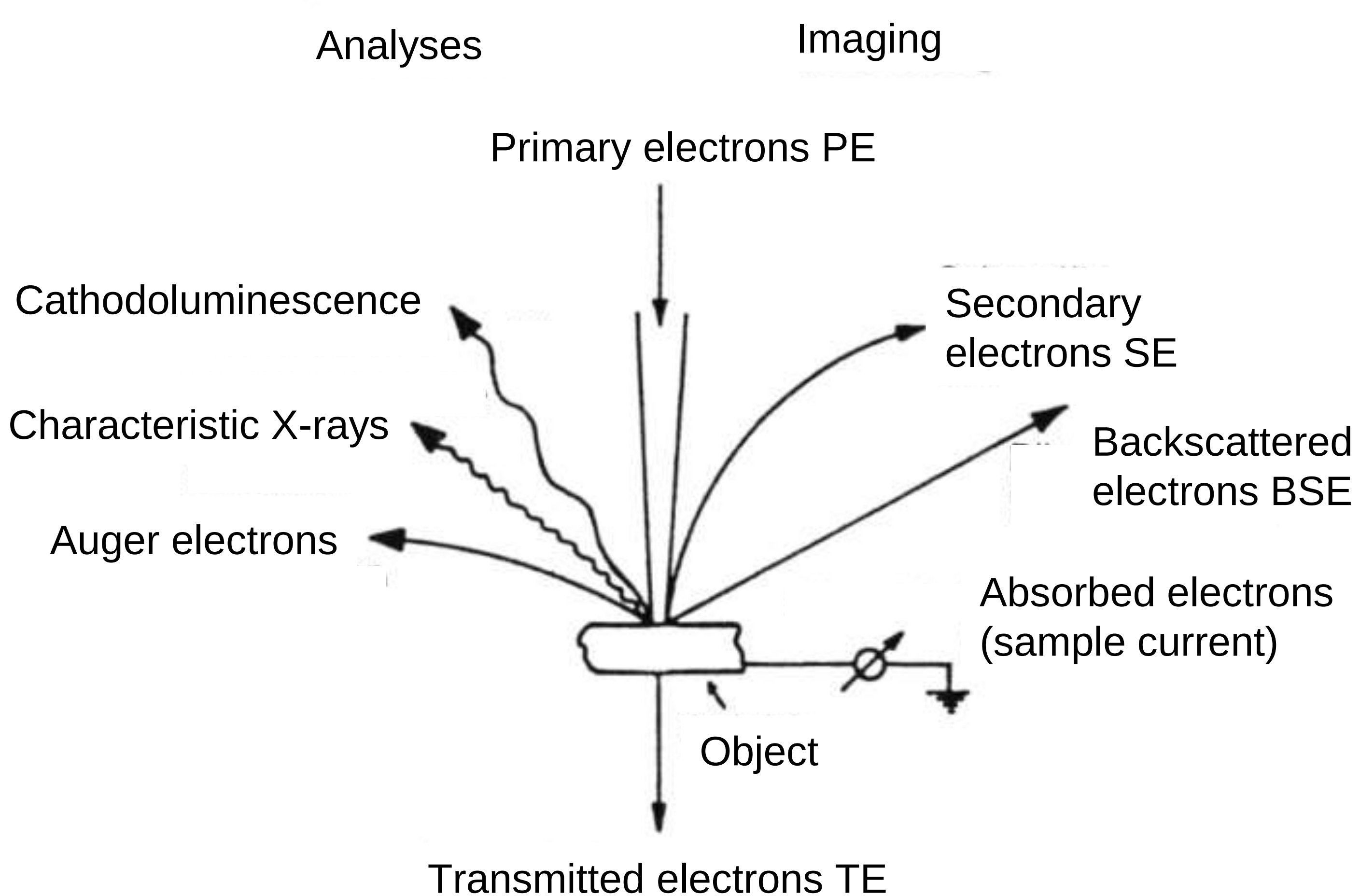


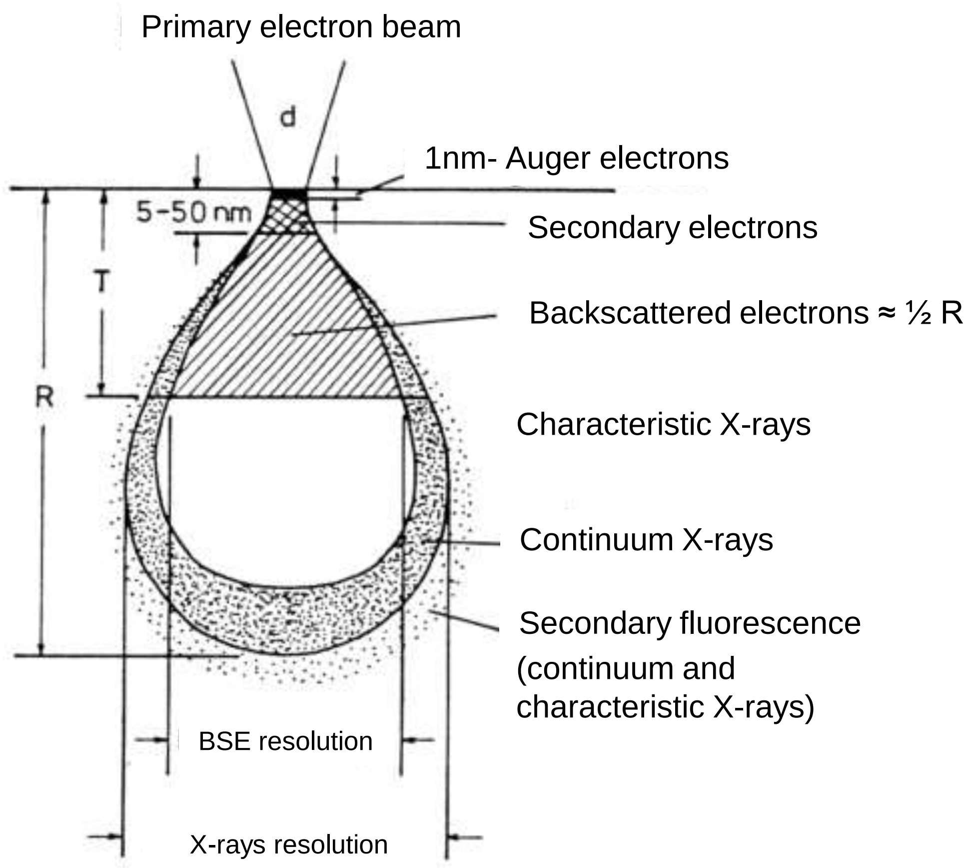
Image Creation in SEM



Which type of signal?



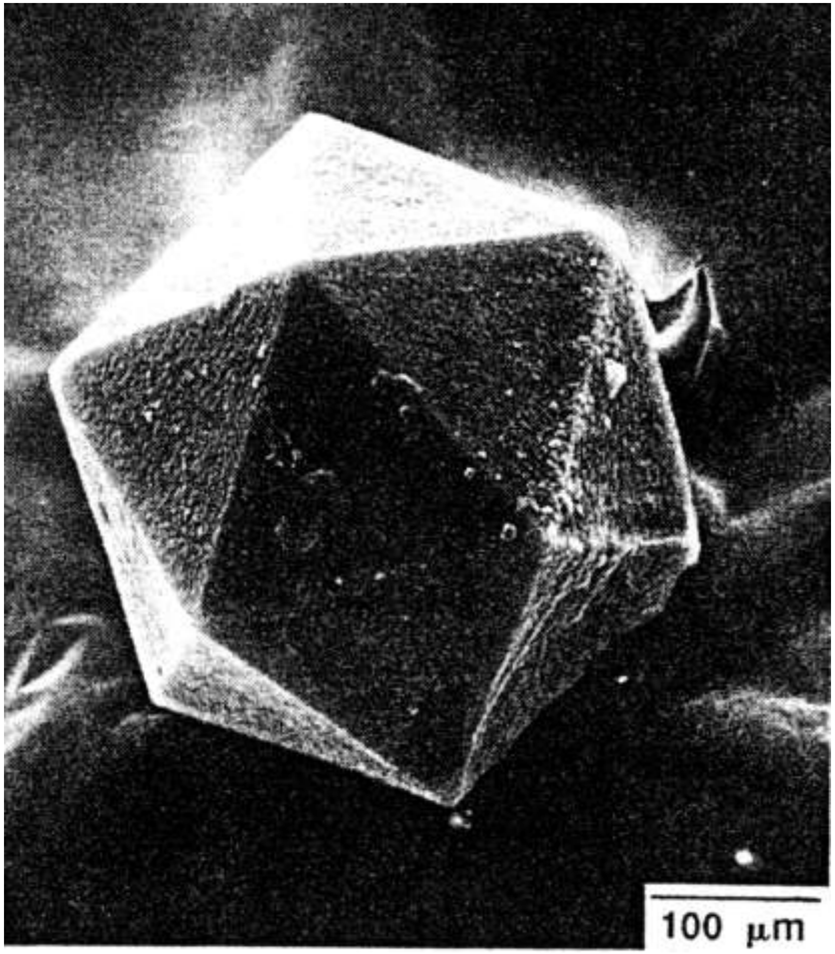
From which volume does the signal come?

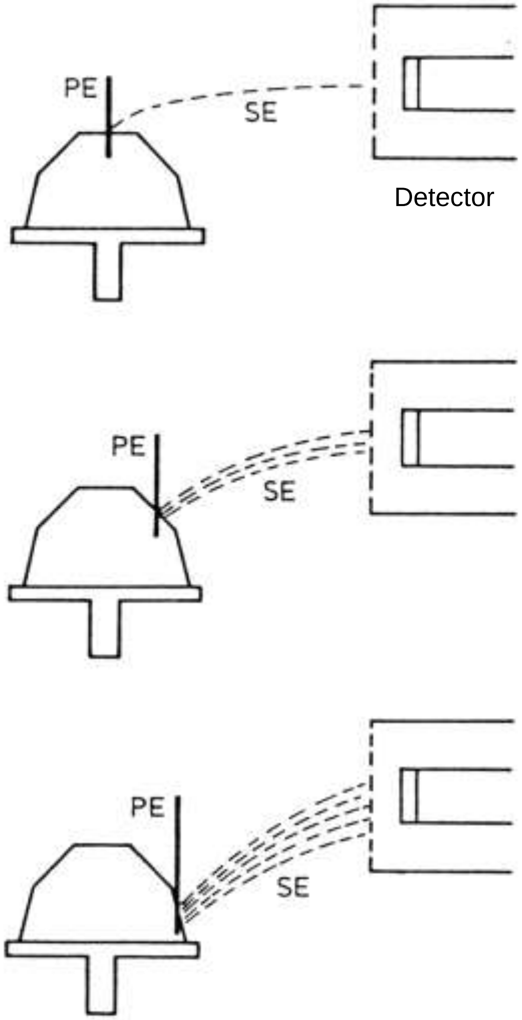


Coverage of the BSE and the PE and depth of the different signals in SEM:

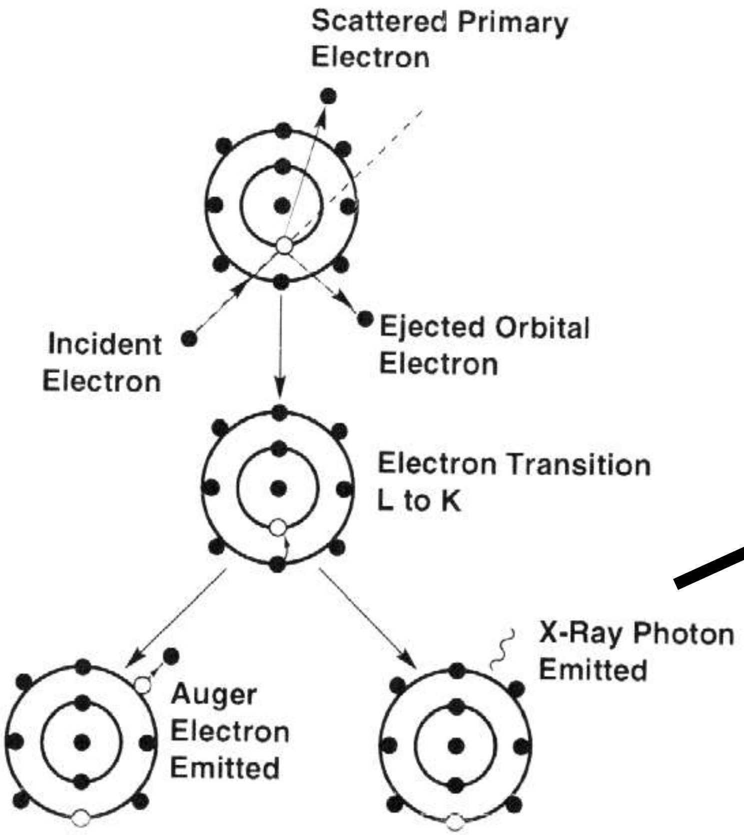
- R: Coverage of the PE
-T: Signal depth of the BSE

Application: Topography contrast





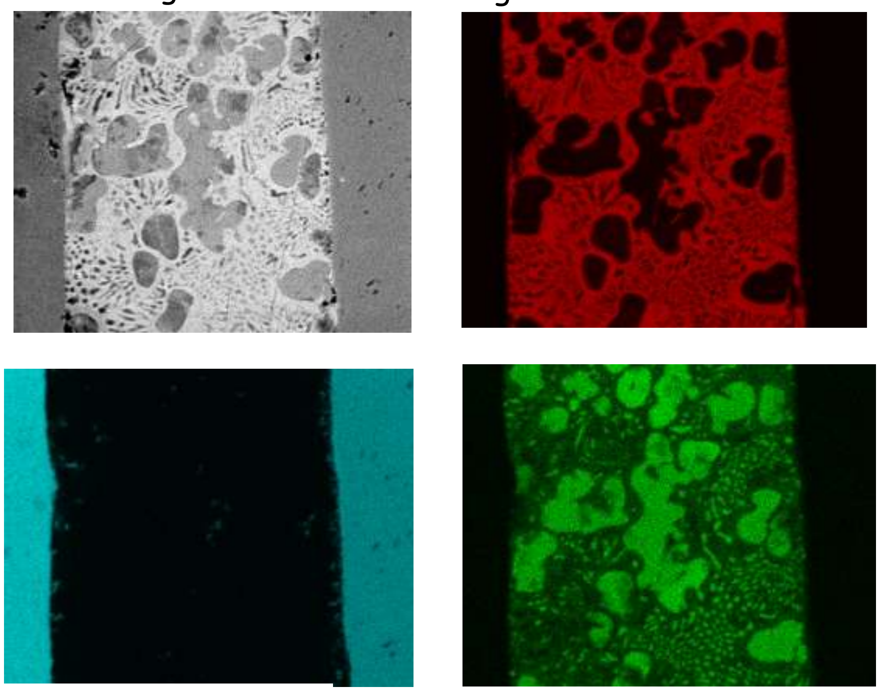
Application: X-rays distribution image



Example: soldering of stainless steel tube

BSE image

Ag distribution

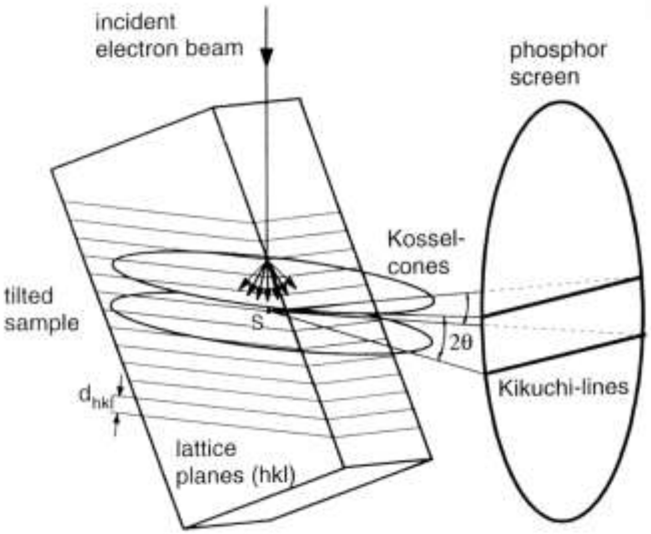


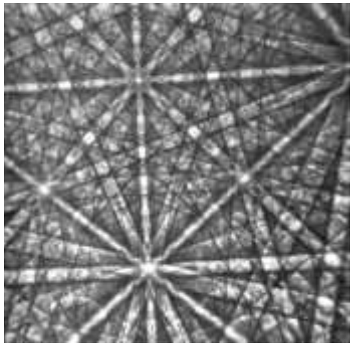
Fe distribution

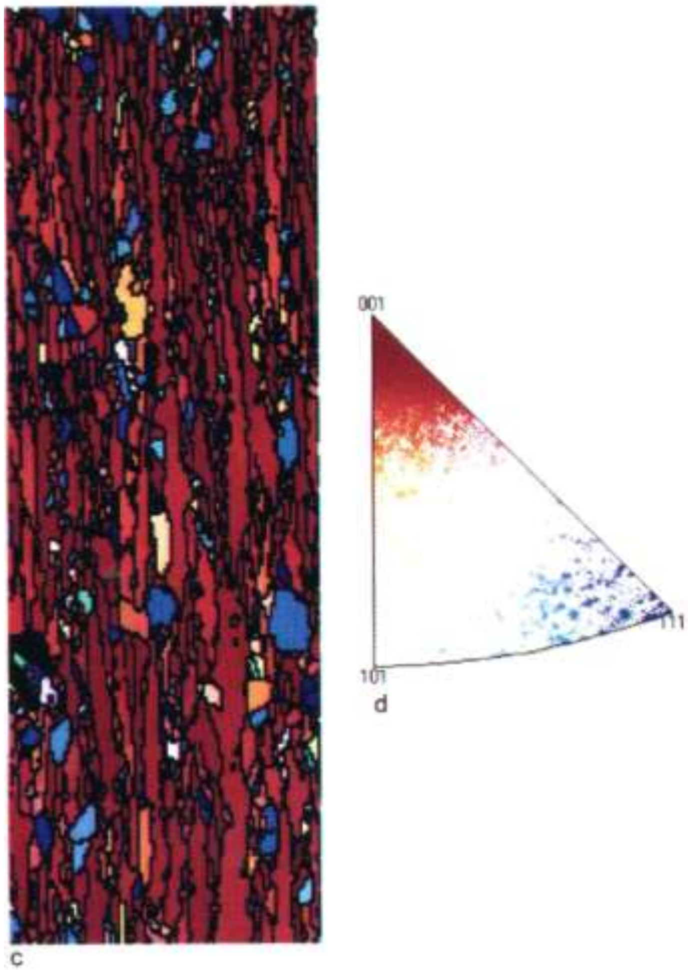
Zn distribution

Application: Orientation distribution image

Electron backscatter diffraction diagram (EBSD):







Orientation map of electrolytically separated nickel

outline

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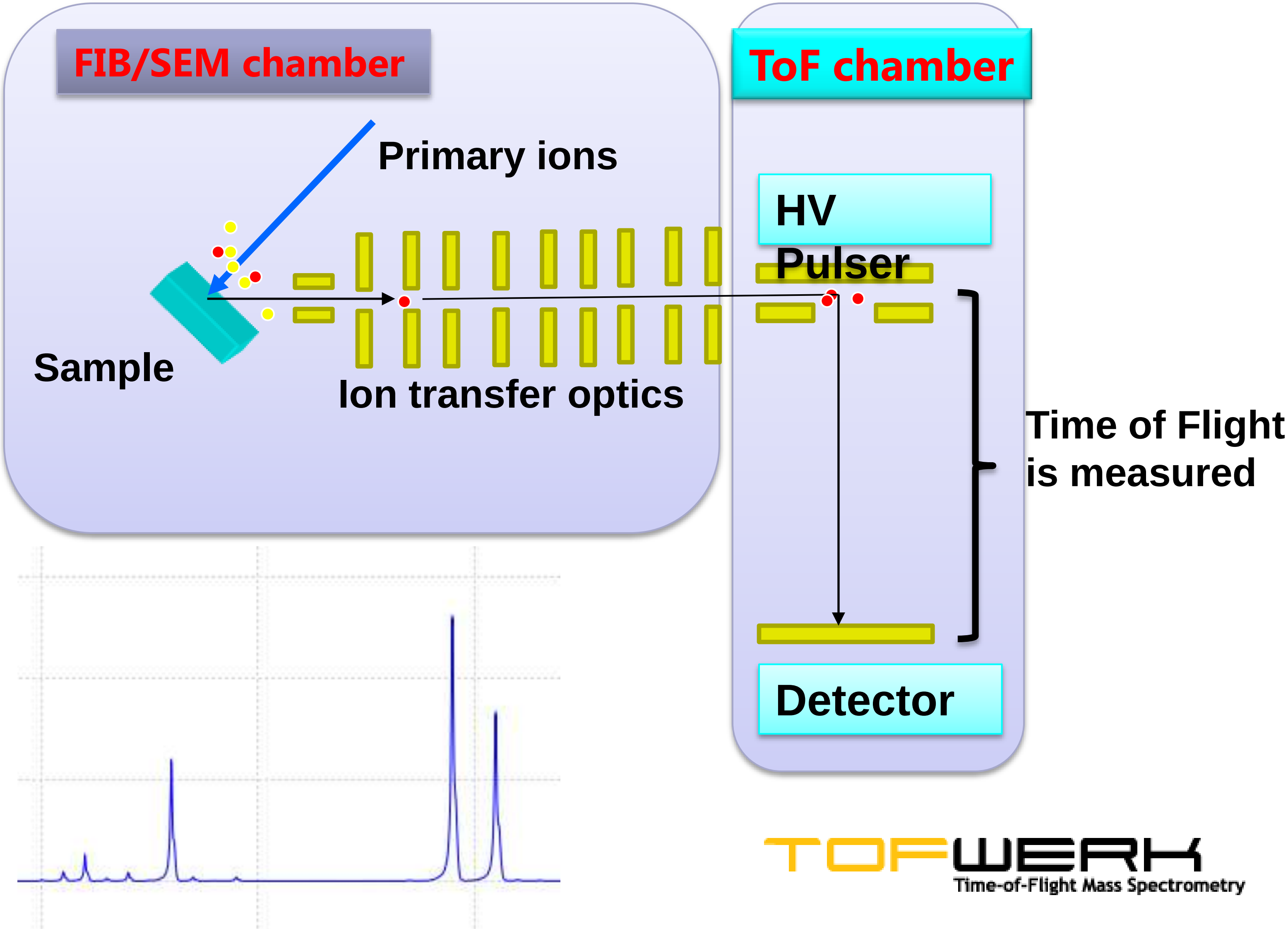
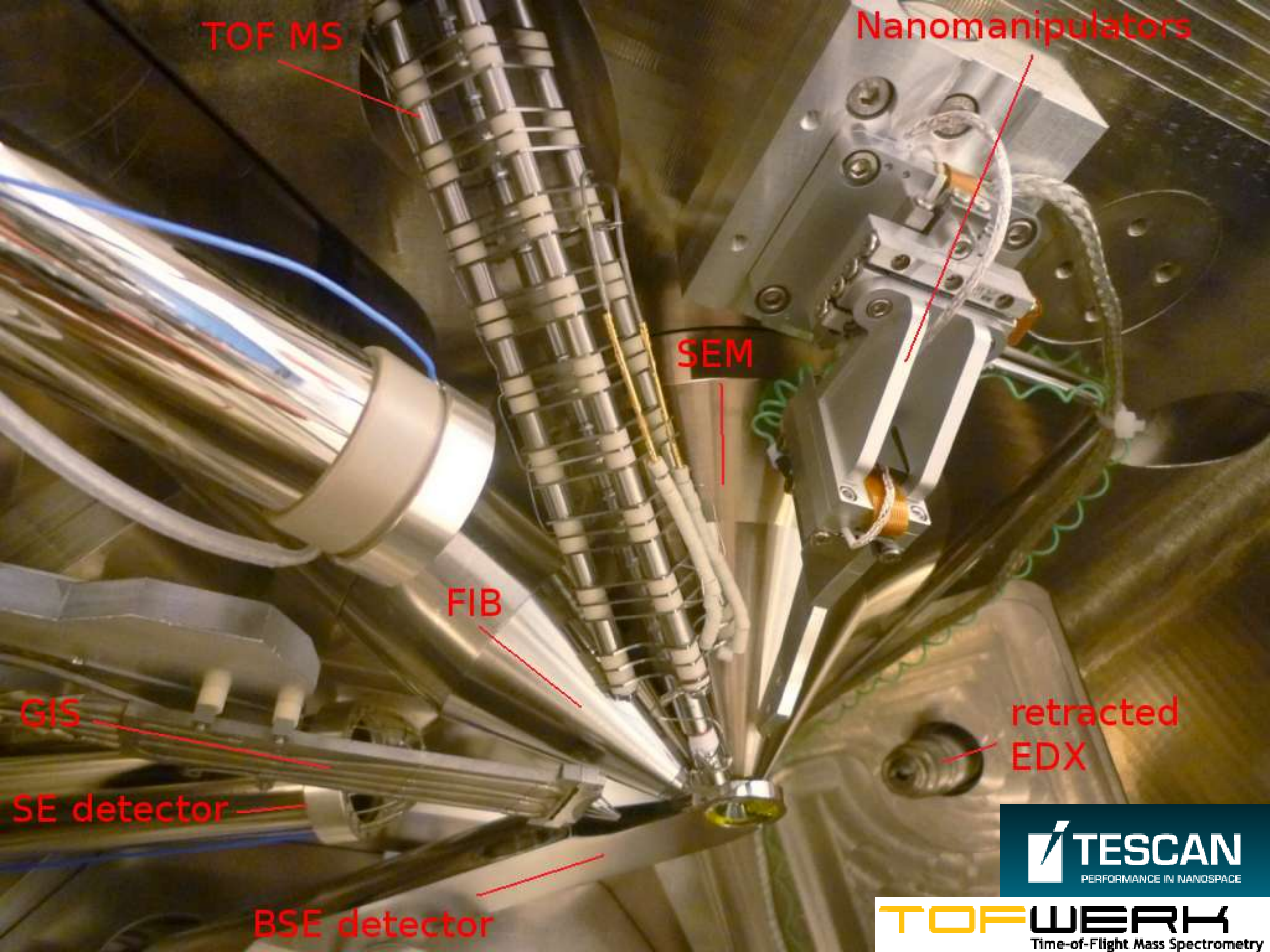
Orthogonal ToF-SIMS



TOFWERK
Time-of-Flight Mass Spectrometry

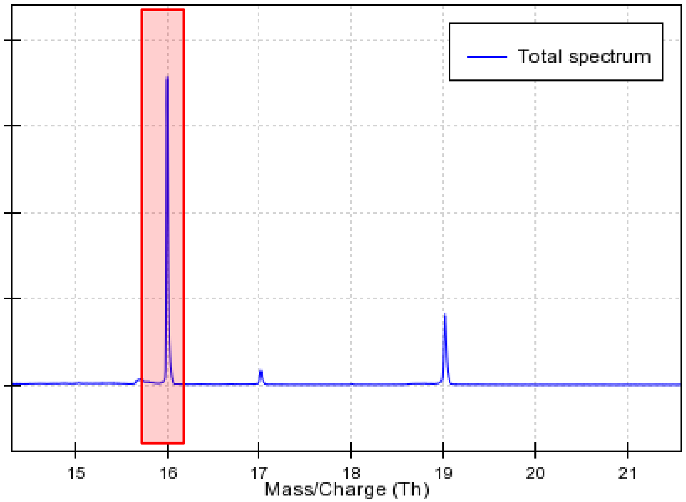


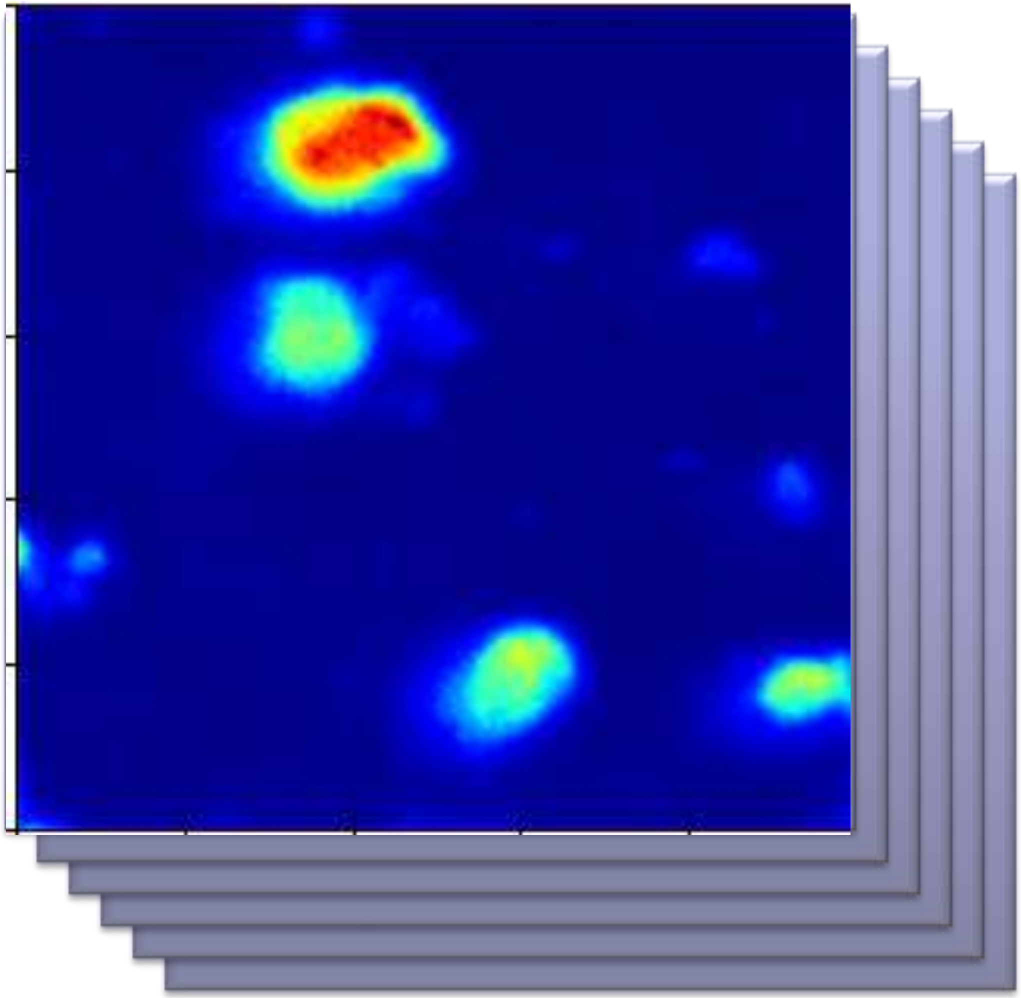
TESCAN
PERFORMANCE IN NANOSPACE
TOFWERK
Time-of-Flight Mass Spectrometry

Orthogonal ToF-SIMS

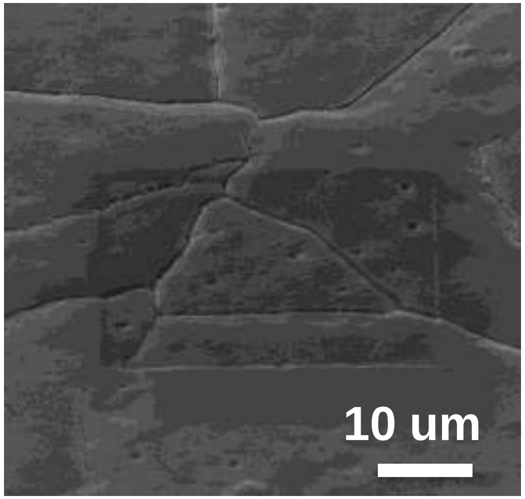
Synchronization of the ToF pulser with the FIB beam raster:
One spectrum per pixel

Averaging laterally and in depth



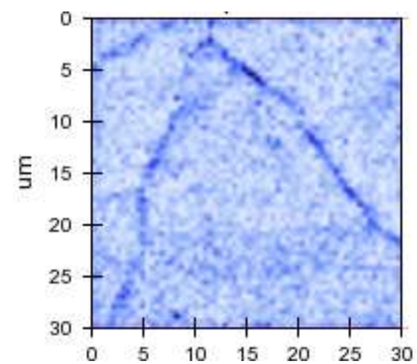


Imaging and Depth Profiling

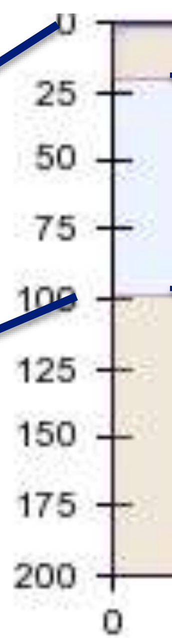


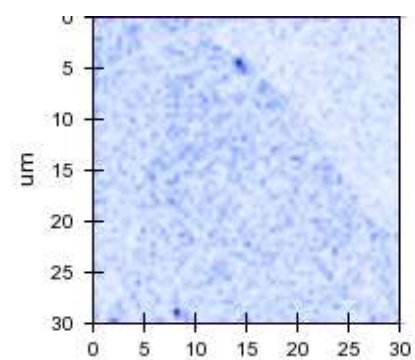
SEM image after SIMS

Chlorine map

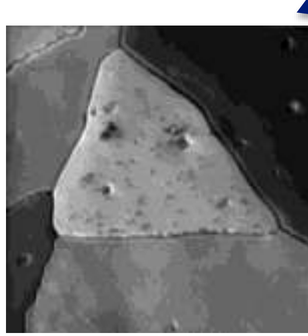


Depth (frames)

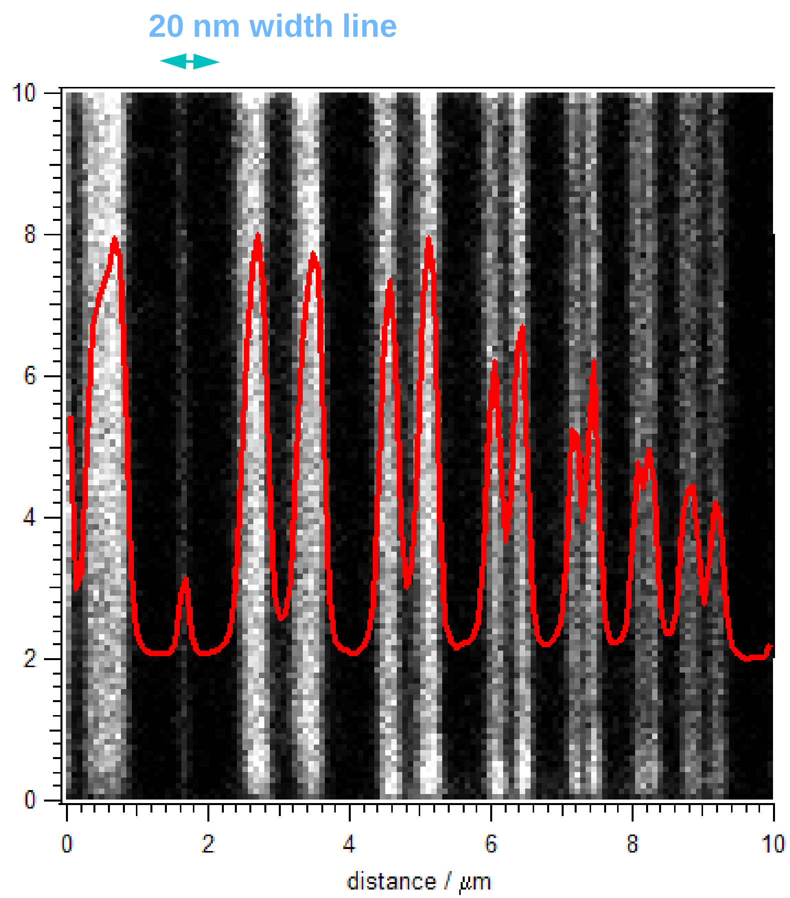


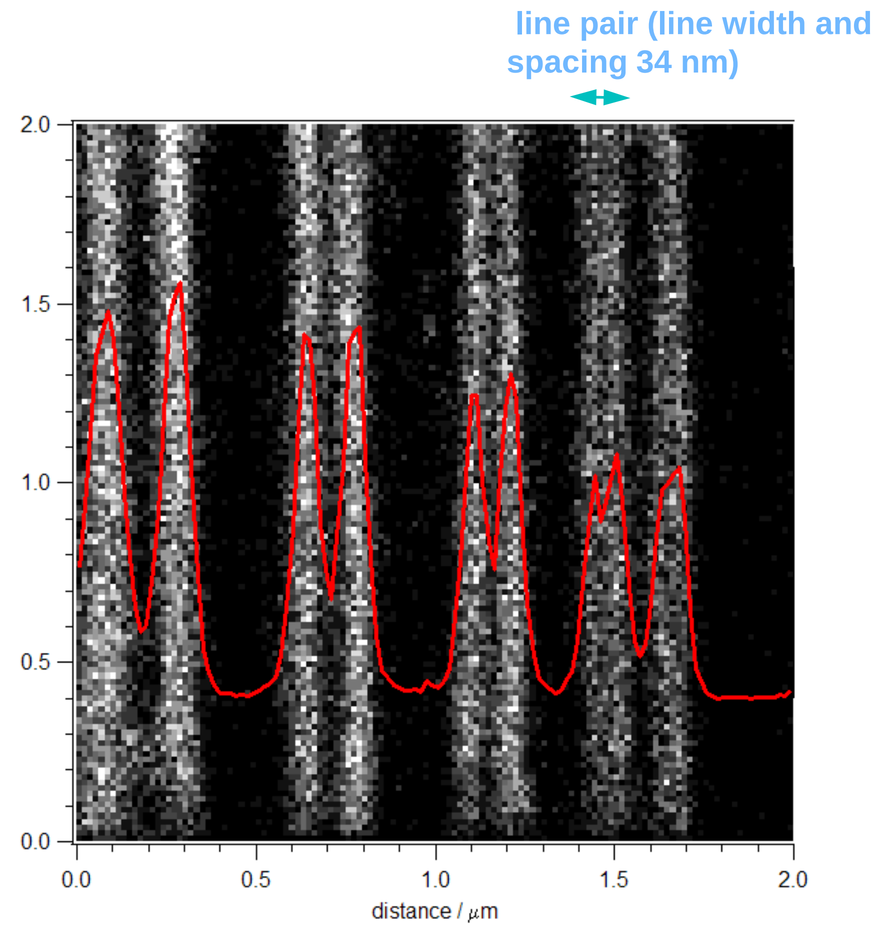


FIB Images at different depth



Multilayer reference sample resolution



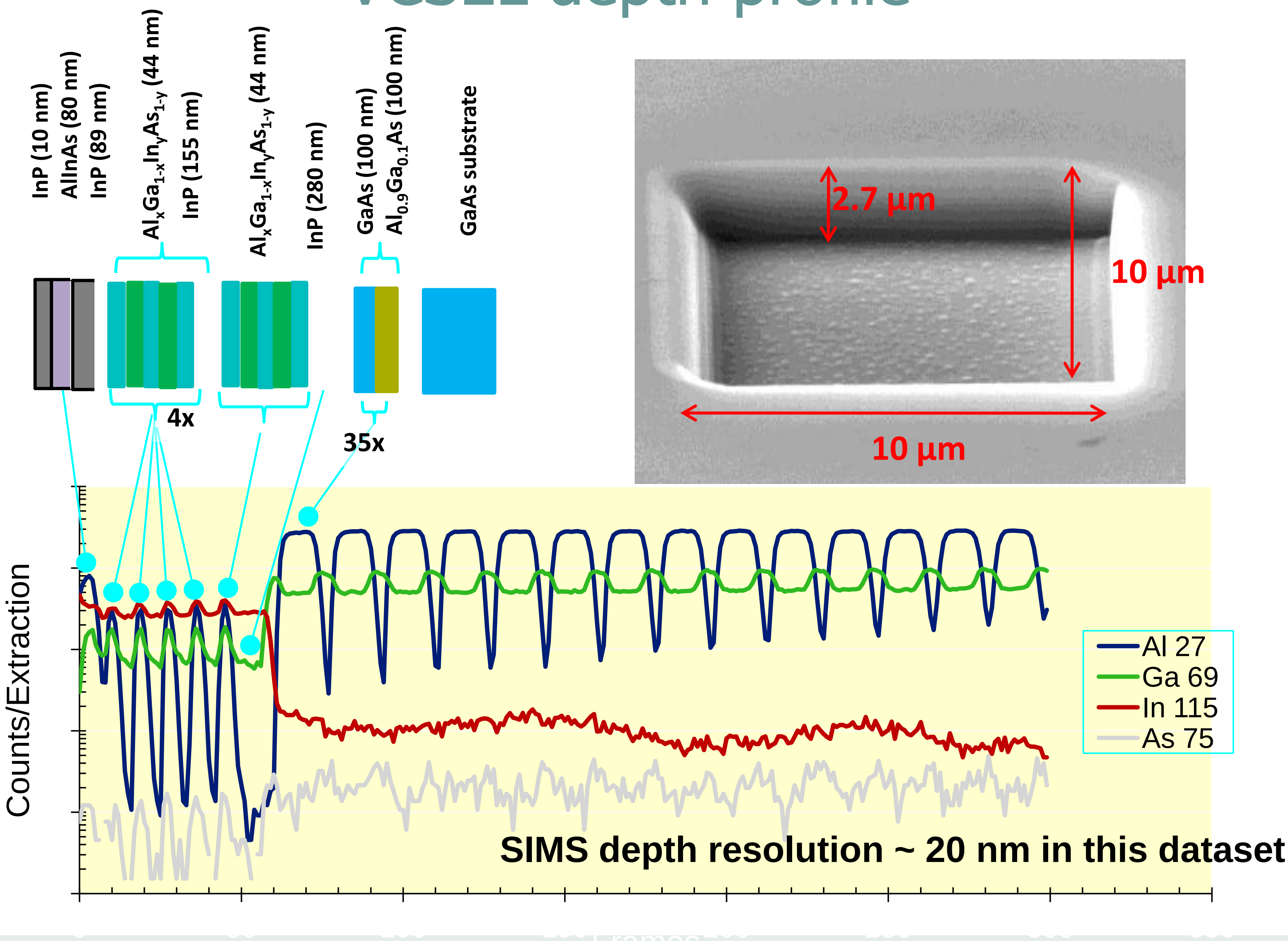


Aluminium ion image of AlGaAs stripes in GaAs

Lateral resolution ~ 50 nm

Features 20 nm across easily detected (not resolved)

VCSEL depth profile



Summary: Focussed Ion Beam Secondary Ion Mass Spectrometry (FIBSIMS)

- Analytical technique exploiting sputtered ions
  - Use Ga^+ Focused Ion Beam (FIB) as primary ion source
- Much better spatial resolution and detection limits than SEM/EDX
  - <50 nm, < 1 ppm (instead of 1000 nm, 10000 ppm)
- 3D chemical images
  - Depth resolution <20 nm (comparable to glow discharge)
  - Retrospective analysis (e.g. depth profile) possible
  - Simultaneous secondary electron imaging
- FIB-TOFSIMS much cheaper than dedicated TOFSIMS instrument
  - If you already have a FIB!

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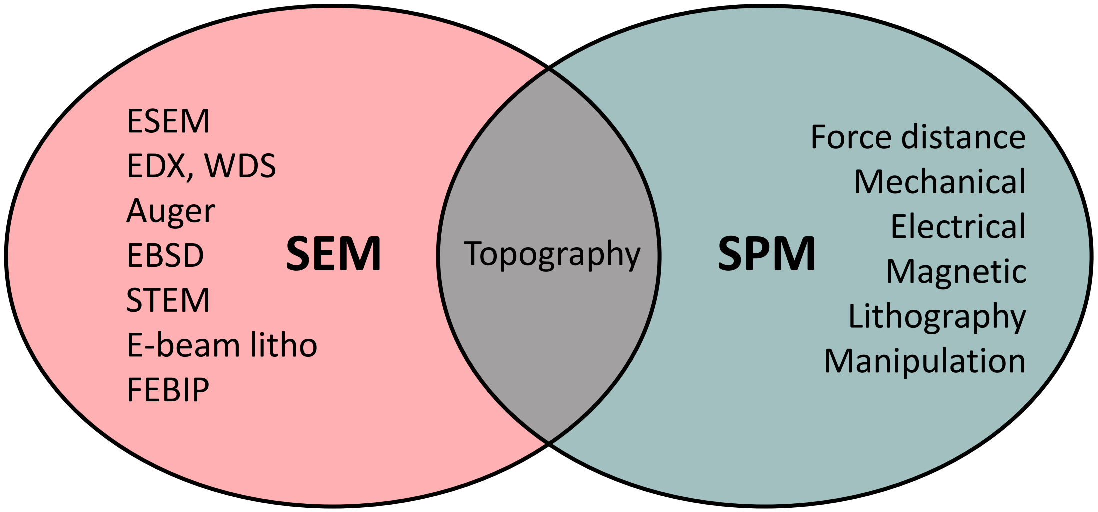
AFM in SEM



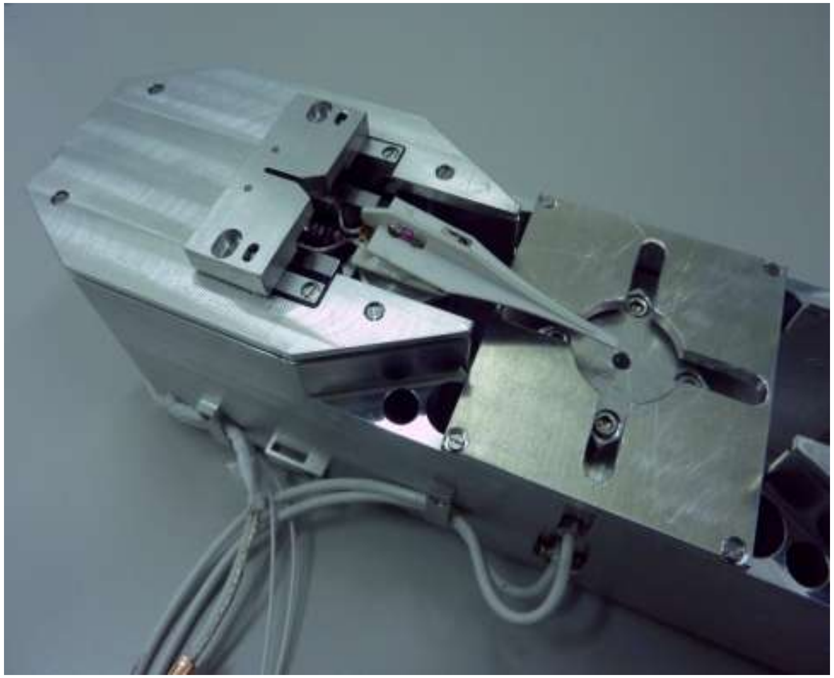
Strength of SEM:
- Chemical composition

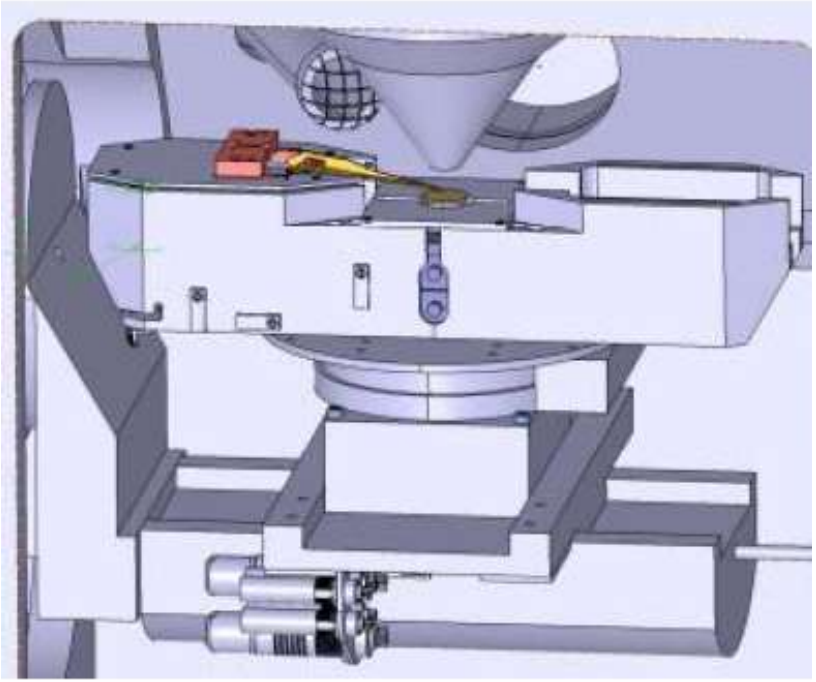
Strength of SPM:
- Mechanical/Electrical properties of surfaces

Limitations of SEM:
- no depth resolution
- Insulating samples
- Metrology

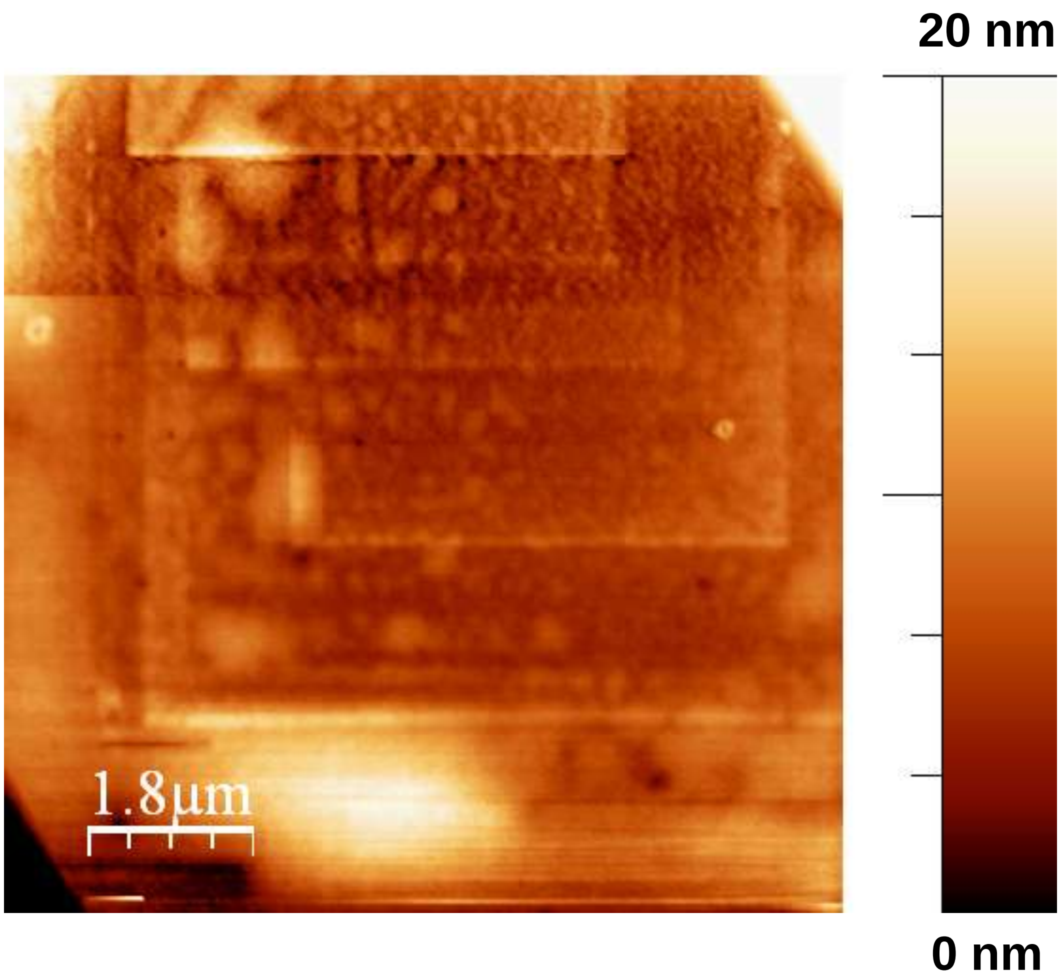
Limitations of SPM:
- Slow acquisition
- Small field of view
- Small depth of field

AFM in SEM

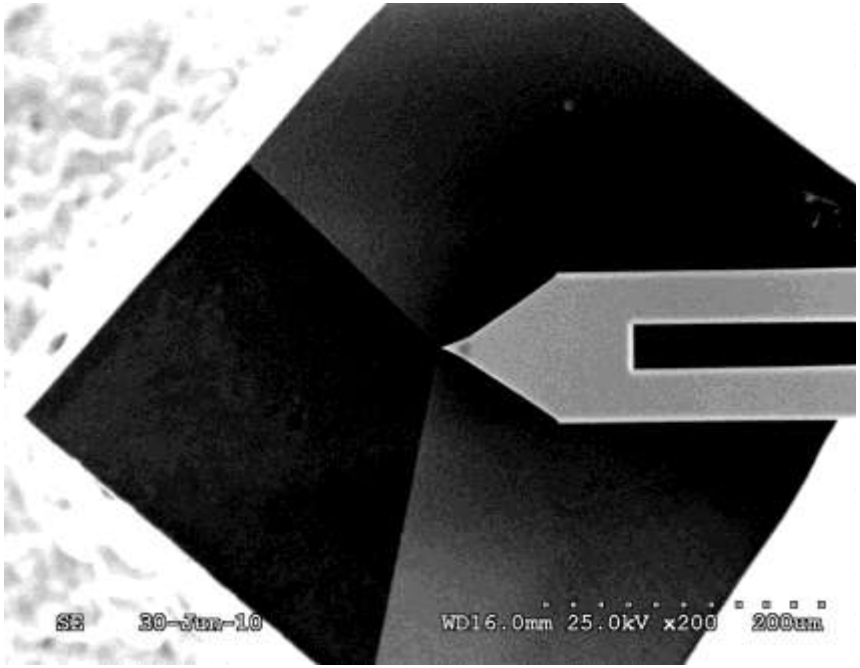


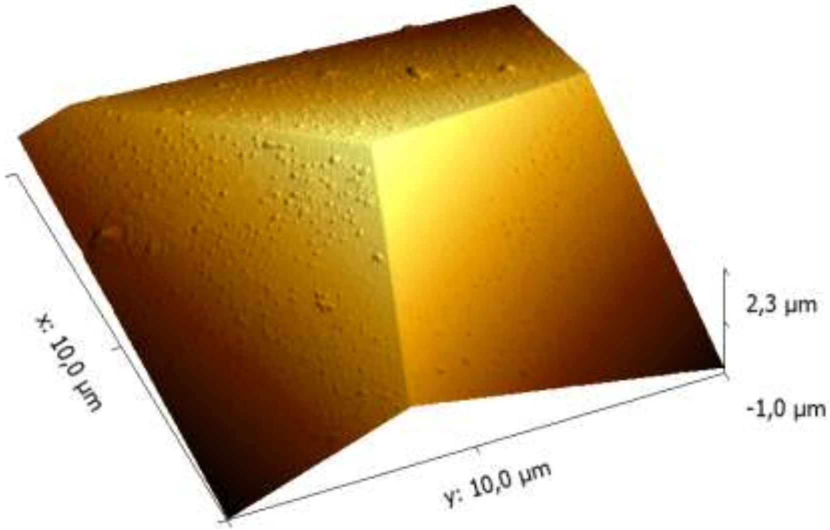


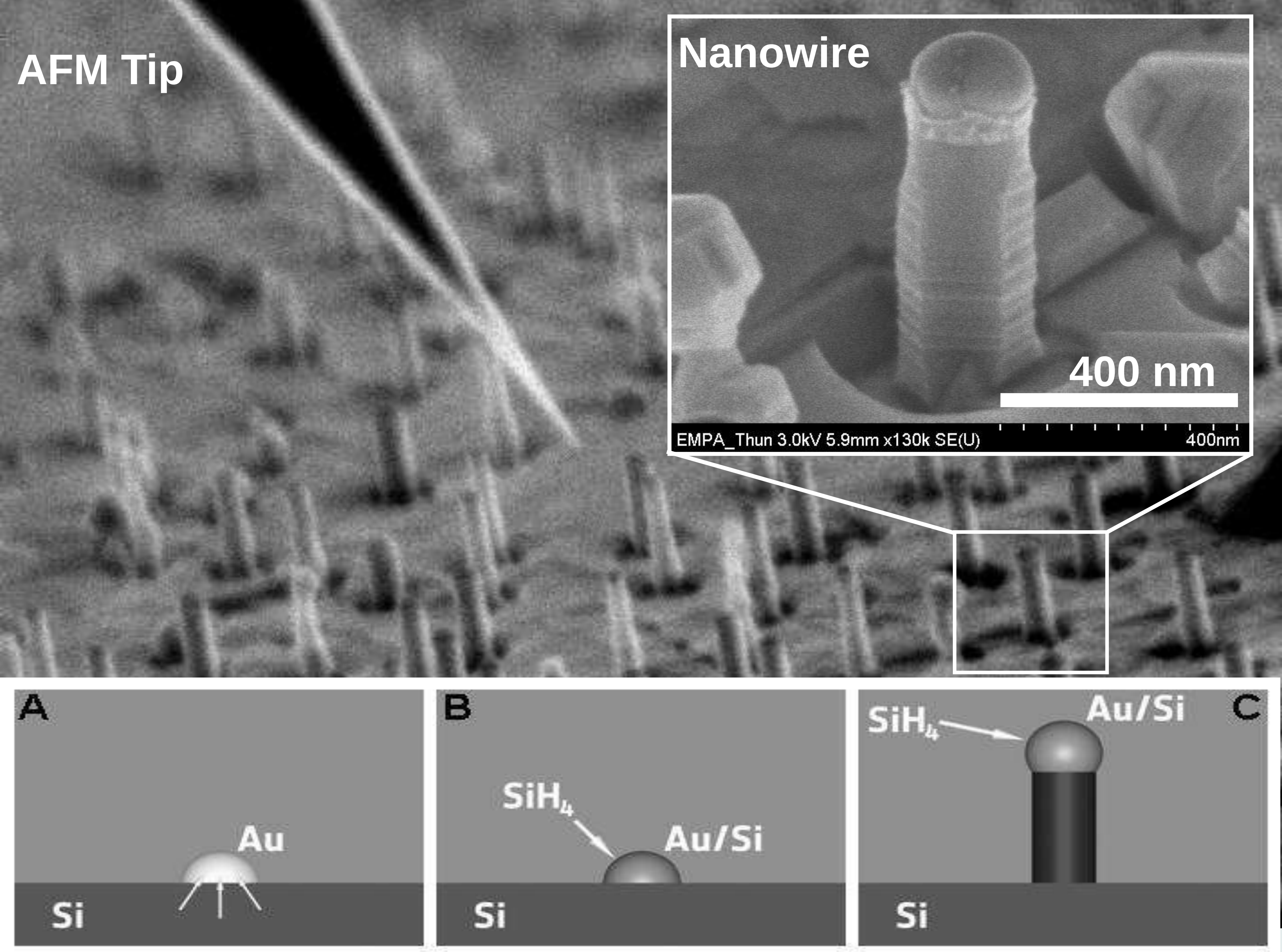
applications: contamination boxes



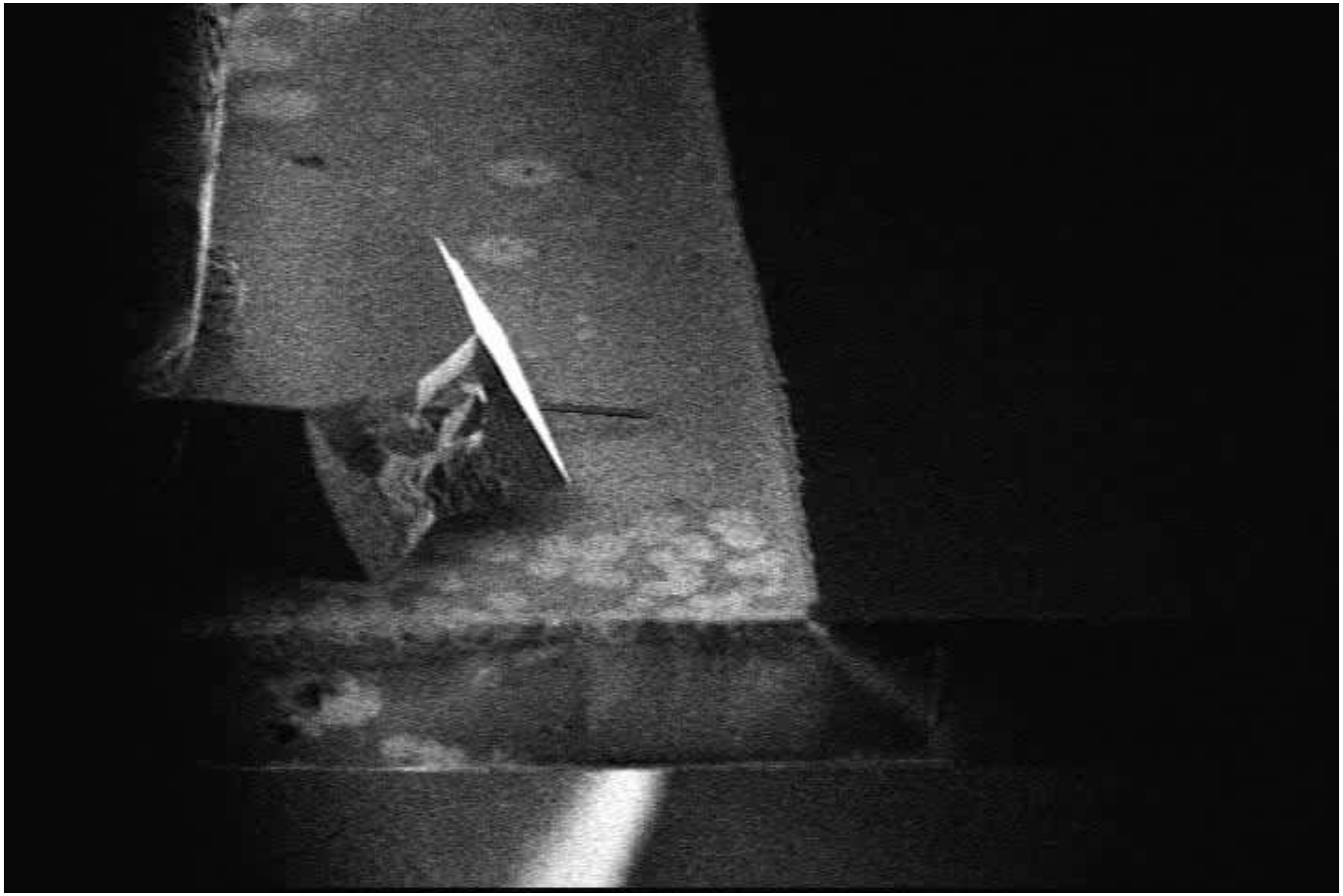
applications: diamond tips for nanoindenters



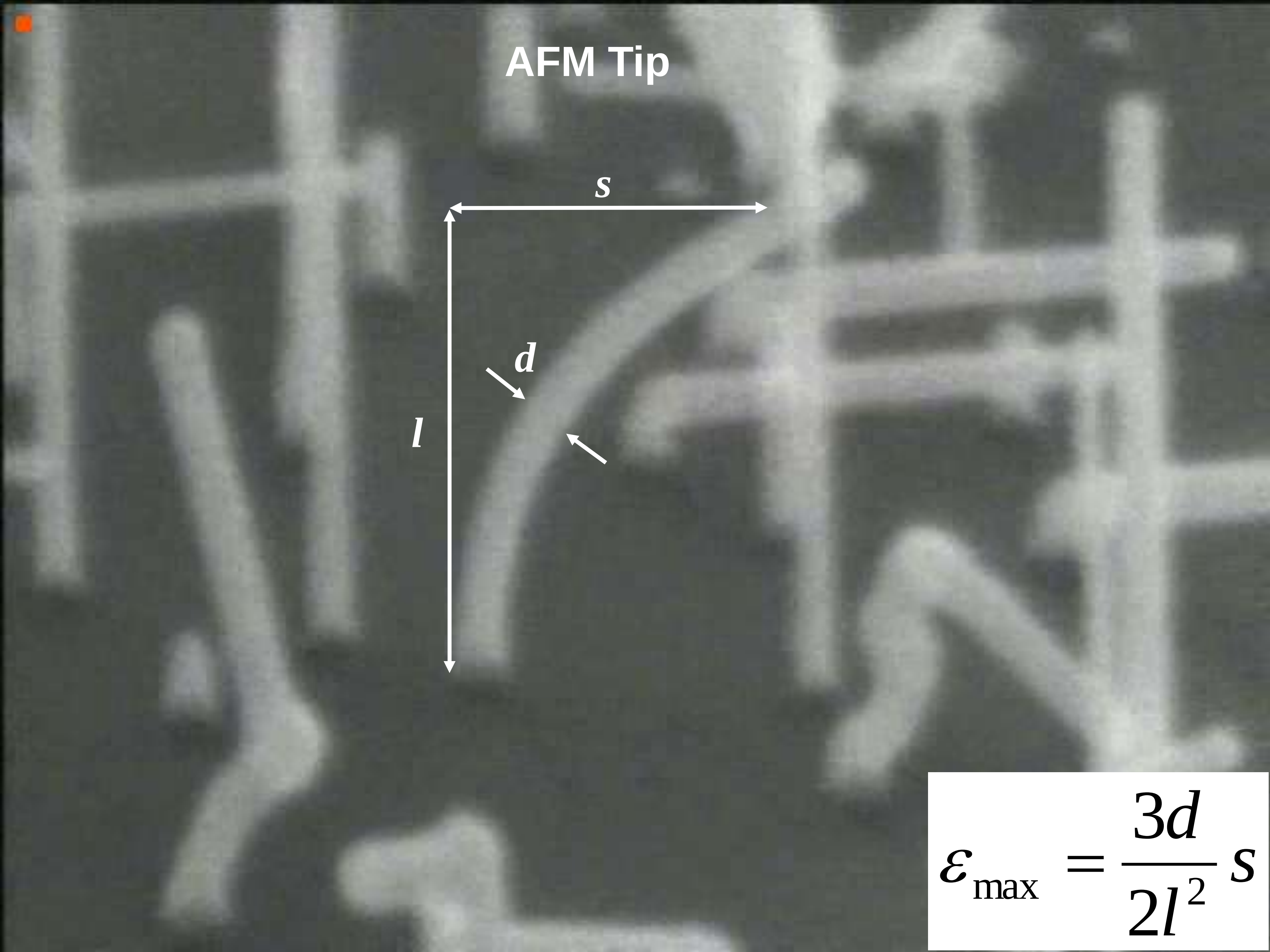



AFM Tip
Nanowire
400 nm
EMPA_Thun 3.0kV 5.9mm x130k SE(U)
400nm
A
Au
Si
B
SiH4
Au/Si
Si
C
SiH4
Au/Si
Si

AFM-in-SEM nanobending

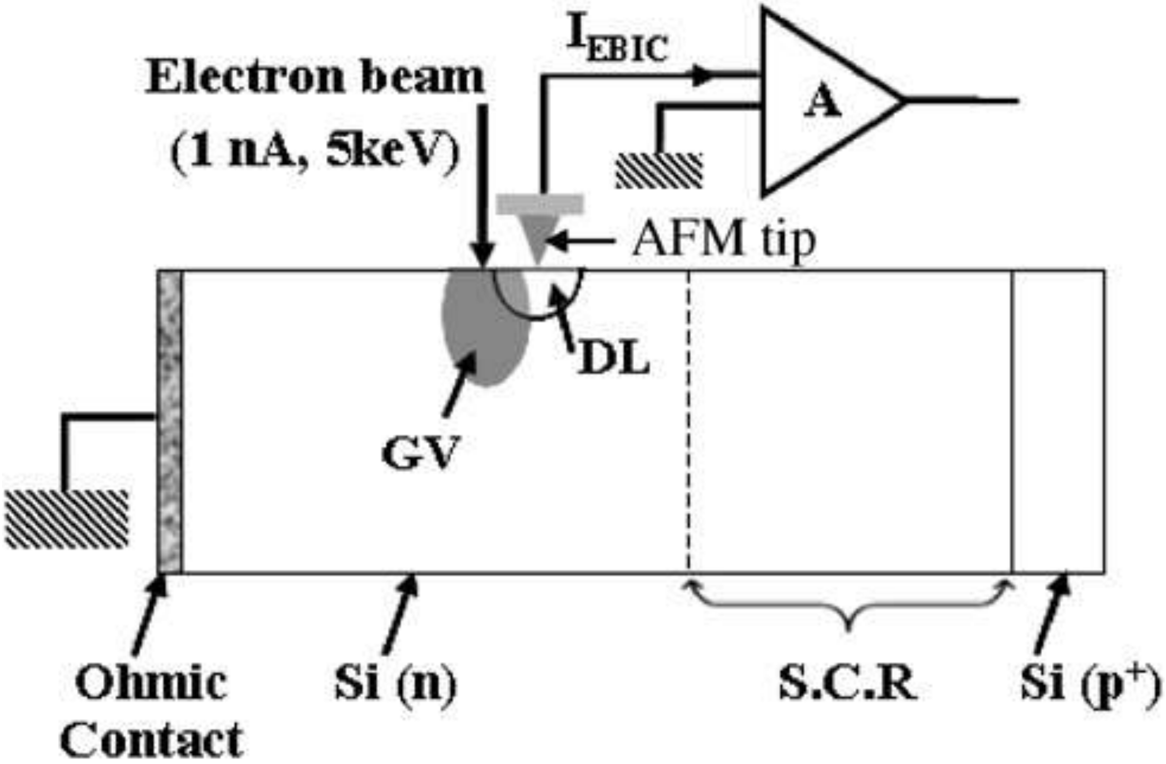


Hoffmann et al (2006) Nanoletters



$$\varepsilon_{\max} = \frac{3d}{2l^2} s$$

AFM-in-SEM: nanoebic



Troyon M. et al, Ultramicroscopy 108 (2008) 605–612

Summary: AFM in SEM

SEM: sample topography overview, crystallography, chemistry

AFM: imaging (all modes)

Combined:

nanomanipulation, probing

hybrid operation (EBIC, SNOM)

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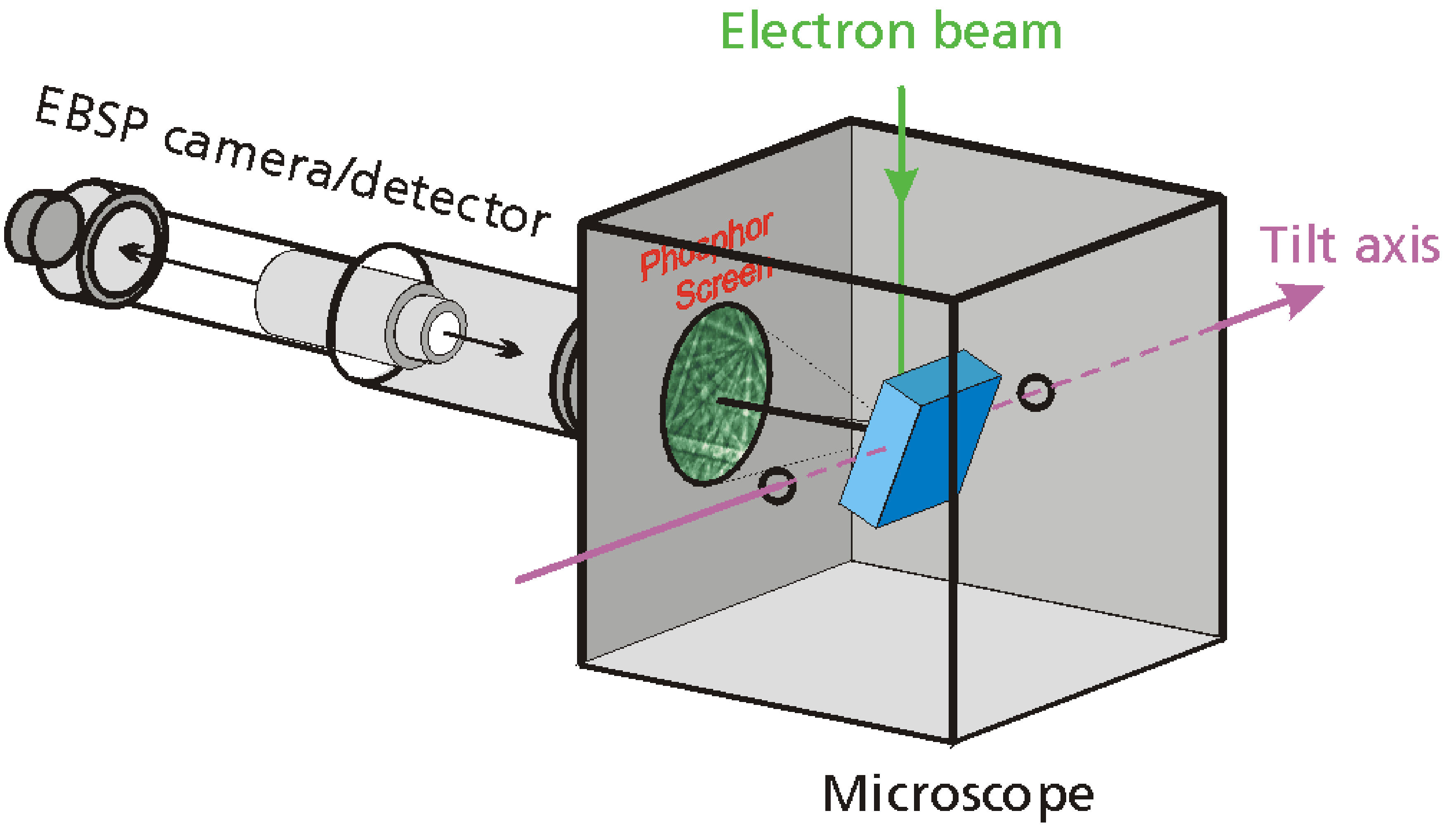
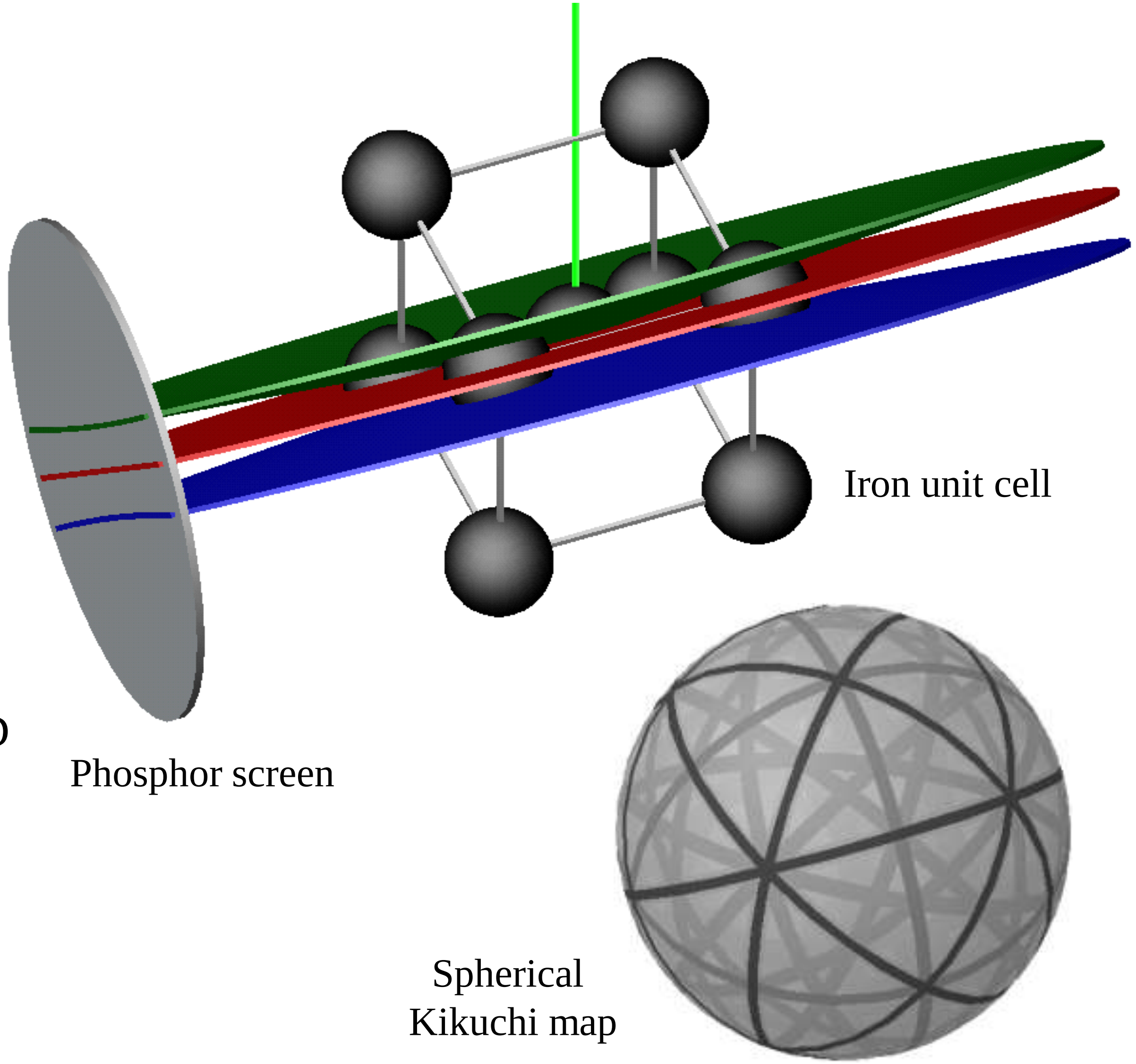
rapid prototyping

Electron Backscatter Diffraction

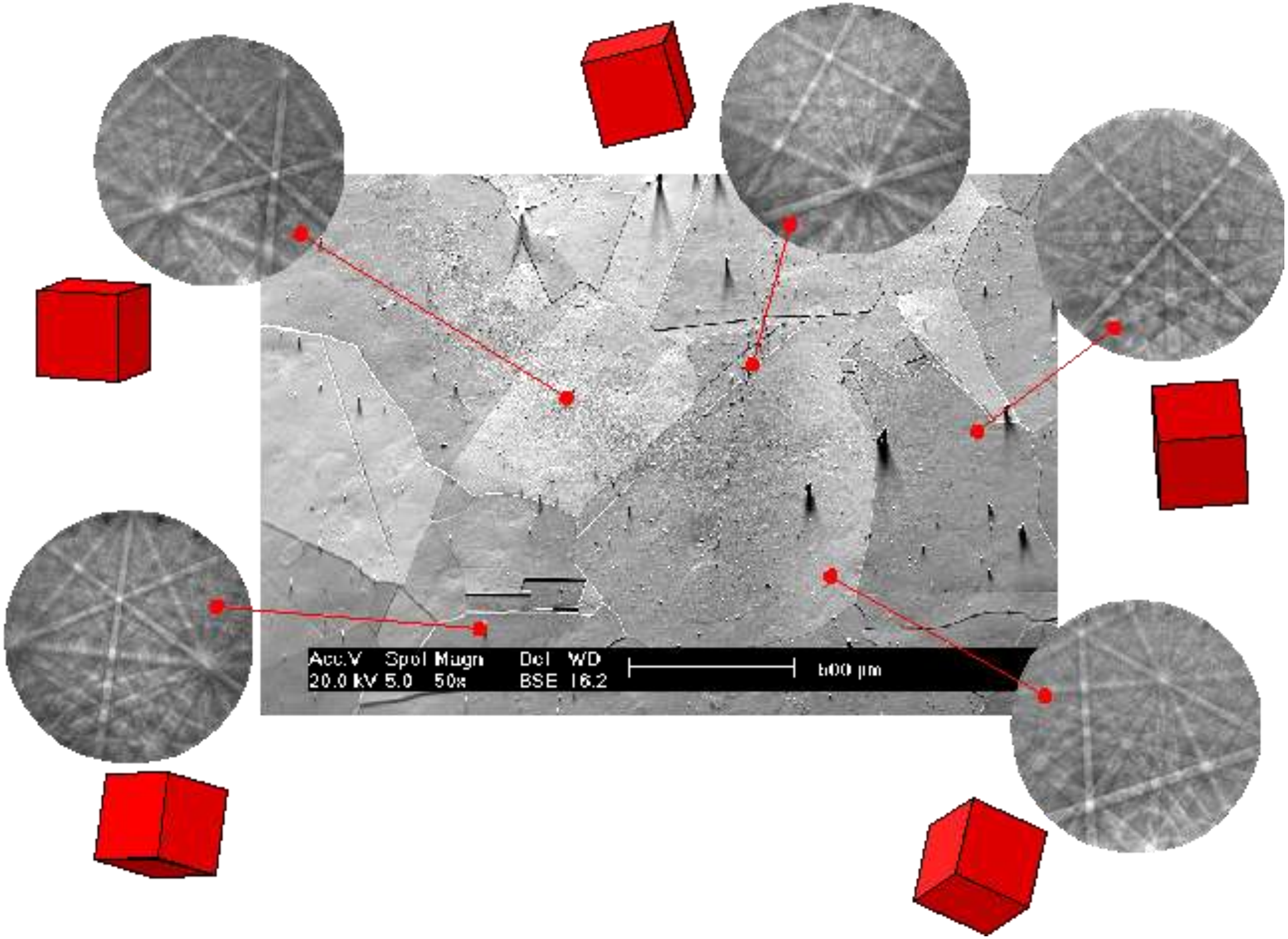


Formation of EBSD patterns: Geometry

- Electron beam strikes specimen
- Scattering produces electrons travelling in all directions
- Electrons that satisfy the Bragg condition ($n\lambda=2d.\sin\theta$) for a plane (hkl) are channeled ⇒ Kikuchi bands
- Electrons strike the phosphor and produce light
- Which is detected by a CCD camera and digitised
- The resulting EBSP is automatically analysed and indexed...



Formation of EBSD patterns: image formation



Large scale, multicrystalline thin film silicon

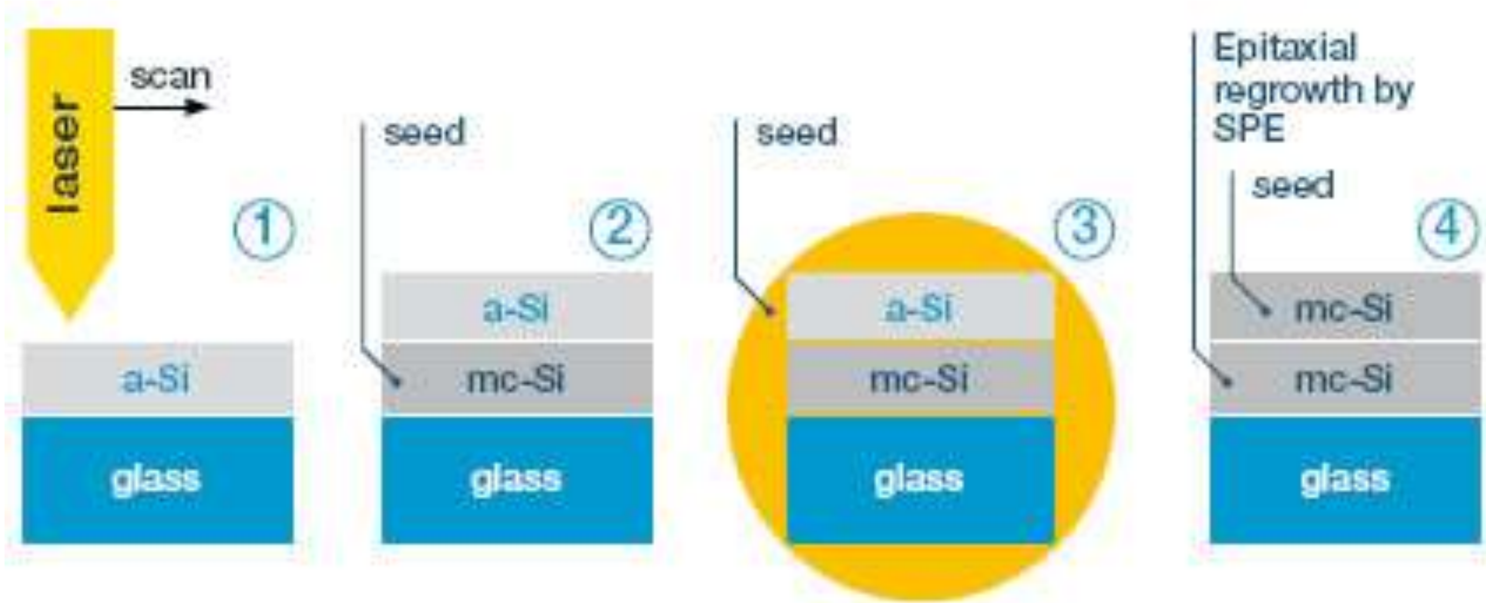
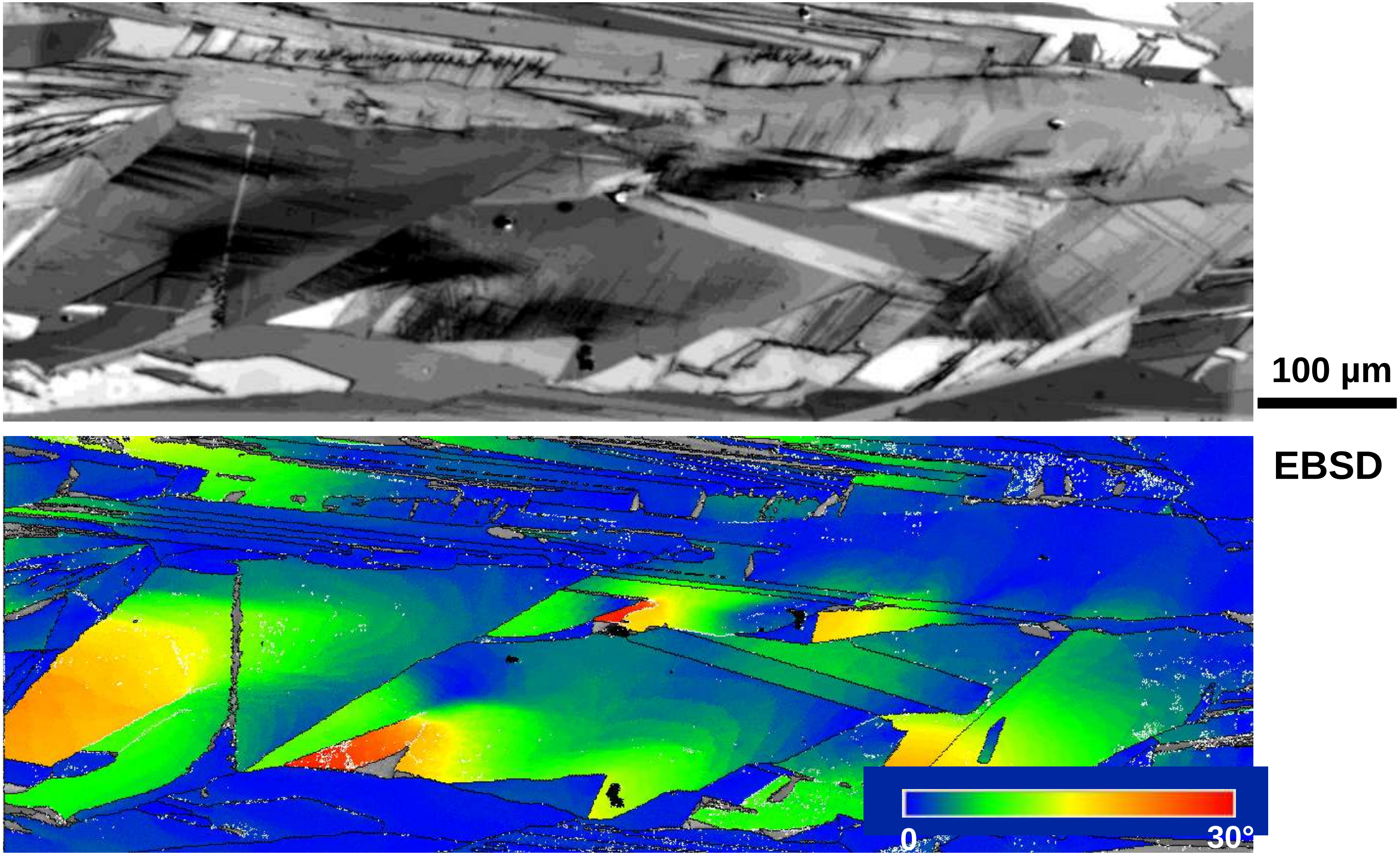


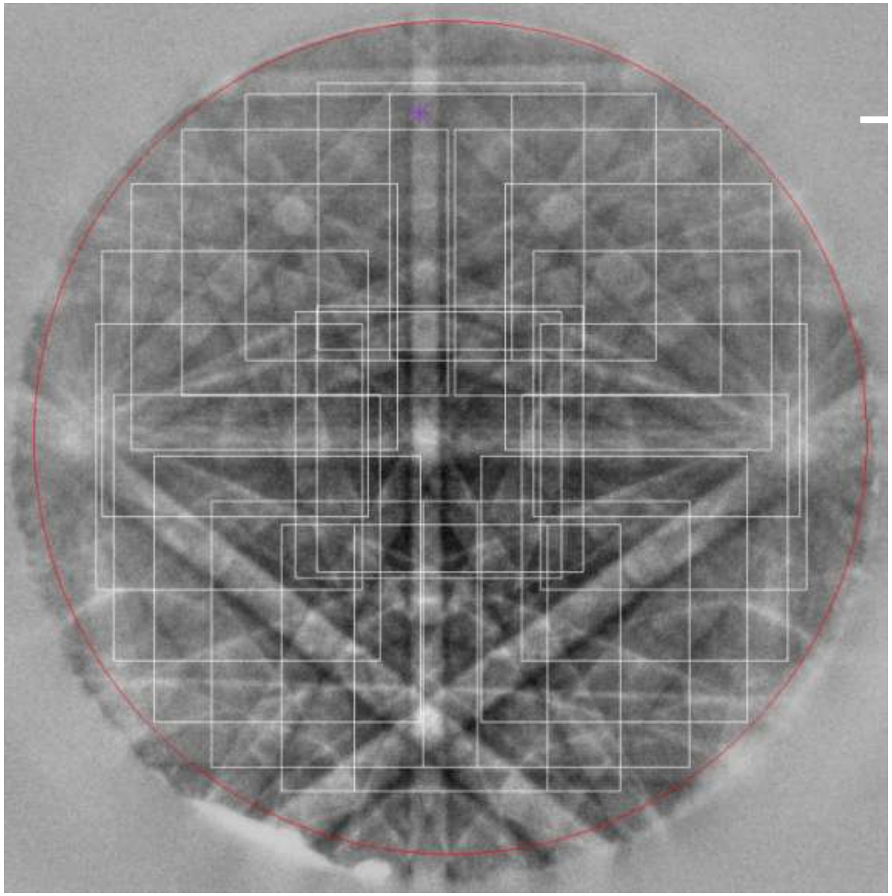
Figure 1:
Schematic of the laser-SPE process which will be established to realize large grained, low defective silicon layers on glass that have the potential for >10% efficiencies.

dislocation distribution: EBIC and OIM



Strain – stress analysis with EBSD: Cross Court

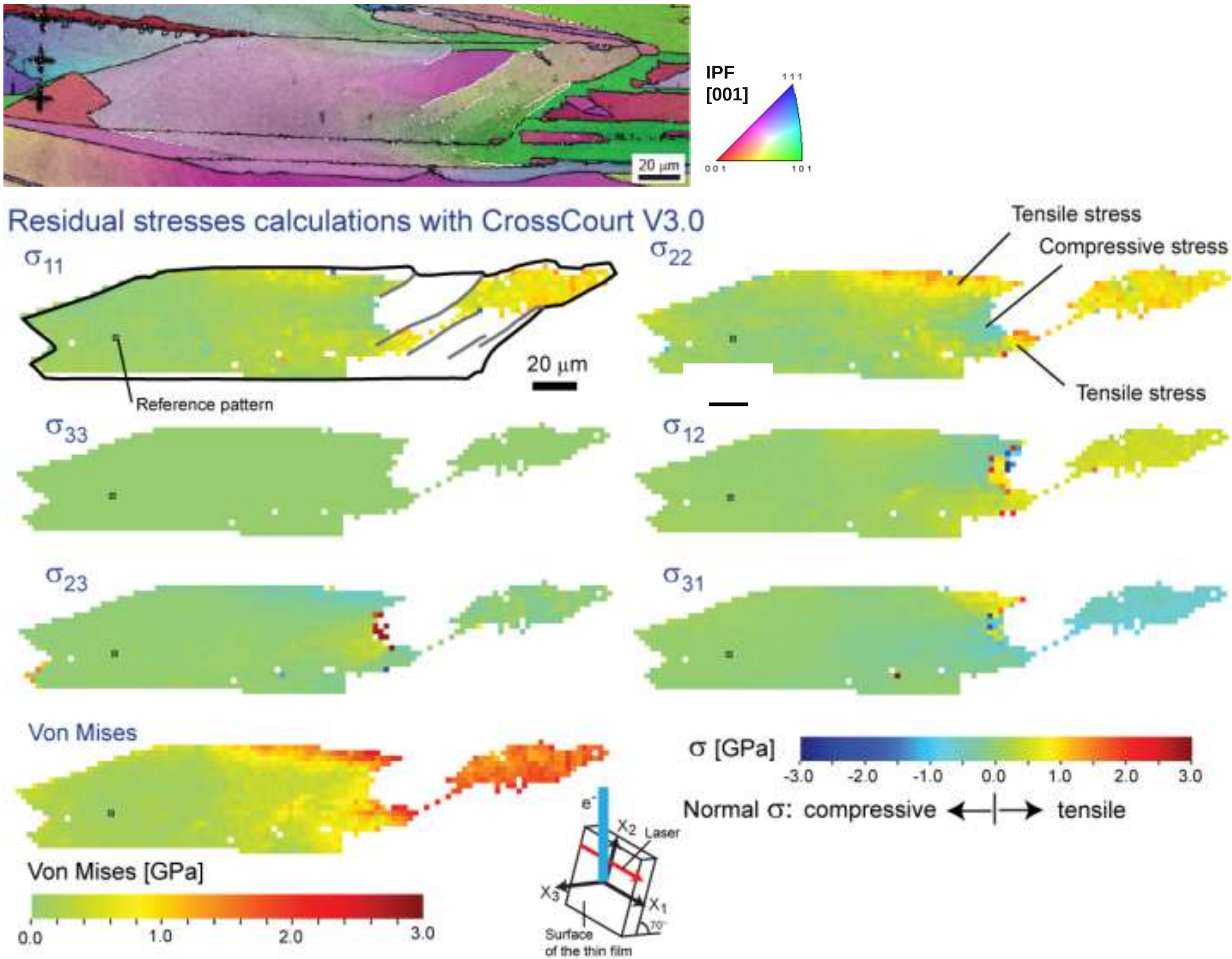
Cross-correlation between a strain free reference pattern and the pattern at the point of interest



- Rotation precision: 0.01°
- Strain resolution: 10^{-4}

A.J. Wilkinson, G. Meaden and D.J. Dingley, Ultramicroscopy 106 (2006); Mater. Sci. & Tech. 22 (2006)

stress concentrations: EBSD



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size effects in materials

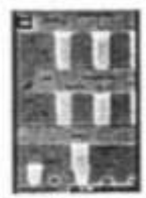
10^{-9} m   10^{-6} m   10^{-3} m   1 m

plasticity   fracture

<= intrinsic length scale

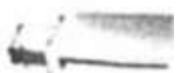
elasticity   fatigue

Nano-Devices



microelectronics

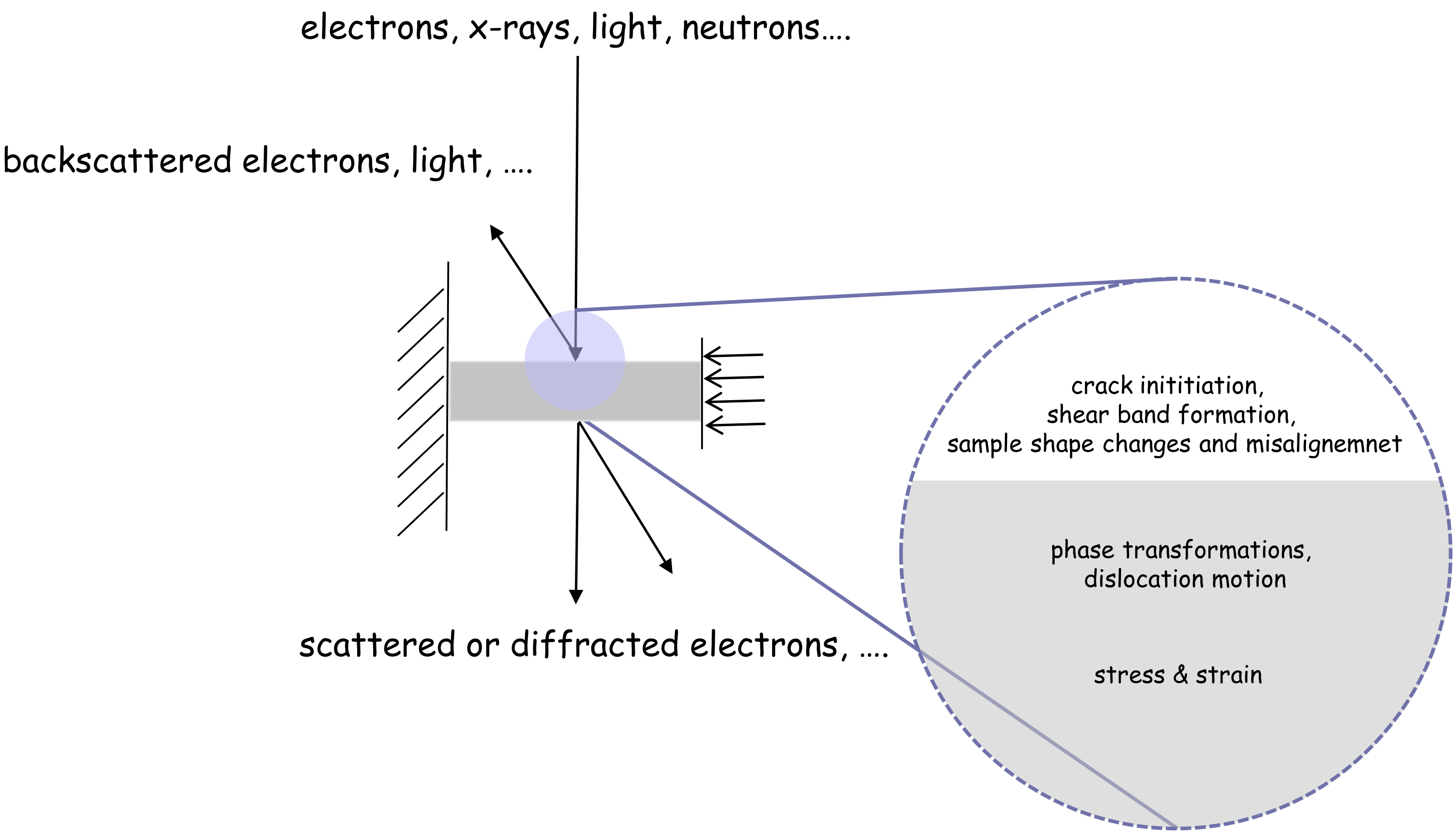
MEMS



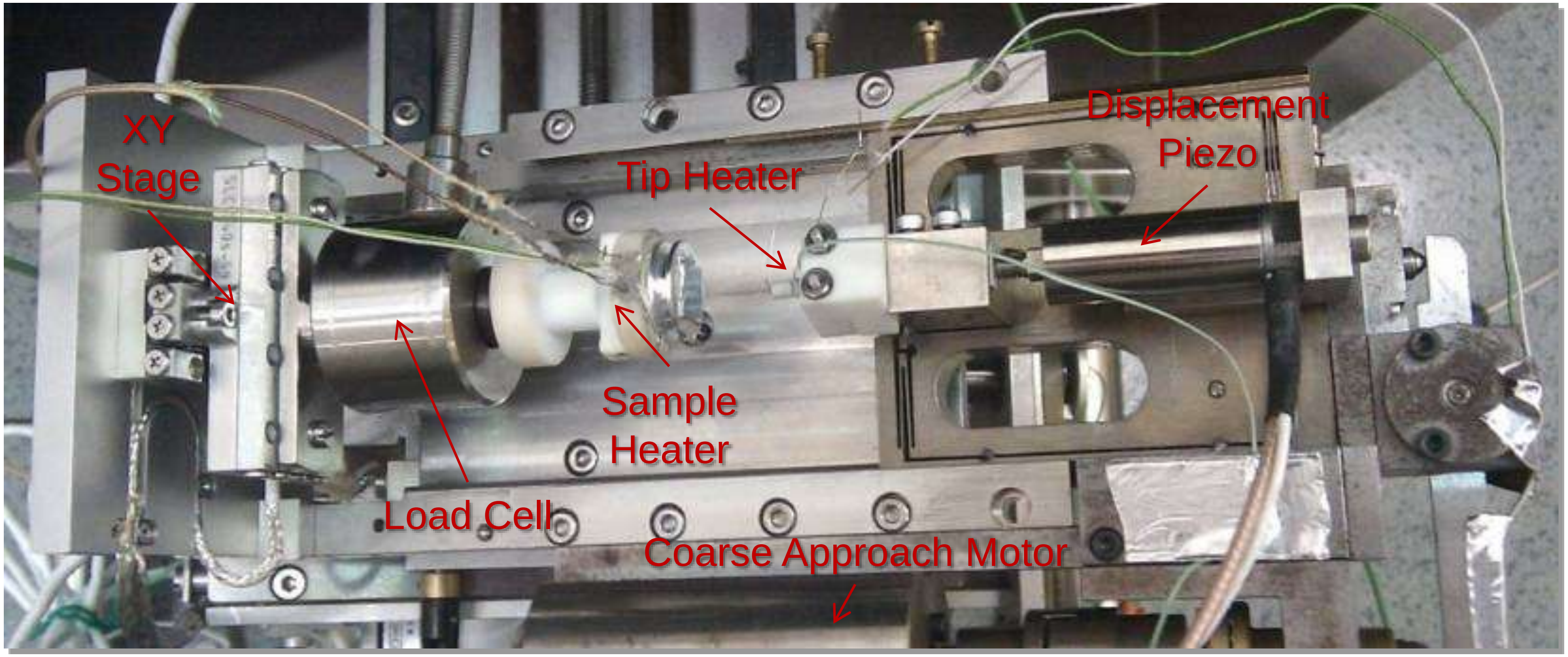
engines etc.

conventional testing

in-situ experiments



in-situ high temperature indentation system



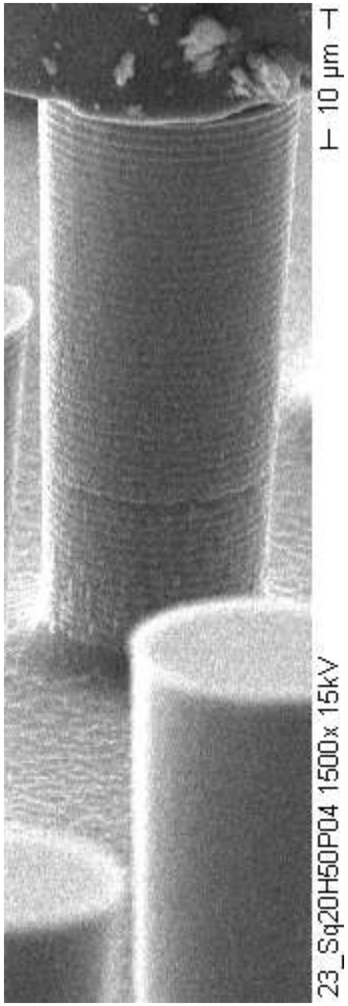
Custom *In Situ* , Elevated Temperature SEM Indentation System

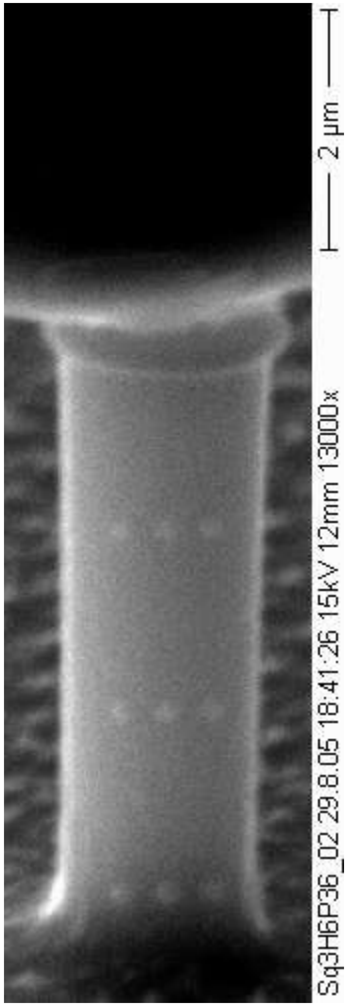
Current max temperature: 700°C

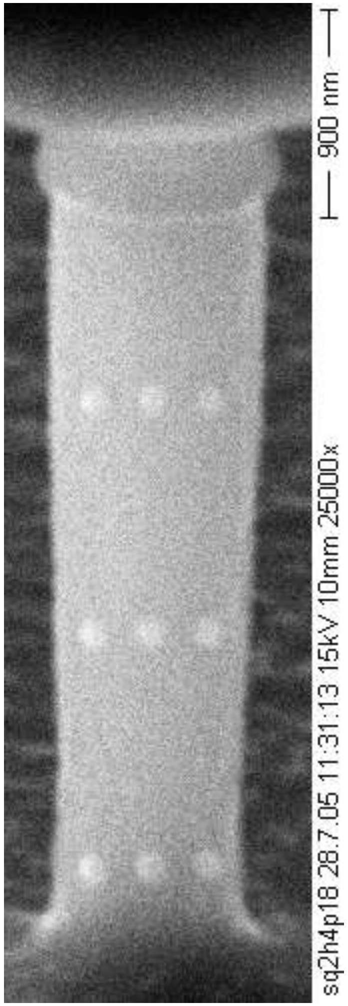
Independent Indenter Tip and Sample heating with feedback loop control thermocouples.

Contact drift tunable to < 0.1 nm/s

fracture of silicon micropillars



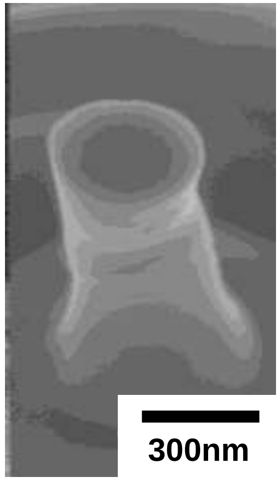




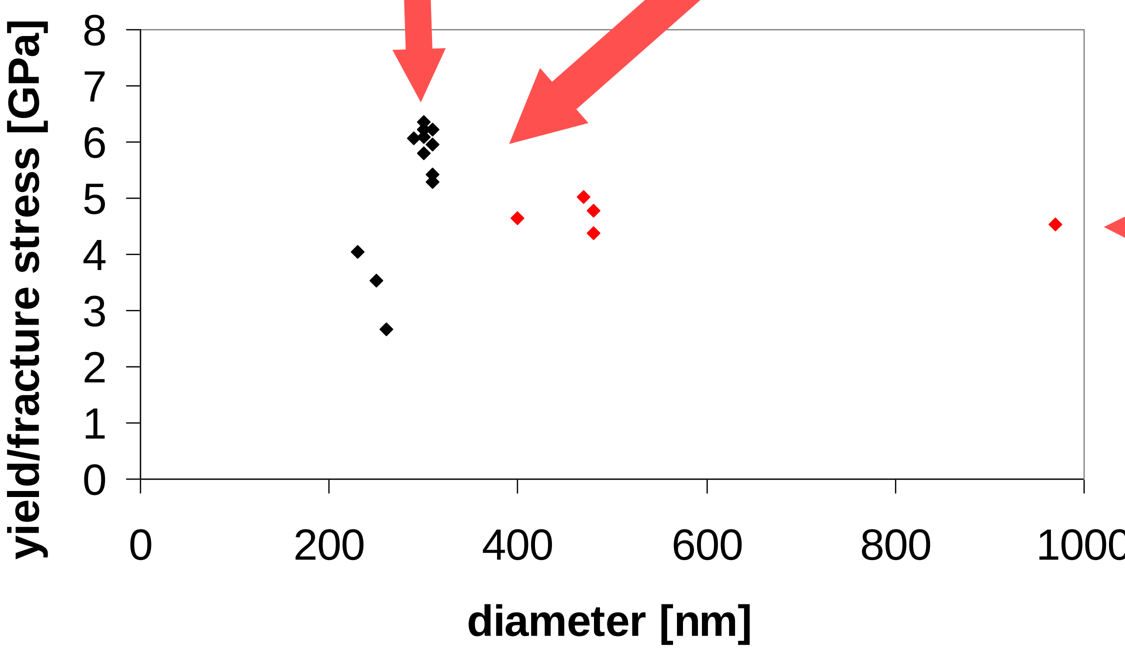
Moser B. et al (2007) J. Mater Res.

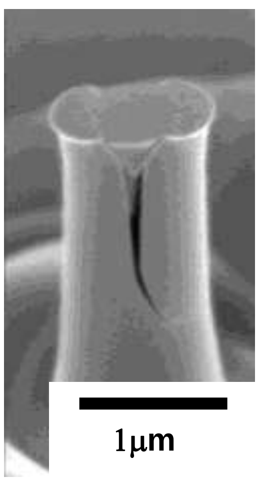
Si (001)

plasticity of silicon in uniaxial compression



ductile – brittle transition

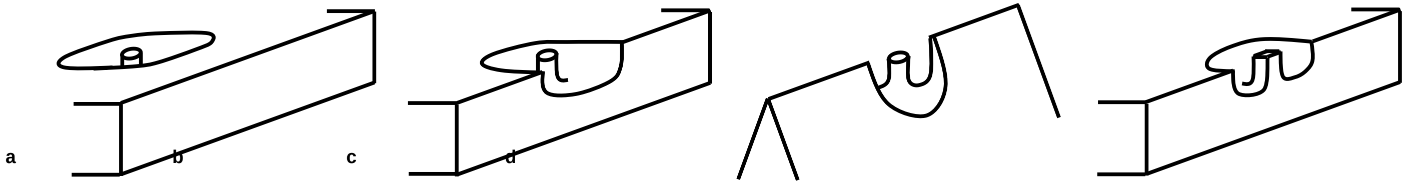




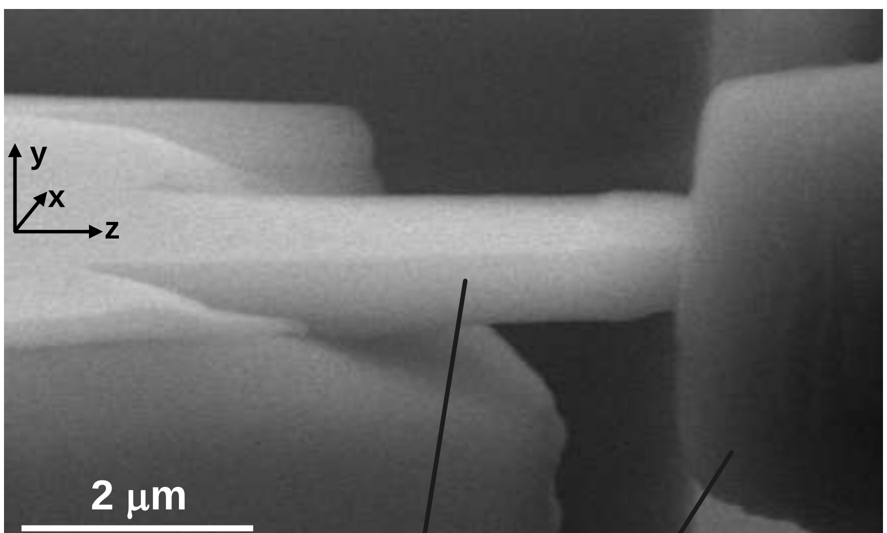
Oestlund (2009) Adv. Functional. Mater.,

in-situ EBSD compression setup

sample preparation

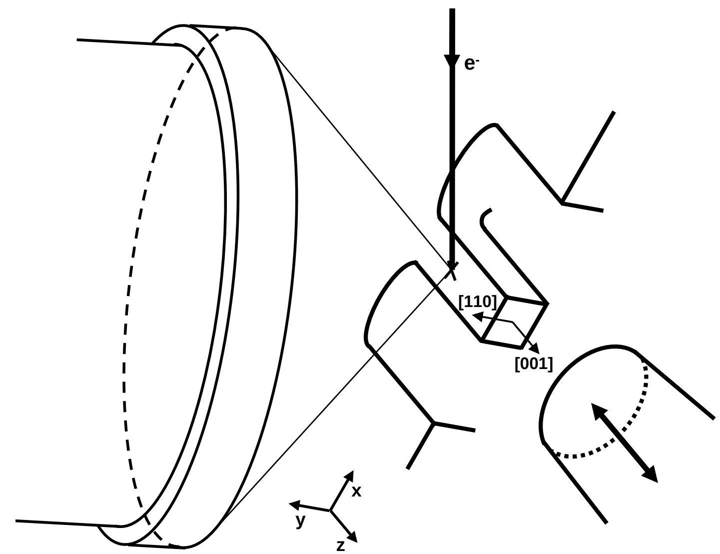


EBSD geometry

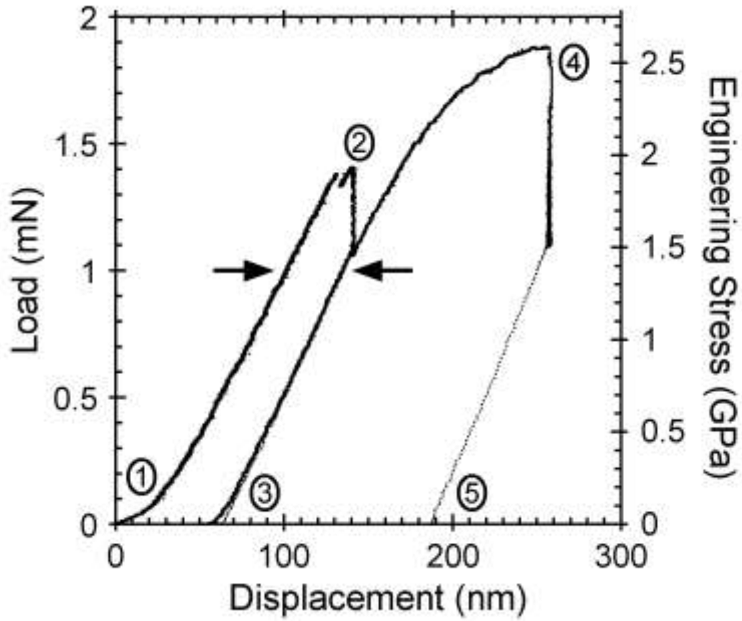


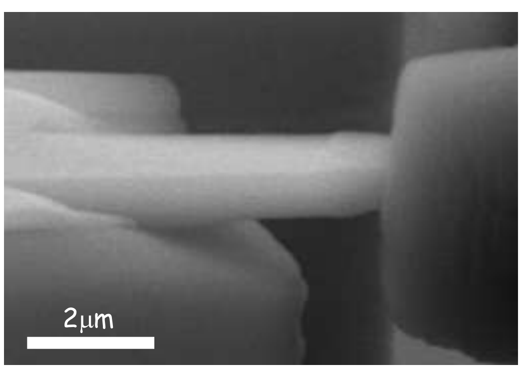
micropillar

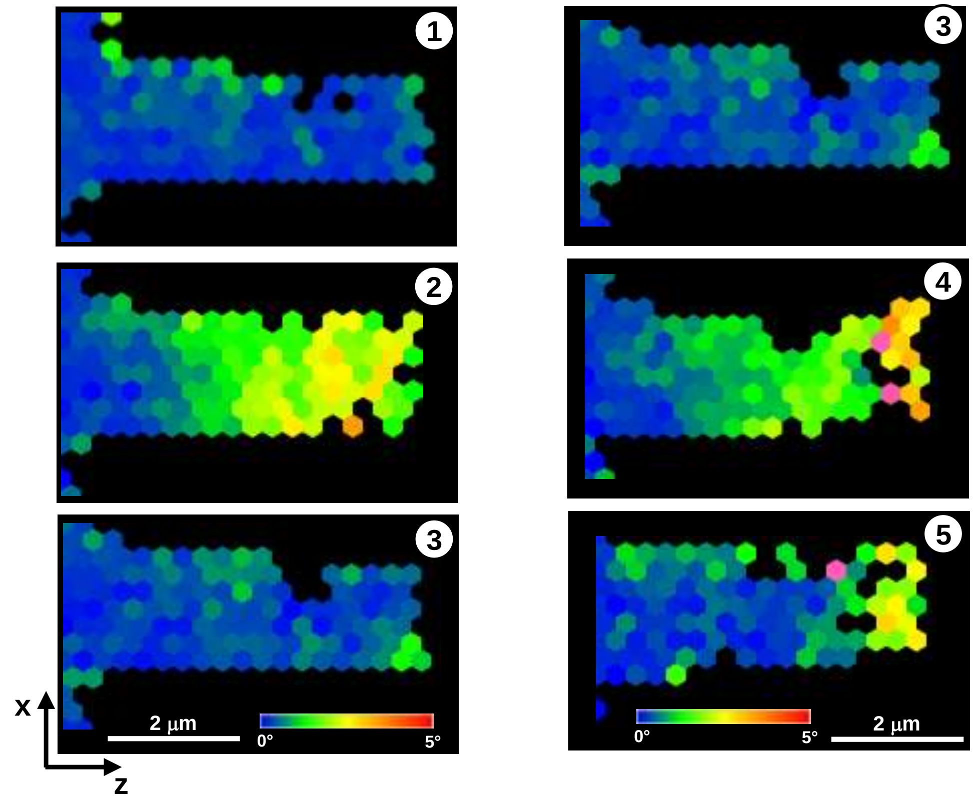
flat punch
indenter



in-situ EBSD compression of GaAs







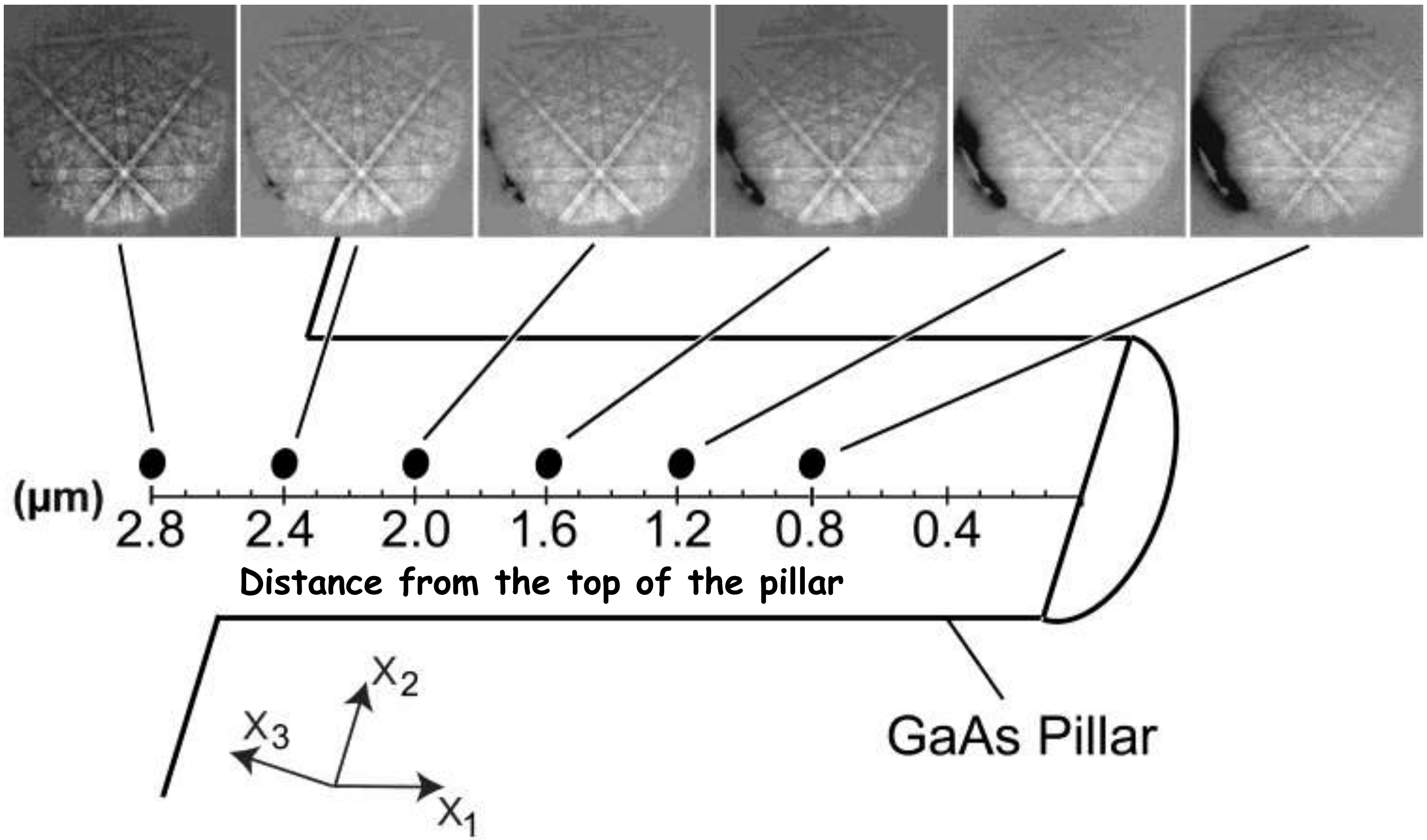
reversal elastic bending

remaining orientation changes

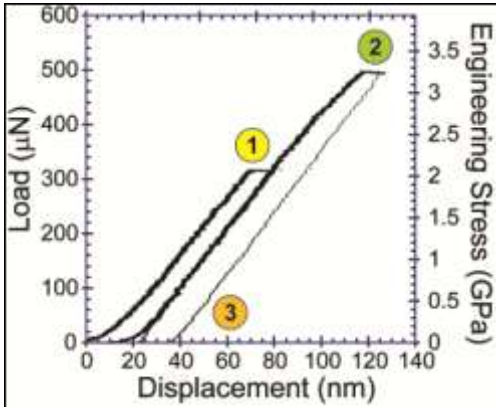
Niederberger et al.,(2010) J. Mat.Sci. Eng. A

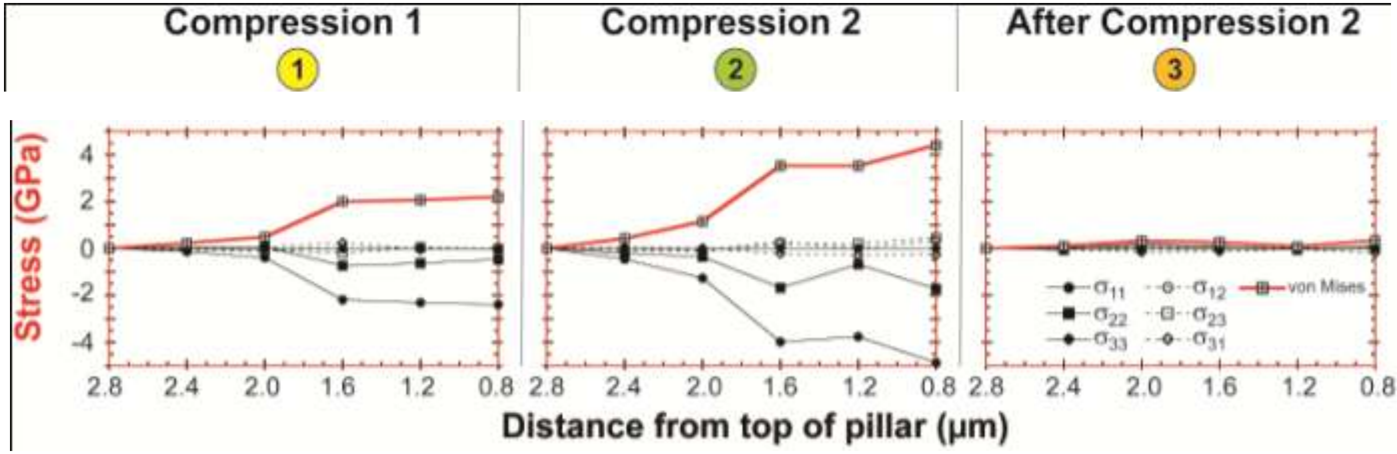
Strain / stress measurement during compression experiment

High resolution EBSD pattern recorded along the pillar

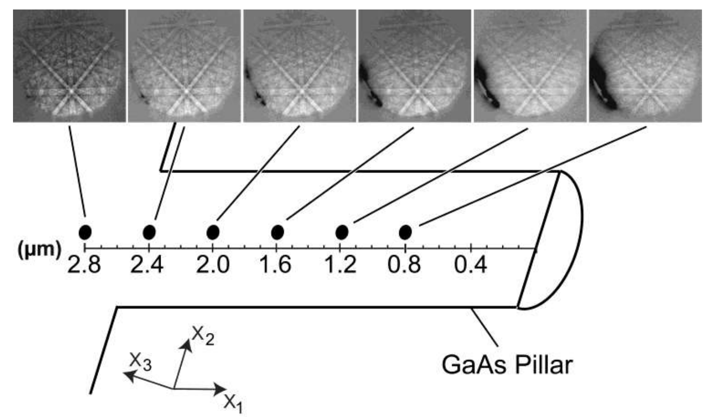


strain / stress measurement during compression





Measures	EBSD cross-correlation ε_{11} [%]	σ_{11}[GPa]	PicoIndenter® engineering stress [GPa]
1	1.66	2.06	2.07
2	2.79	3.42	3.25
3	0.05	0.09	0

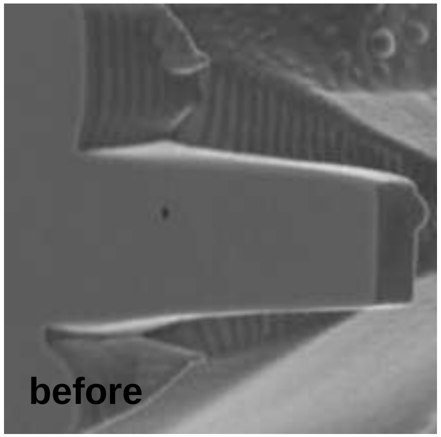


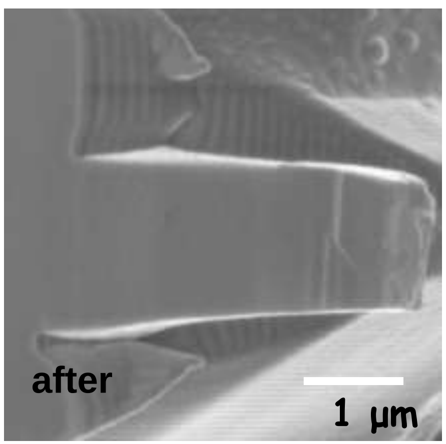
- EBSD stress values compare well with the engineering stress
- Off-axis strain/stress are small but not negligible (not completely uniaxial)
- In particular, at pillar root and flat punch indenter non-uniaxial stress components

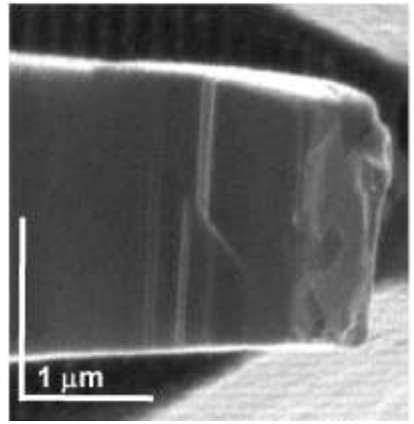
Crosscourt from BLG Productions

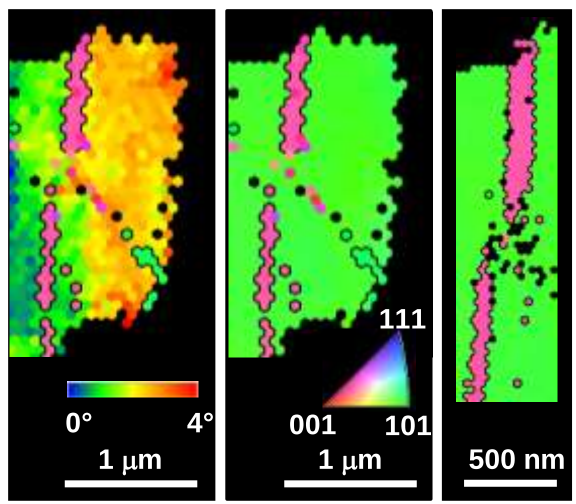
in-situ EBSD compression of GaAs

Deformed microstructure

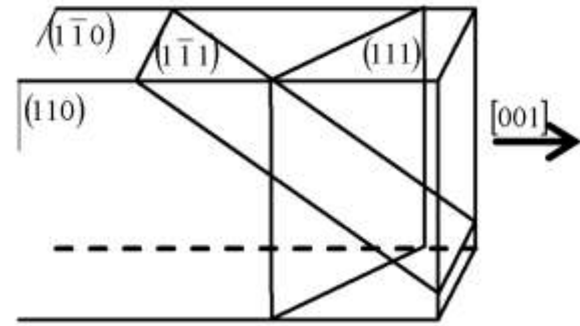




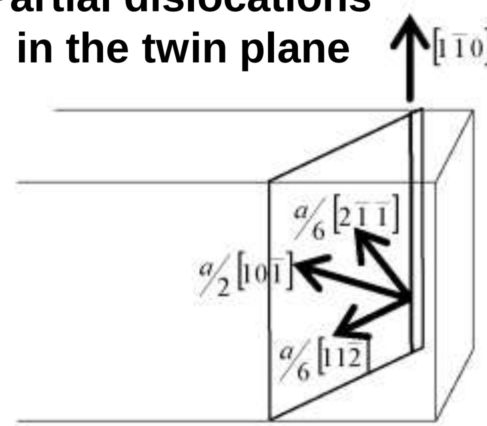




Active Slip Planes



Partial dislocations in the twin plane



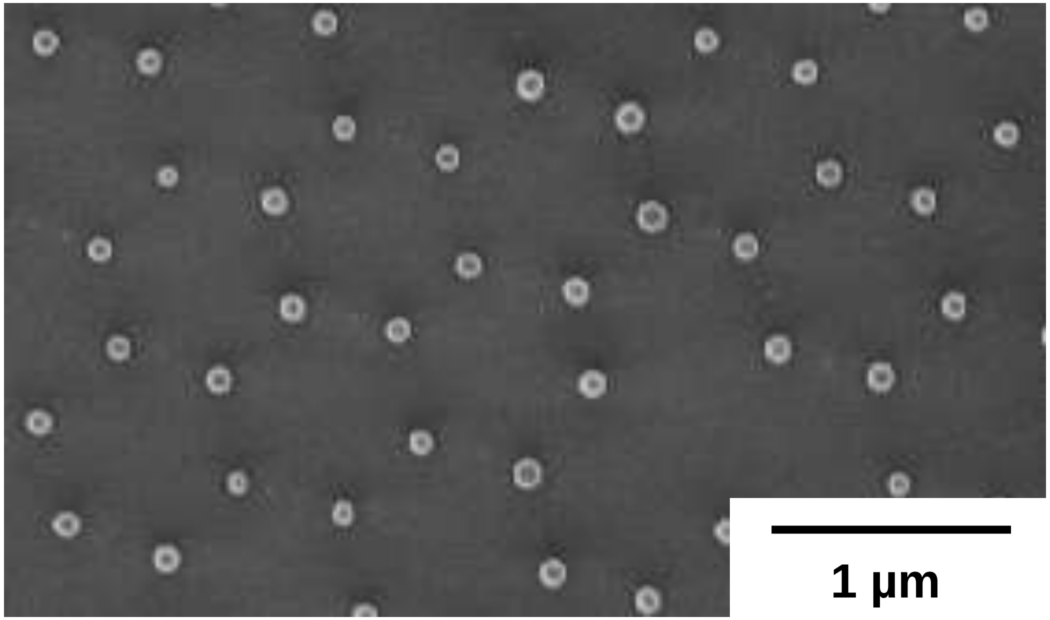
X.J. Ning et al. Phi. Mag. Lett. 74 (1996) 241-245.

- Twinning indicates deformation via individual partial dislocations

ordered arrays of gold dots

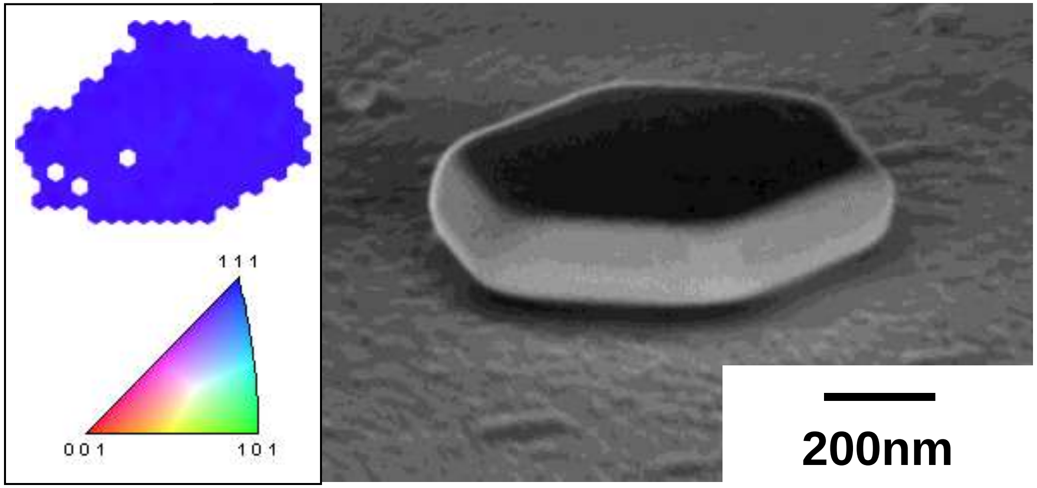
Fabrication steps:

- Colloidal templating
- Au PVD
- annealing at 1000°C/1h



EBSD:

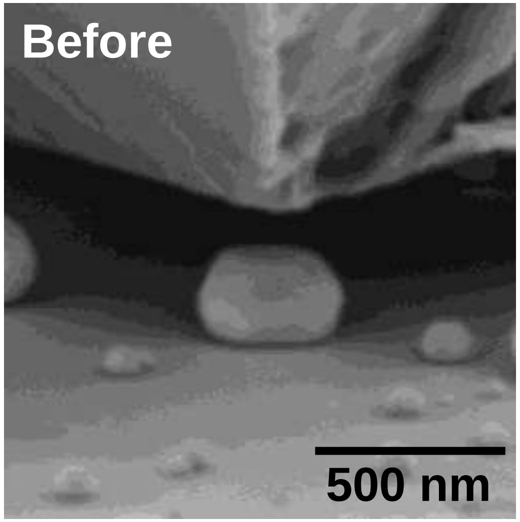
- single crystal <111>

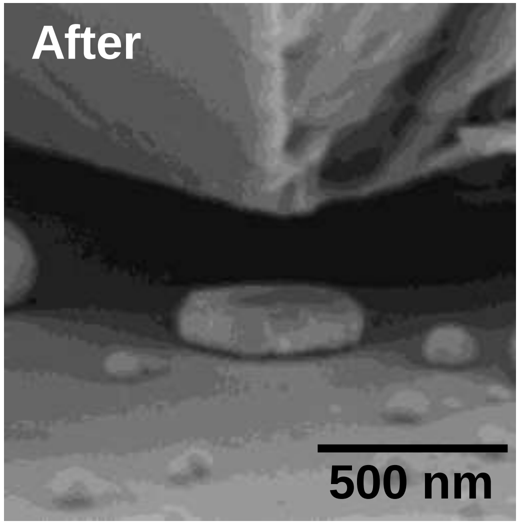


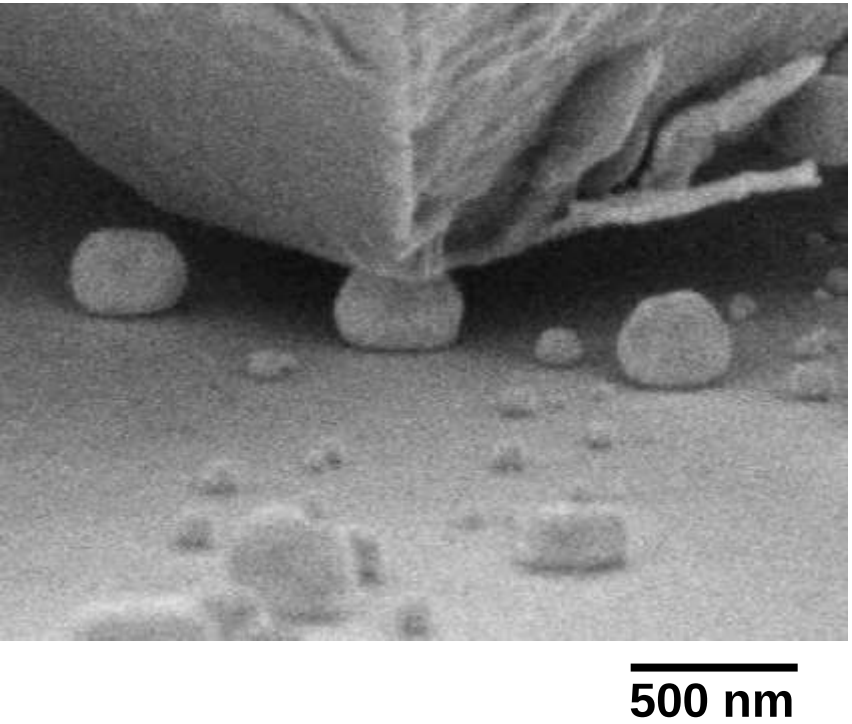
displacement excursions & load drops

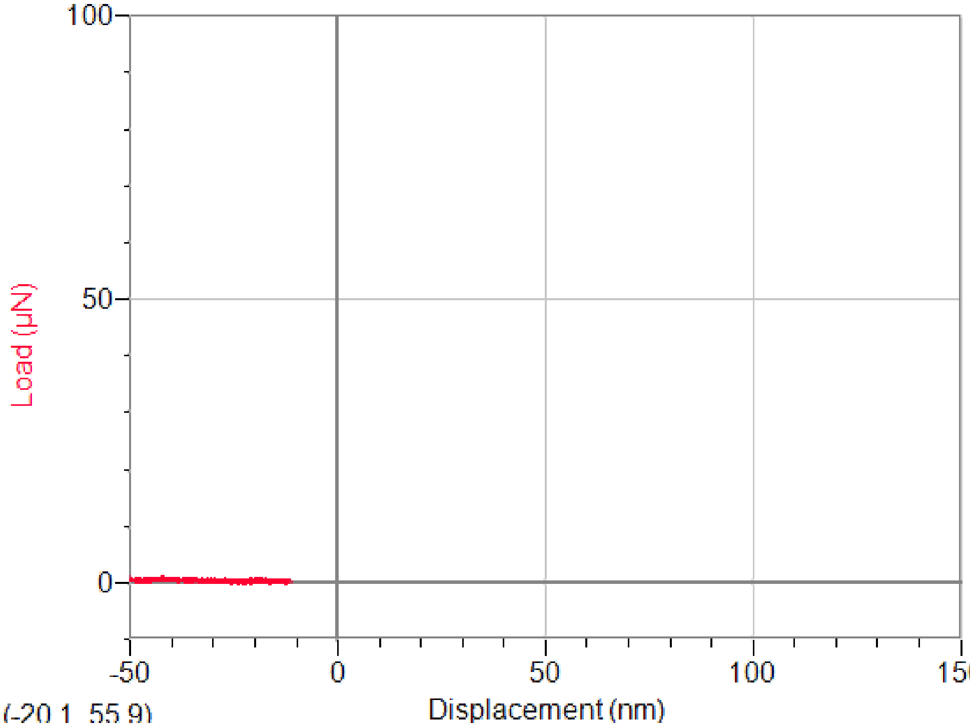
$h_0 = 240$ nm

$r_0 = 150$ nm



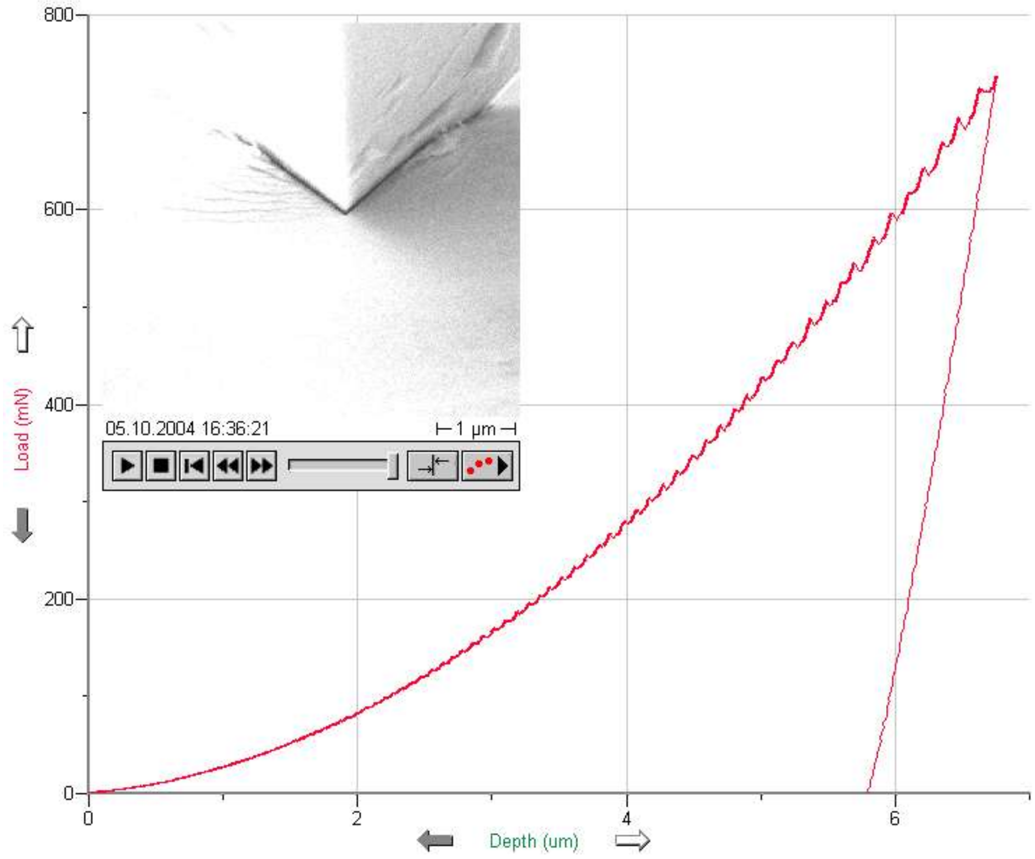






High temperature plasticity of GaN microprisms and of a-Me

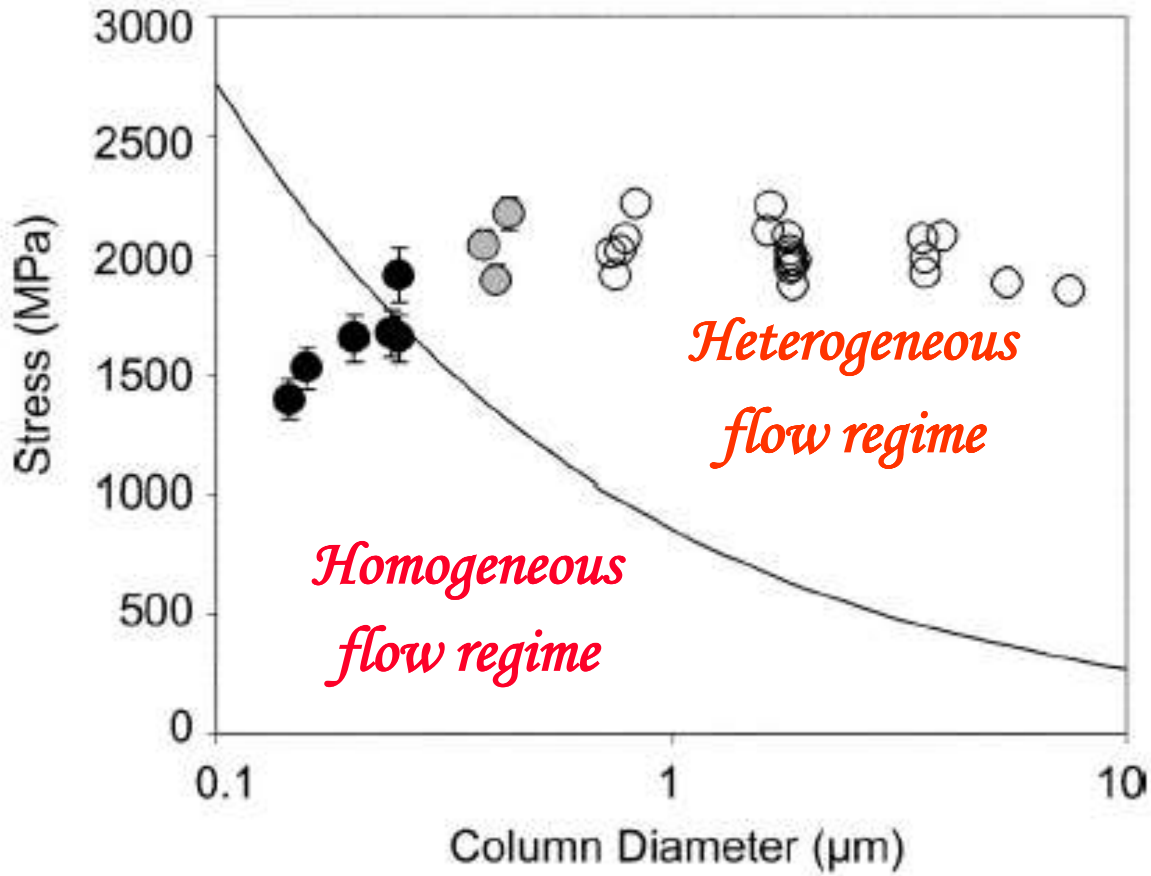
in-situ SEM indentation in Vitreloy-105



$Zr_{52.5}Ti_5Cu_{17.9}Ni_{14.6}Al_{10}$

B. Moser (2005) **Adv. Eng. Mater. 7,** 388-392

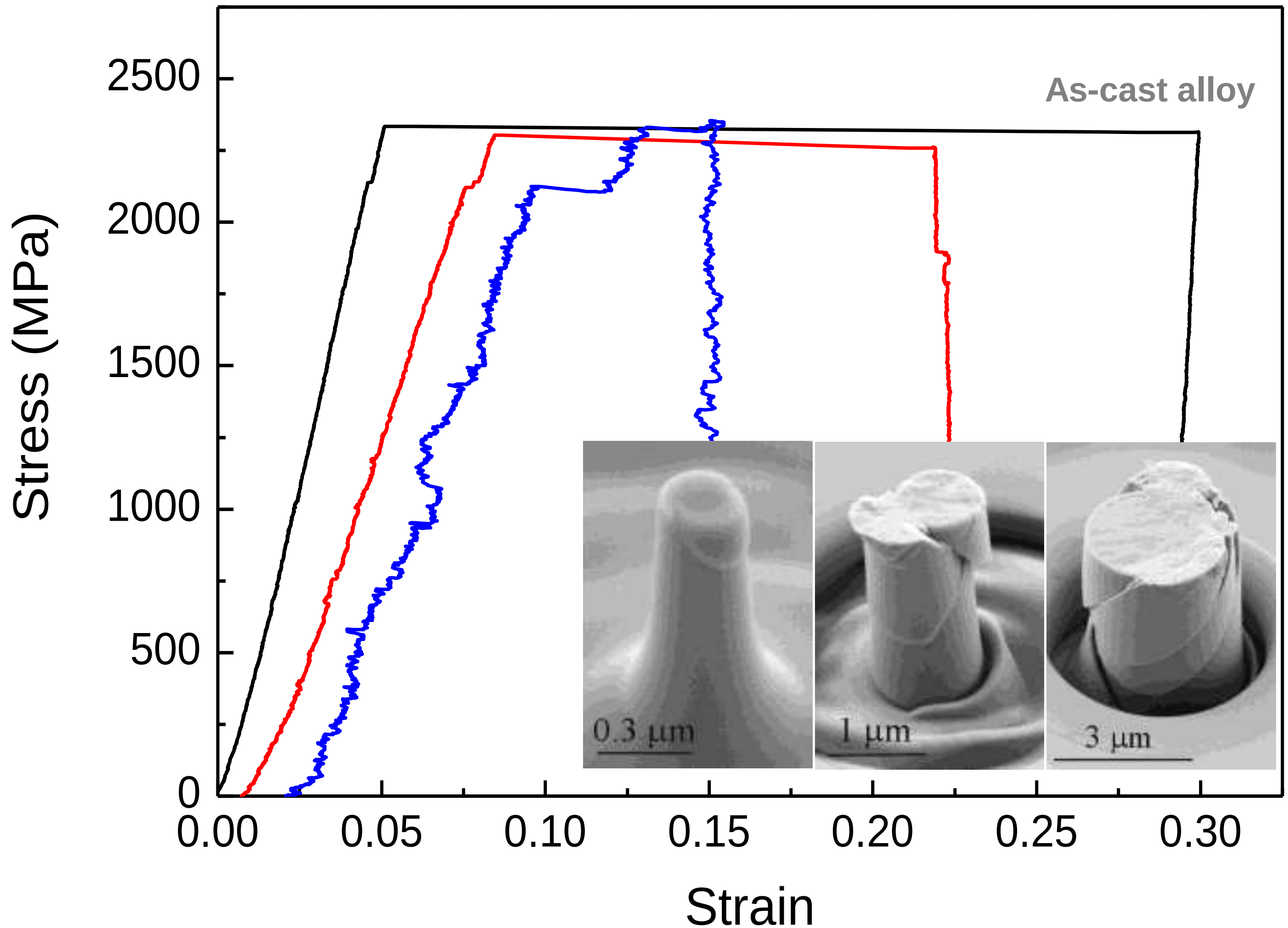
effect of free volume?



C. Volkert, JAP, 2008

How does free volume influence such transition?

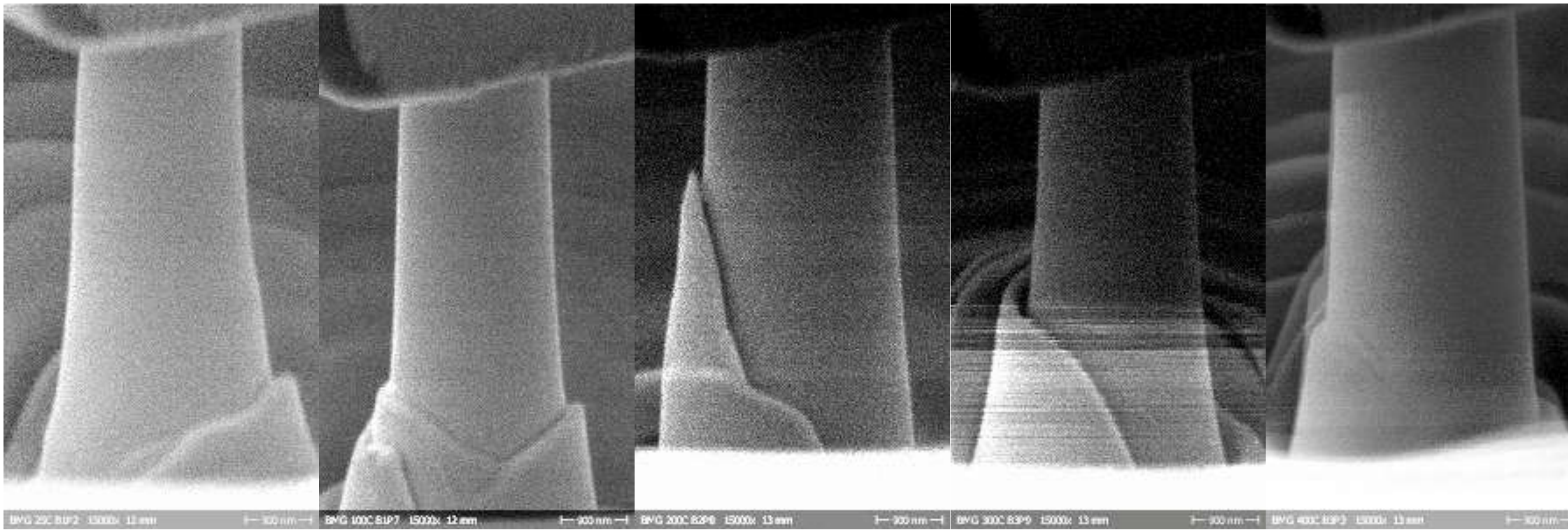
load controlled compression of micropillars



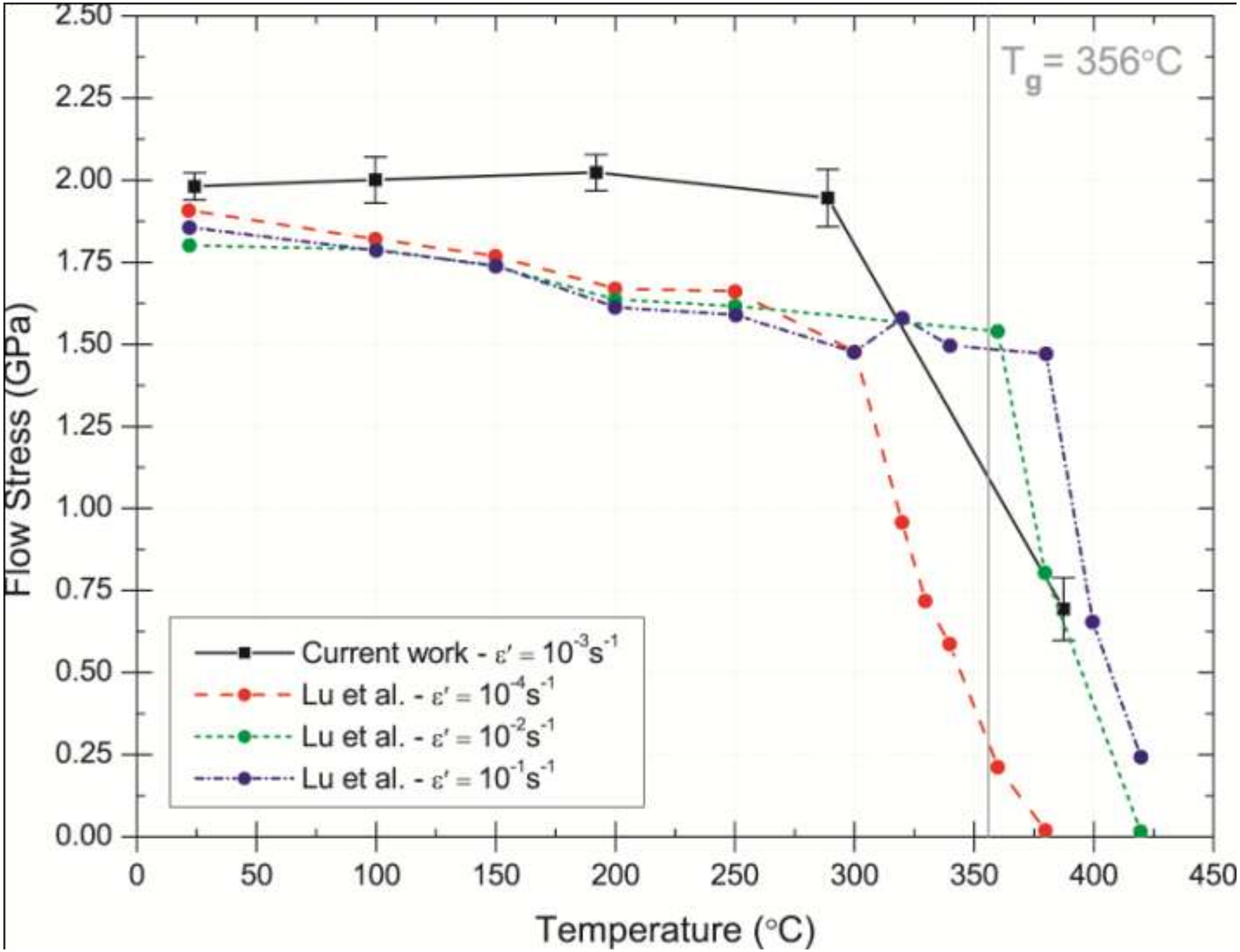
Dubach et al. Scripta Mat. 60, 567 (2009)

In-situ elevated temperature micro-compression

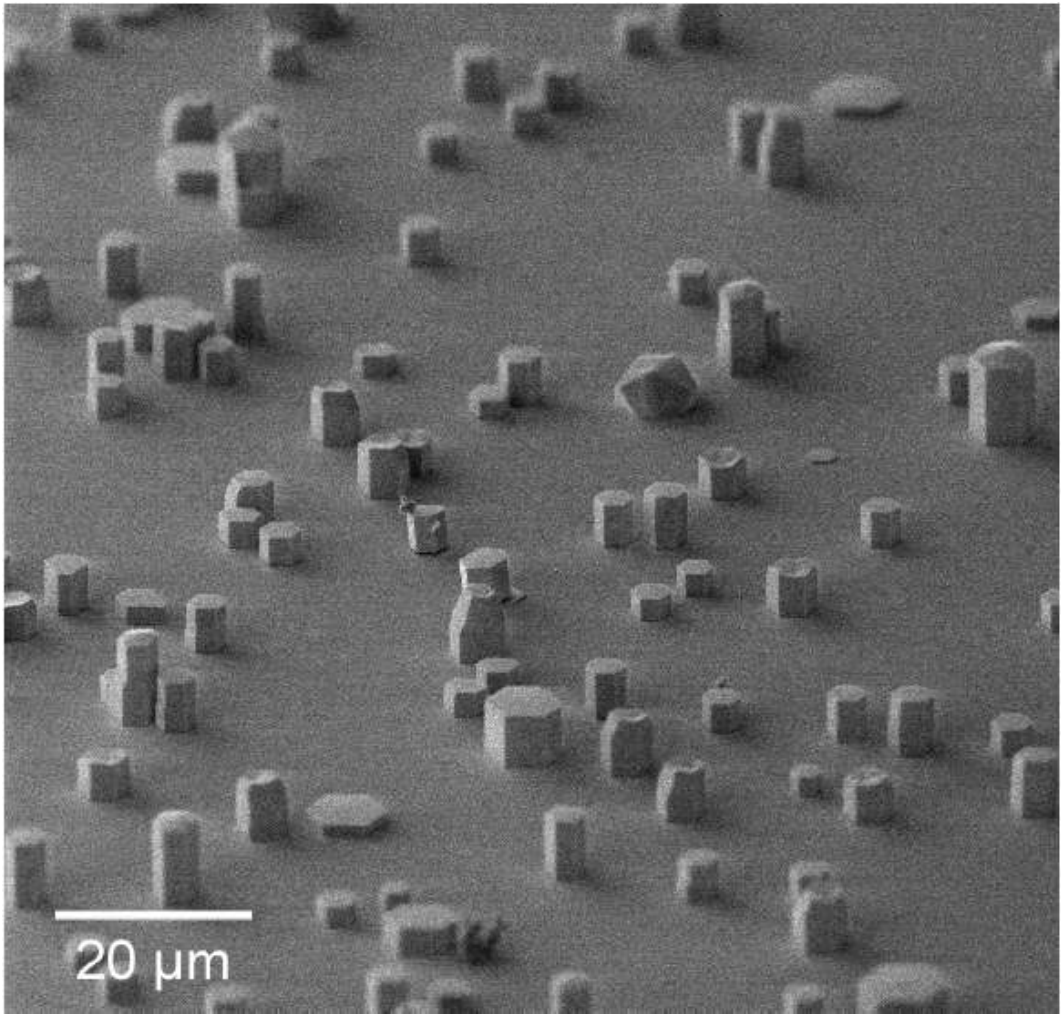
24°C 100°C 192°C 289°C 387°C



Plasticity in a-Me at HT: flow stress

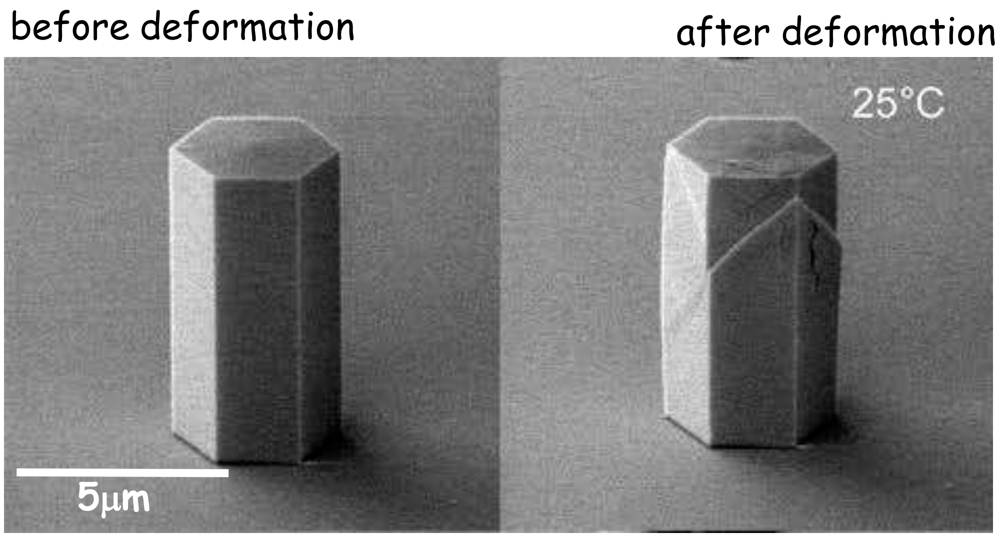


plasticity of GaN



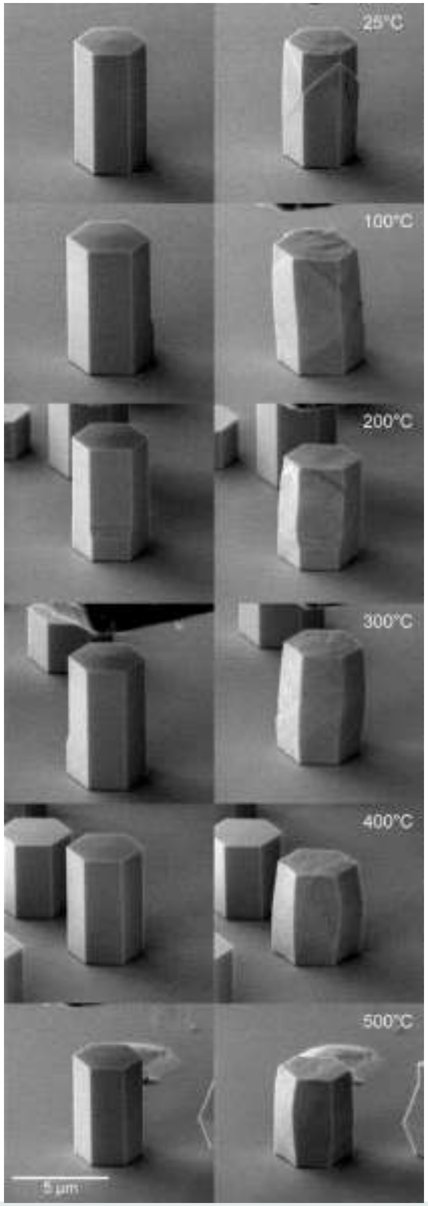
GaN prisms grown by MOVPE on a sapphire substrate,
collaboration with S. Chrsitiansen, MPI Science of the Light, Erlangen, Germany.

plasticity of GaN - 2nd order pyramidal slip

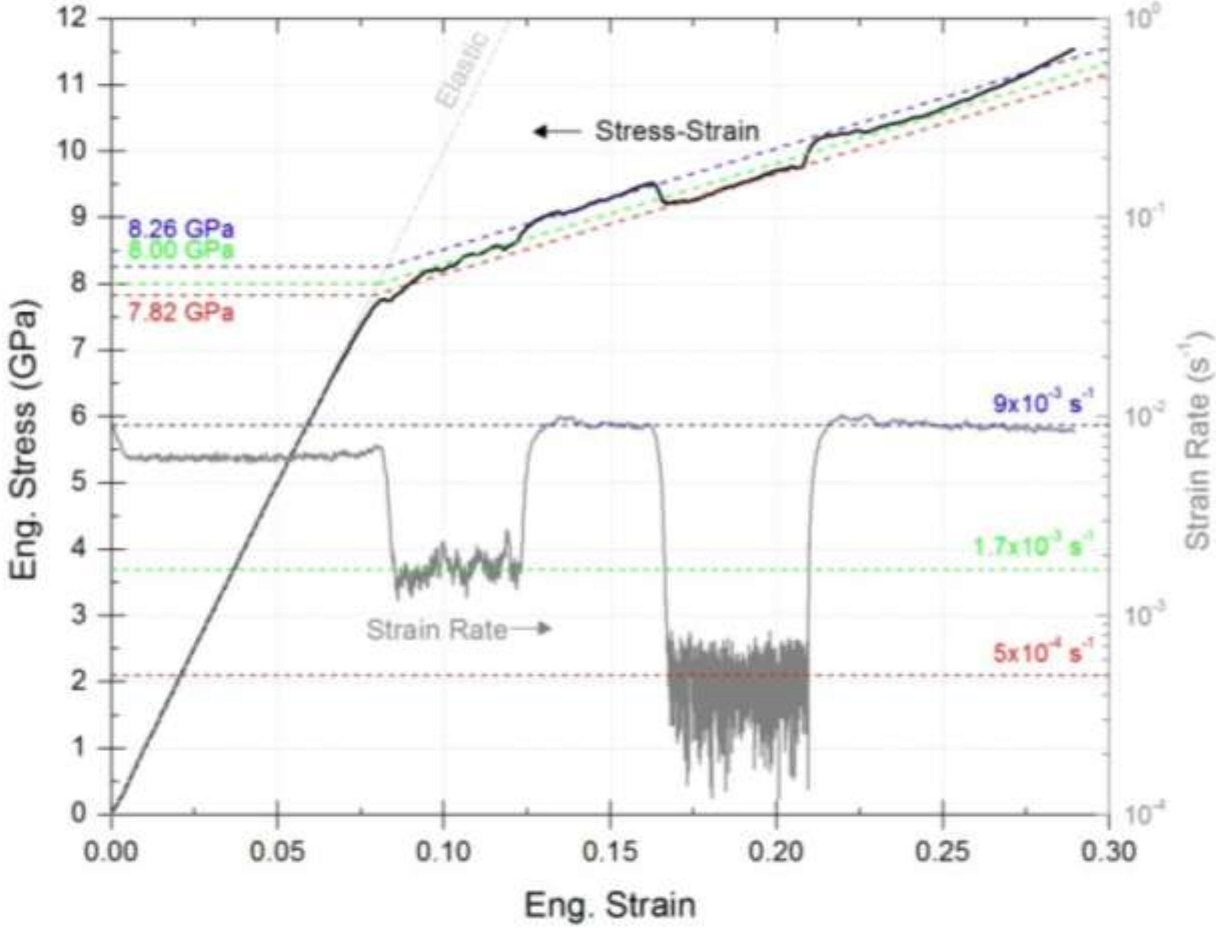


Slip system	Plane angle, ϑ	Direction angle, γ	Schmid factor
$\{11\bar{2}2\}<11\bar{2}3>$	58.41°	47.31°	0.355
$\{1\bar{1}01\}<11\bar{2}3>$	61.96°	47.31°	0.319
$\{1\bar{1}02\}<1\bar{1}01>$	43.19°	61.96°	0.343

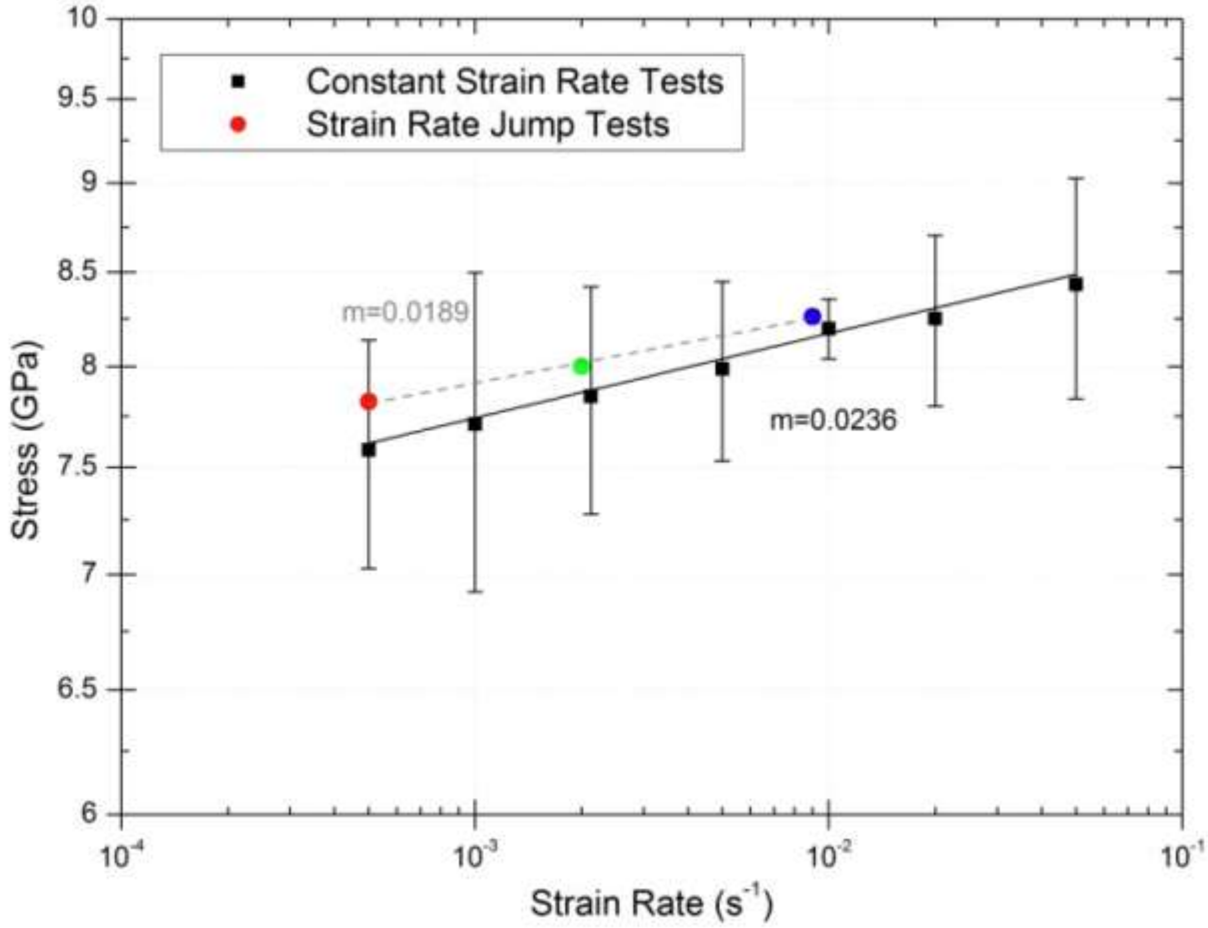
plasticity of GaN



plasticity of GaN - strain rate jump tests



plasticity of GaN – activation volume



the strain rate sensitivity exponent

$$m = \frac{d(\ln \sigma_f)}{d(\ln \dot{\varepsilon})}$$

the activation volume

$$V = \frac{\sqrt{3} k_B T}{m \cdot \sigma_f}$$

this yields an activation volume equal to

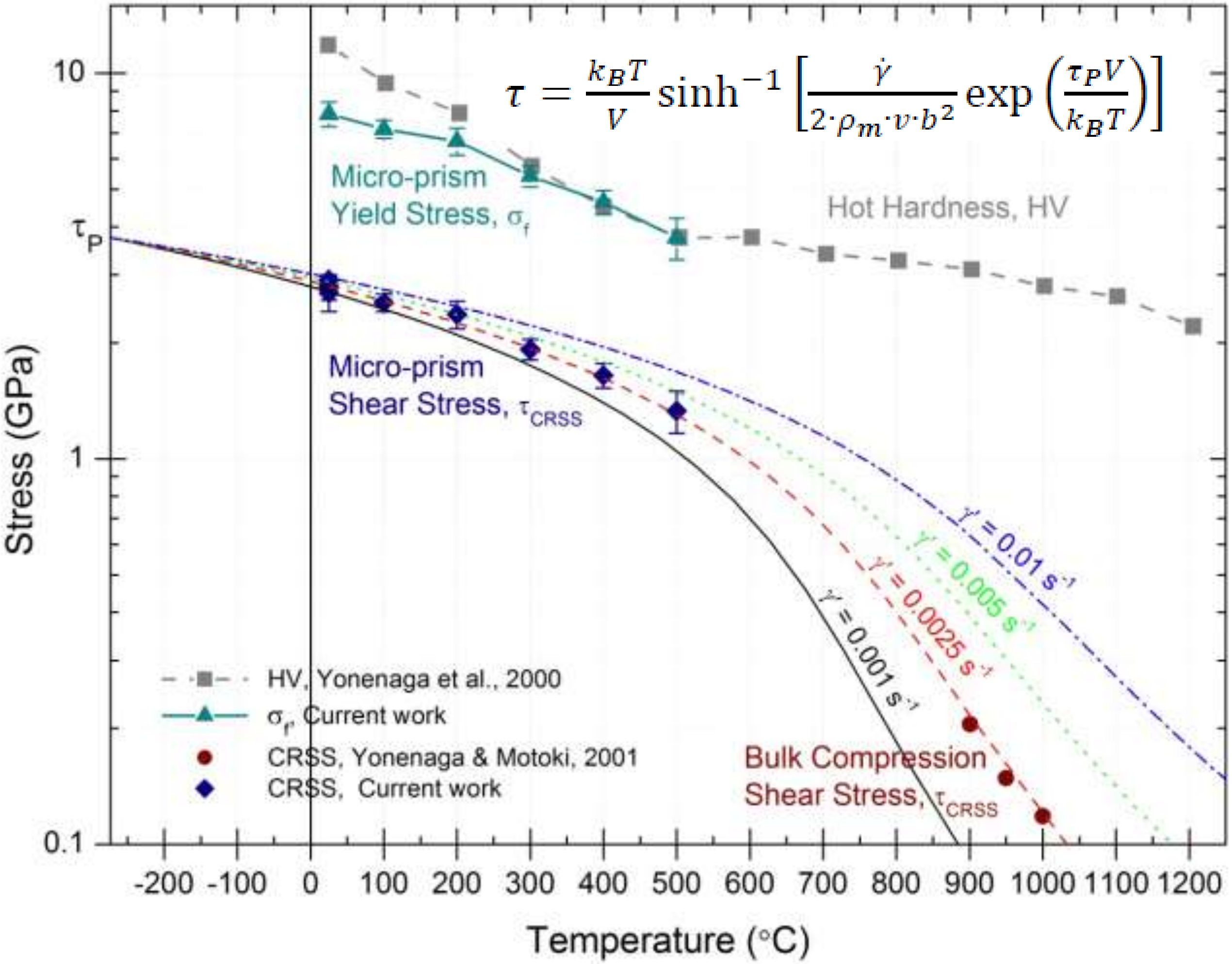
$3.9 \times 10^{-29} m^3$

or

1.2 ± 0.5 *b^3,*

where b is the Burger's vector, 0.3186 nm

plasticity of GaN – glide planes



The Peierl's stress was determined to be 3.75 GPa by linear extrapolation of the critical resolved shear to 0K

$Q = \tau_P V$, the activation energy over this temperature range was determined to be 0.91 ± 0.2 eV.

Problems in the analysis: size effects? Dislocation nucleation versus propagation of dislocations? Initial dislocation density?

Schlussfolgerungen

TOFSIMS im FIB: höhere räumliche Auflösung, molekulare Information, bessere Nachweisgrenzen als EDX

AFM im REM: bequem, nicht blinde Manipulation, Hybridmethoden

EBSD: Neben Kristallorientierung und Kristallstruktur auch innere Spannungen

Nanomechanik: kleiner ist anders, Hochtemperaturversuche

Das REM ist das "Schweizer Messer" der Materialanalytik!

seeing is believing!

2µm

Achknowledgement: Jeff Wheeler, Bill Mook, Rejin Raghavan, Rudy Ghisleni,Sid Pathak, Matthias Schamel, Christophe Niederberger ….

Open post-doc positions at EMPA!
contact johann.michler@empa.ch

GaAs (001)


2μm
GaAs (001)