

Christina Krywka

# X-ray nanodiffraction meets materials science

Röntgen-Nanodiffraktion in der Materialforschung

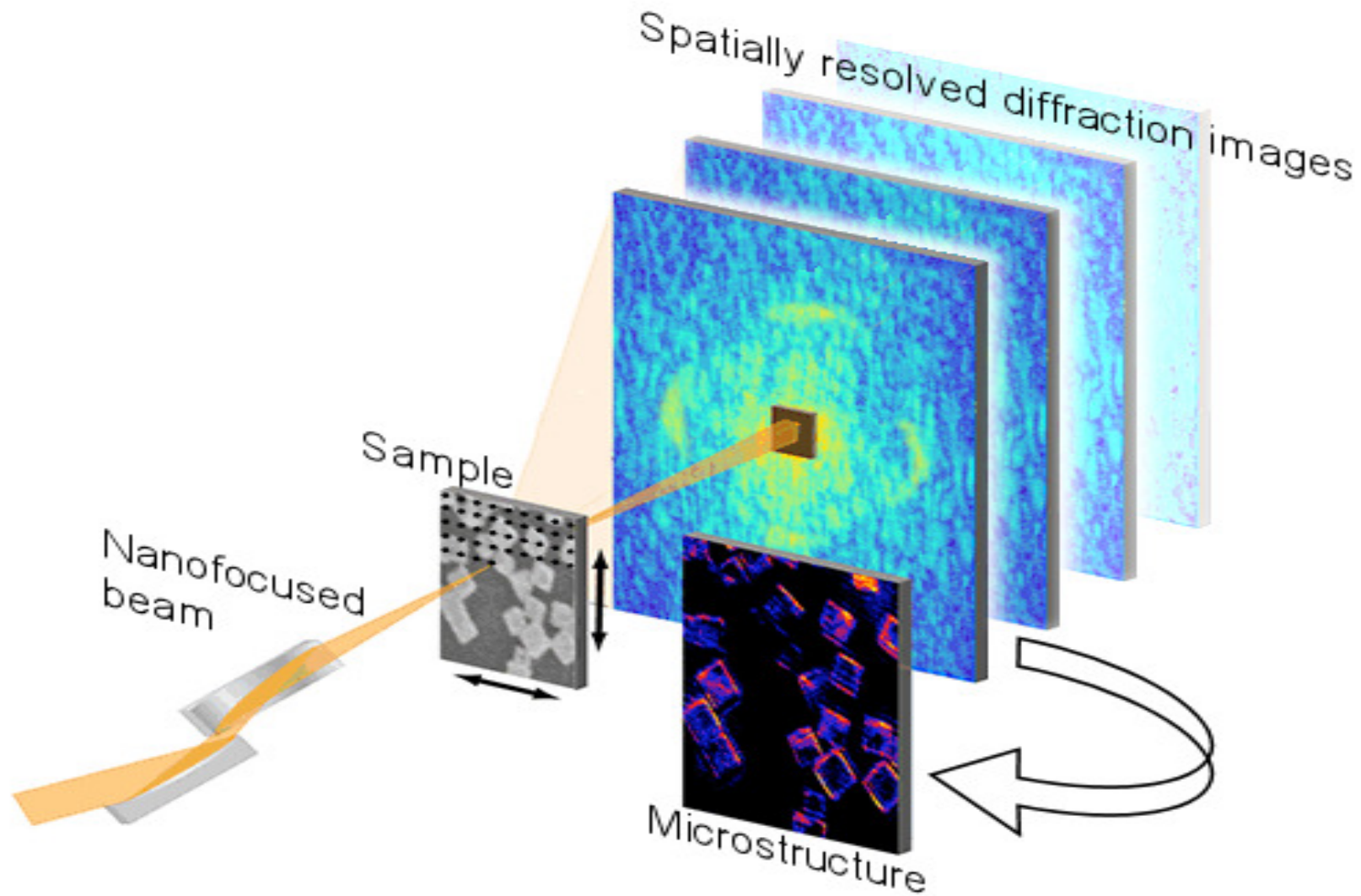


# outline

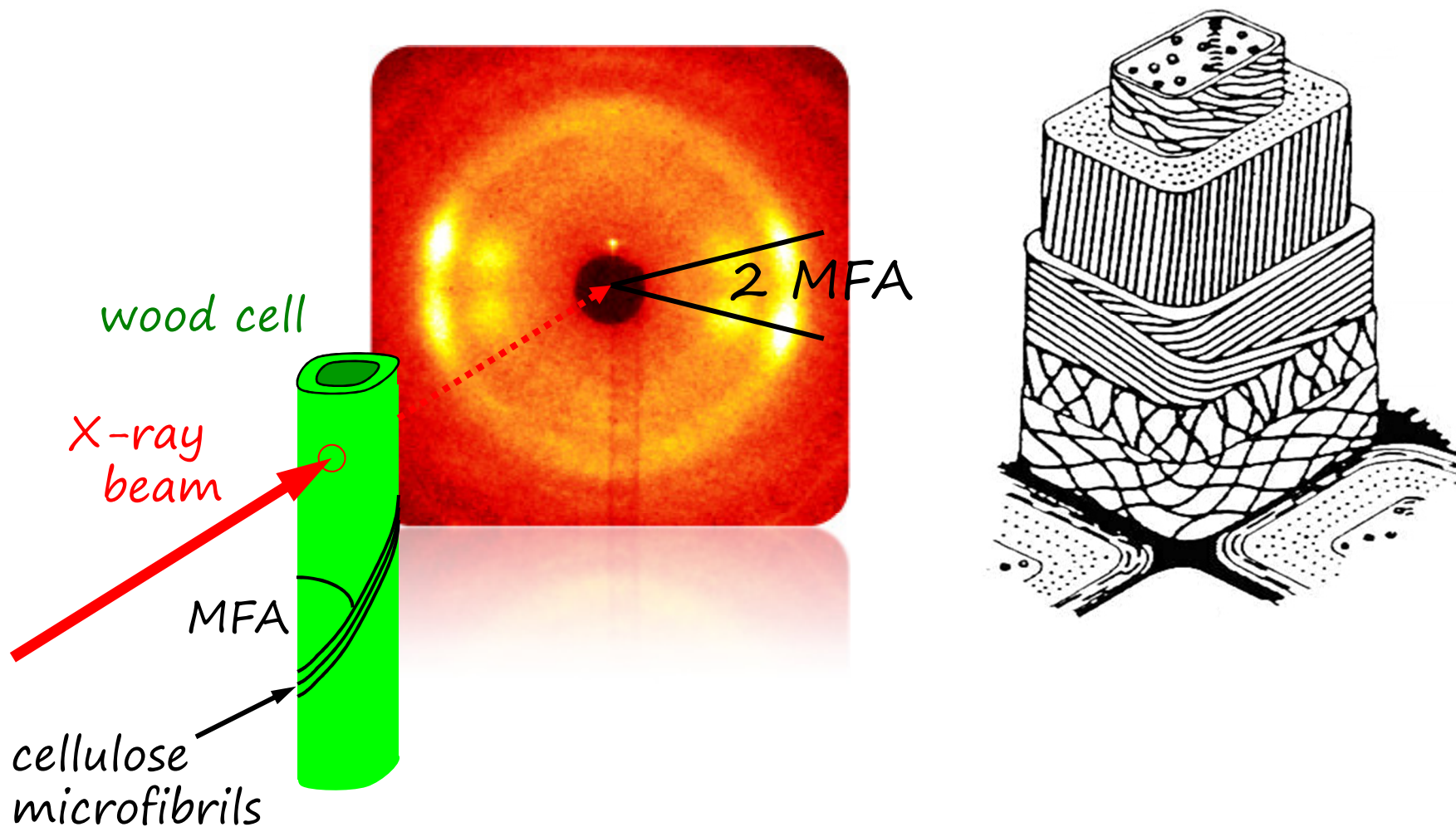
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- „the why“ of nanodiffraction
- „the how“ of nanodiffraction
- nanodiffraction experiments
  
- German Engineering Materials Science Center

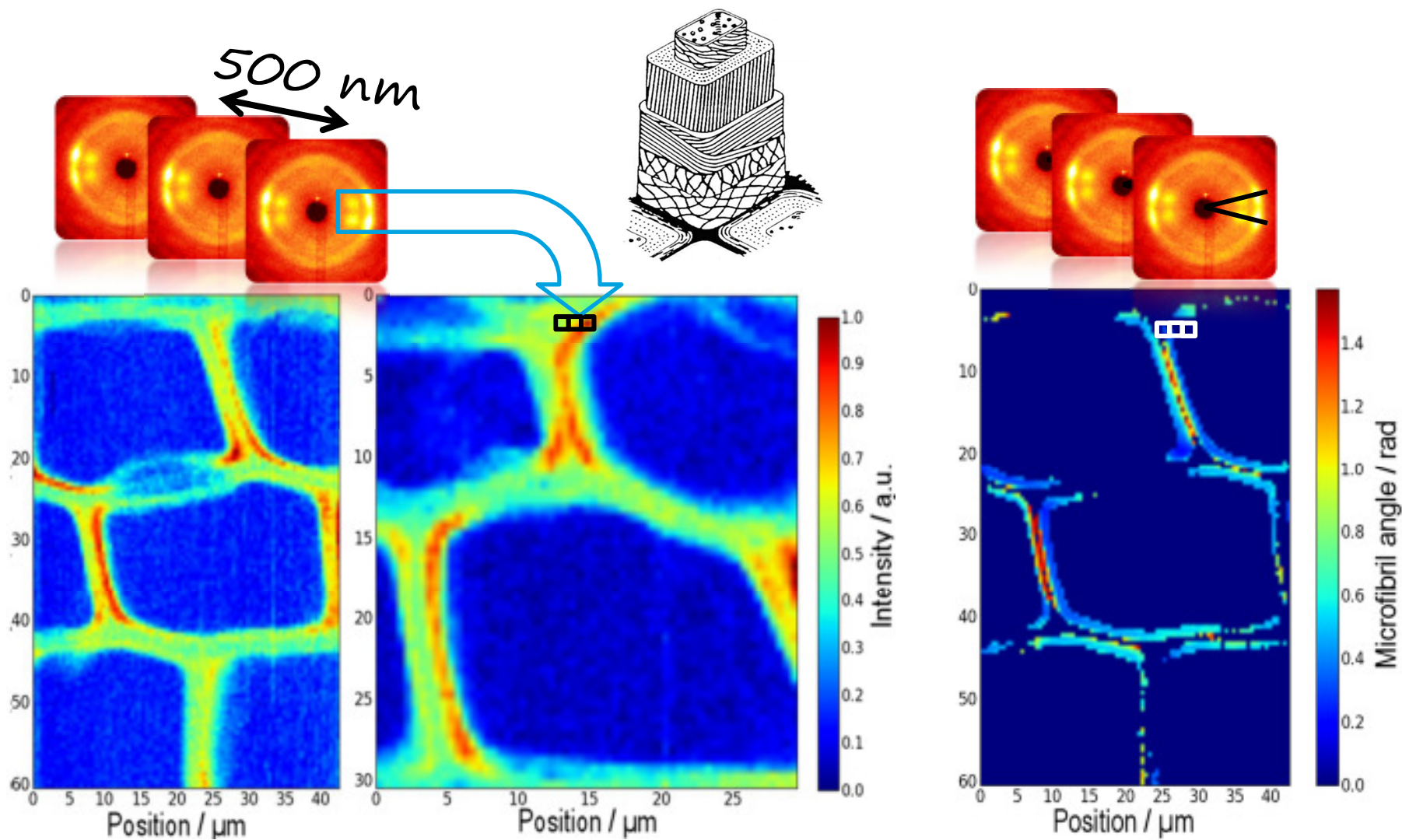
# scanning nanodiffraction



# scanning nanodiffraction

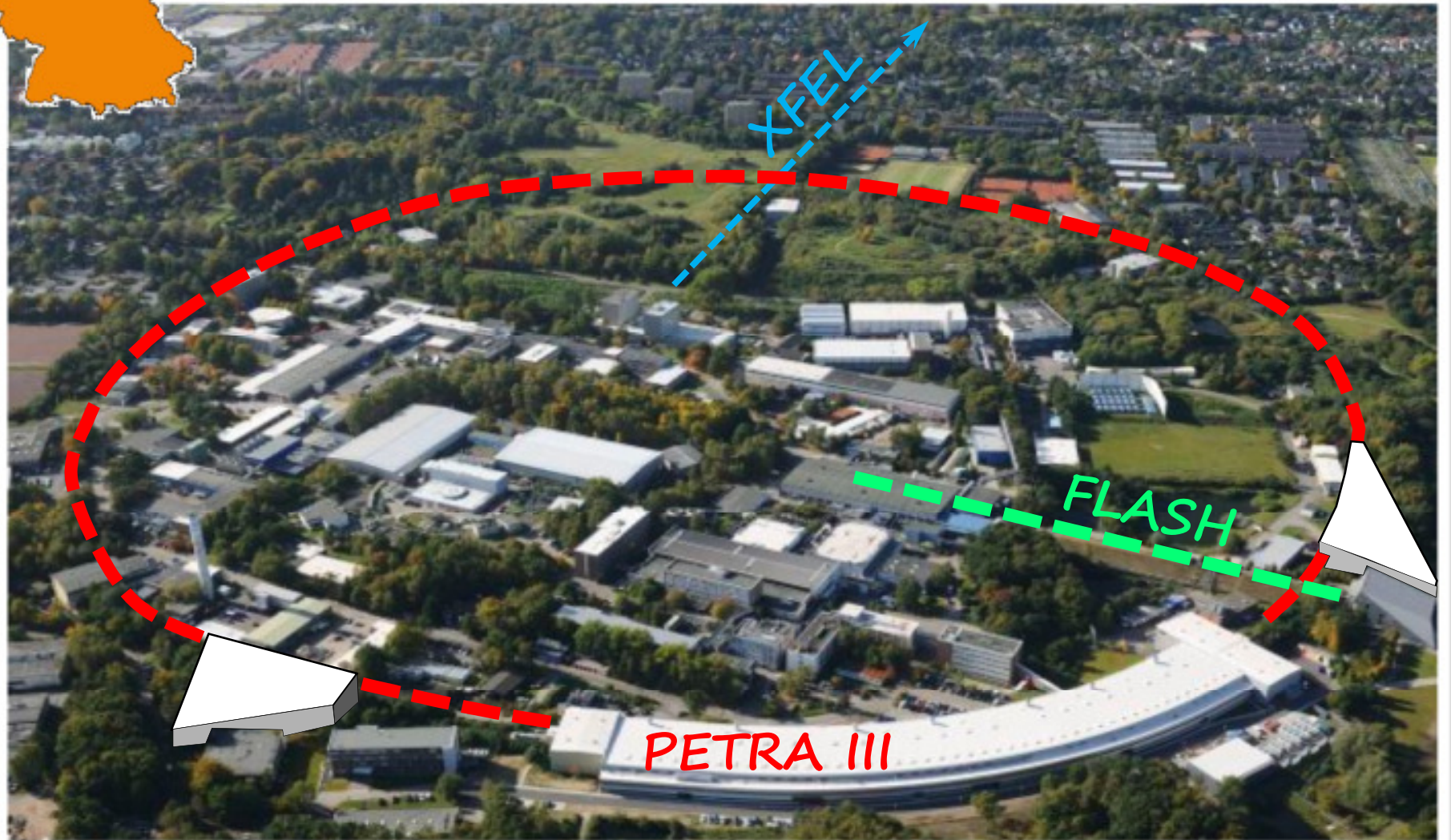


# scanning nanodiffraction



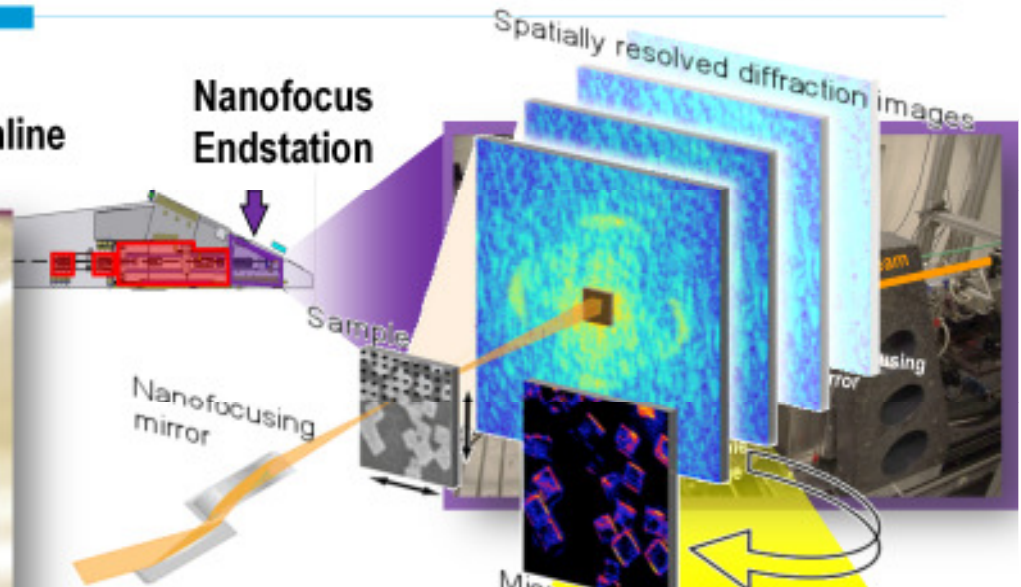
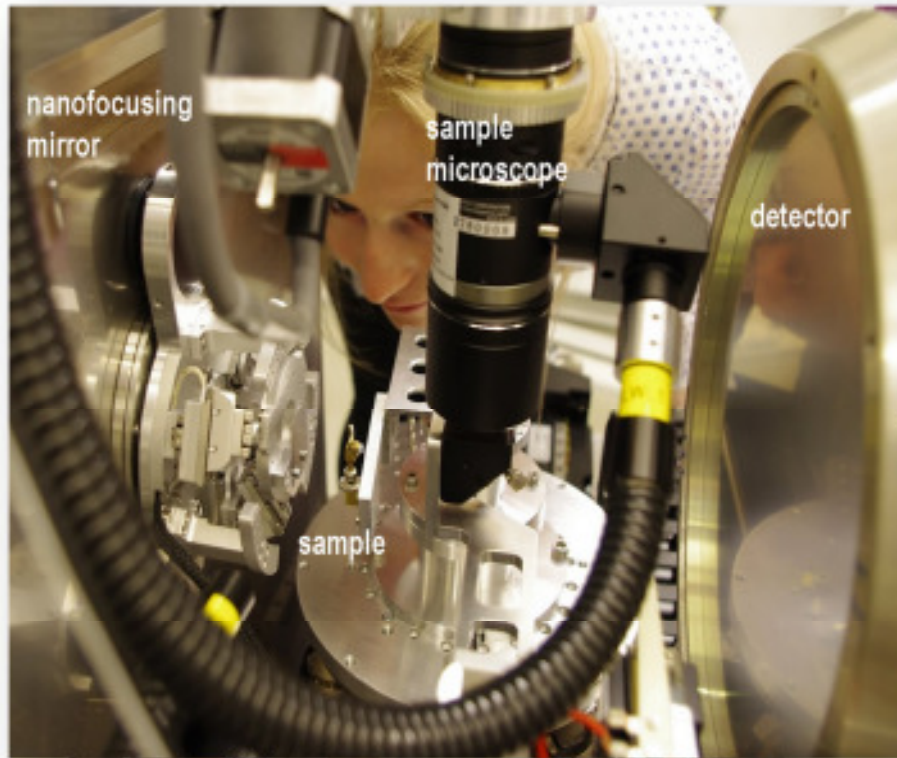
S. Storm, Master Thesis, University of Kiel (2012)

# PETRA III @ DESY (Hamburg)

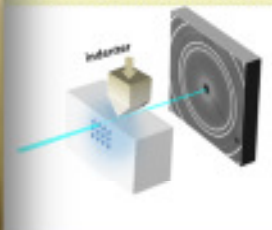


# Nanofocus Endstation @ P03

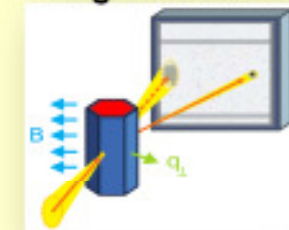
MINAXS (P03) beamline



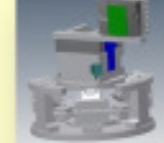
+ nanoindentation



+ magnetic field



...and more



nanofocusing  
mirror

sample  
microscope

detector

sample

Geesthacht  
Material- und Küst...



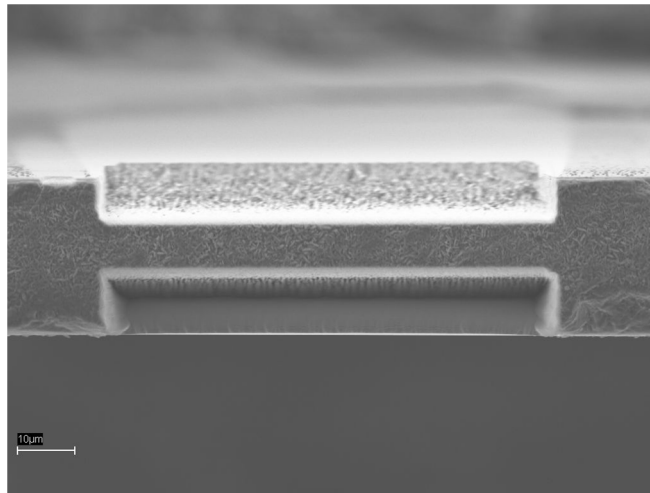
8  
d  
:



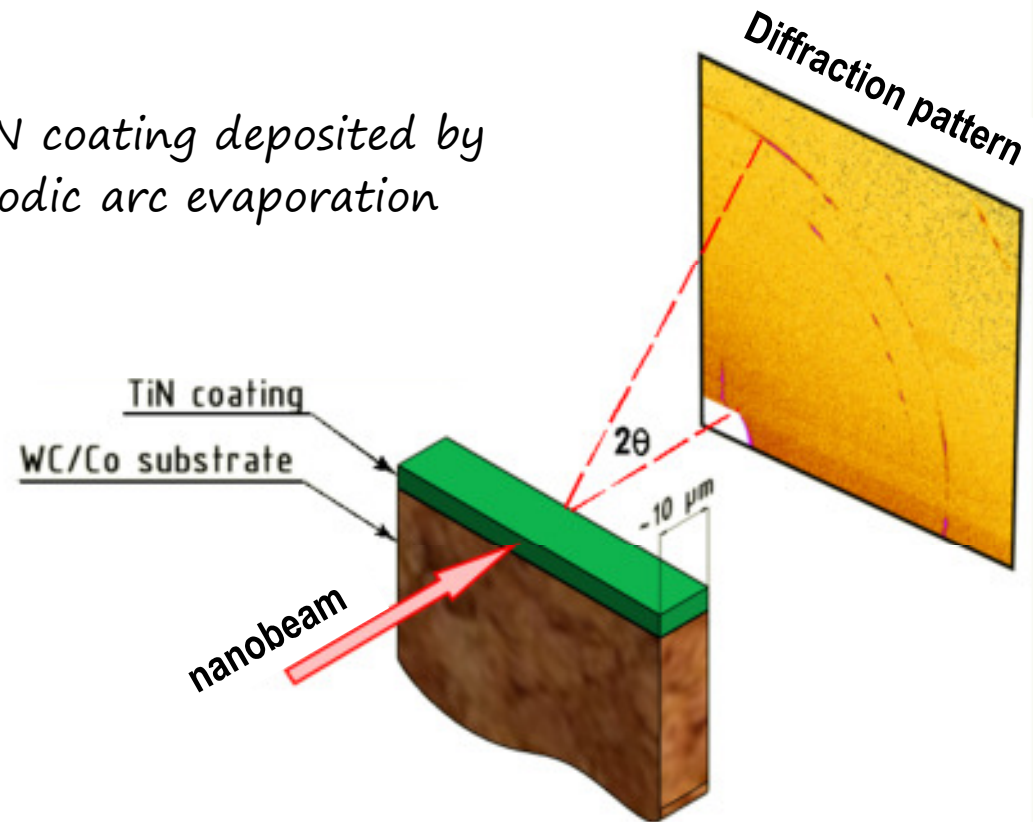
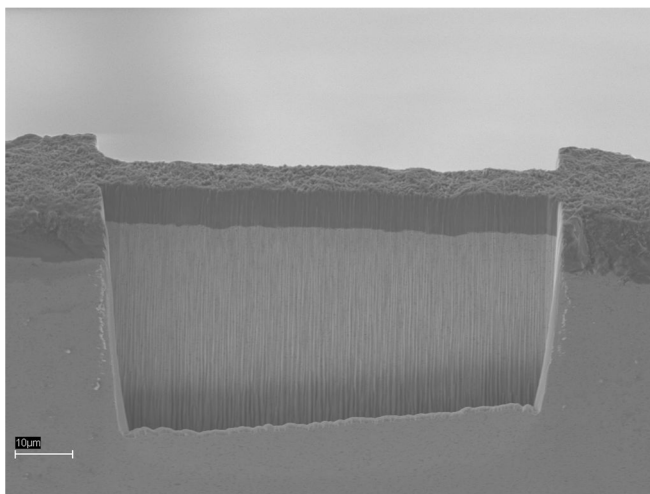
# „static“ nanodiffraction

J. Keckes, A. Riedl, et al.

Montanuniversität Leoben, Erich Schmid Institute

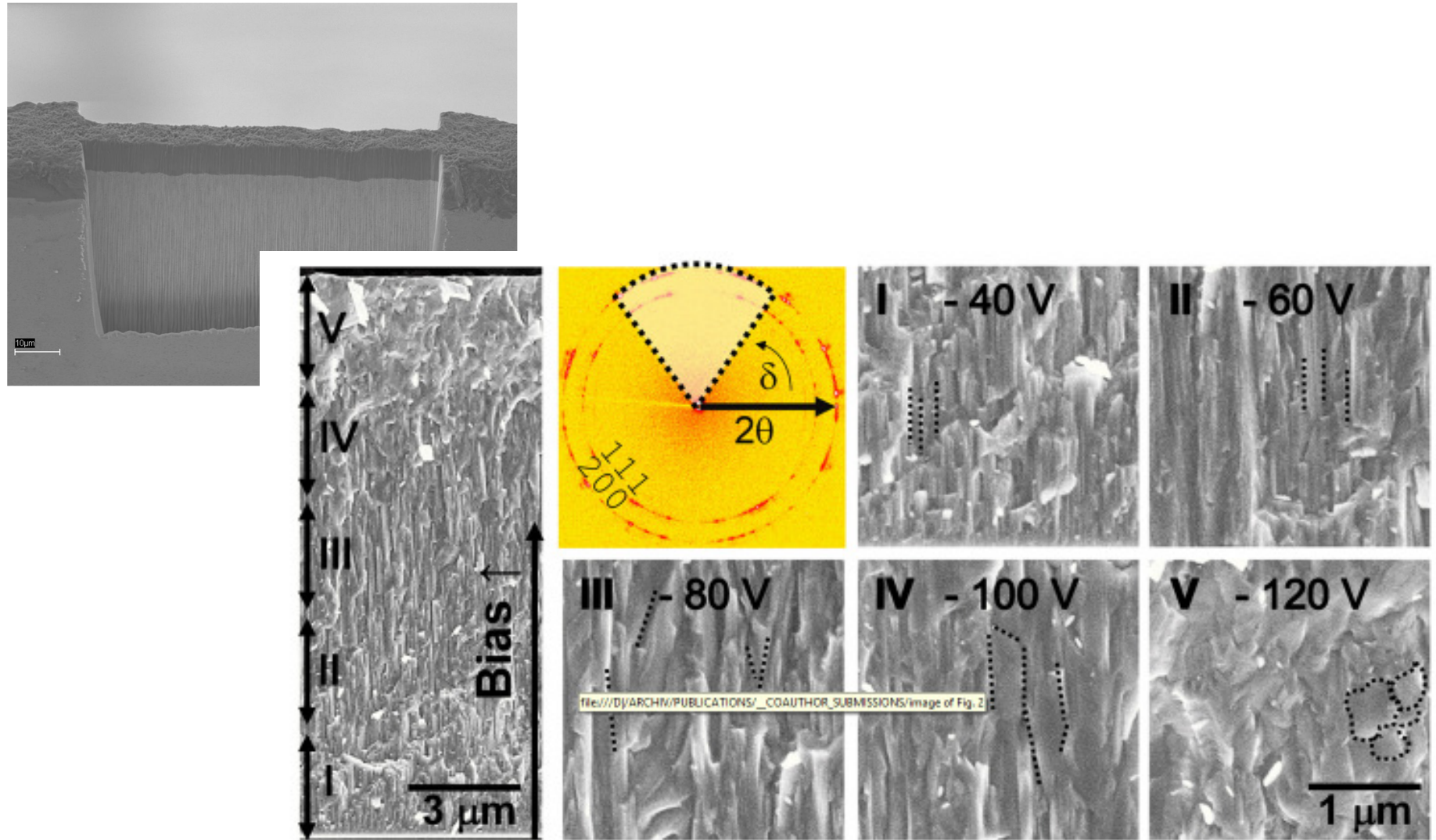


TiAlN coating deposited by cathodic arc evaporation



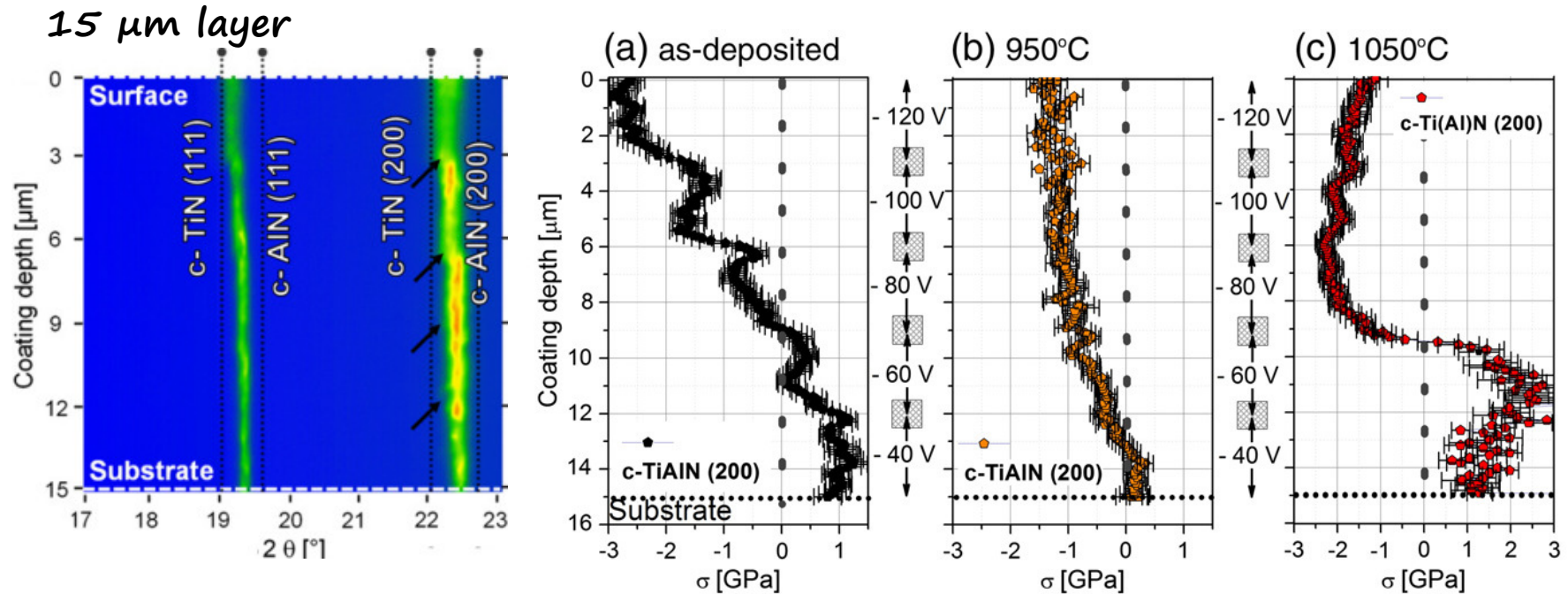
Krywka et al., Journal of Applied Crystallography 45, 85 (2012)

# „static“ nanodiffraction

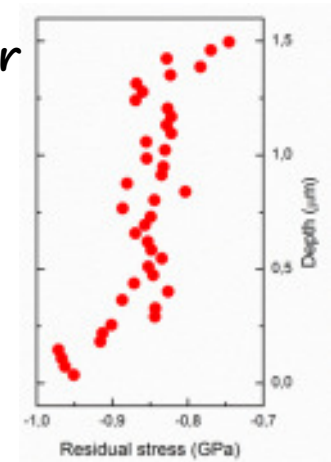


Riedl et al., *Surface & Coatings Technology* 257, 108 (2014)

# „static“ nanodiffraction



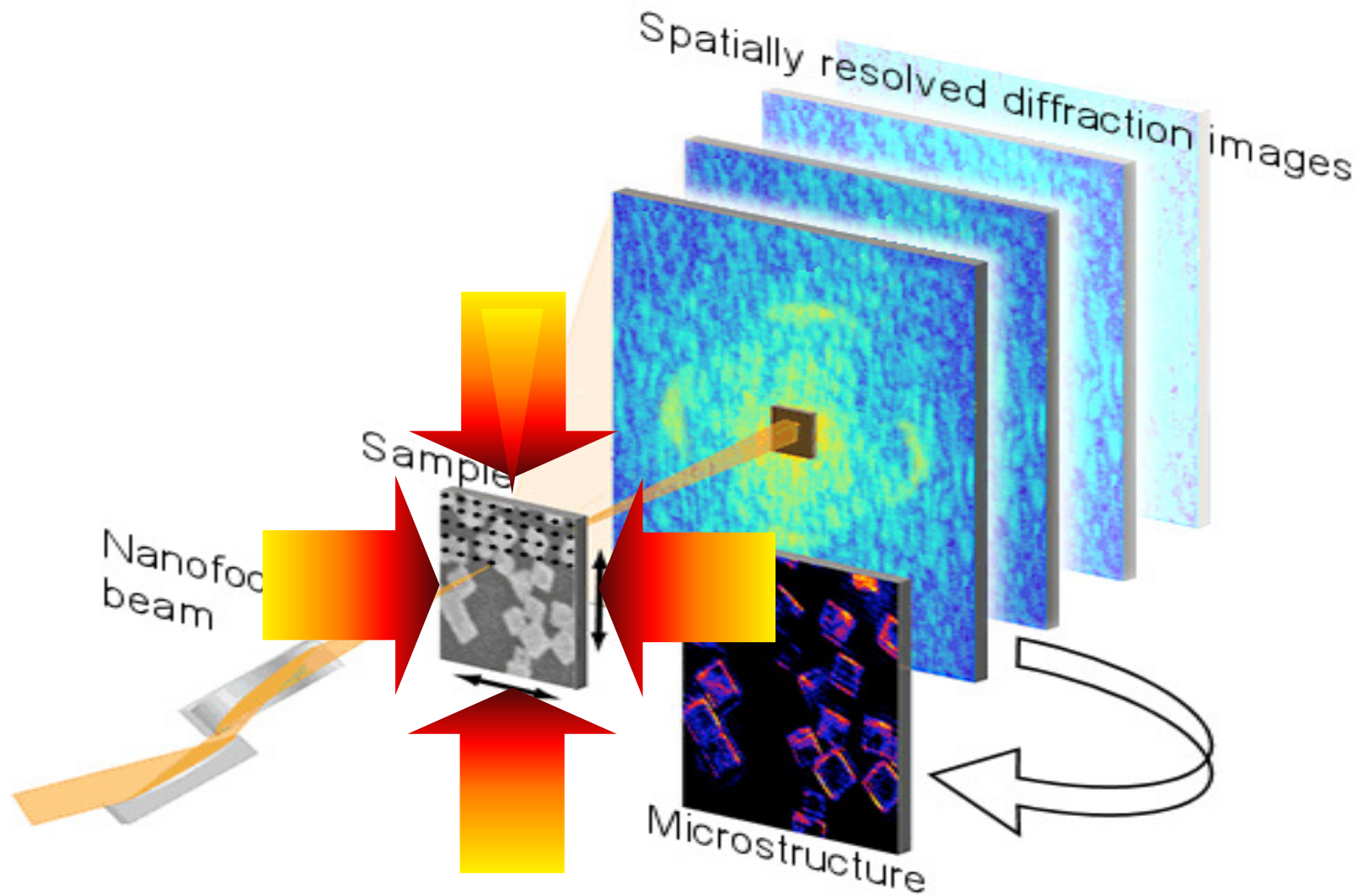
1.5  $\mu\text{m}$  layer



Riedl et al., *Surface & Coatings Technology* 257, 108 (2014)

Hollerweger et al., *Acta Materialia* 83, 108 (2014)

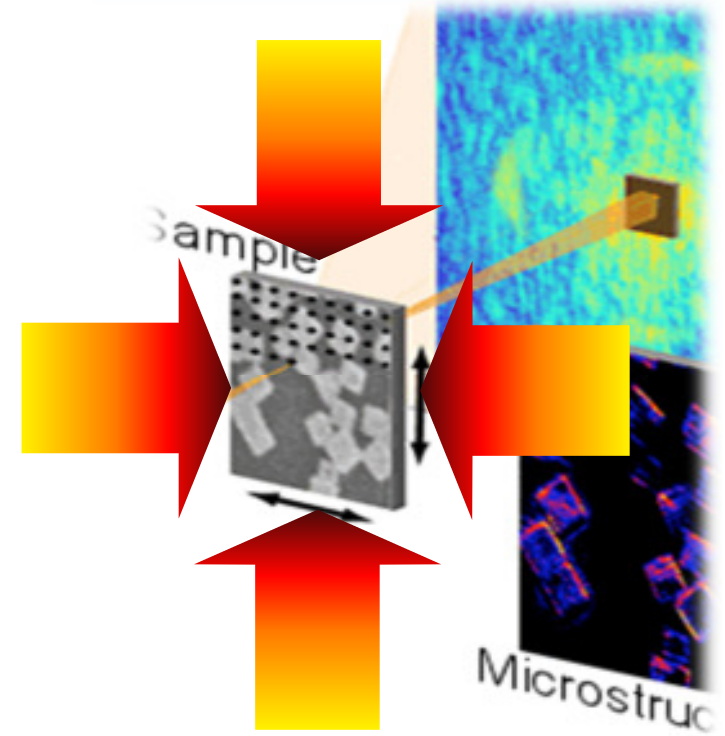
# nanodiffraction with *in-situ* parameters



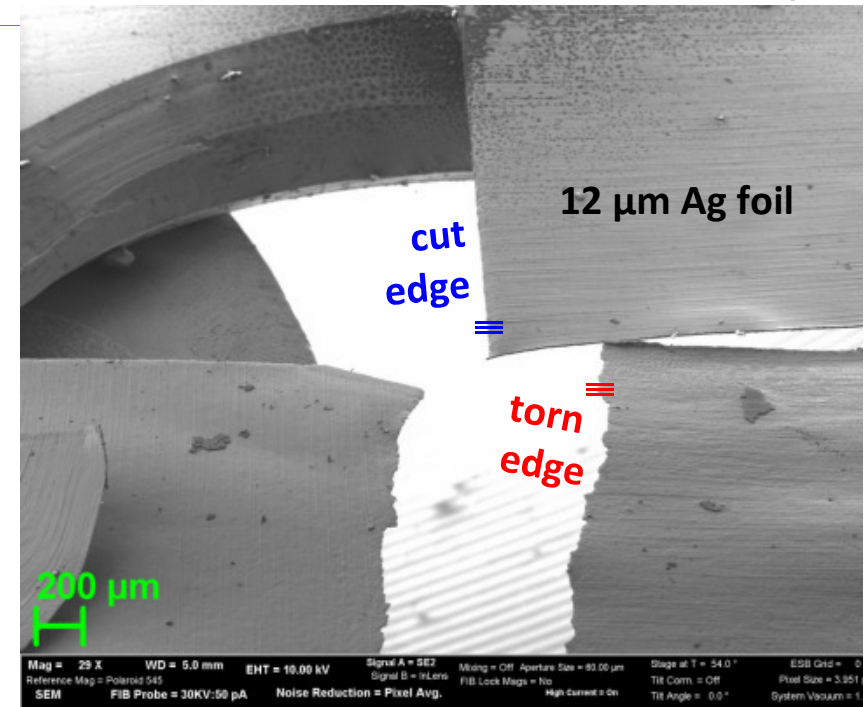
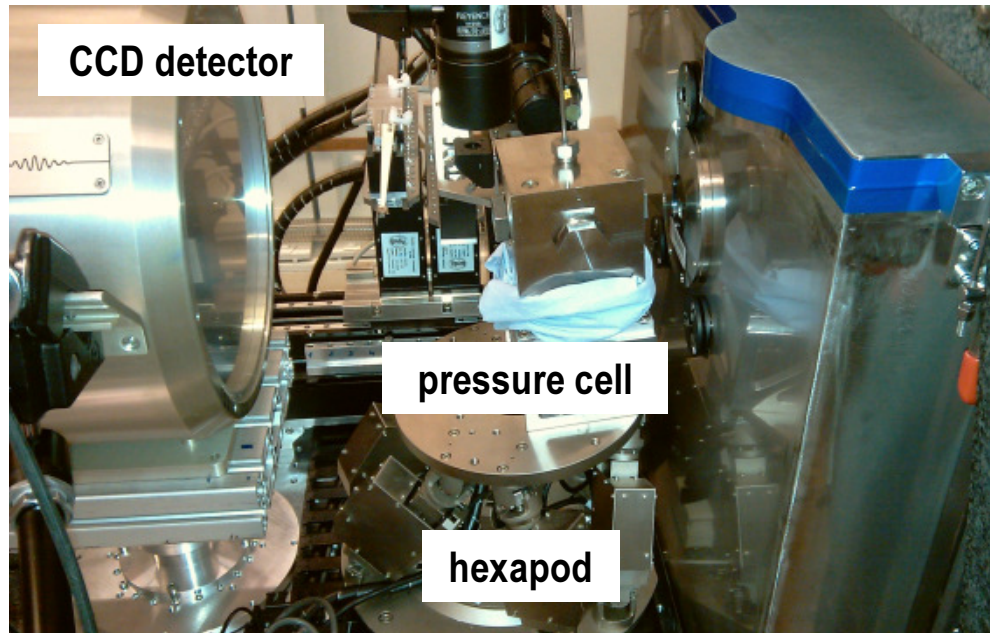
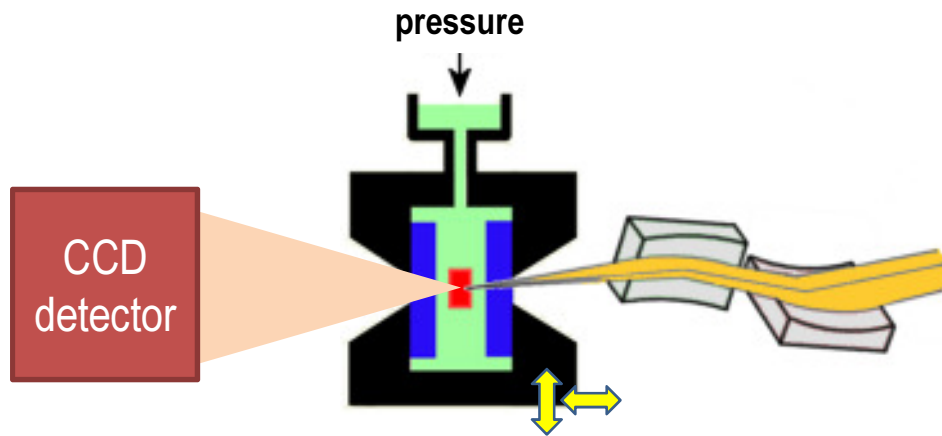
## high pressure nanodiffraction

elastic material properties  
from isotropic load

hydrogen embrittlement can  
be studied „in operando“



# high pressure nanodiffraction



## Applications of HP diffraction:

Krywka et al.

Macromolecules 47, 7187 (2014)

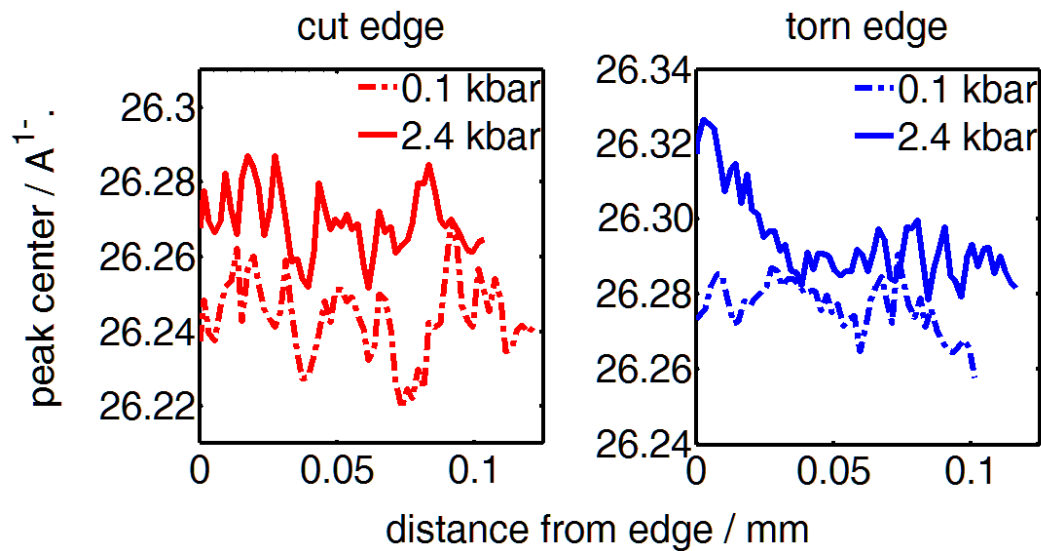
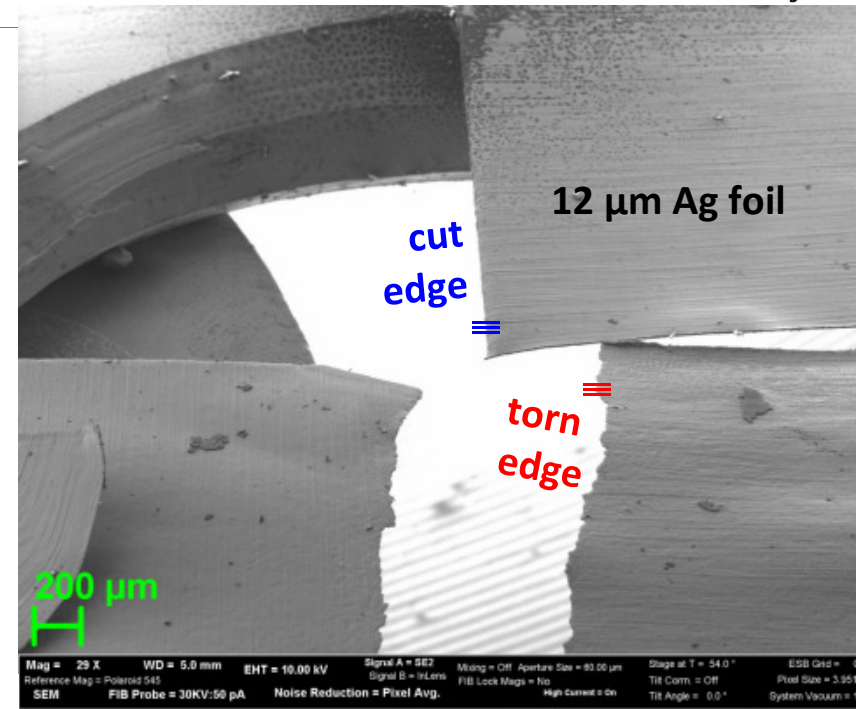
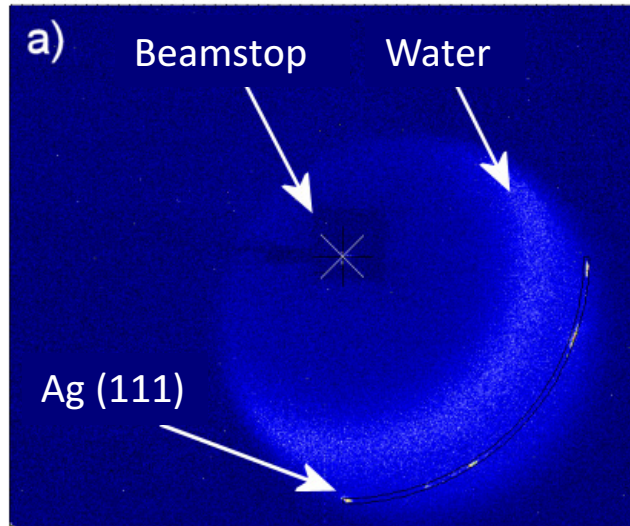
Ene, Krywka et al.

Polymer 53, 5507 (2012)

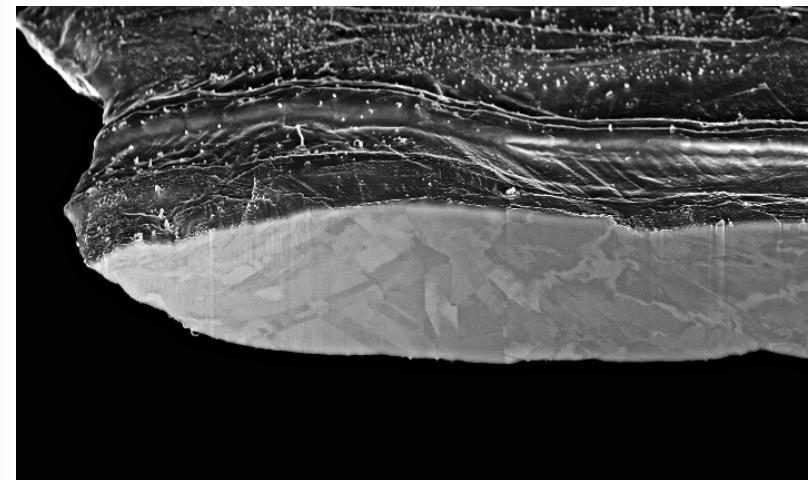
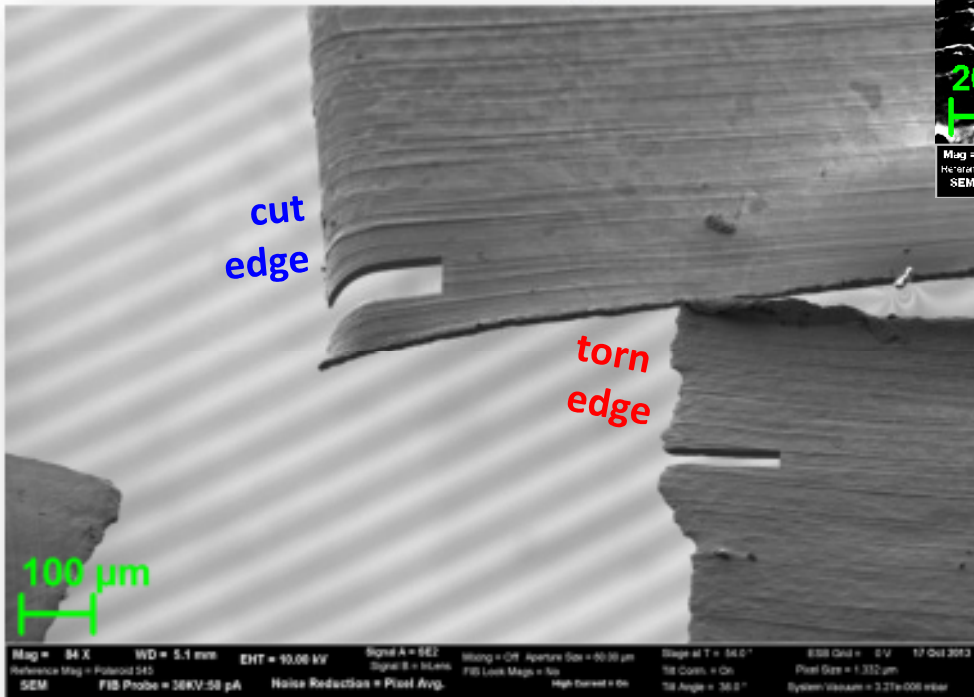
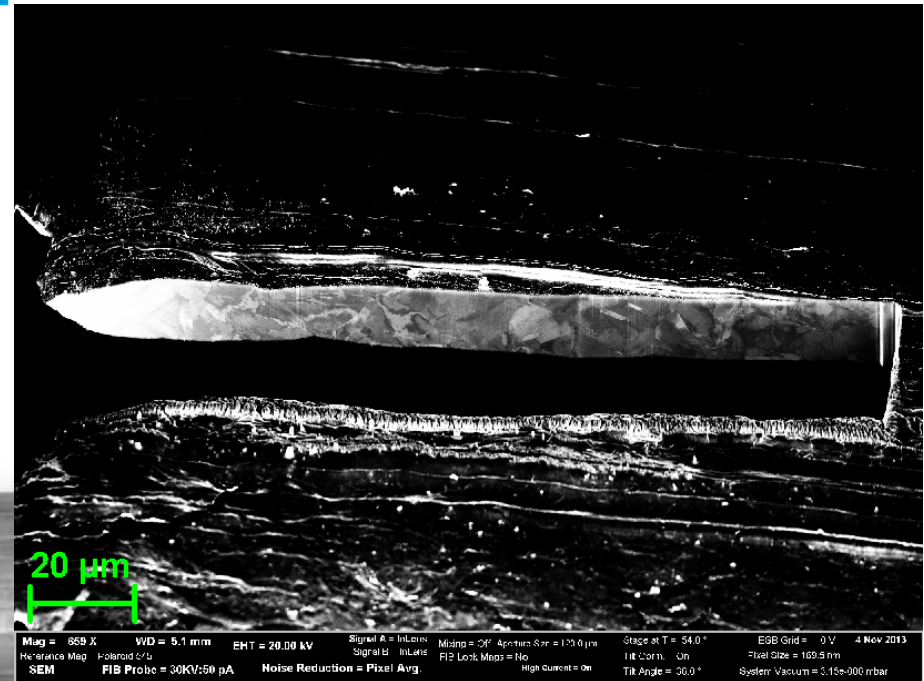
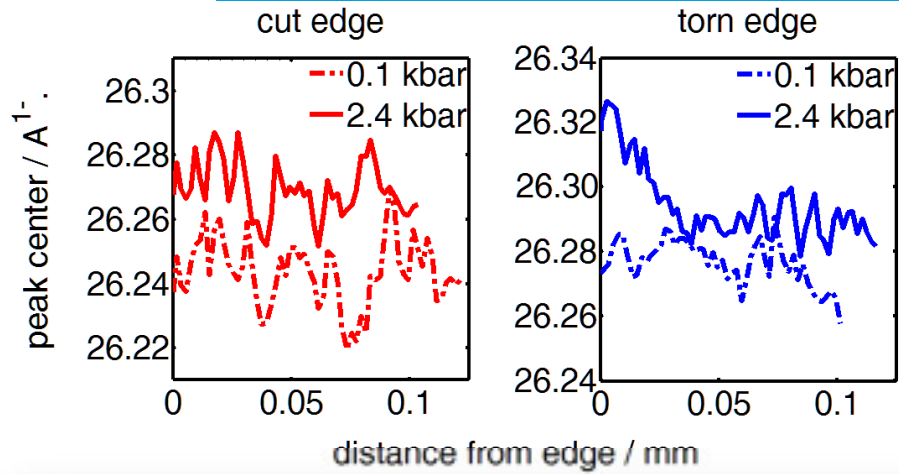
Krywka et al.

Chem. Phys. Chem. 9, 2809 (2008)

# high pressure nanodiffraction



# high pressure nanodiffraction



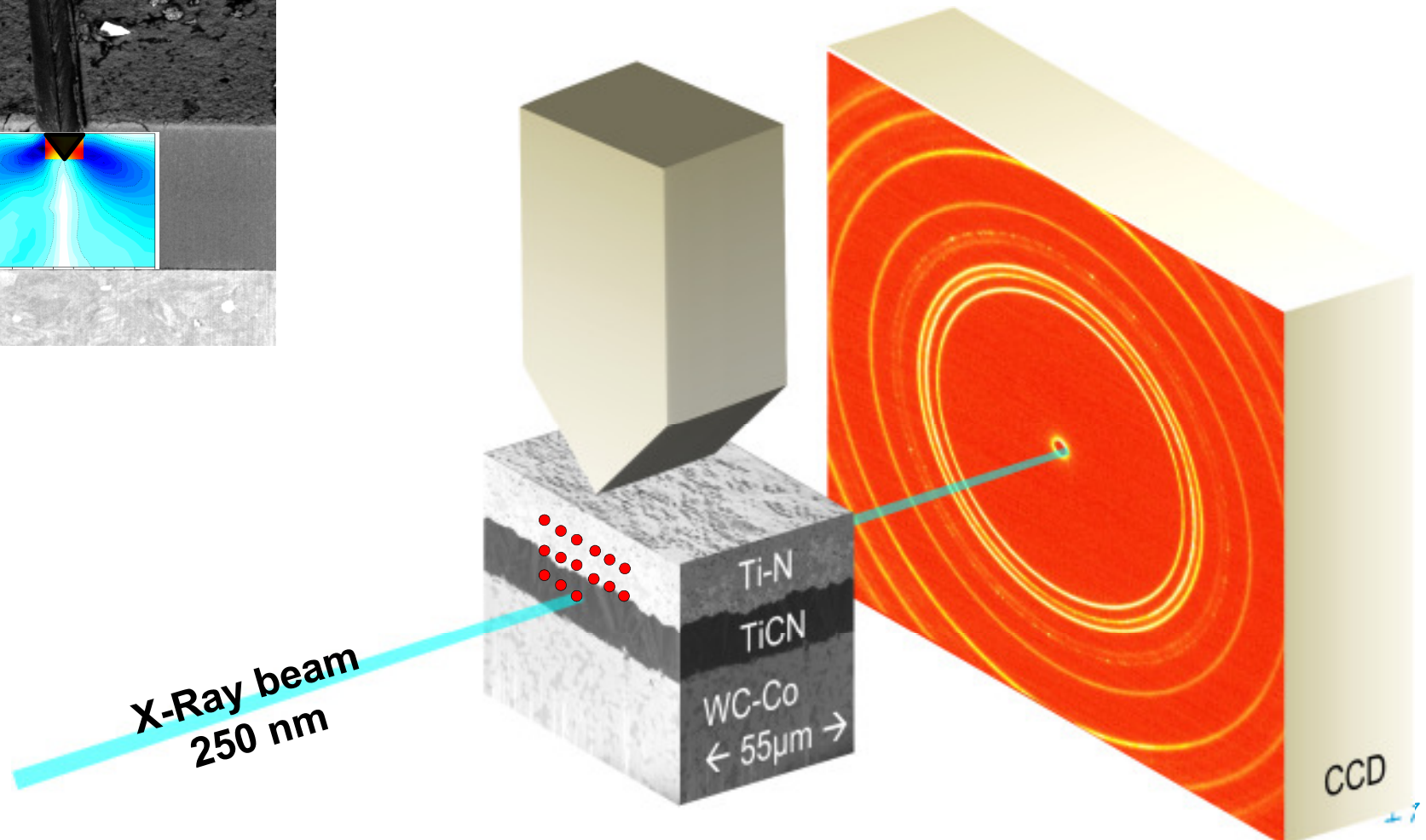
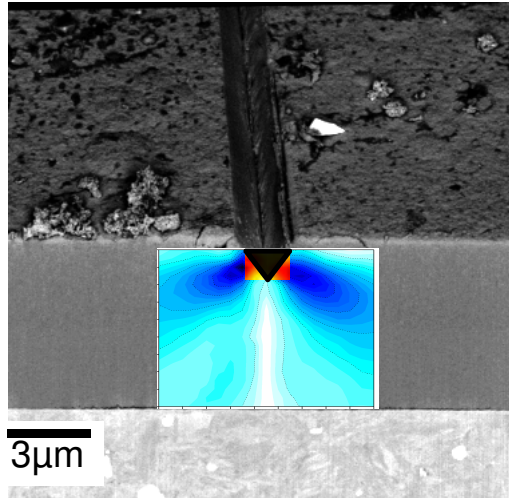


# nanoindentation

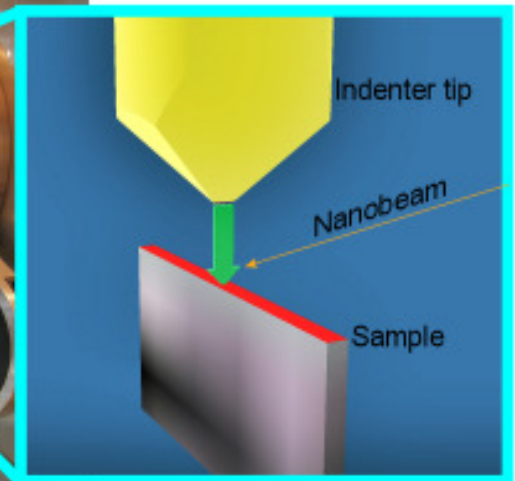
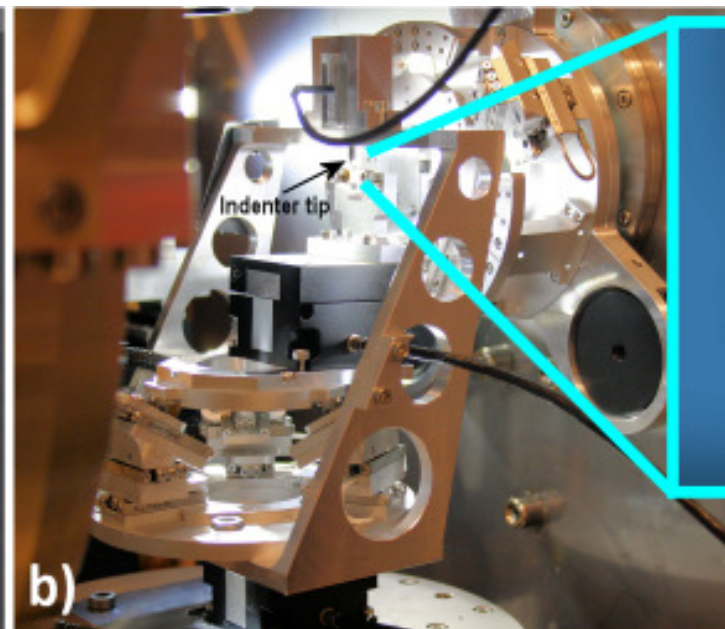
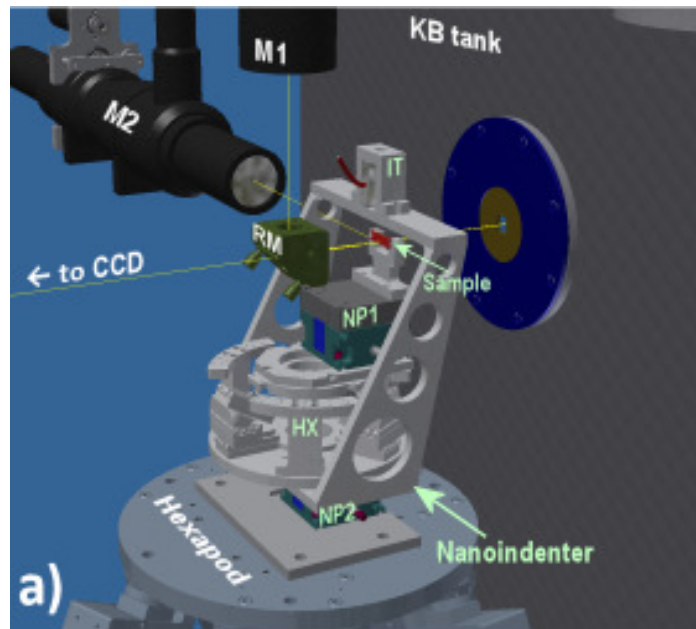


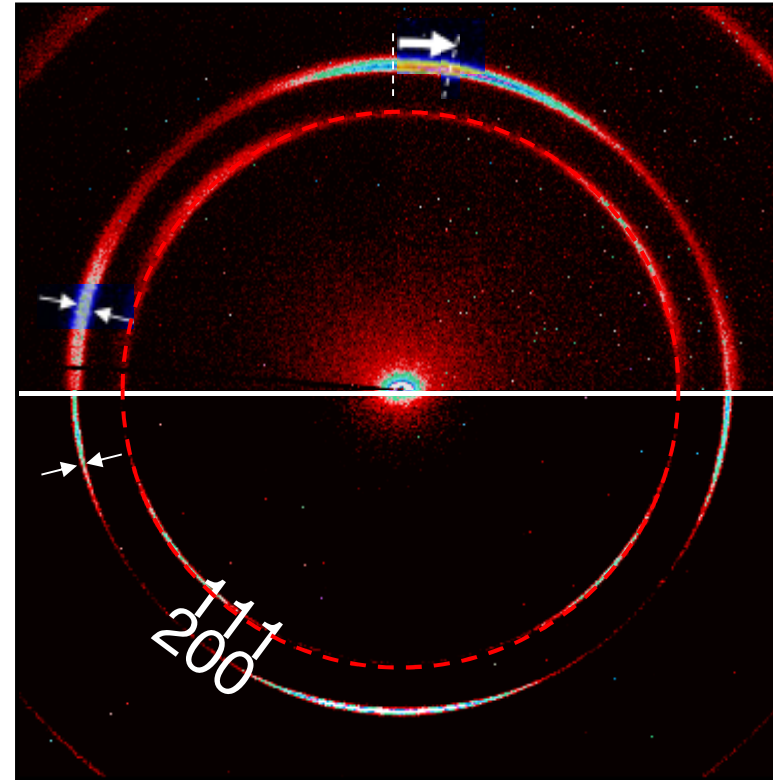
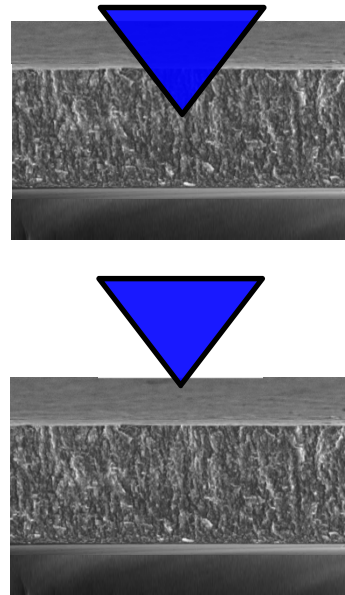
Helmholtz-Zentrum  
Geesthacht  
Zentrum für Material- und Küstenforschung

A. Zeilinger, J. Keckes et al.  
Montanuniversität Leoben, Austria



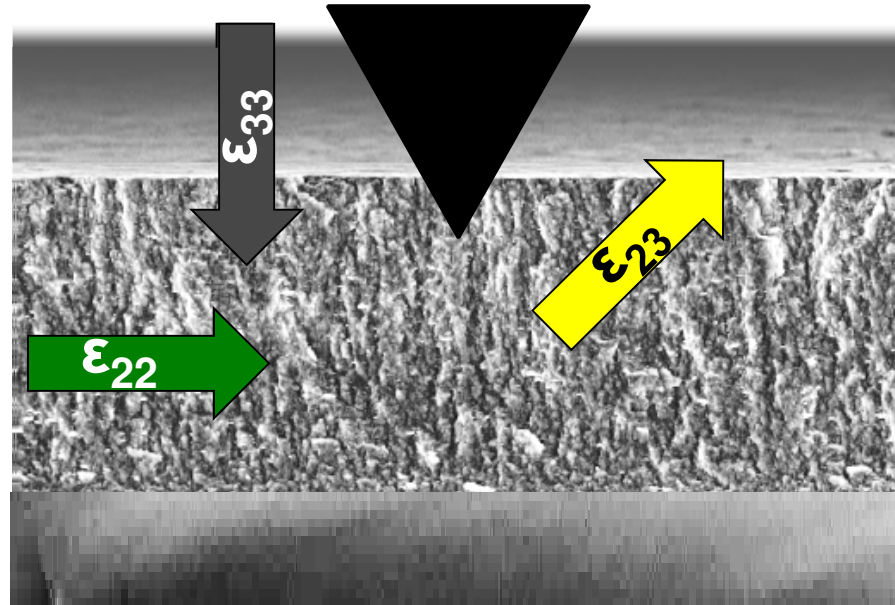
# nanoindentation





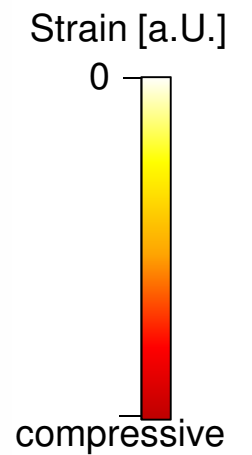
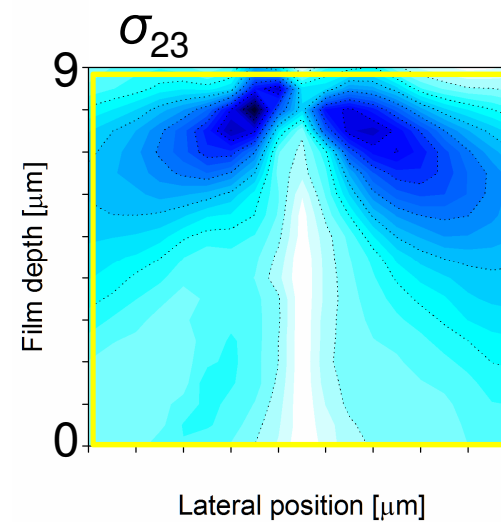
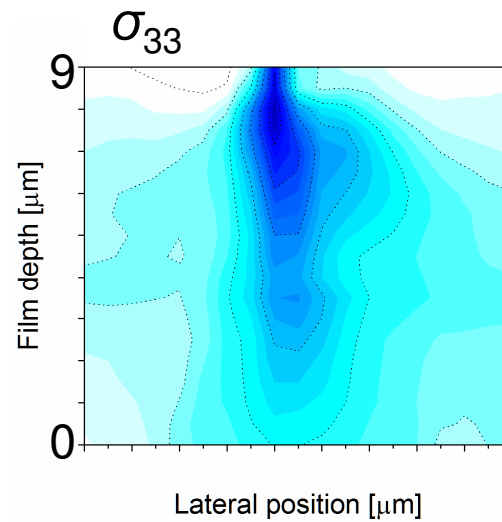
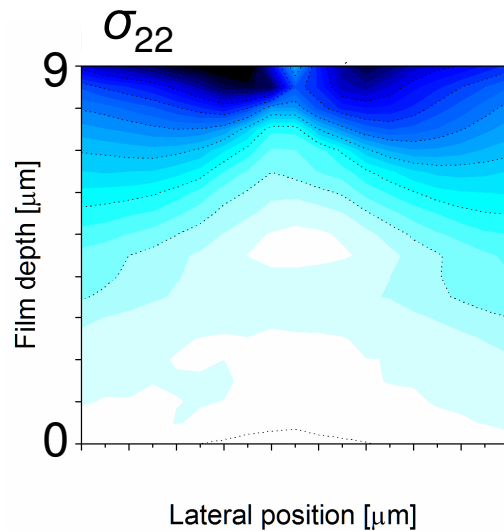
- i) Broadening of the diffraction rings: Strain 2<sup>nd</sup> 3<sup>rd</sup> order
- ii) Shift of the intensity maxima: Crystal rotation
- iii) Changes in ring shape: Macroscopic strain / Elastic response

# nanoindentation

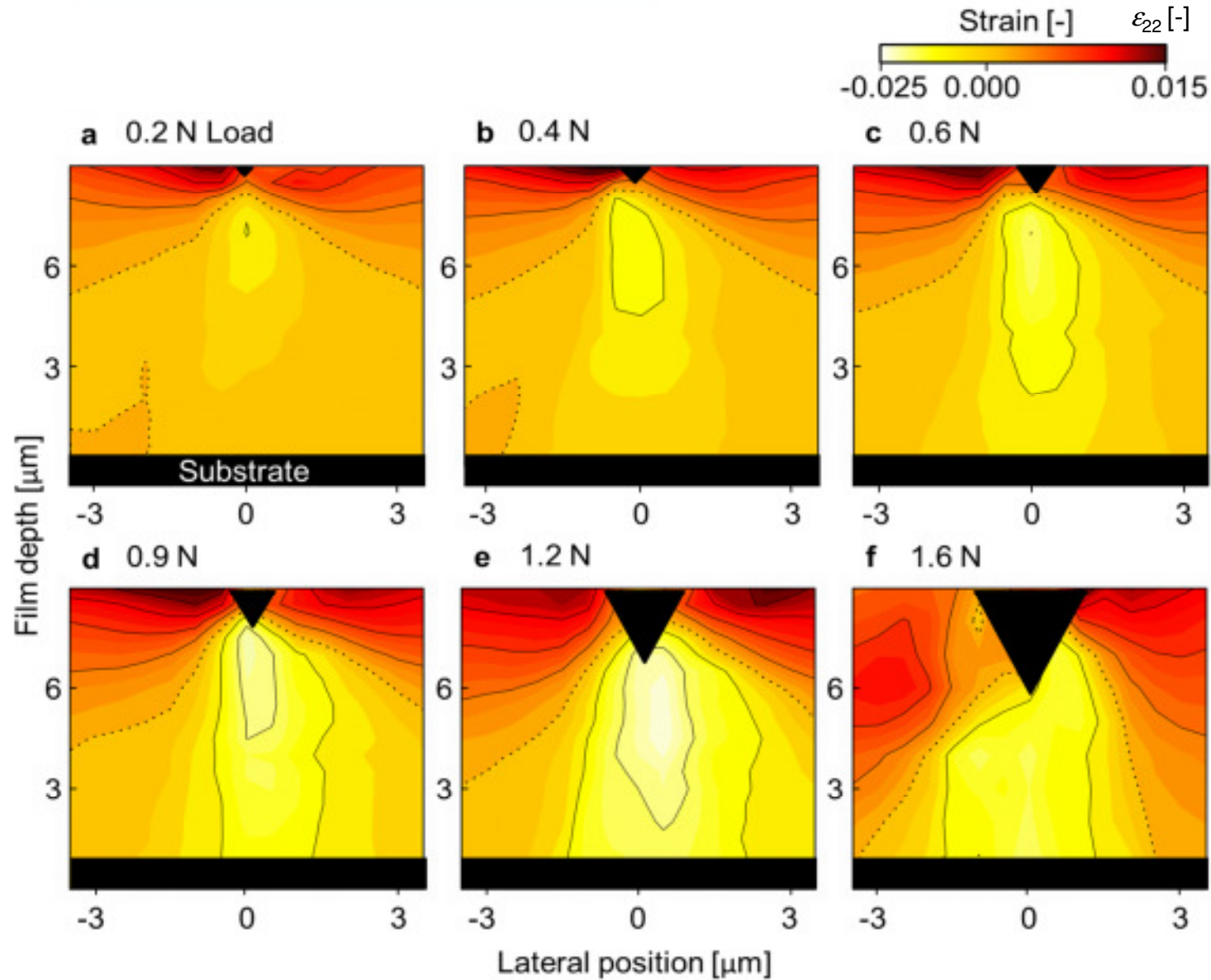


$\epsilon_{ij}$  = strain tensor

$\sigma_{ij}$  = stress tensor

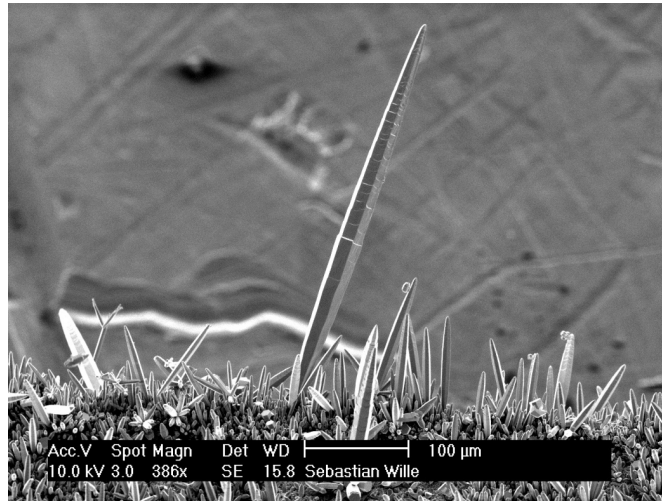


# nanoindentation

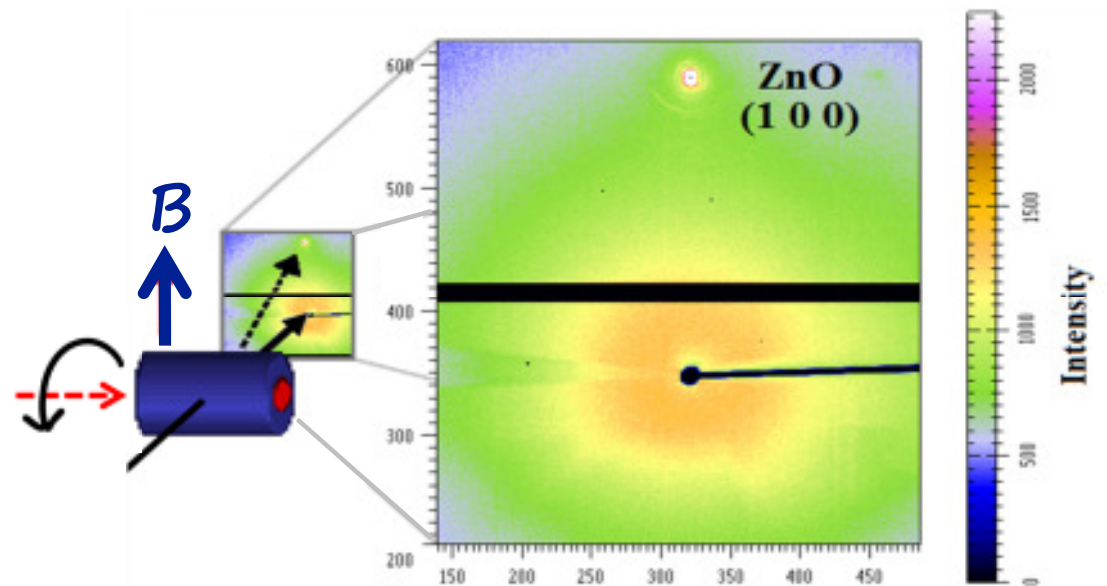


# nanodiffraction in external fields

S. Hrkac, B. Murphy  
Christian-Albrechts-Universität Kiel

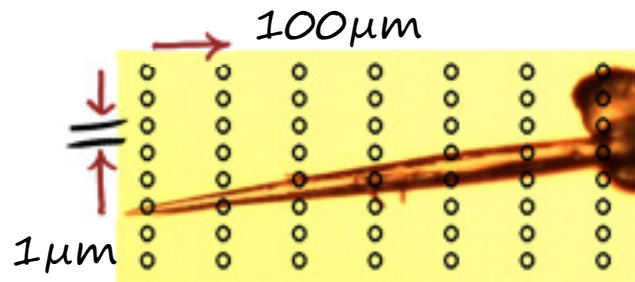


ZnO needles  
(piezoelectric)  
coated with METGLAS<sup>®</sup>  
(magnetostrictive)

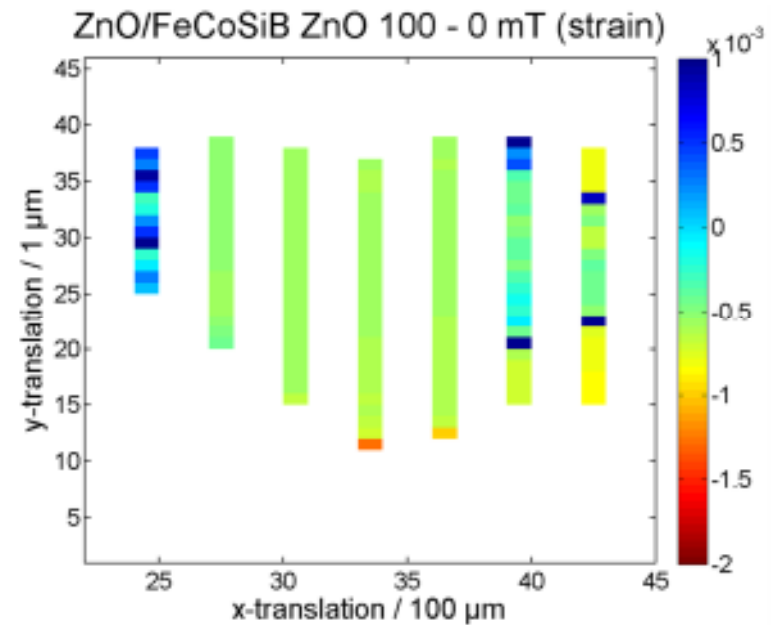
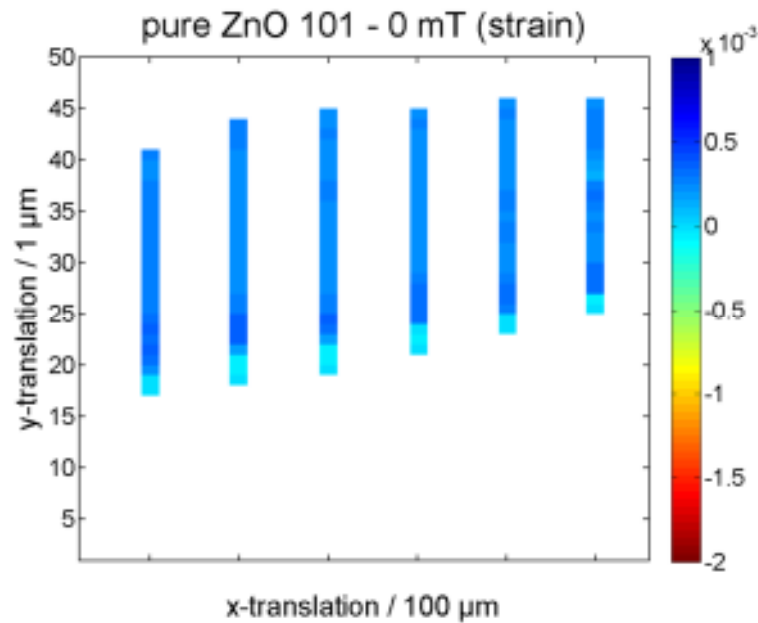


Hrkac et al., Appl. Phys. Lett. 103, 123111 (2013)

# nanodiffraction in external fields



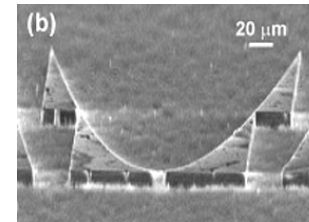
## measured strain



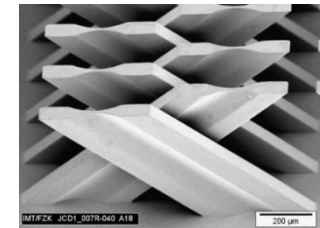
Hrkac et al. Appl. Phys. Lett. 103, 123111 (2013)

# future plans

Small(er) beams:  
kinoform lenses and  
adiabatic lenses

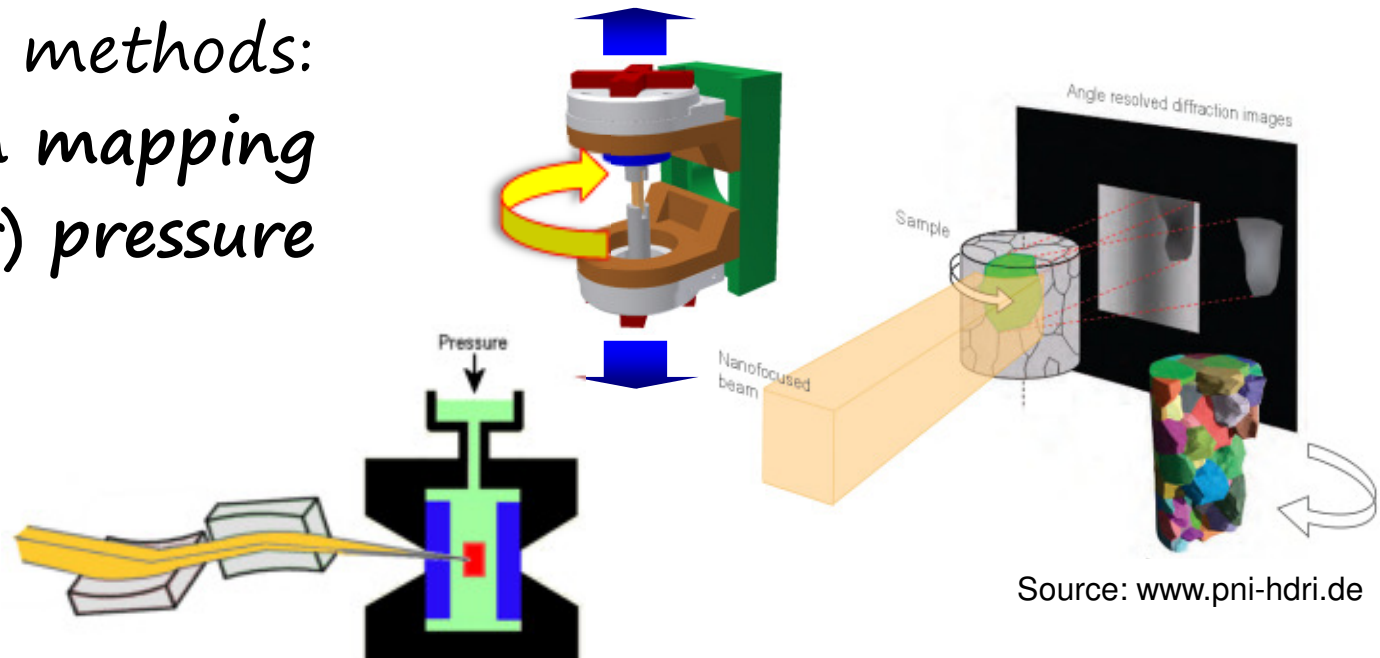


BNL, USA



KIT, Karlsruhe

New methods:  
grain mapping  
high(er) pressure





## summary

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nanodiffraction ♥ materials science

PO3 Nanofocus ♥ in situ methods

we can resolve  
strain fields, texture, crystal structure ...  
with sub- $\mu\text{m}$  resolution

interested ? please contact me

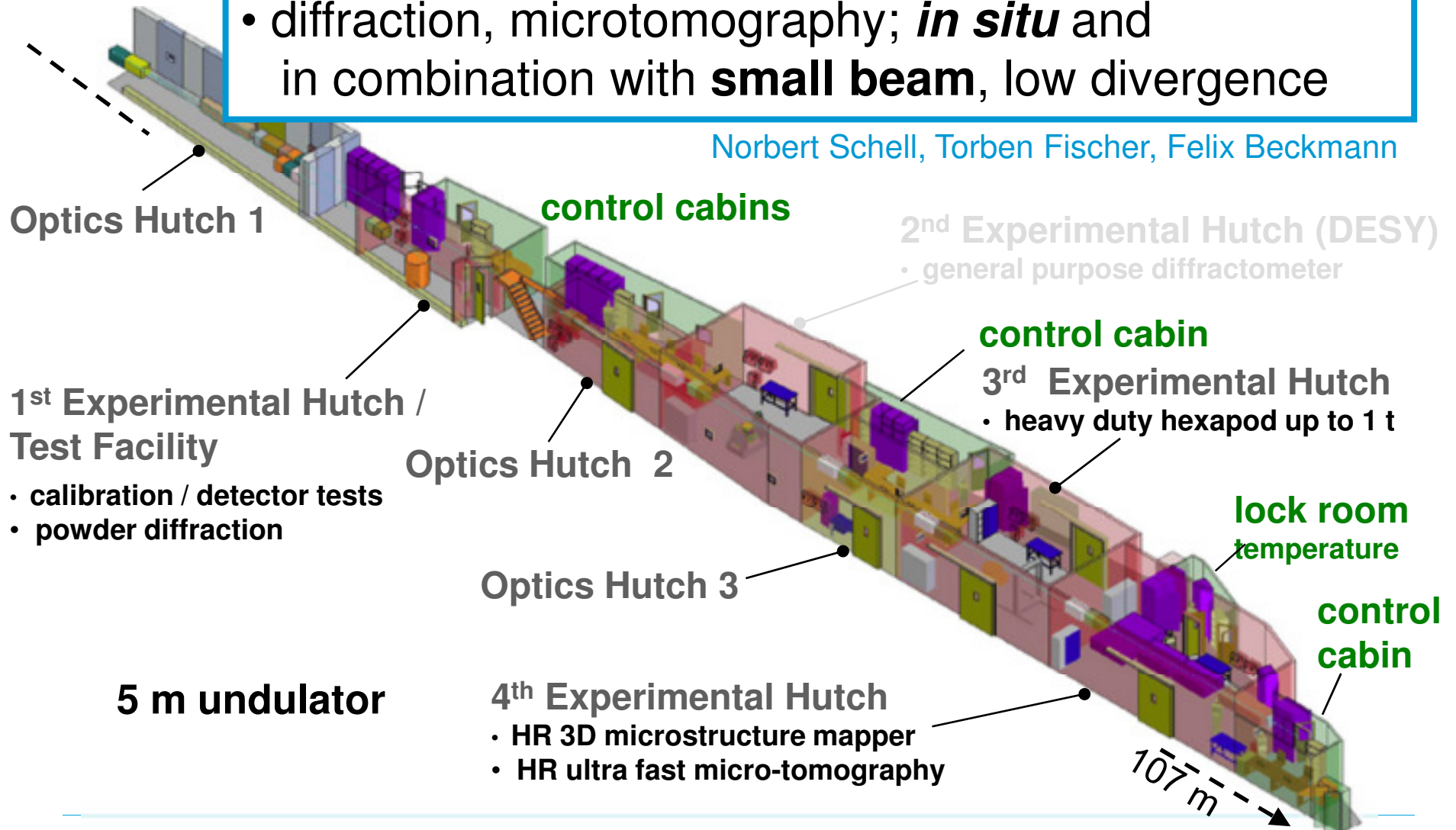
**HEMS (with DESY), IBL, BioSAXS (with EMBL) & P03 NFE (with CAU & DESY)  
at PETRA III**

**HARWI III**



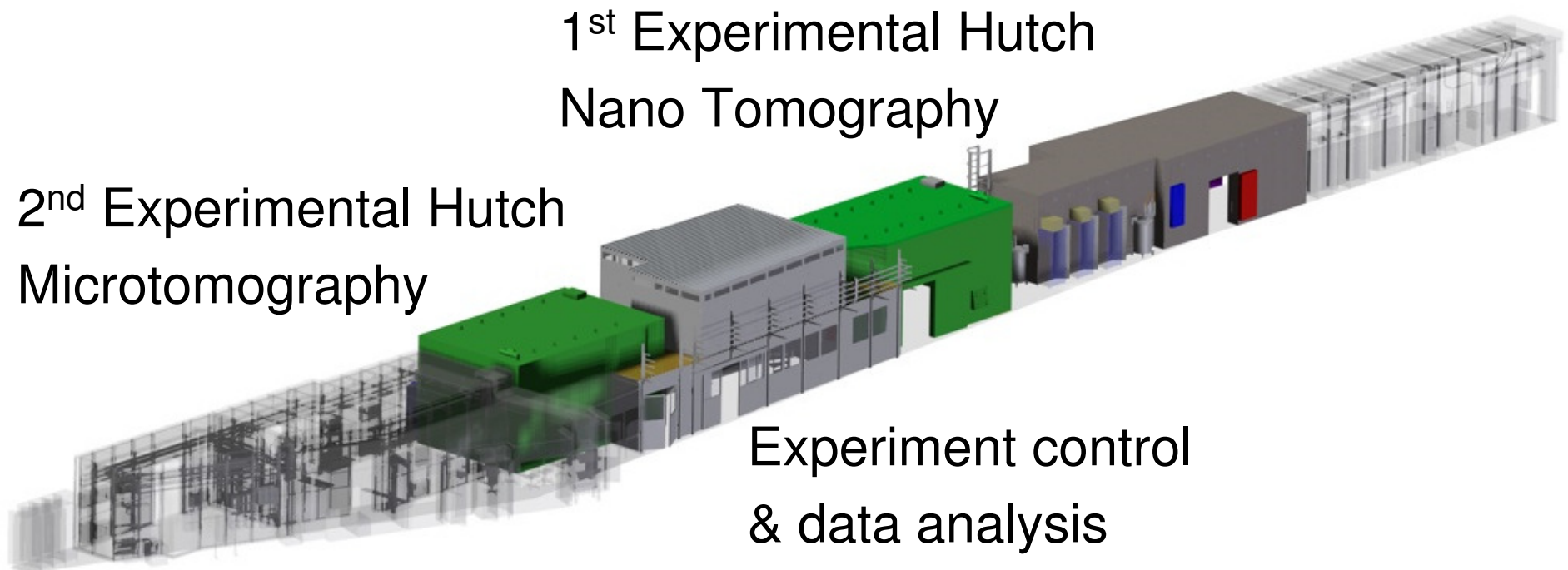
- high energy, P07 at PETRA III, 1/3 DESY (EH 2)
- diffraction, microtomography; *in situ* and in combination with **small beam**, low divergence

Norbert Schell, Torben Fischer, Felix Beckmann

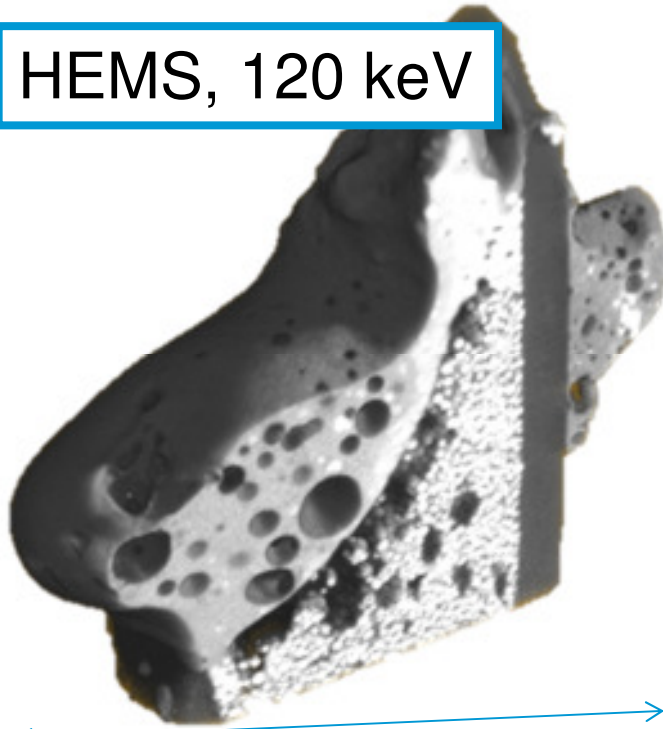


- medium energy, P05 at PETRA III
- micro- and nanotomography  
with mm- and  $\mu\text{m}$ -size samples

Fabian Wilde, Julia Herzen, Malte Ogurreck, Imke Greving, Felix Beckmann



HEMS, 120 keV

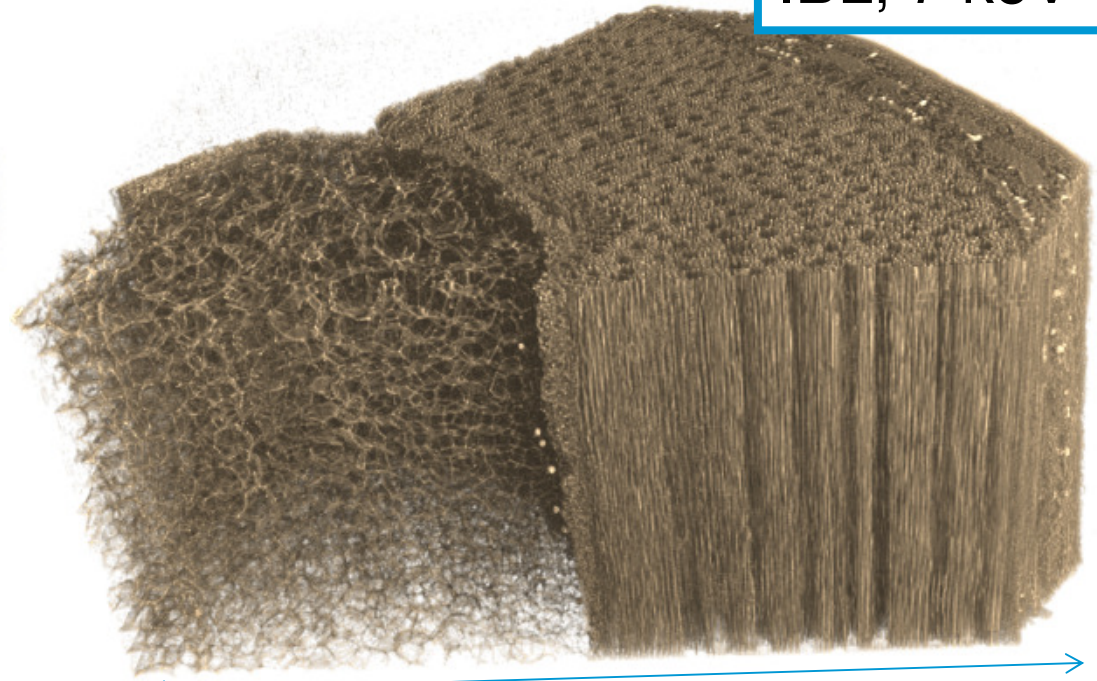


1.5 mm

understanding thermal spraying  
process for WC/Co coatings

J. Nellesen (TU Dortmund), F. Beckmann

IBL, 7 keV



3 mm

microstructure of wood  
on different length scales

S. Lautner, J. Fromm (Uni Hamburg)  
J. Herzen, F. Beckmann, F. Wilde

- medium energy, P12 at PETRA III, 85 % EMBL
- small-angle (solution) scattering, microfluidics

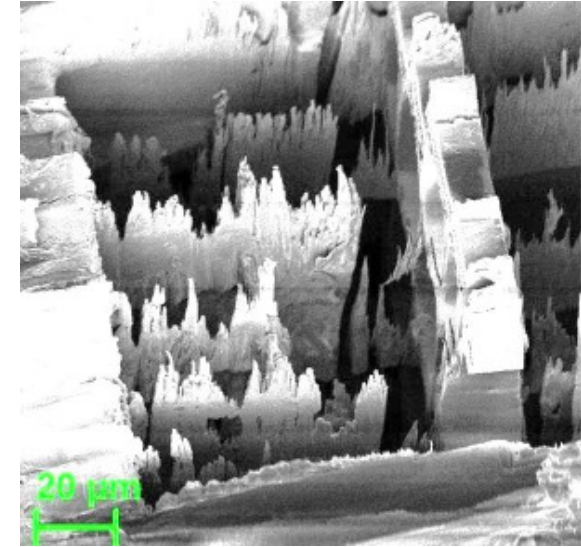
Vasyl Haramus, Regine Willumeit

strong request for  
small-angle scattering  
in biochemistry and  
in materials science  
(SFB 986)





Daniel Laipple



instrument in Geesthacht

preparation  
of thin wood sections



S. Storm, *Master's thesis*, Kiel (2012)

Experiments with  
**synchrotron radiation**  
and **neutrons**

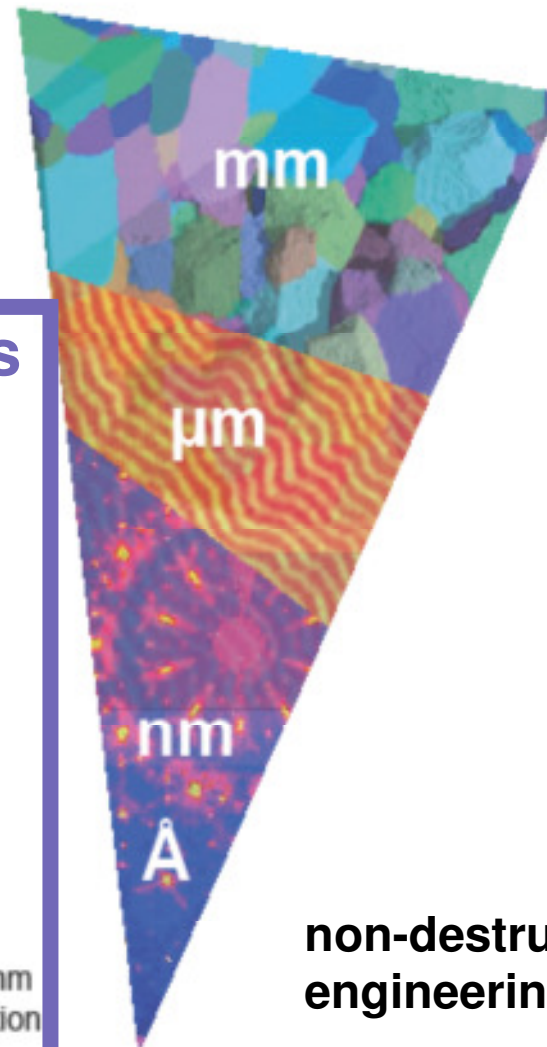
## Scattering Methods

### Reciprocal Space

- Small-angle scattering
- Diffraction
- High spatial resolution with small beams on the nm length scale

Resolution:

- Beam sizes down to 20 nm (scanning)
  - Diffraction  $10^{-4}$  Å
  - Small-angle scattering: particles and pore sizes ~ nm – 2000 nm
- *in situ* experiments with ms time resolution



## Imaging methods

### Real Space

- Micro and nano tomography
- Absorption contrast
- Phase contrast
- Diffraction contrast

Resolution:

< µm (700 nm)

Nano tomography:

~ 60 nm, smaller until 2016

**non-destructive characterisation**  
**engineering-specific in-situ devices**



# acknowledgements

for the great teamwork

S. Hrkac, B. Murphy, S. Storm

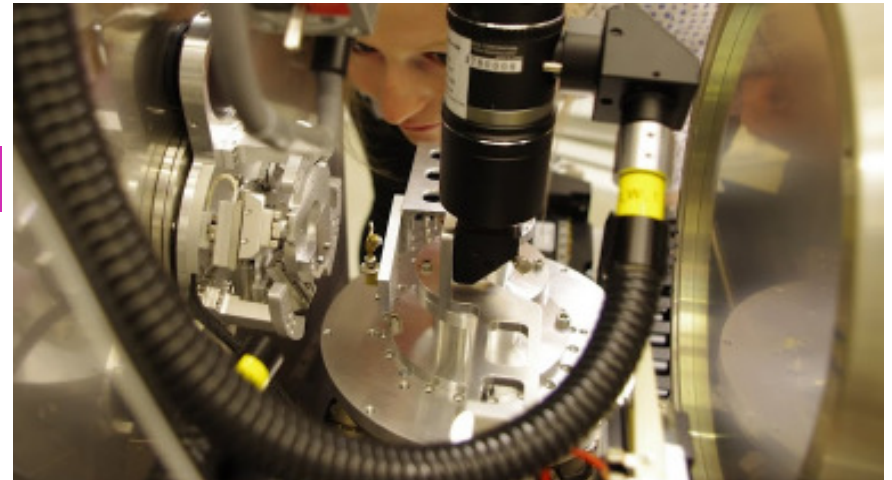


D. Laipple, M. Ogurreck



J. Keckes, J. Todt

A. Zeilinger, M. Stefenelli

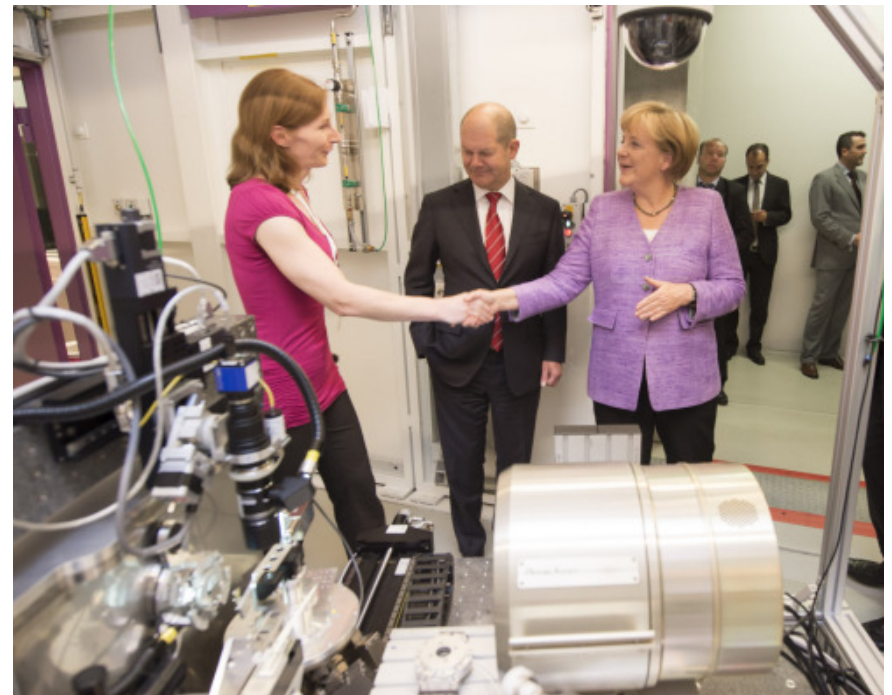


for the photons

P03 beamline team

A. Buffet, R. Doehrmann,

T. Boese, S. Roth et al.



for the dough

05KS7FK1 + 05K10FK3

