Recent Advances in Nanoscale Strain Mapping using 4D STEM

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ÖAW ÖSTERREICHISCHE AKADEMIE DER WISSENSCHAFTEN

Outline

- **4D-STEM**

  - Nanodiffraction strain mapping

- **In situ deformation**

  - Strain mapping during *in situ* deformation
  - Measuring electrical properties during deformation

- **Metallic glasses**

  - Strain mapping in amorphous materials
  - Strain field of a shear band

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4D-STEM

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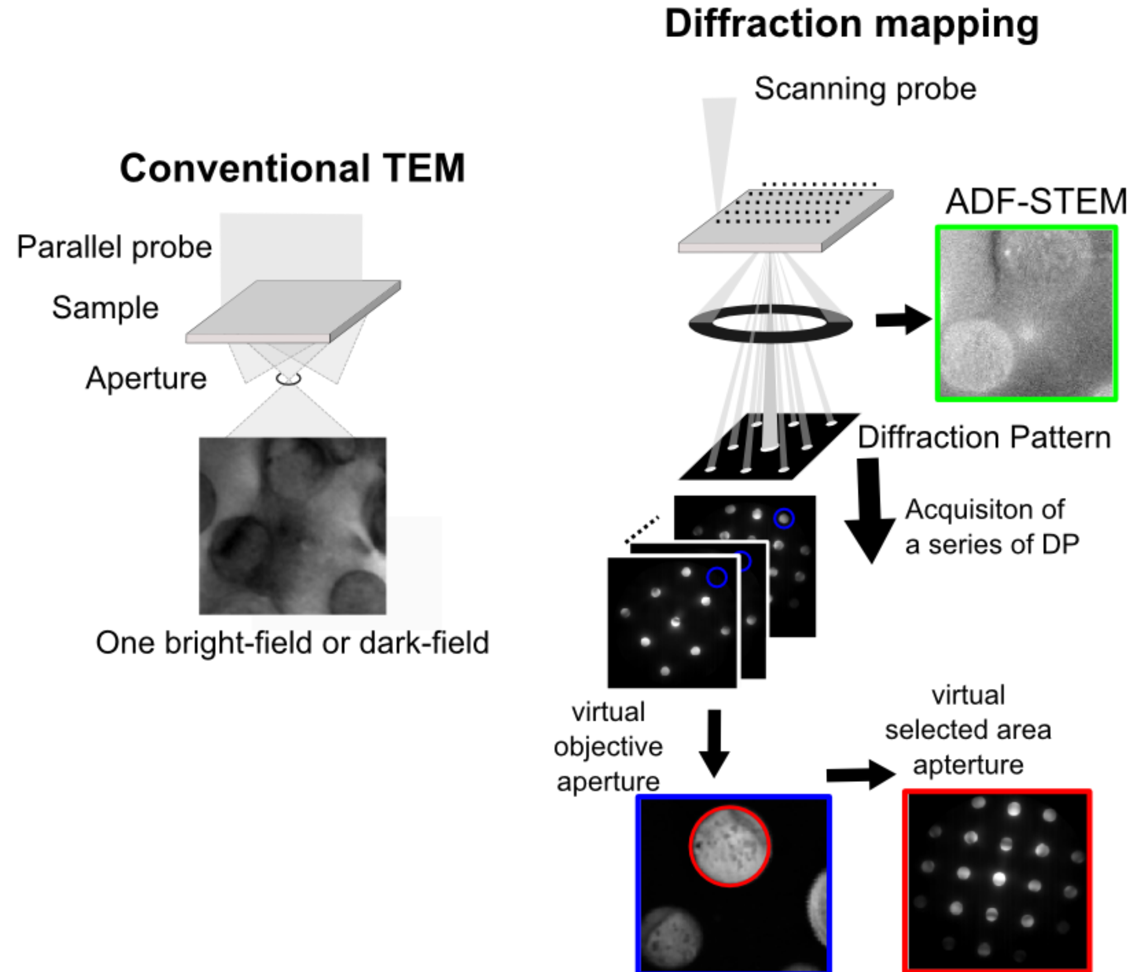
Experimental setup



Collect maximum available information and extract desired information afterwards

C. Gammer, V. B. Ozdol, C. H. Liebscher, A. M. Minor. Ultramicroscopy 155, 1 (2015).

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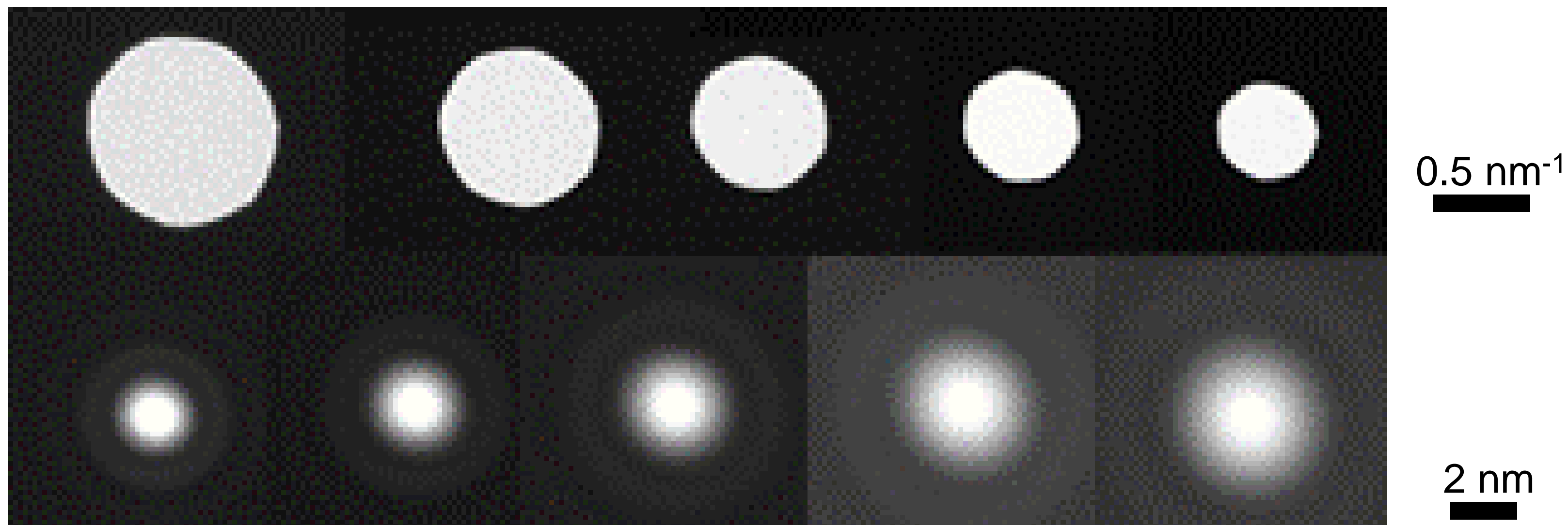
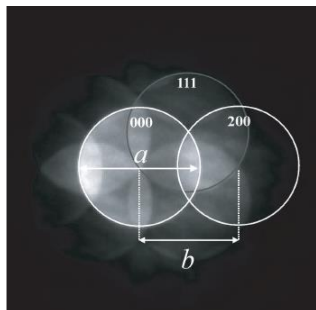
Convergence angle



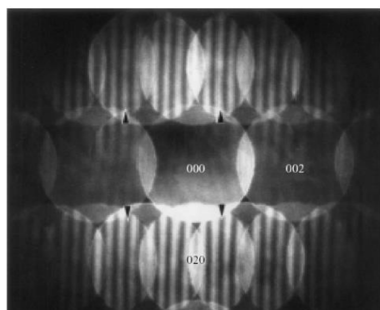
JEOL 2100F

High resolution → Nanoscale information

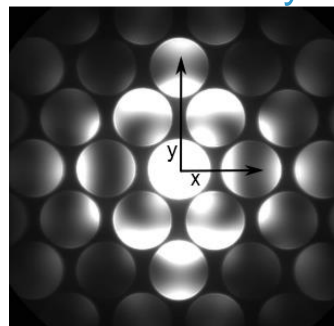
Interference → Easily detectable

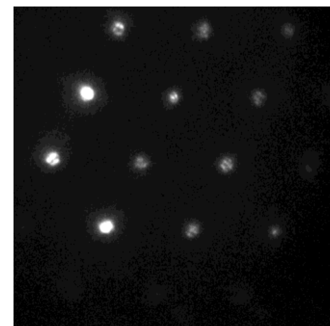


M.Weyland and D.A. Muller. Tuning the convergence angle for optimum STEM performance



John C H Spence. Reports on Progress in Physics 69, 3





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Nanodiffraction strain mapping

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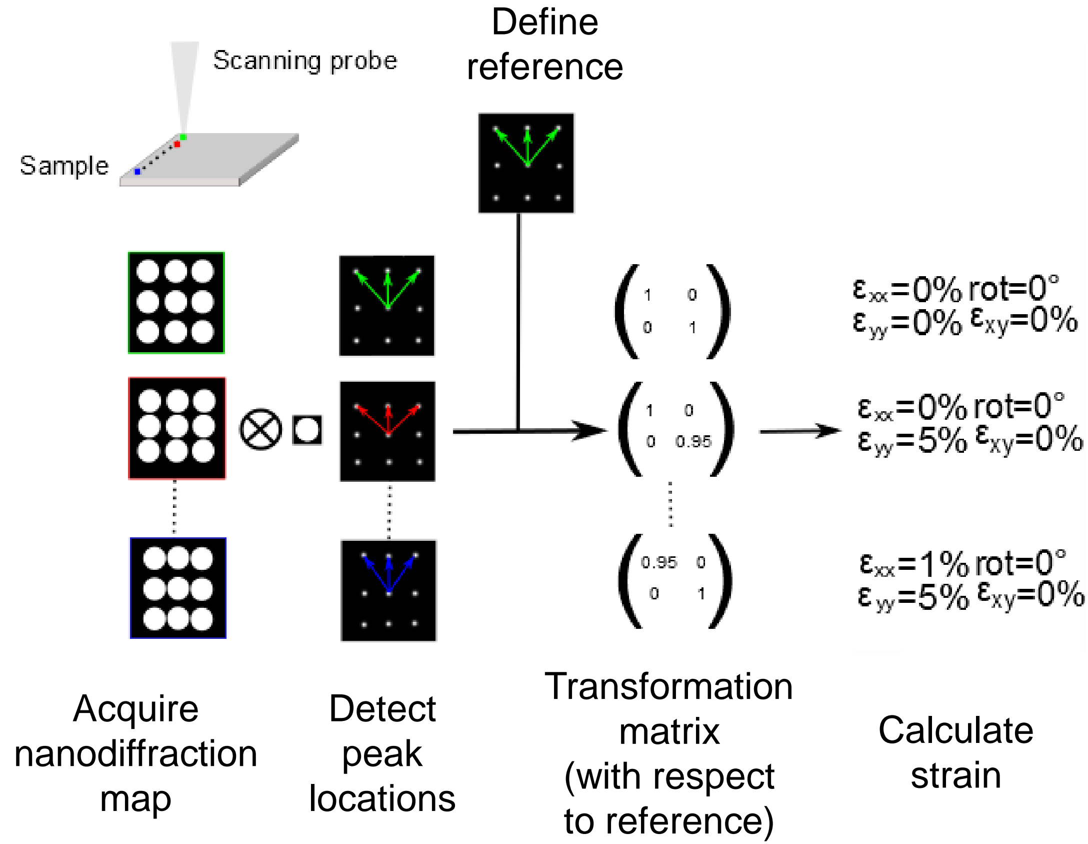
Nanodiffraction strain mapping



Use **all peak positions**
correlation value (confidence for peak location) used to weigh peaks
→ **very precise and robust results**

V. B. Ozdol, C. Gammer, X.G. Jin, P. Ercius, C. Ophus, J. Ciston and A.M. Minor.
Applied Physics Letters 106, 253107 (2015).

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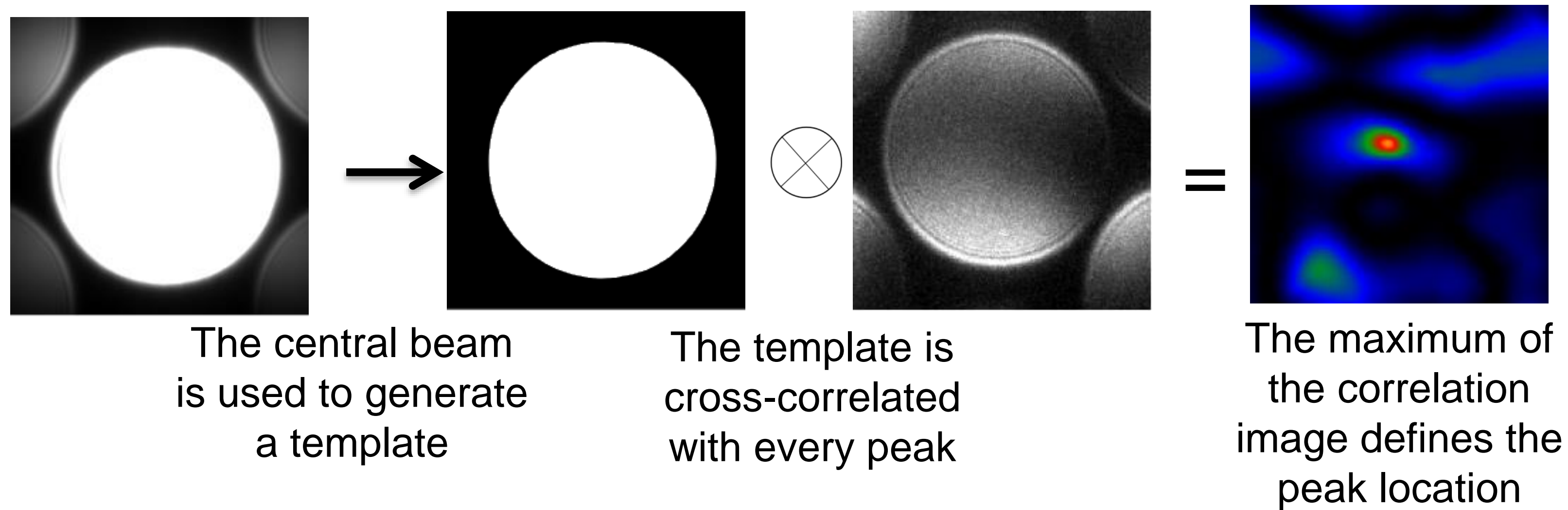
Peak detection



Cross correlation is a fast method to detect the peak position with sub-pixel precision

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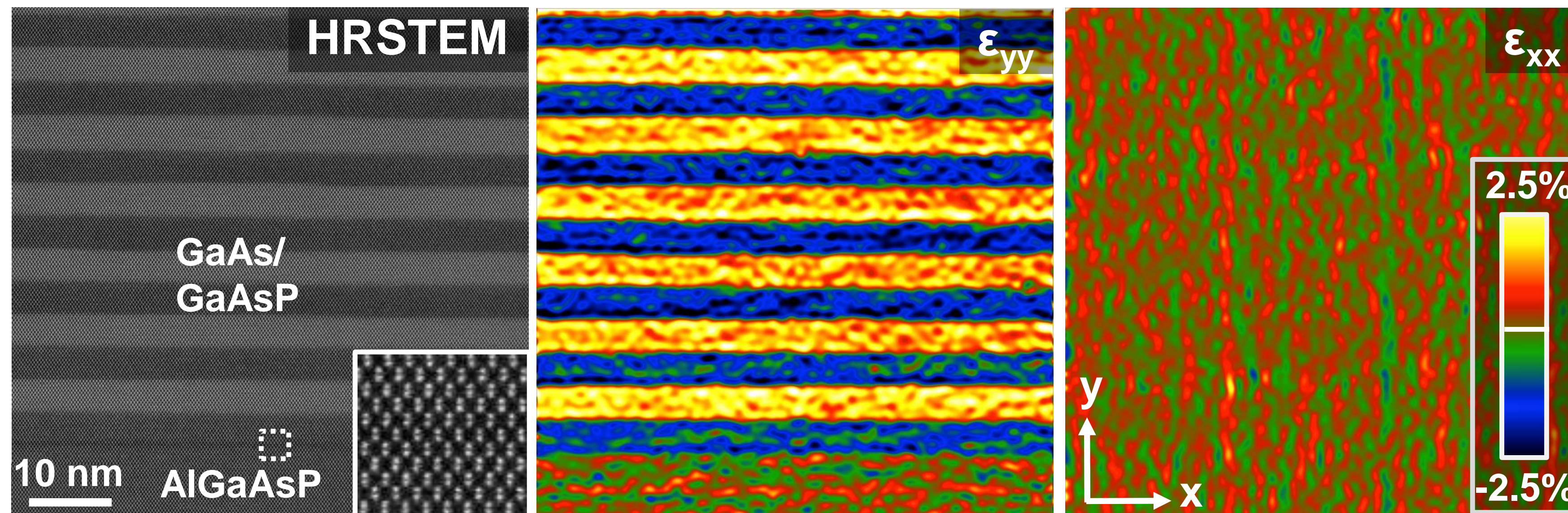
Atomic resolution STEM

GaAs/GaAsP multilayer



Actual photocathode device consists of 90 pairs of layers (~4 nm)

V. B. Ozdol, C. Gammer, X.G. Jin, P. Ercius, C. Ophus, J. Ciston and A.M. Minor.
Applied Physics Letters 106, 253107 (2015).

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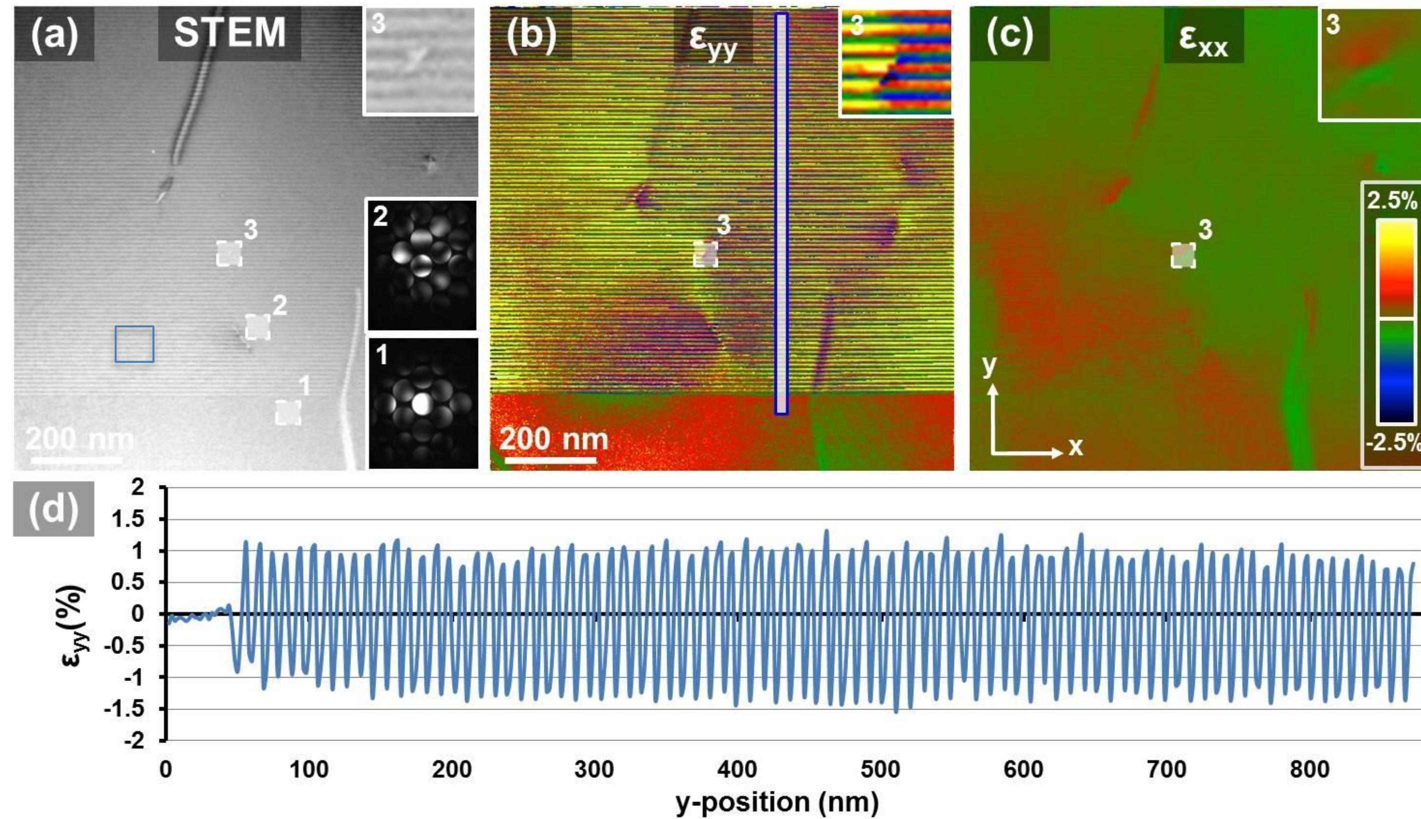
Large field of view

The actual device photocathode device consisting of 90 pairs of layers and contains dislocations, impossible to measure with other techniques.



Diffraction map 512x512 consisting of more that **260,000** diffraction patterns (>1TB data)

Strain map with a field of view >1µm at <2nm resolution and <10^{-3} precision

V. B. Ozdol, C. Gammer, X.G. Jin, P. Ercius, C. Ophus, J. Ciston and A.M. Minor. Applied Physics Letters 106, 253107 (2015).

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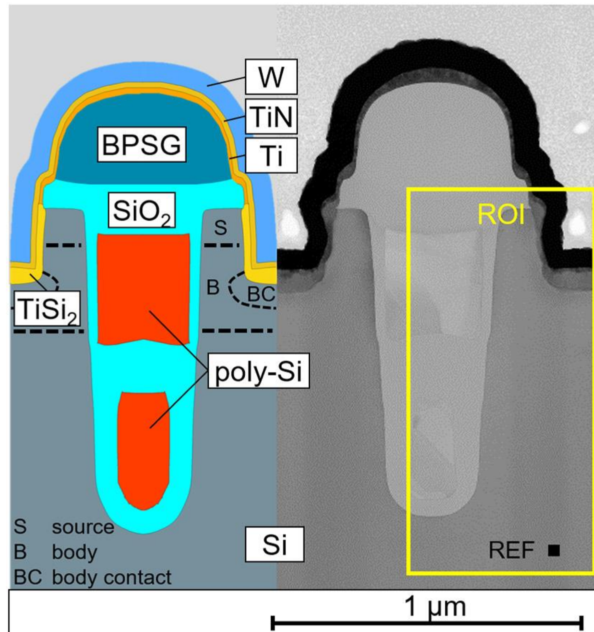
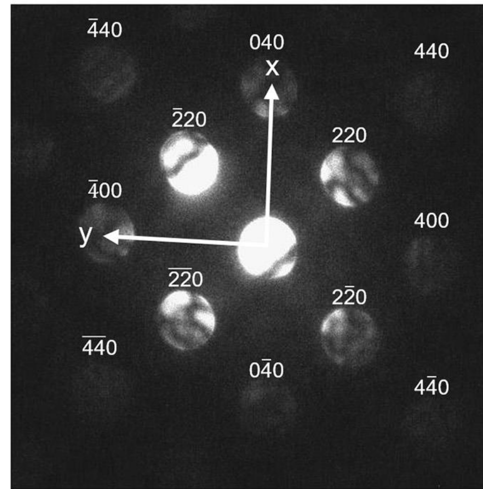
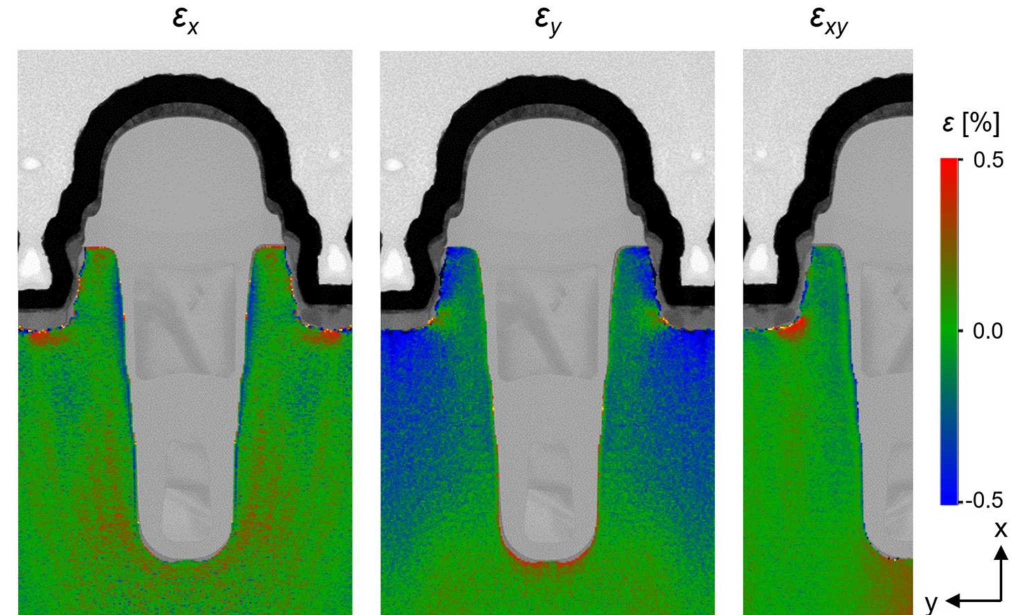
Strain in trench power MOSFETs







Complex strain fields in actual devices can be measured.

Stefan Karner, Oliver Blank, Maximilian Rösch, Jakub Zalesak, Jozef Keckes, Christoph Gammer.
Microelectronic Engineering 264,111870 (2022).

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In situ strain mapping

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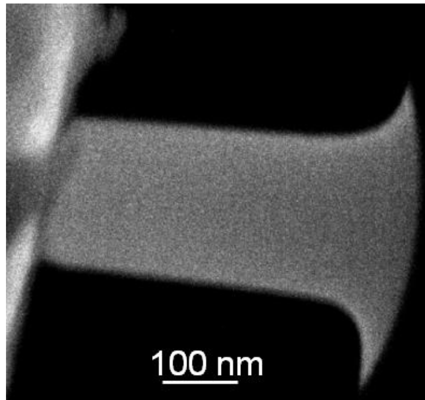
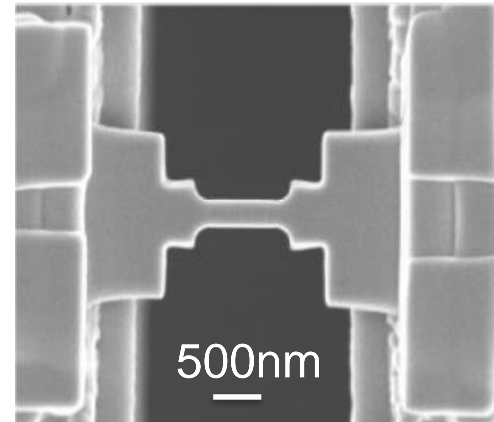
In situ TEM deformation

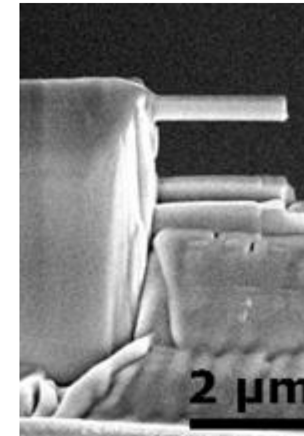
Compression



Tension



Bending



Well defined deformation geometries in combination with modern deformation holders
→Quantitative stress strain curves correlated with real-time videos

Bruker Hysitron PI 95 picoindenter

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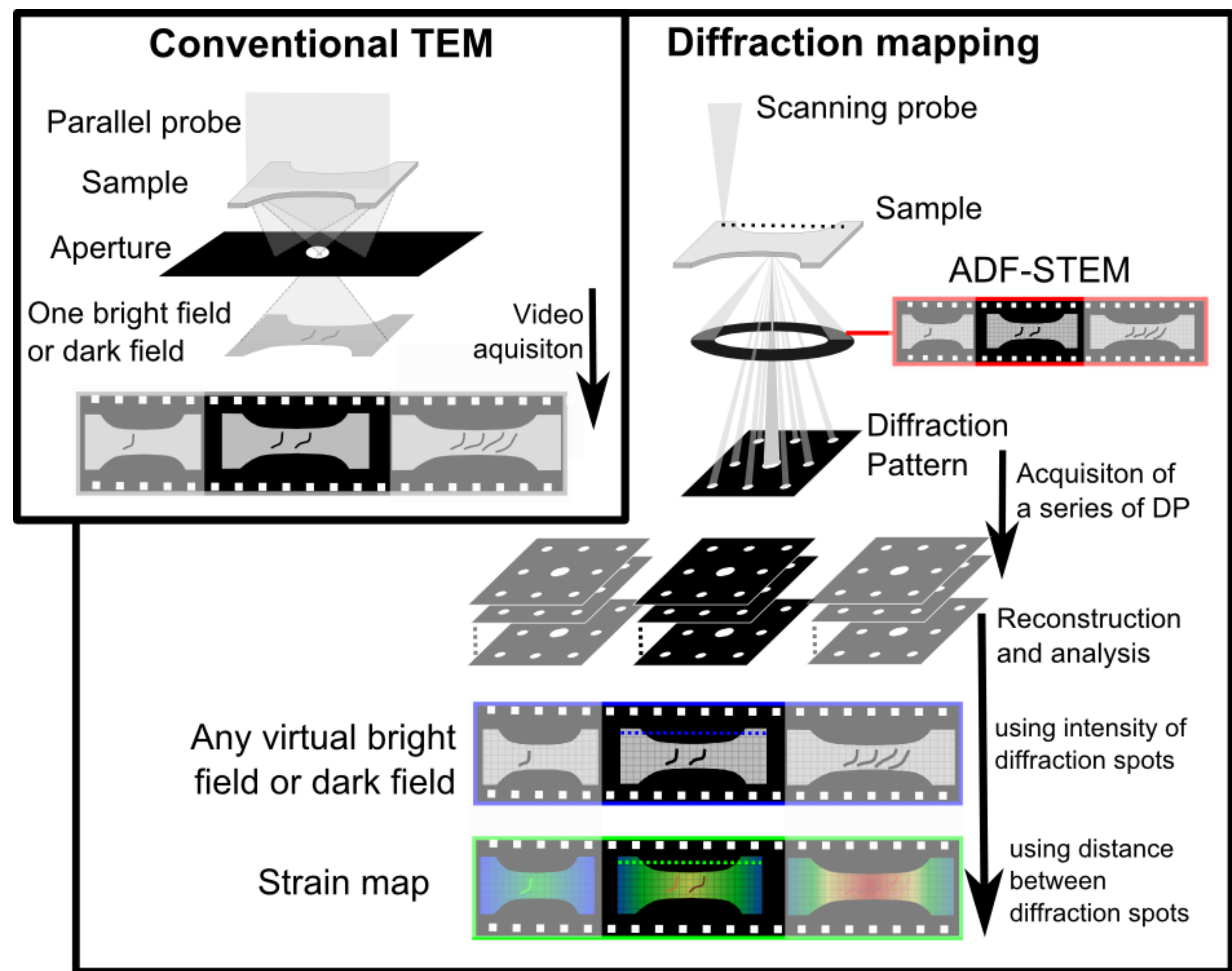
ESI

In situ nanodiffraction mapping



A series of diffraction patterns is recorded for every step in time.

Possible by using ultrafast electron detectors

C. Gammer, J. Kacher, C. Czarnik, O.L. Warren, J. Ciston, A.M. Minor,
Applied Physics Letters, 109:081906, 2016.

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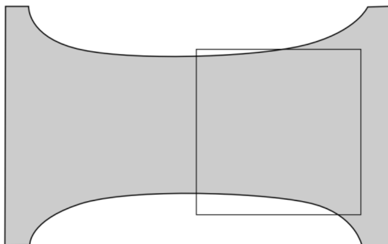
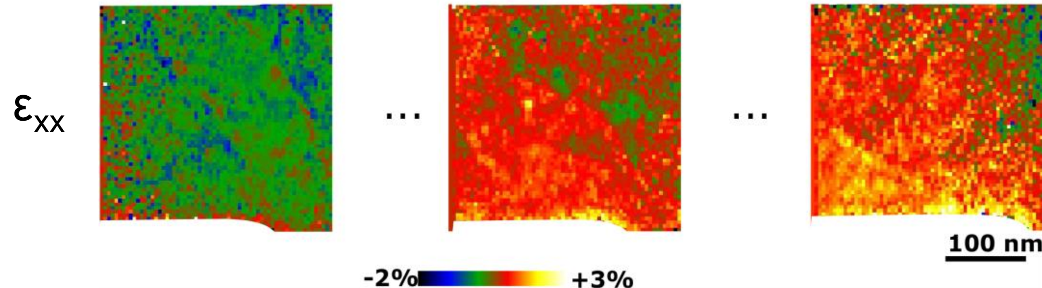
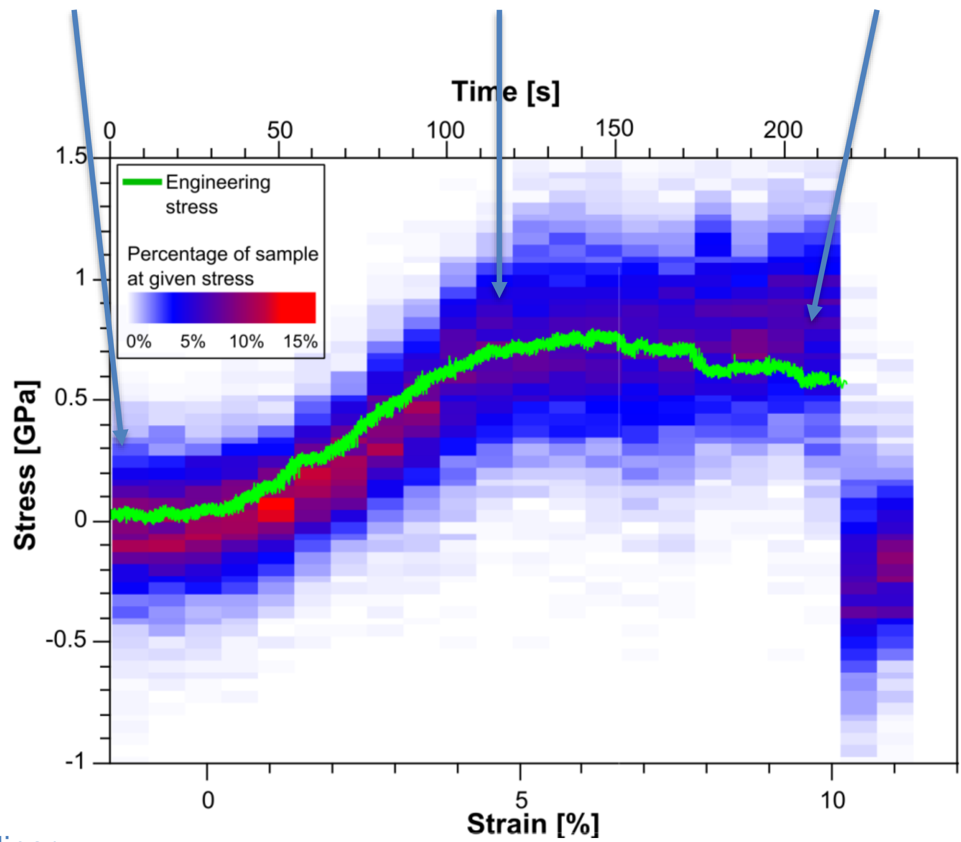
Local vs. global strain





Local stress histogram
Calculated from
local strains using:
σ = E ε
(E=70GPa)

Global stress curve
from indenter



C. Gammer, J. Kacher, C. Czarnik, O.L. Warren, J. Ciston, A.M. Minor,
Applied Physics Letters, 109:081906, 2016.

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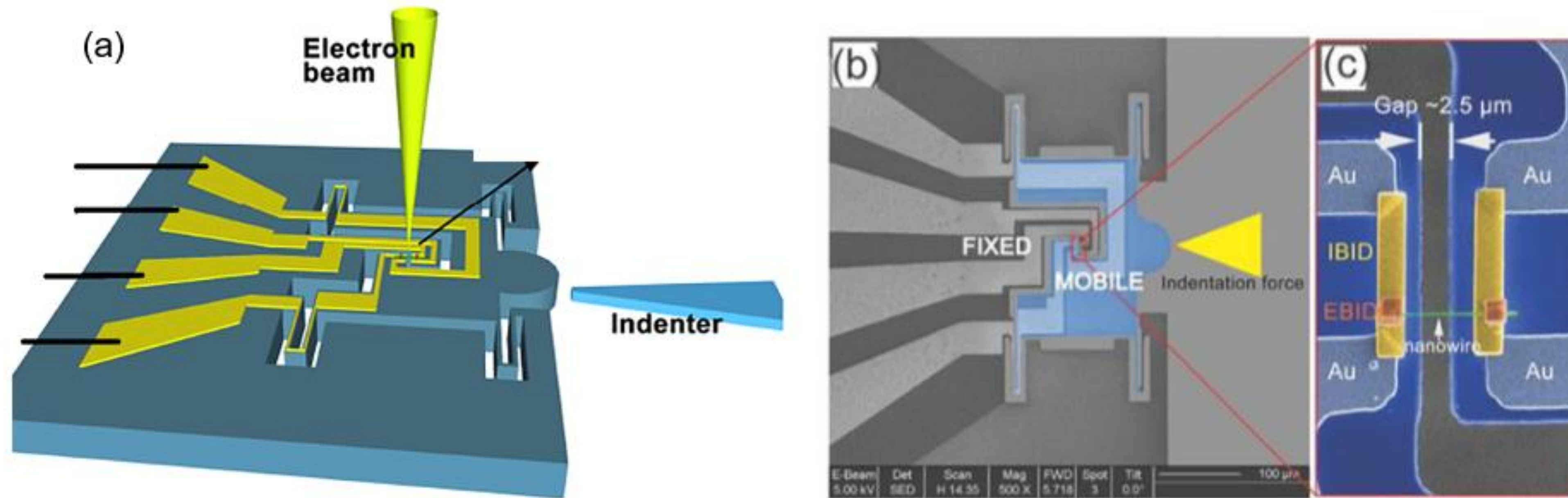
Electrical testing

$InAs/In_{0.6}Ga_{0.4}As$ Core–Shell Nanowires



E-PTP device allows to measure electrical properties during deformation

L. Zeng, C. Gammer, B. Ozdol, T. Nordqvist, J. Nygård, P. Krogstrup, A.M. Minor, W. Jäger, E. Olsson. Nano Letters 18, 4949 (2018).

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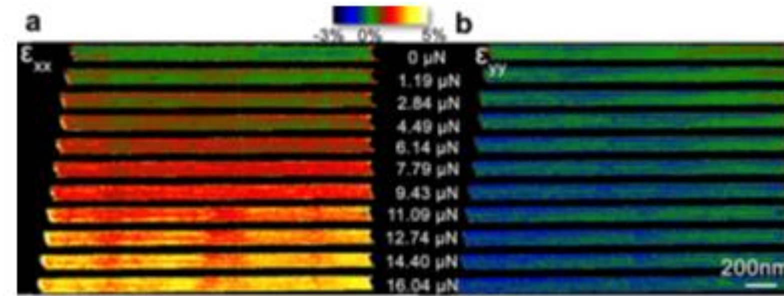
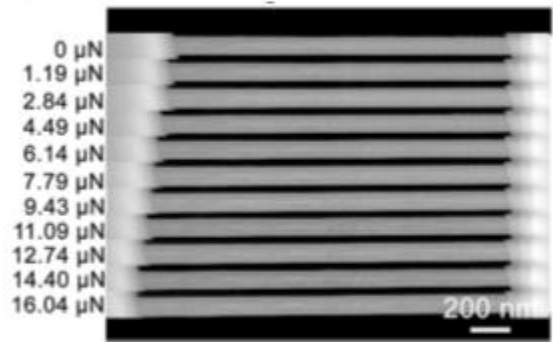
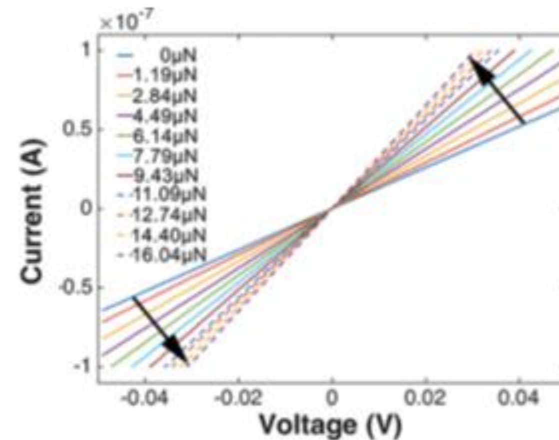
$InAs/In_{0.6}Ga_{0.4}As$ Core−Shell Nanowires



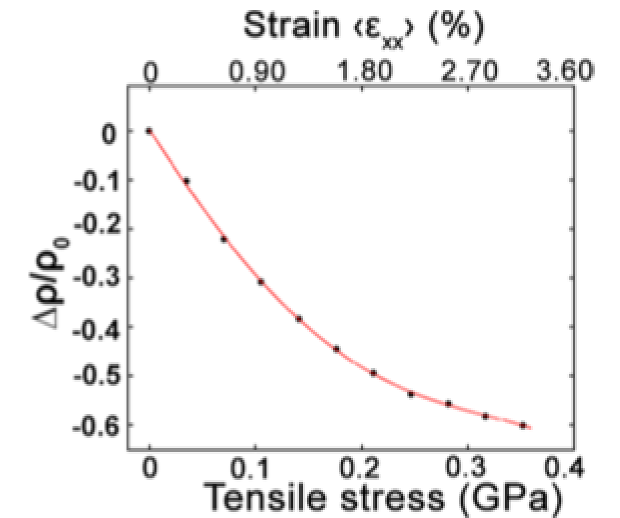
Measure strain



apply load



Measure electrical properties



piezoresistance can be obtained

L. Zeng, C. Gammer, B. Ozdol, T. Nordqvist, J. Nygård, P. Krogstrup, A.M. Minor, W. Jäger, E. Olsson. Nano Letters 18, 4949 (2018).

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Metallic Glasses

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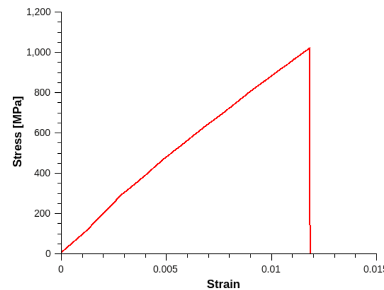
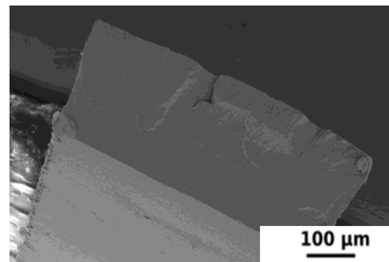
Bulk metallic glass

- CuZrAl bulk metallic glass
- High hardness, brittle fracture
- Localized deformation in the form of shear bands

Bulk tensile test

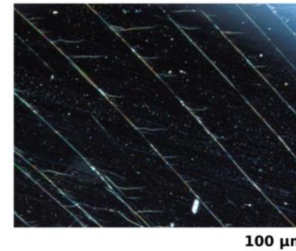


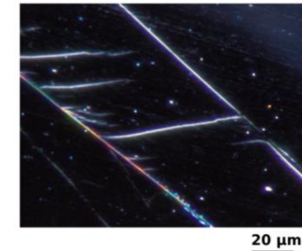
Stress-strain curve



Fracture surface

Rolling (5%)





Light microscopic images showing shear bands

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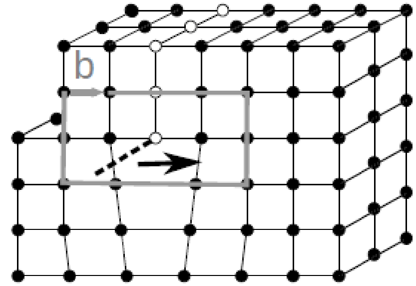
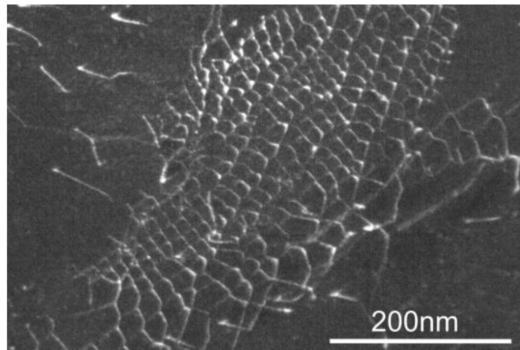
Deformation of bulk metallic glasses

Crystalline structure

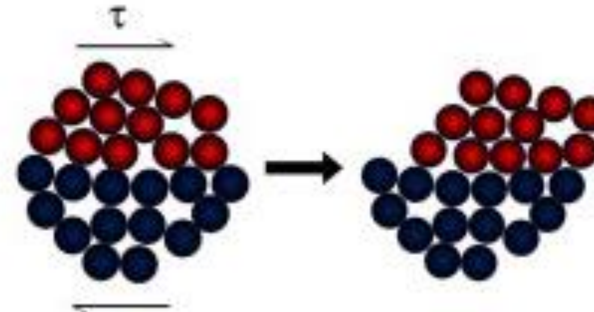


Dislocations

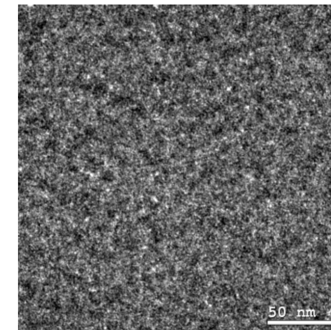


Visible in TEM

Amorphous structure



Shear transformation zone
A. S. Argon, Acta Metall. 27 (1979), 47–58



Not visible in TEM,
indications in MD simulations

Formation and propagation of localized deformation not well understood

→ TEM at nanometer resolution needed

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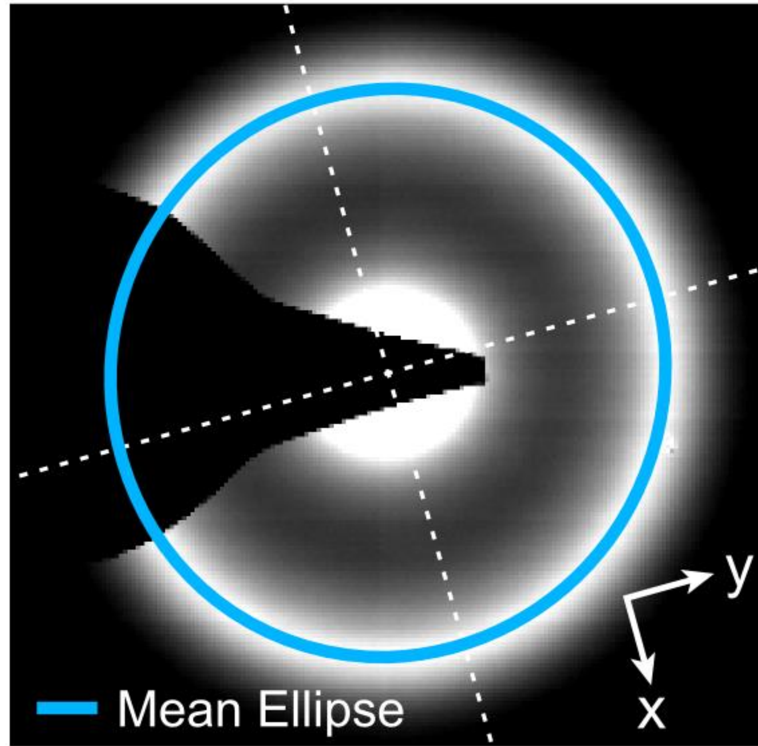
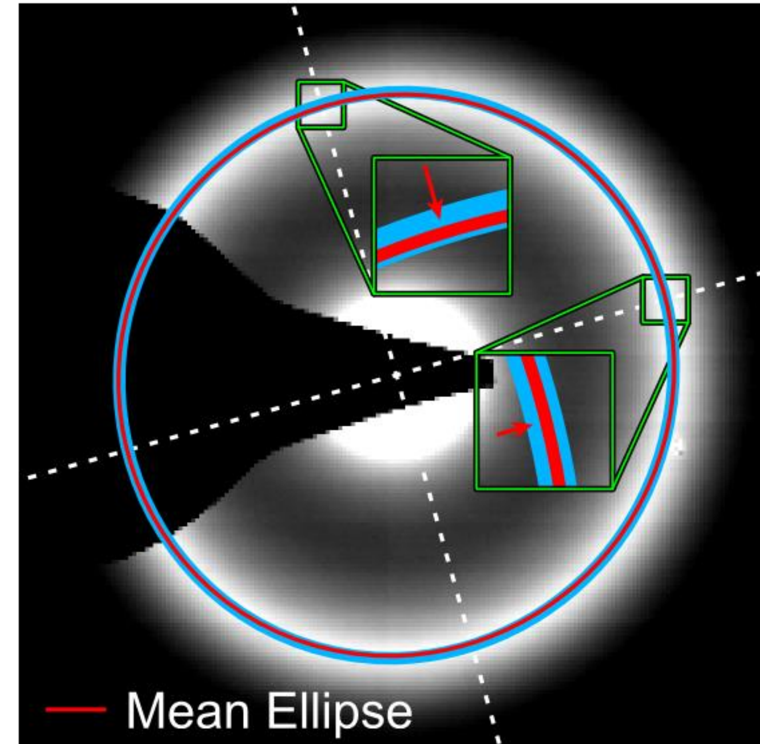
Strain – elliptic distortion

Unstrained



Strained (1%)



Elastic (atomic) strain

C. Gammer, C. Ophus, T.C. Pekin, J. Eckert, A.M. Minor. Applied Physics Letters 112 (2018) 171905.

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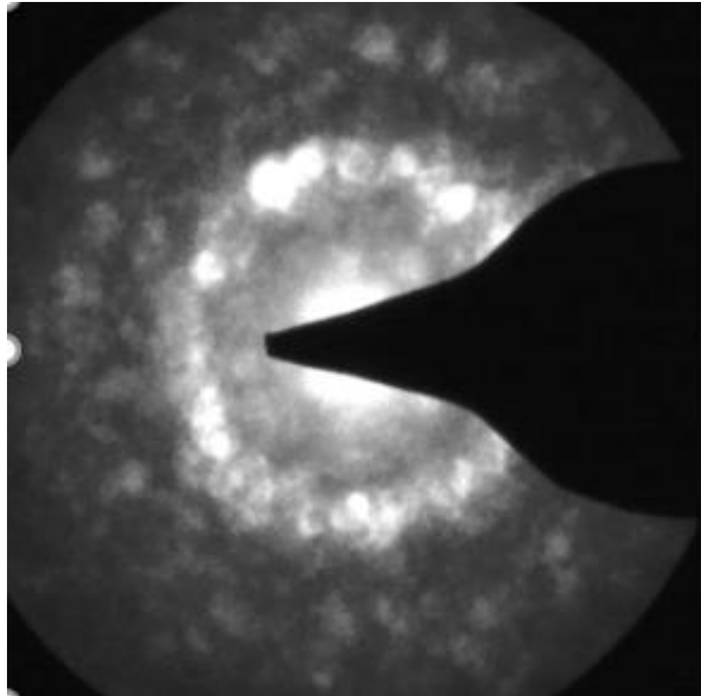
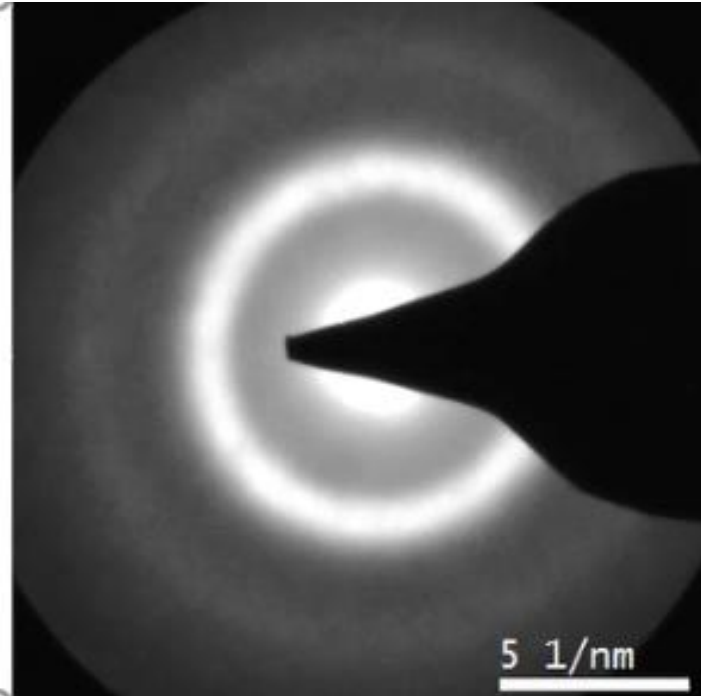
Strain mapping from nanovolumes

Nanodiffraction map

Average of all diffraction patterns





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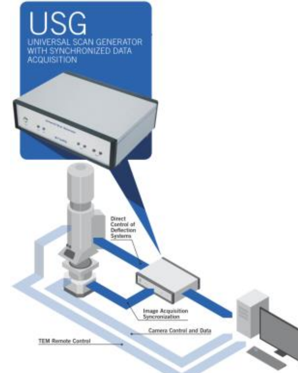
Nanomap transmission electron microscope

www.jeol.de

TEM with in-column energy filter



www.tvips.com

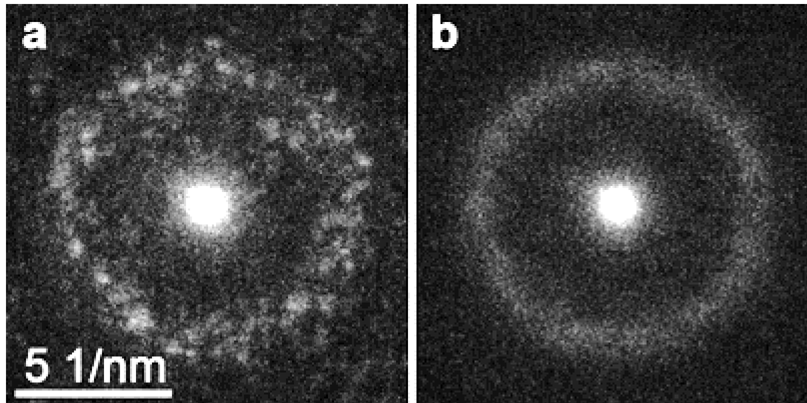
Universal scan generator

www.tvips.com

High speed CMOS detector (4096x4096 pixel, up to 400fps)

quantumdetectors.com

Direct electron detector (512x512pixel, 2kHz/12bit)



Precession:
< 2 nm resolution
< 0.1% strain precision

H. Sheng, D. Sopu, S. Fellner, J. Eckert, C. Gammer, Physical Review Letters 128 (2022) 245501.

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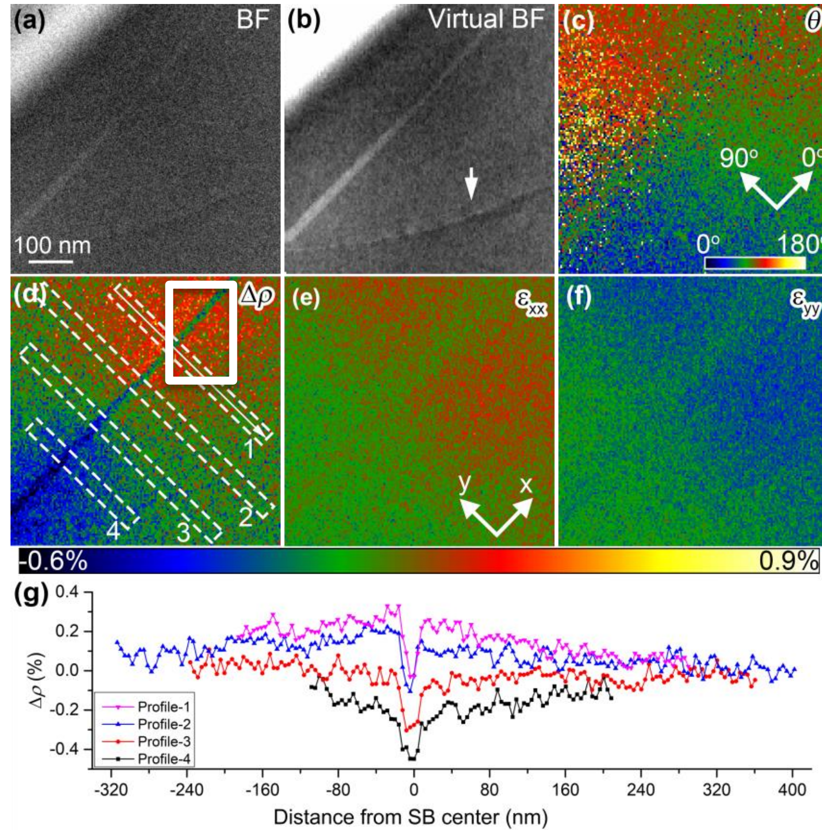
Shear band

Quantify the elastic strain field (atomic strains) with < 2 nm resolution.



Shear band has lower density
Long-range strains associated with shear band segments

H. Sheng, D. Sopu, S. Fellner, J. Eckert, C. Gammer, Physical Review Letters 128 (2022) 245501.

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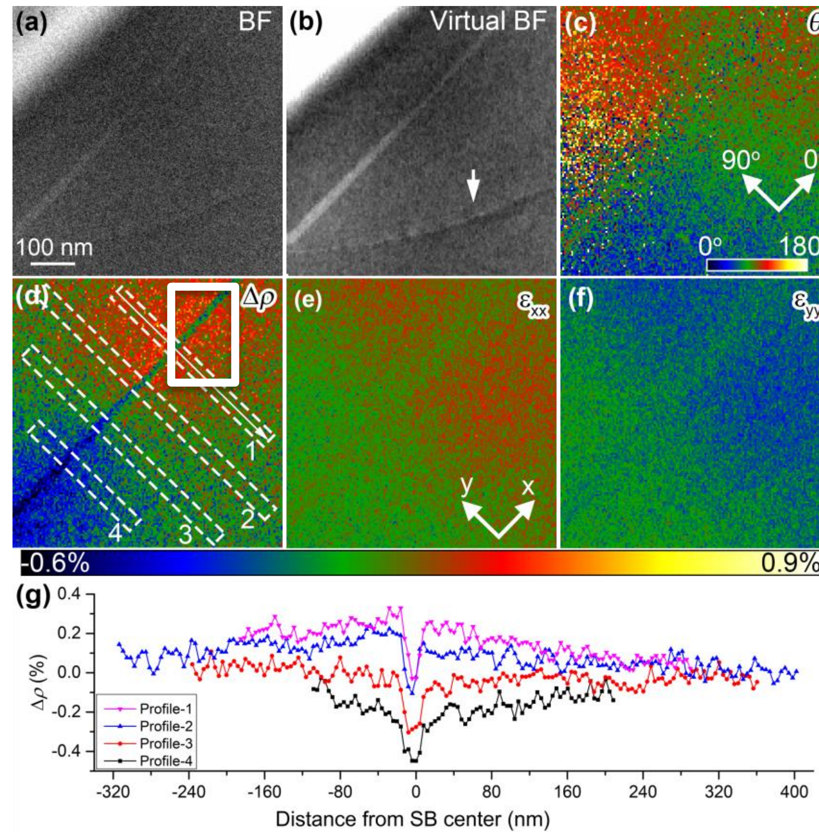
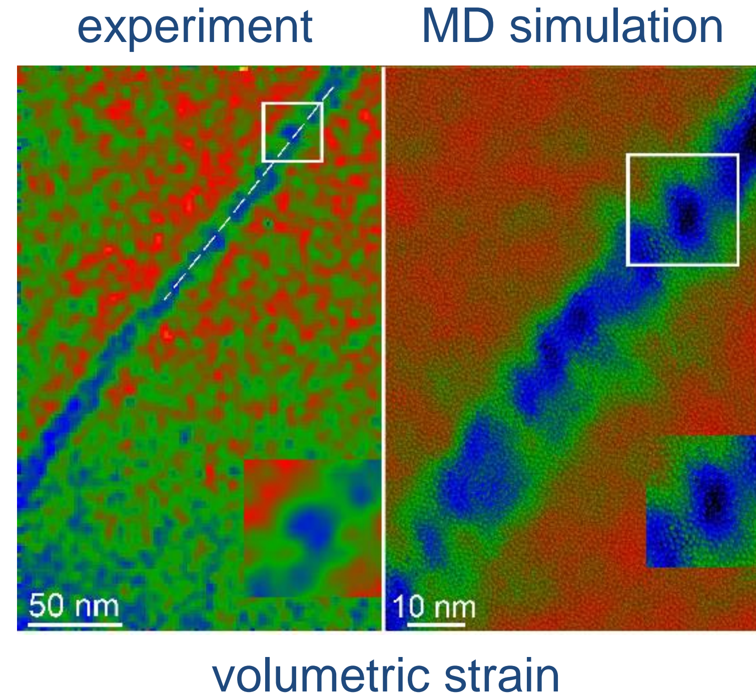
Shear band





volumetric strain

Shear band has lower density
Long-range strains associated with shear band segments
Nanoscale density variations can be linked to MD simulations

H. Sheng, D. Sopu, S. Fellner, J. Eckert, C. Gammer, Physical Review Letters 128 (2022) 245501.

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Summary

Strain mapping using 4D-STEM

- Local elastic strain in real devices
- Elastic strain can be measured for crystalline and amorphous materials
- Enables correlation with simulations
- Transient local stain can be measured during in situ experiments
- Recent advances allow to combine electrical and mechanical measurements

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BERKELEY LAB

Andy Minor, Burak Ozdol

FWF Der Wissenschaftsfonds

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