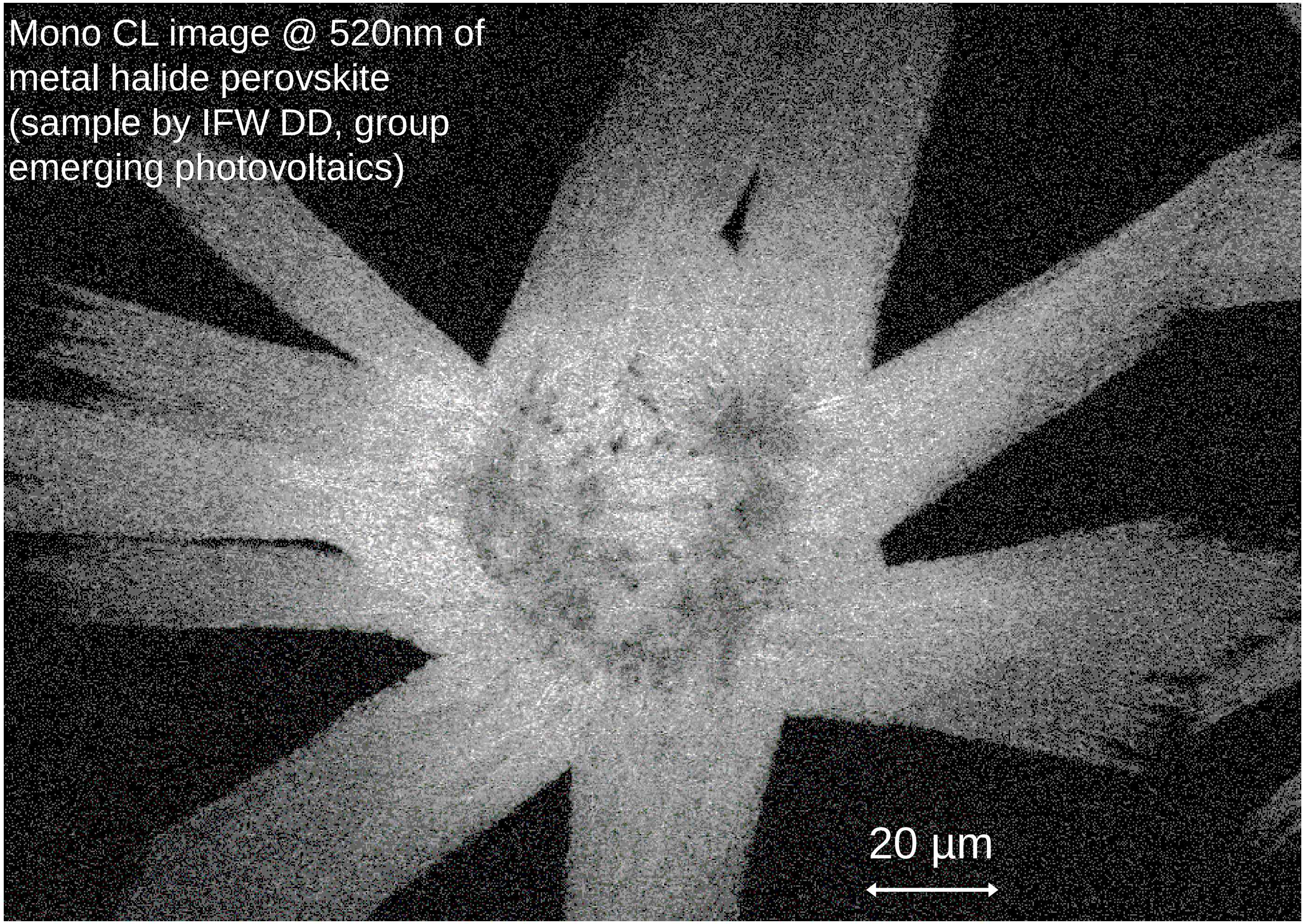

Mono CL image @ 520nm of metal halide perovskite (sample by IFW DD, group emerging photovoltaics)

A happy New Year 2025 to all !

Available experimental methods on the FEG-SEM Ultra55 and examples of application

IFMP / TUD, Institute Seminar

Ellen Hieckmann, 06. 01. 2025

Outline

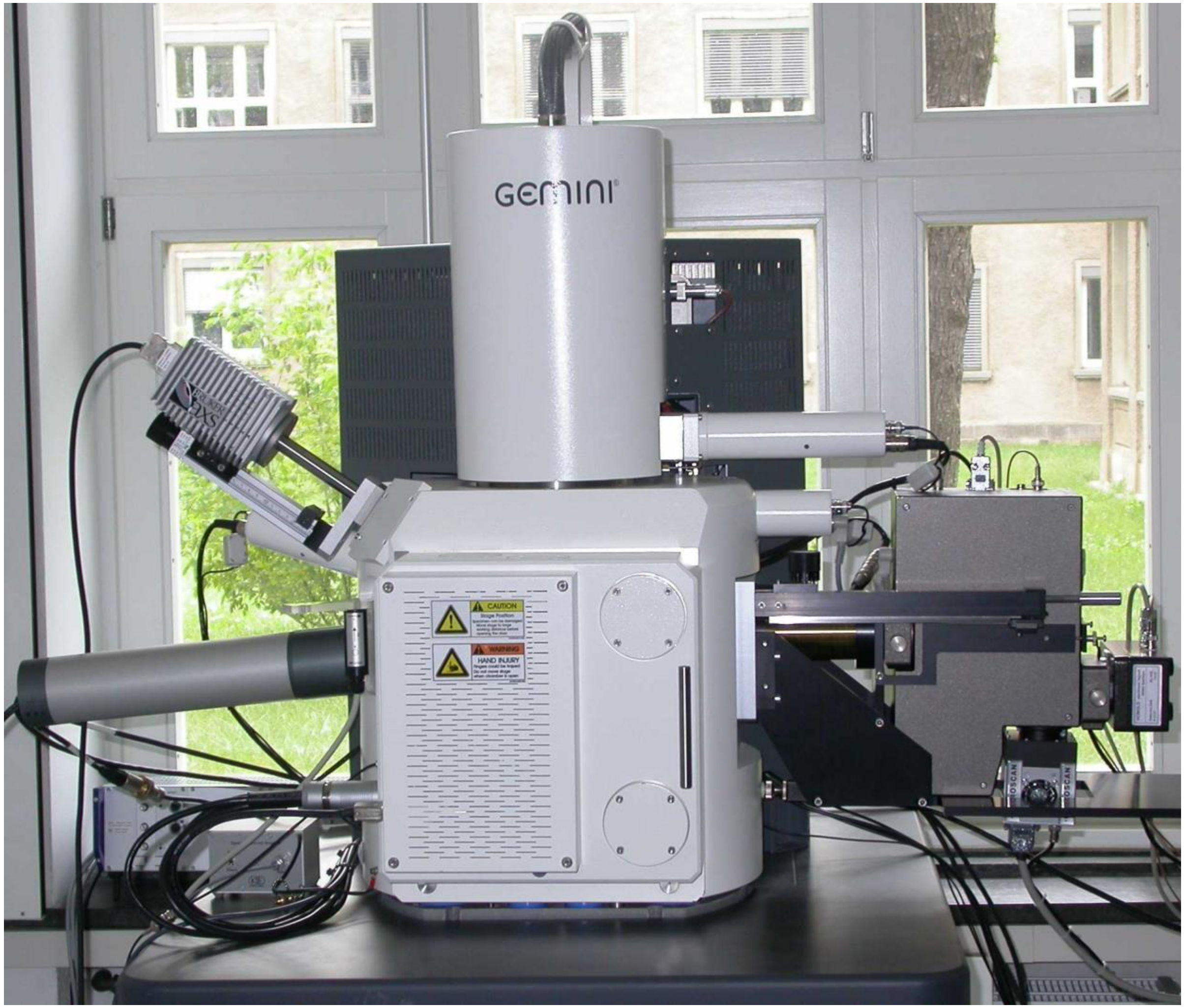
1 The SEM setup

2 Experimental methods and application examples

- 2.1 Imaging structures
- 2.2 Determination of crystal orientation, phases, textures & internal stresses
- 2.3 Analysis of chemical composition and spatial element distribution
- 2.4 Investigation of the luminescence behaviour
- 2.5 Implementation of experiments at low temperatures
- 2.6 Examination of electrical properties
- 2.7 Deformation of materials and *in-situ* observation

3 Access to the lab and future activities

1 The SEM setup



Zeiss Ultra55

with Schottky FEG

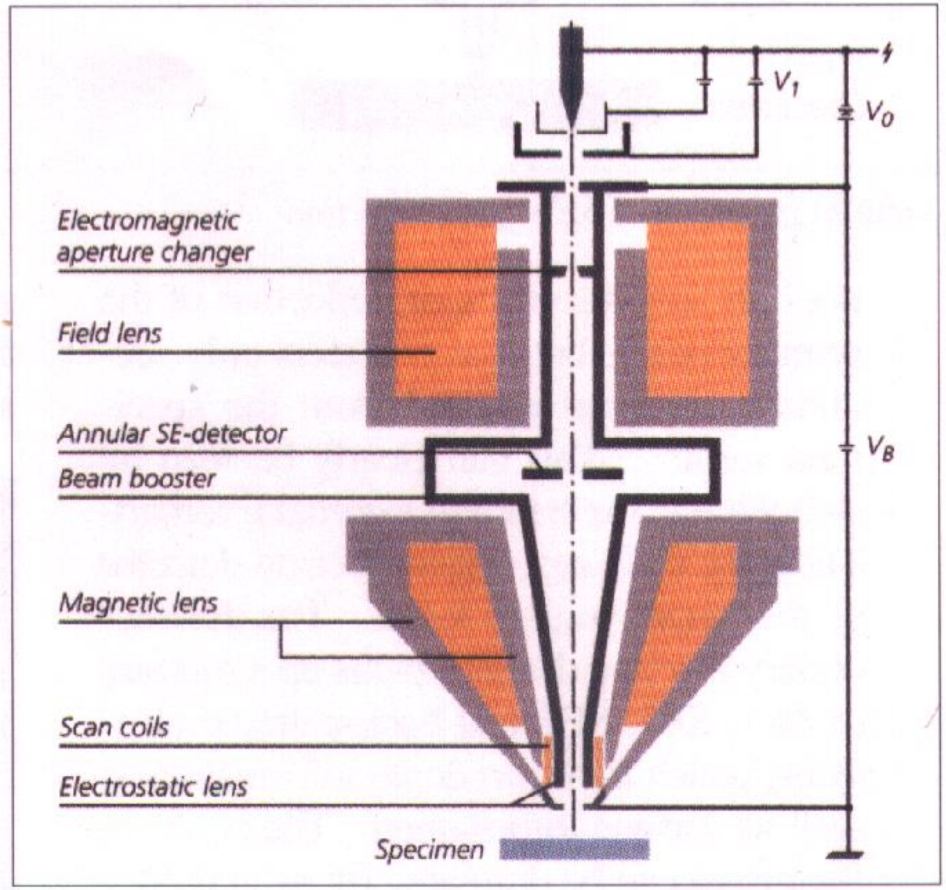
REC B1: SEM with active magnetic field compensation and low-vibration ground floor

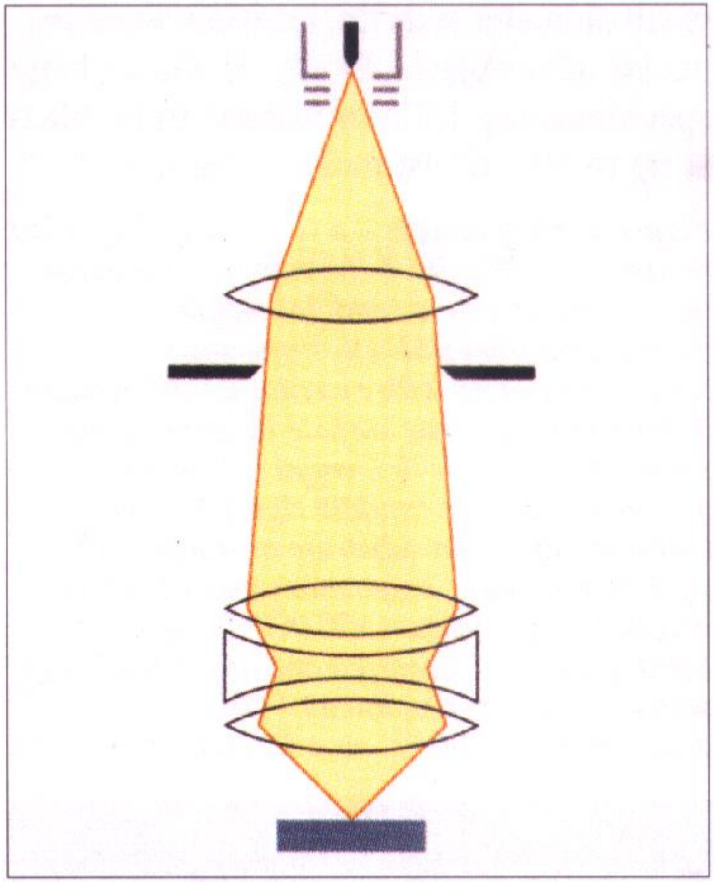
REC B15: cooling water aggregate, backing pump, compressor

REC B2: N gas supply (reconstruction planned)

1 The SEM setup

Electron optics: Gemini principle by Zeiss

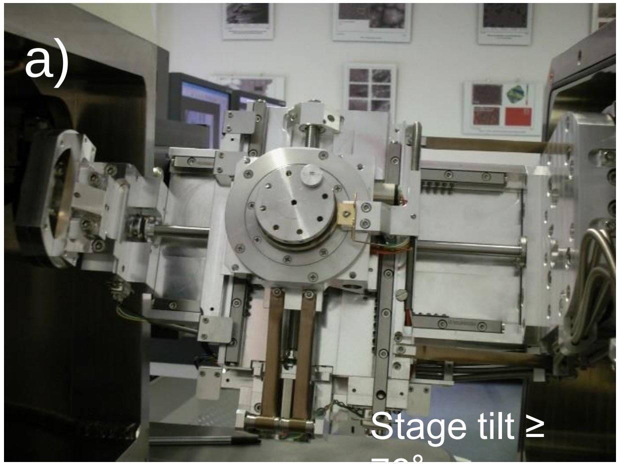


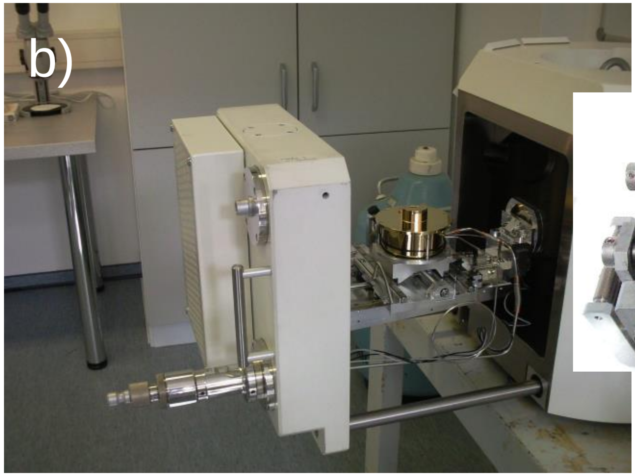


primary electron energy between 30 keV and 100 eV (cross-over free), maximum probe current 40 nA

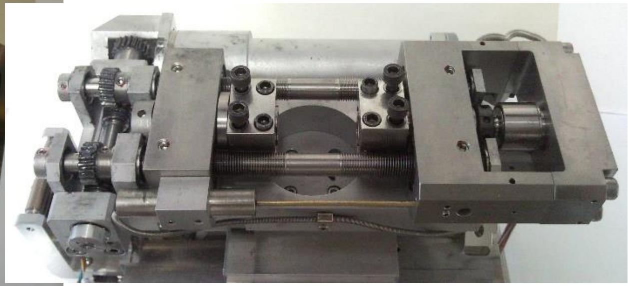
1 The SEM setup

3 Stages:





c)



a) 6 - axes stage for eucentric tilt at room temperature, maximum sample displacement: ± 65 mm in *x* and *y* direction

b) 3 - axes stage with cryostat for investigations at low temperatures (5 K)

c) 3 - axes stage with push-pull device for *in-situ* uniaxial mechanical deformation (force up to 10 kN)

2 Experimental methods and application examples

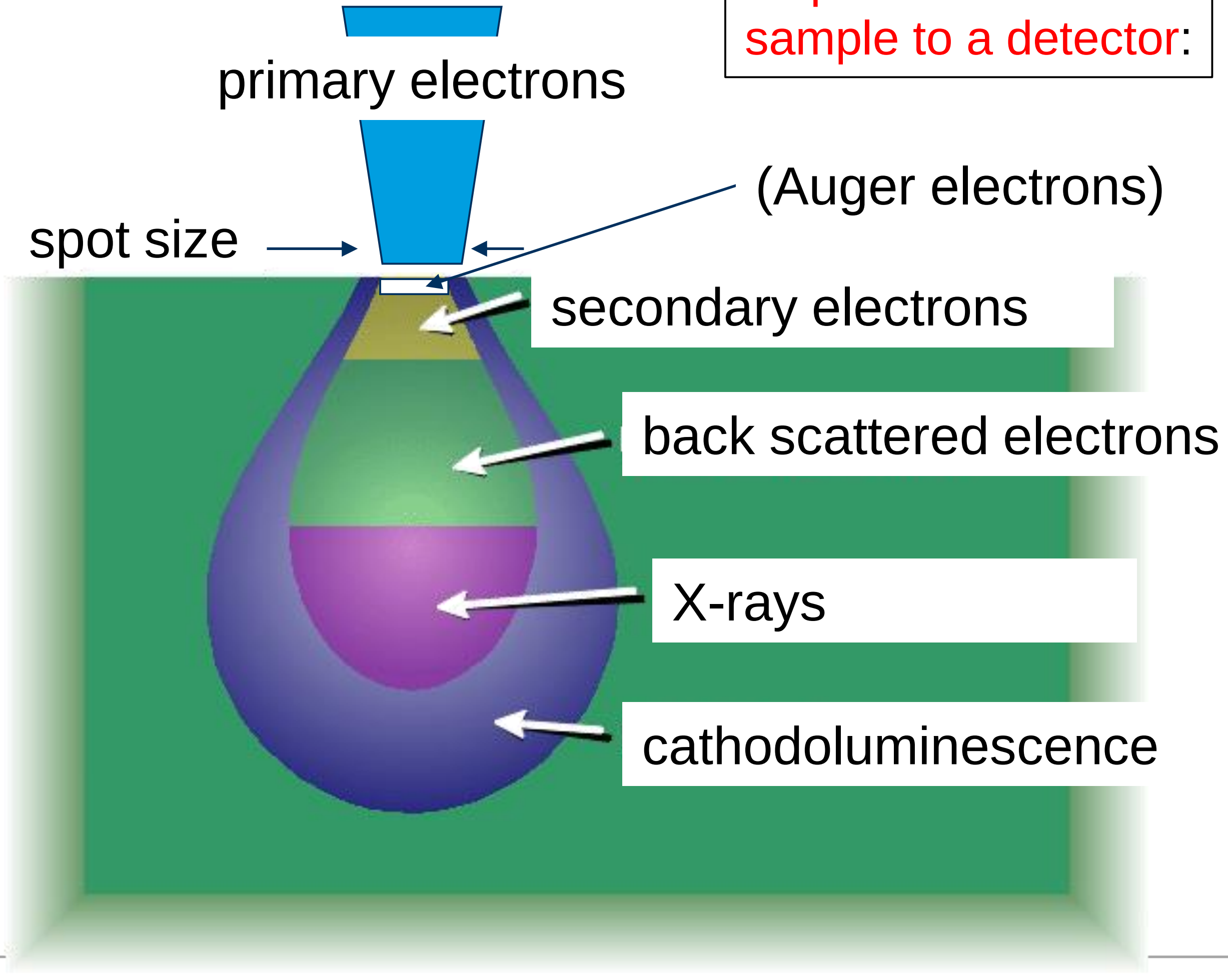
2.1 Imaging structures

Elastic and in-elastic scattering processes in the interaction volume (depending on acceleration voltage EHT, *Z* and mass density of the sample)

➔ different information depth for different interaction products

➔ generally the spatial resolution is lower than the spot size diameter (about 1 nm) !

Interaction products that can escape upwards from the sample to a detector:



primary electrons

spot size

(Auger electrons)

secondary electrons

back scattered electrons

X-rays

cathodoluminescence

2.1 Imaging structures

- by secondary electrons with SE2 and InLense detector
- by backscattered electrons with

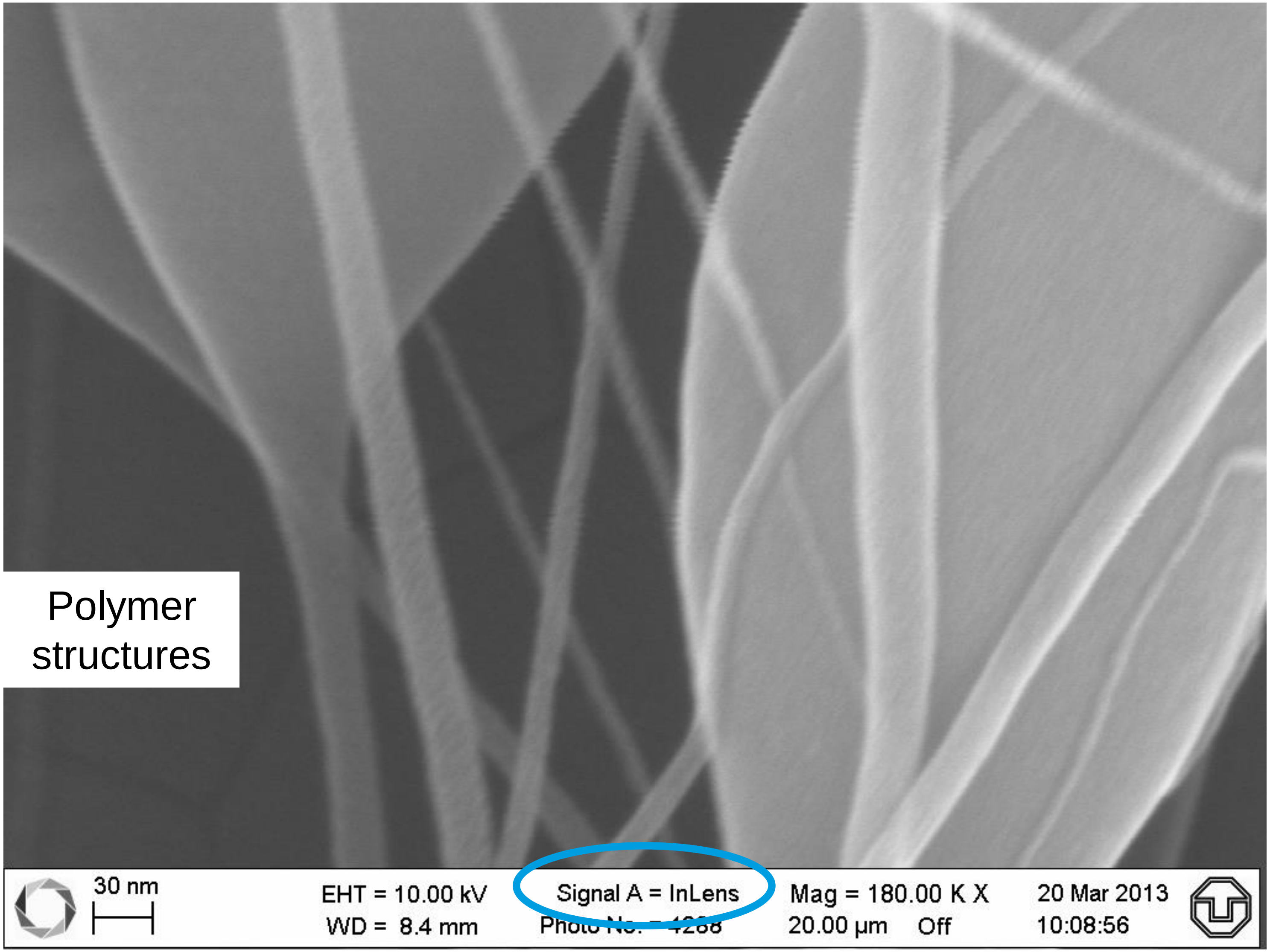
  * ASB (angle selective backscatter detector with 4 segments, also suited for topography contrast imaging for EHT > 7 kV)

  * ESB („in-lense“ energy selective backscatter detector for EHT < 7 kV)

  * FSD (forward scatter detector with 4 diodes for investigations of tilted samples)

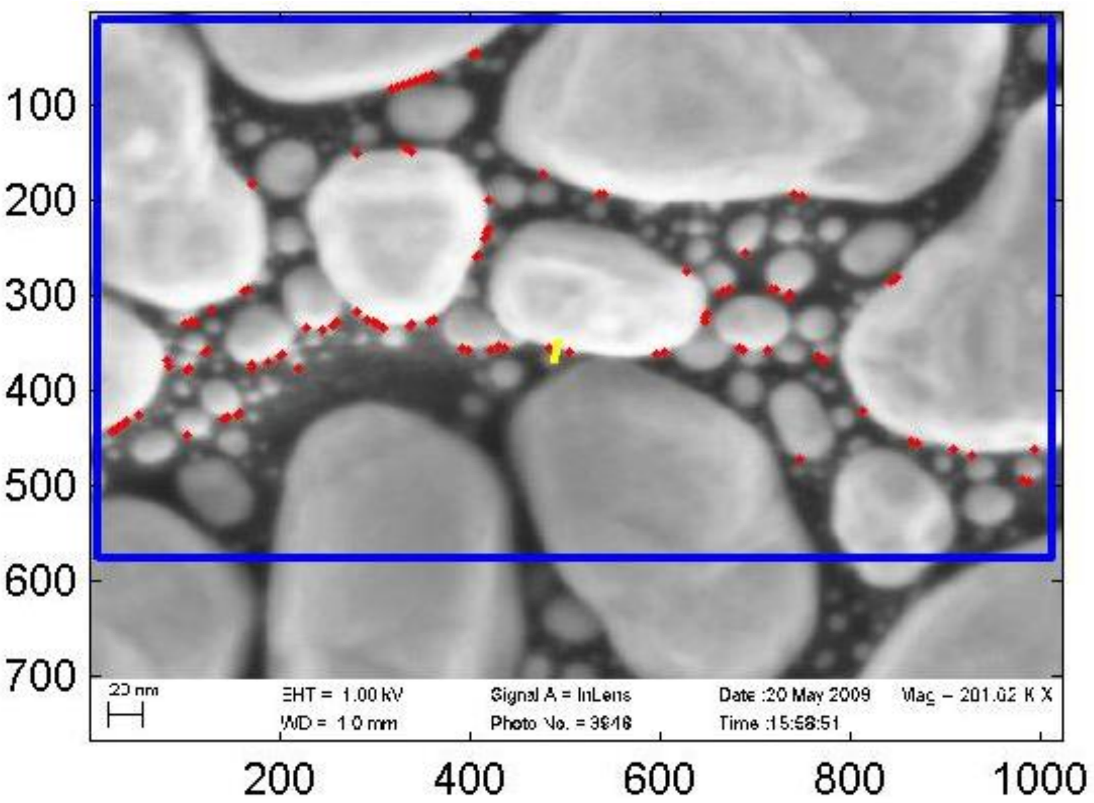
- by transmitted high energy electrons with STEM detector
- by leakage current with sample current detector
- by cathodoluminescence with photomultiplier detectors

InLense detector



Topography contrast

Resolution limit (Au on carbon substrate @ 1 kV)



Results

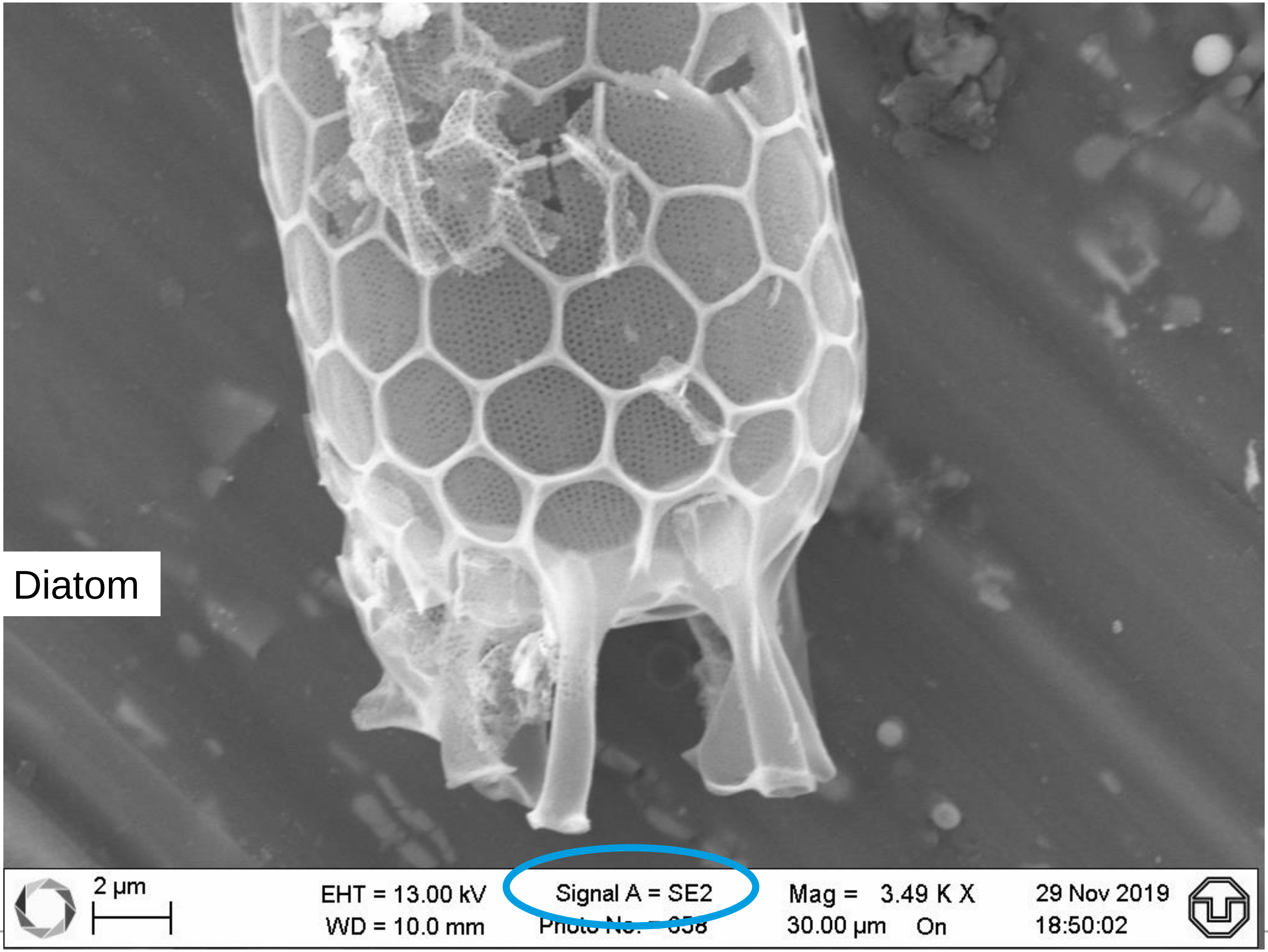
Resolution=1.8±0.27nm

110 edges evaluated

S/N ratio=349.1

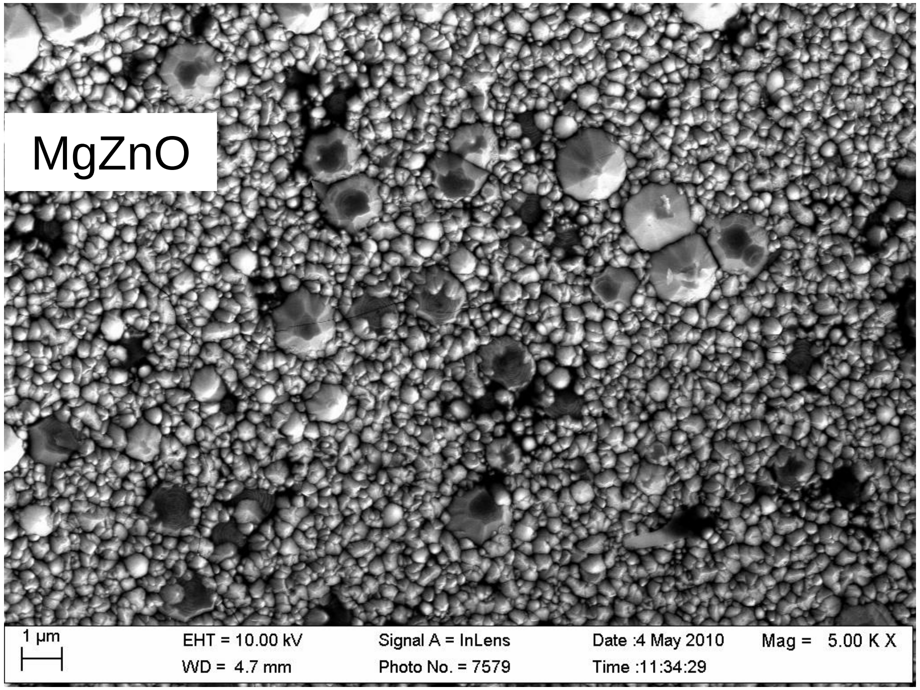
PATE V3.3 03-Mar-2009

Everhardt - Thornley detector (SE2)



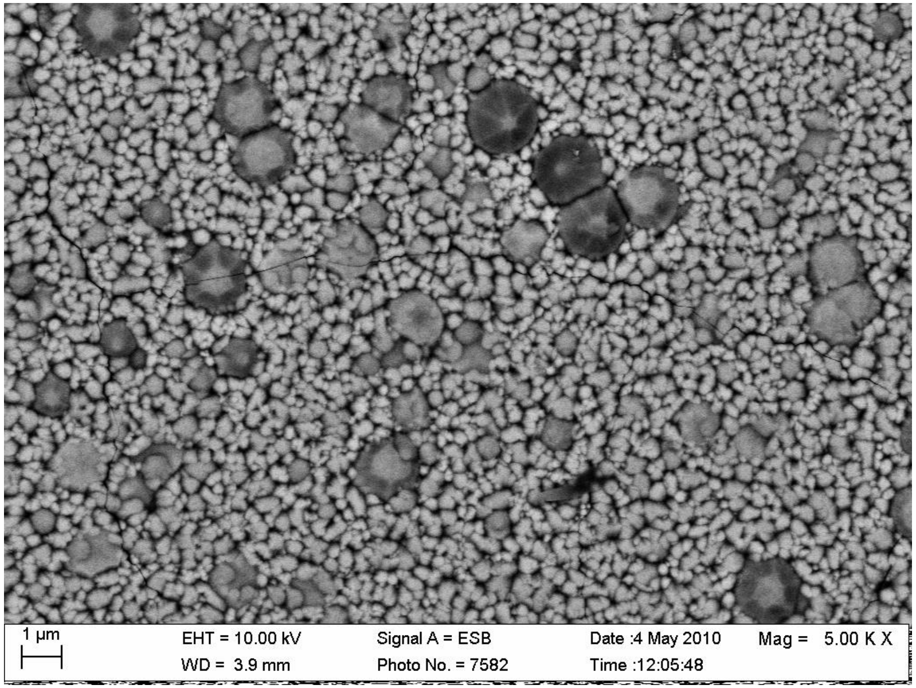
Topography contrast

InLense detector



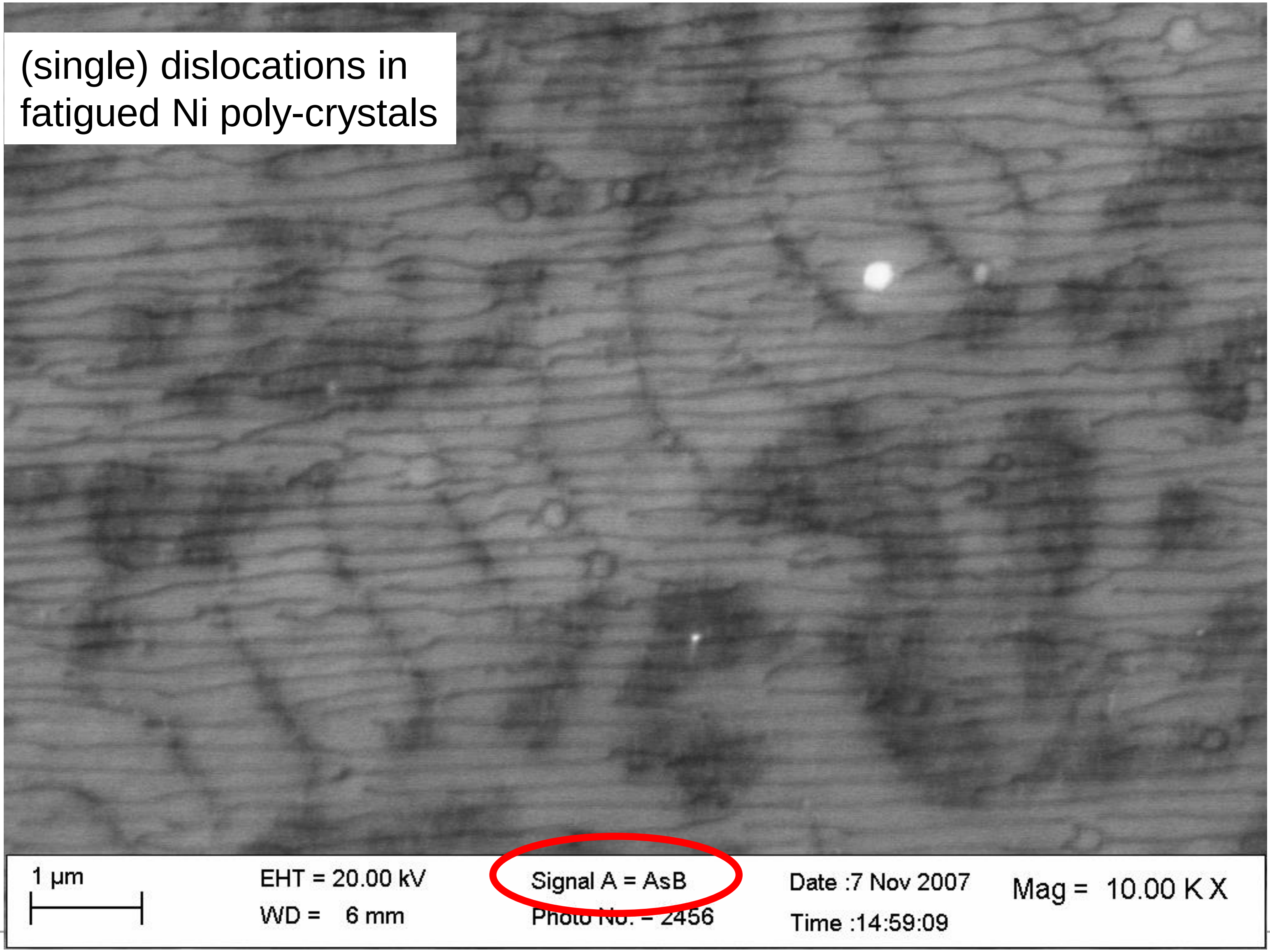
Topography contrast

ESB detector



Z contrast

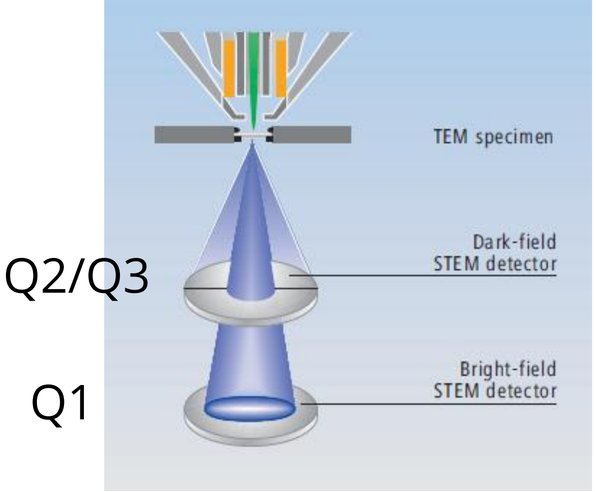
ASB detector



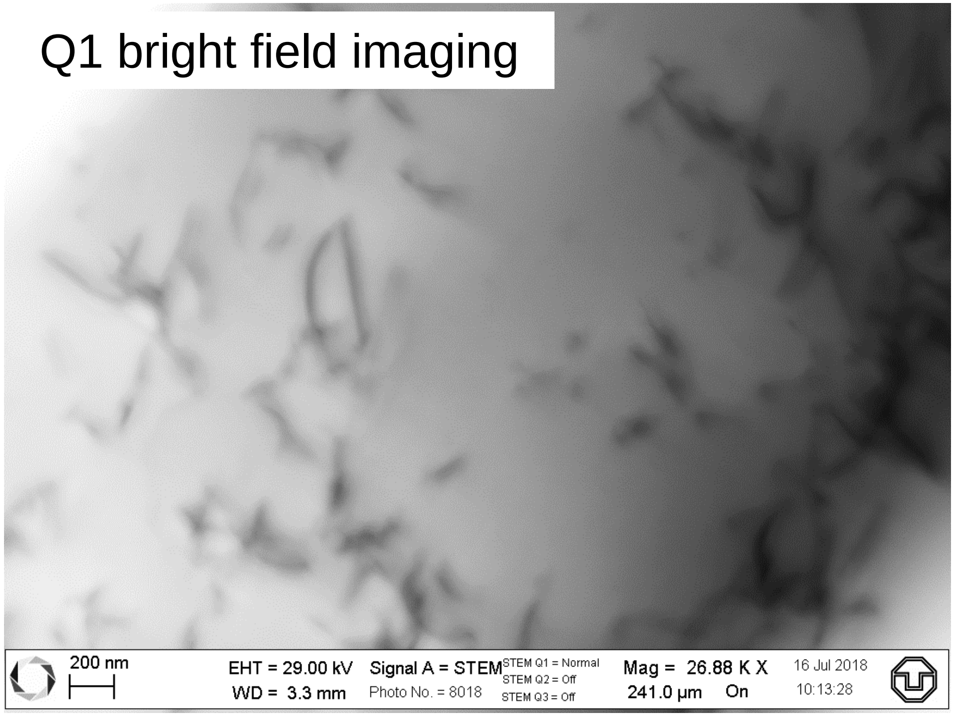
(single) dislocations in fatigued Ni poly-crystals

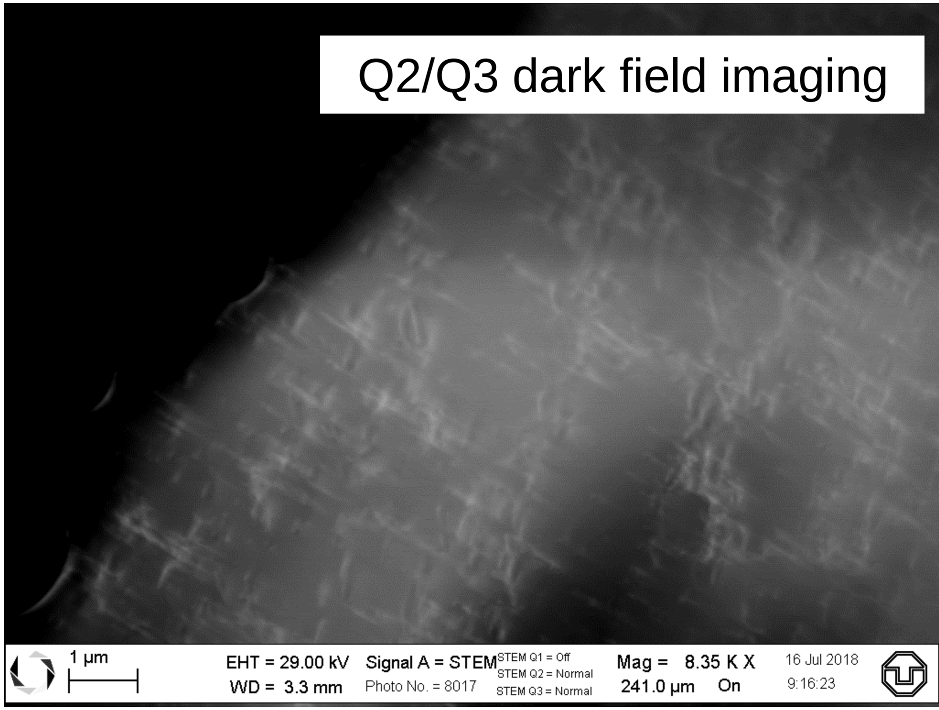
Channelling contrast

STEM detector



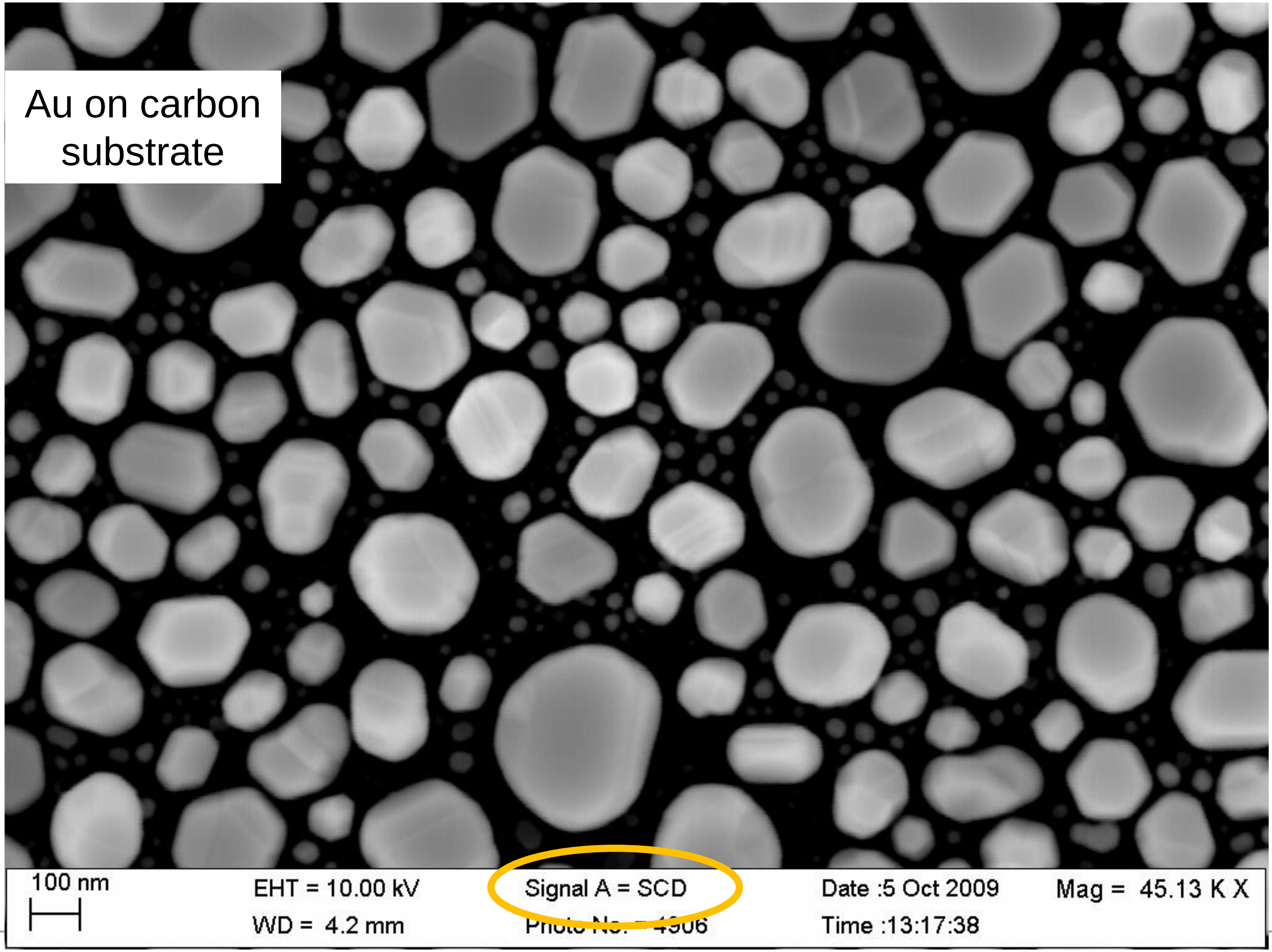
Dislocations in Si single crystals
(thinned sample for transmission geometry)


Q1 bright field imaging


Q2/Q3 dark field imaging

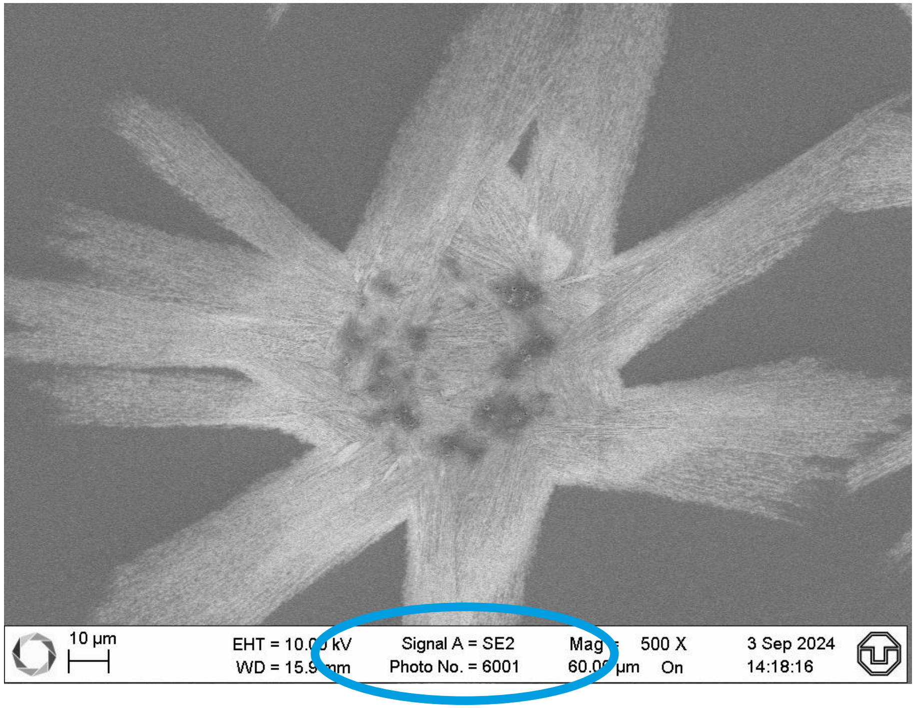
scattering contrast

SCD detector

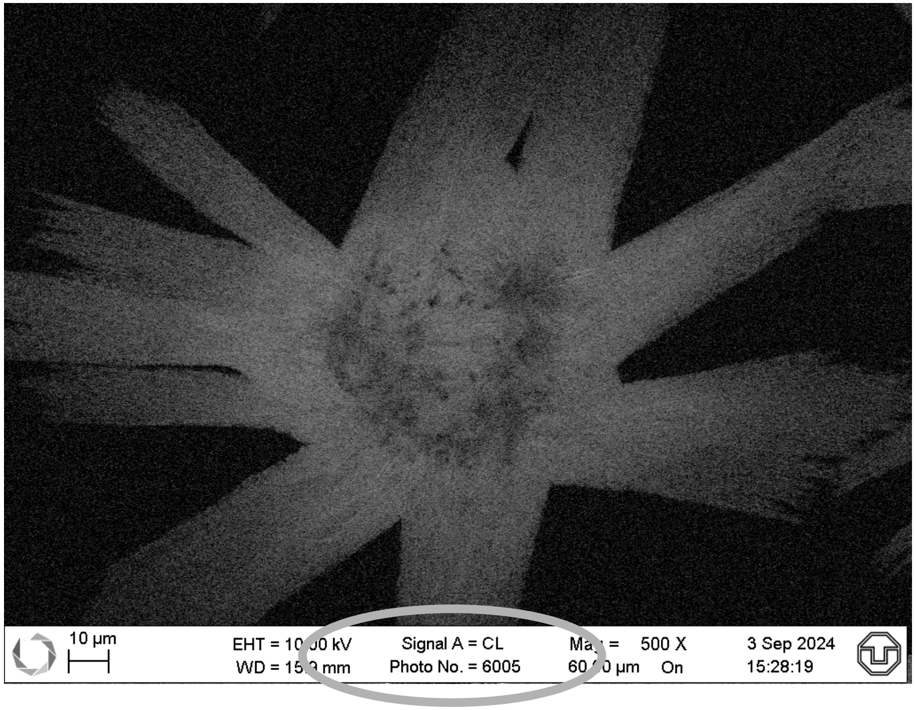


Z contrast

SE2 detector



Photomultiplier detector



metal halide perovskite (CsPbJ)

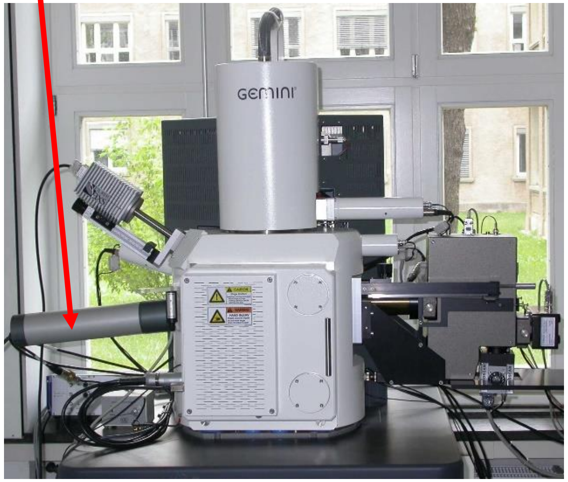
Topography contrast

Defect contrast in luminescence intensity @ 520 nm

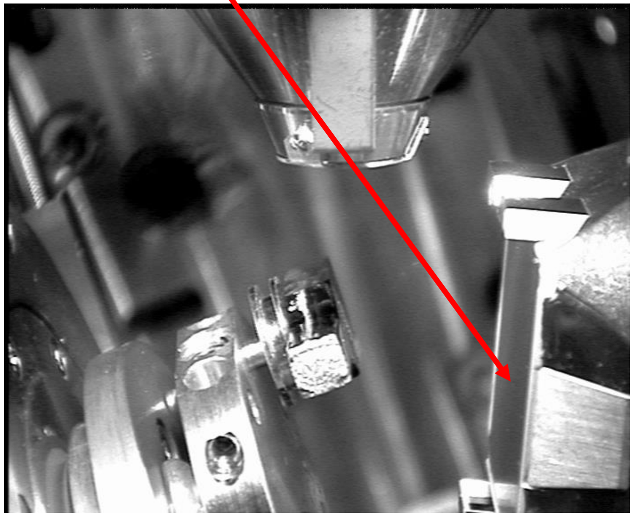
2.2 Determination of crystal orientation, textures, phases & internal stresses

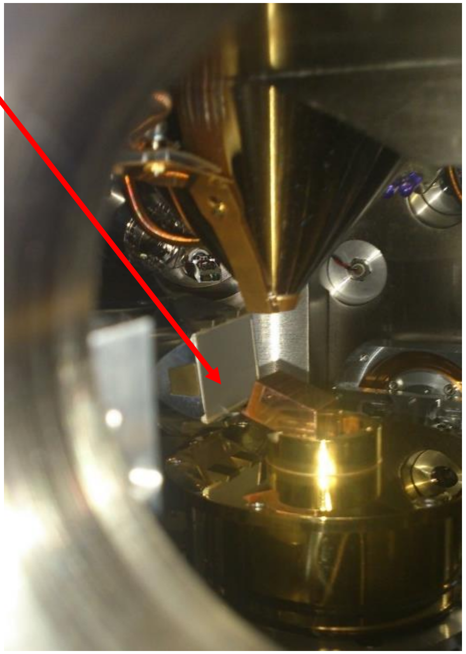
EBSD detector

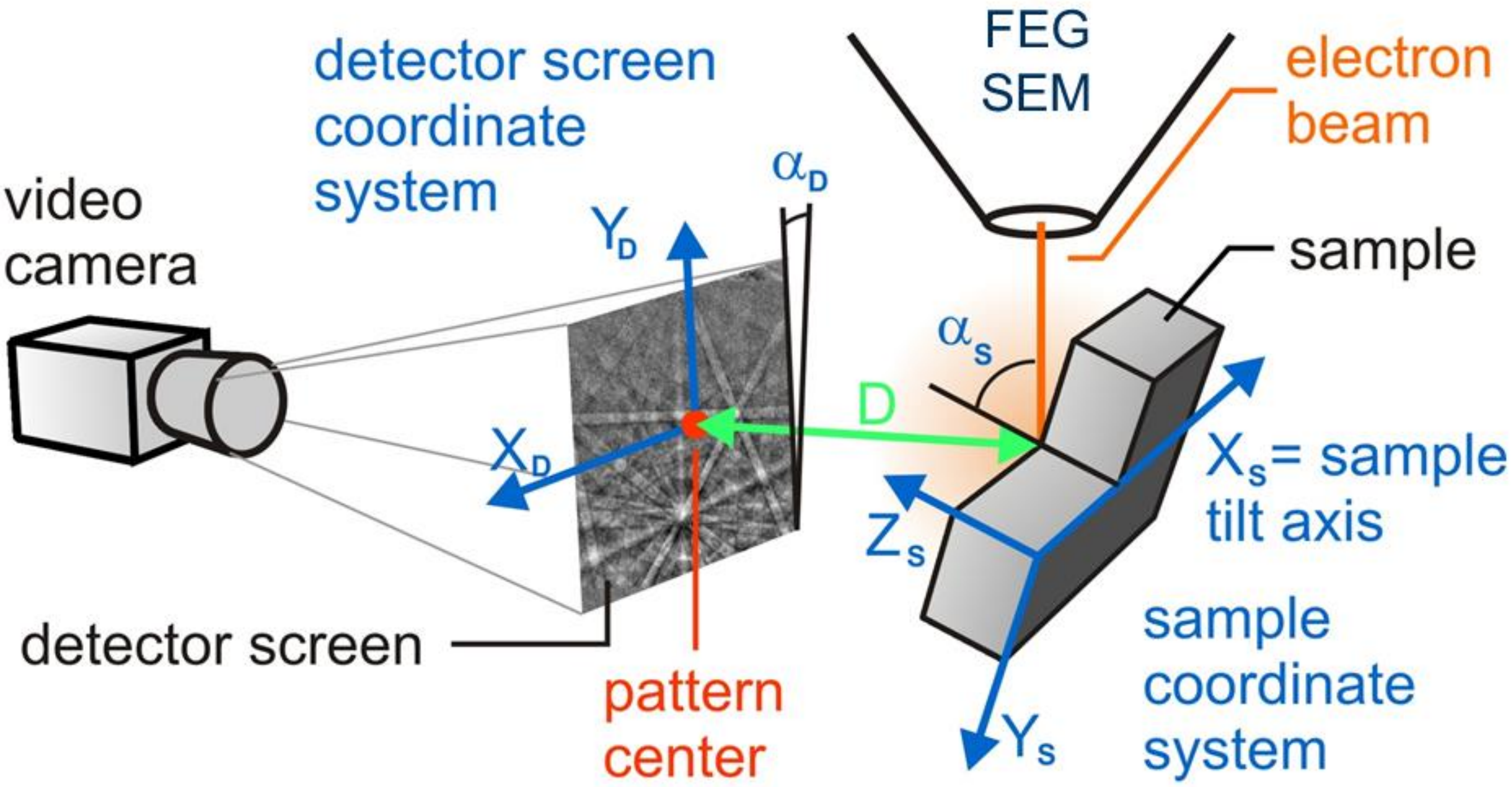
Nordlys EBSD detector, channel 5 and ccEBSD software



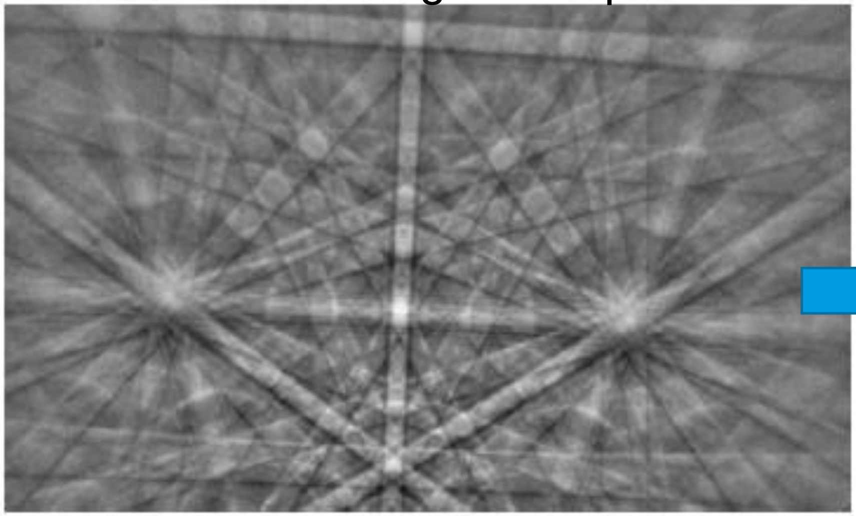
Electron backscatter diffraction (EBSD)
@ RT and low temperatures



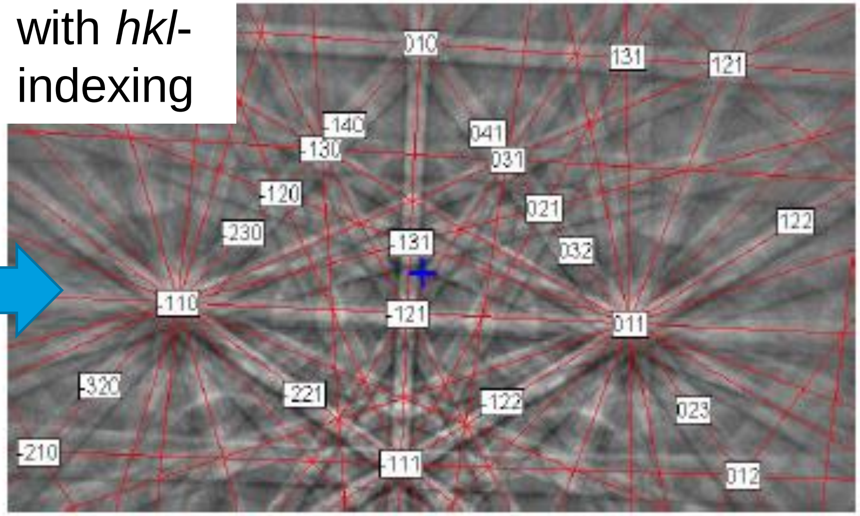




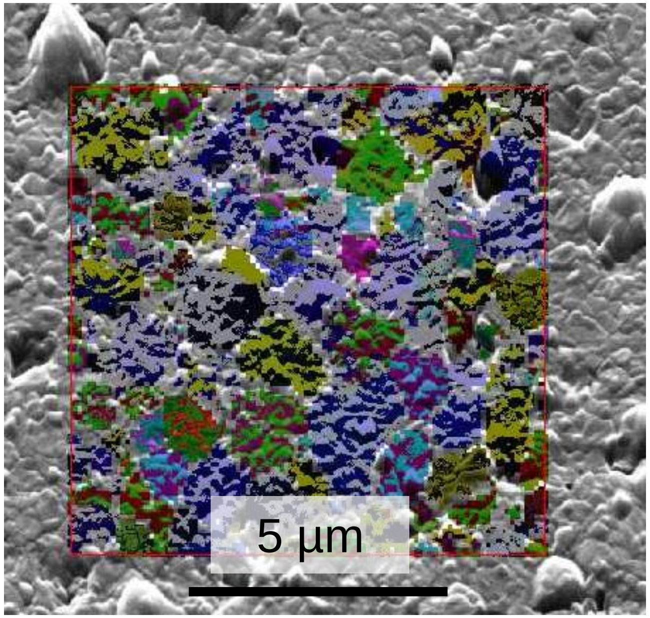
Hardware: recording EBSD pattern



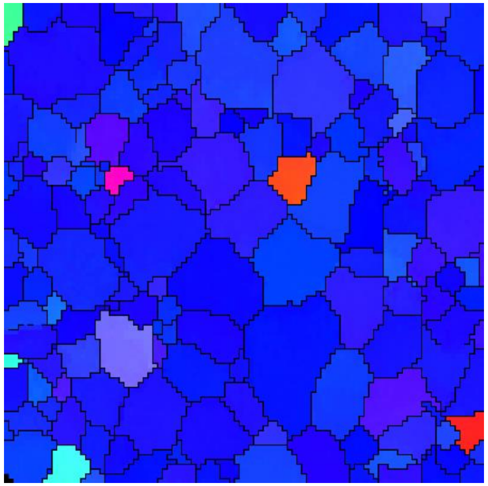
Software: detection of Kikuchi bands with *hkl*-indexing



Orientation mapping on sample surface

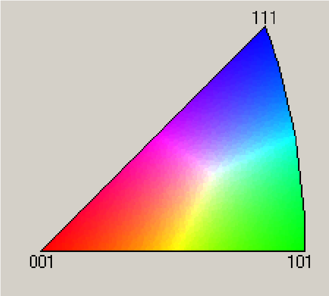


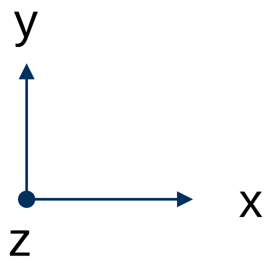
Orientation map of
z axis with GB



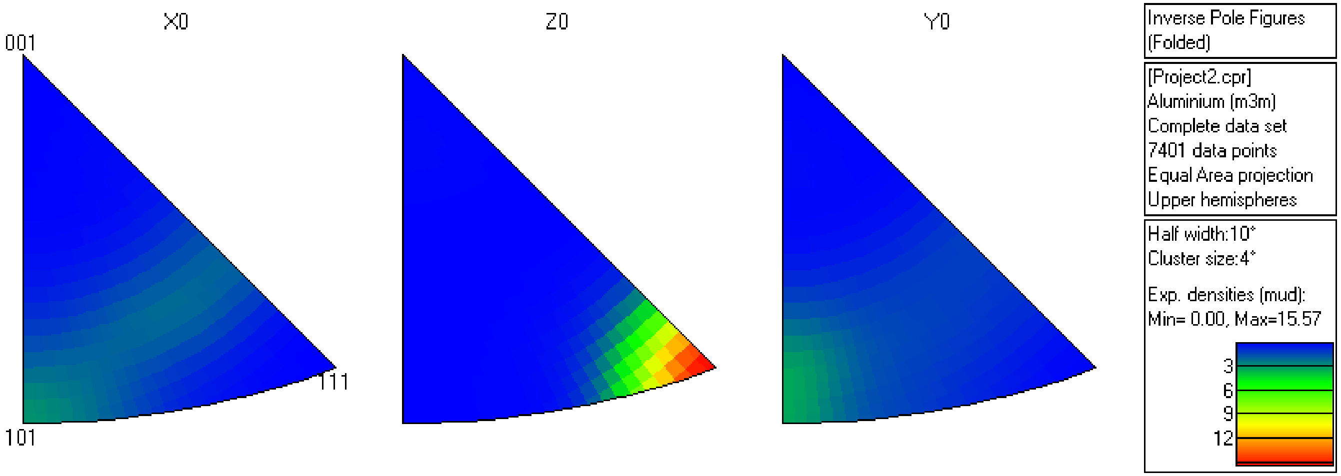
mc AlSi (1%) layer
on n-type Si-Wafer <100>

Orientation colour coding





Orientation distribution
of sample axes

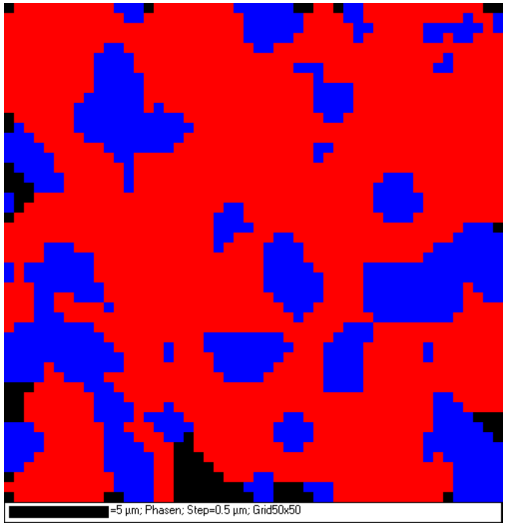


Inverse Pole Figures
(Folded)

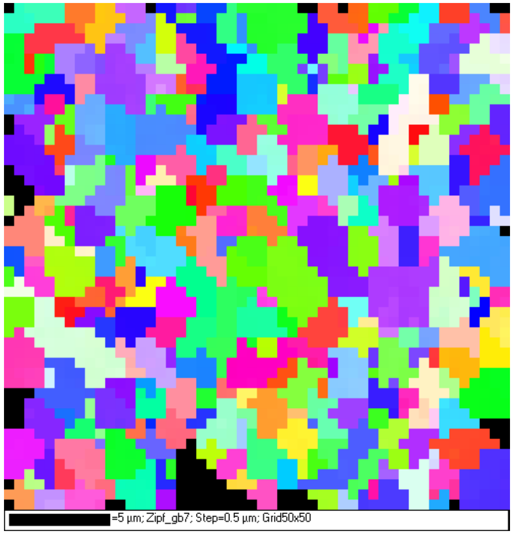
[Project2.cpr]
Aluminium (m3m)
Complete data set
7401 data points
Equal Area projection
Upper hemispheres

Half width:10°
Cluster size:4°
Exp. densities (mud):
Min= 0.00, Max=15.57

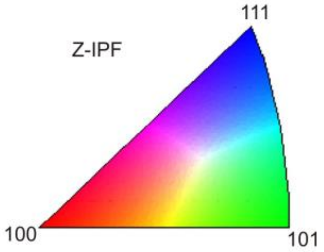
Phase mapping on sample surface



phase
mapping



orientation
mapping
(z axis)



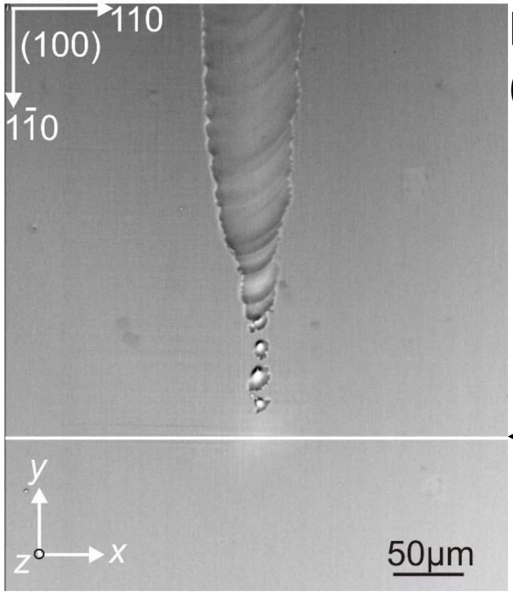
Phase determination from known units cells

Hydroxyl-Apatite ($Ca_{10}(OH)_2(PO_4)_6$
hexagonal: space group hex 176
a = 0,9352 nm, c = 0,6882 nm,
42 atoms

Beta-TCP (Tricalcium phosphate $Ca_3(PO_4)_2$)
trigonal: space group H 166
a = 0,52479 nm, c = 1,86910 nm,
39 atoms

Internal mechanical stresses from cross-correlation EBSD

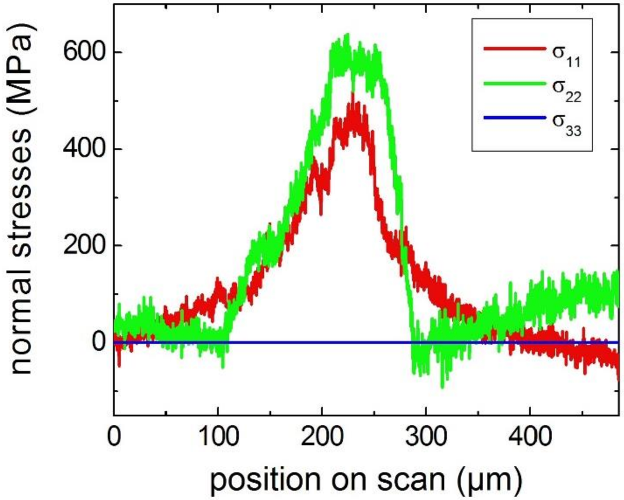
Determined from Fourier analysis of the diffraction patterns and cross-correlation with a pattern from a stress-free region



Liquid phase re-crystallization (LPR) on a Si wafer

EBSD line scan with step size of 50 nm

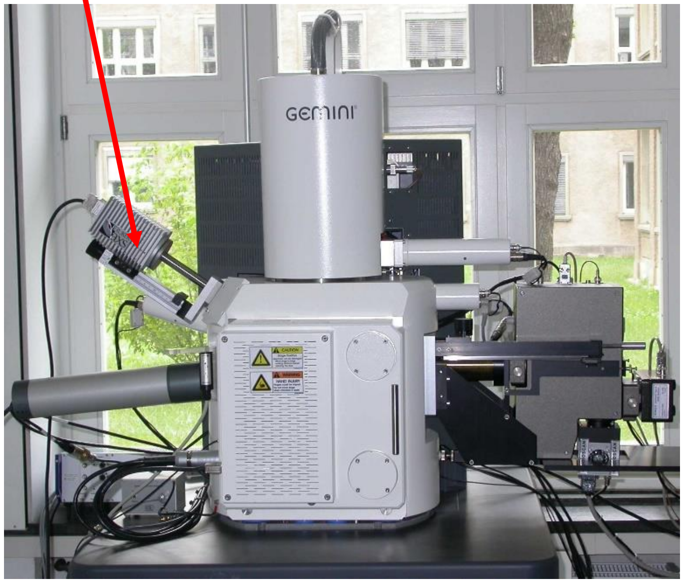
Here: plane bi-axial tensile stress state in the vicinity of the LPR track where dislocation activity is observed

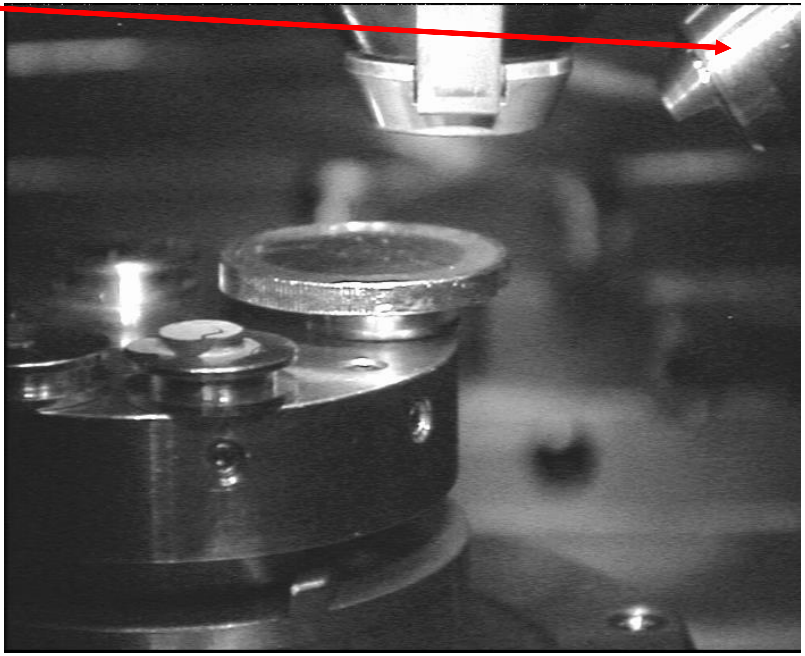


2.3 Analysis of chemical composition and spatial element distribution

EDX

Bruker Quantax 400 with XFlash 5010 SDD and ESPRIT software

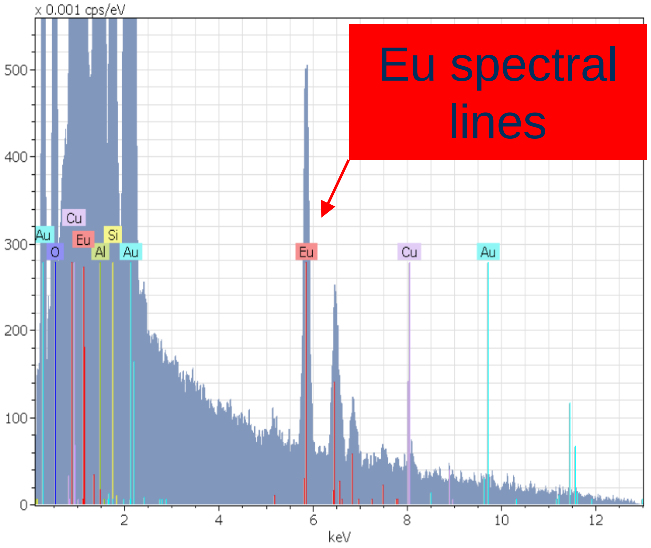




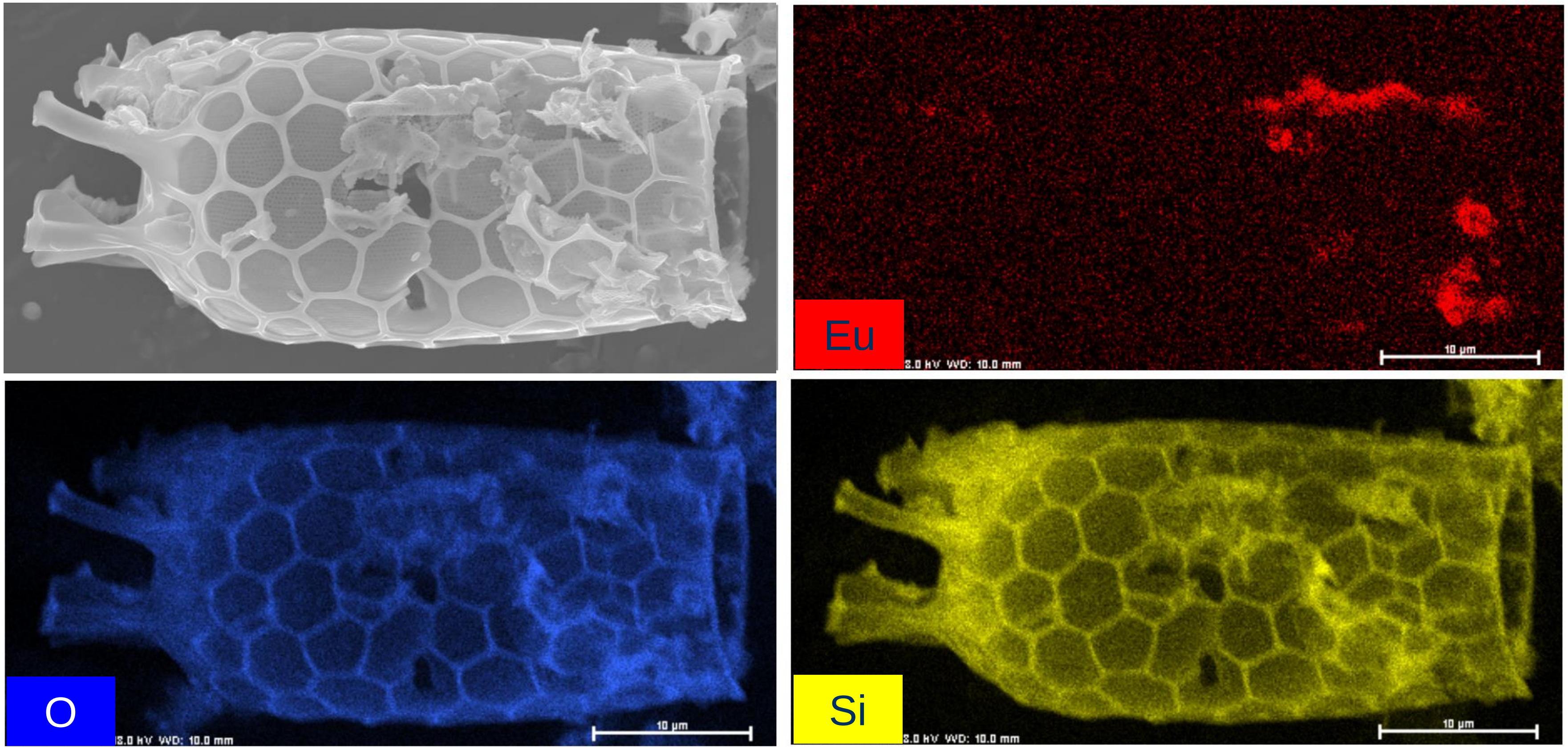
standard-free quantitative determination of element concentration and spatial element distribution by energy dispersive X-ray analysis (EDX)

Element identification and spatial distribution

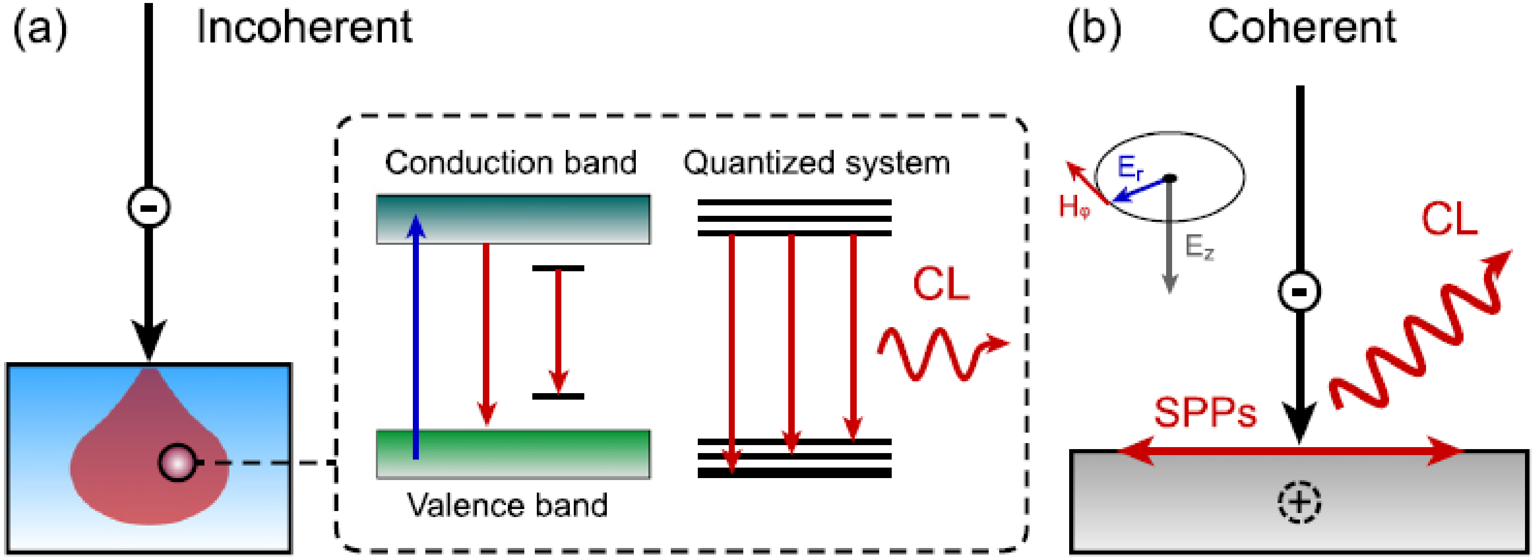
Calcined diatoms after Europium sorption



Eu spectral lines



2.4 Investigation of the luminescence behaviour



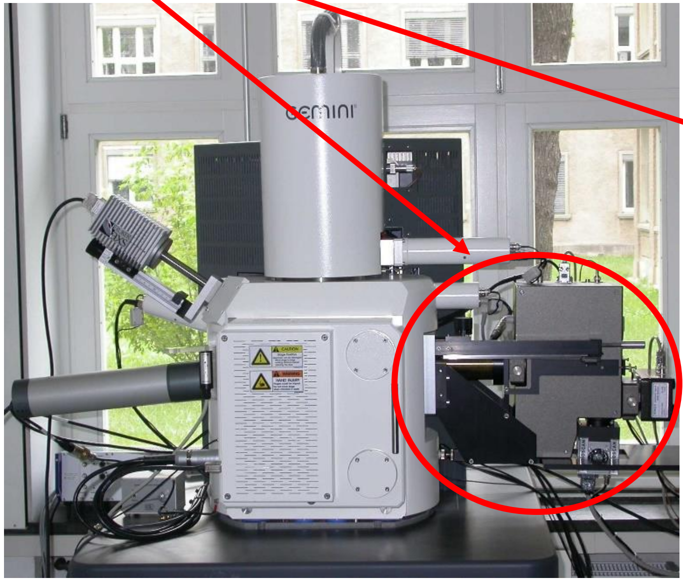
Koenen, Haegel 2017

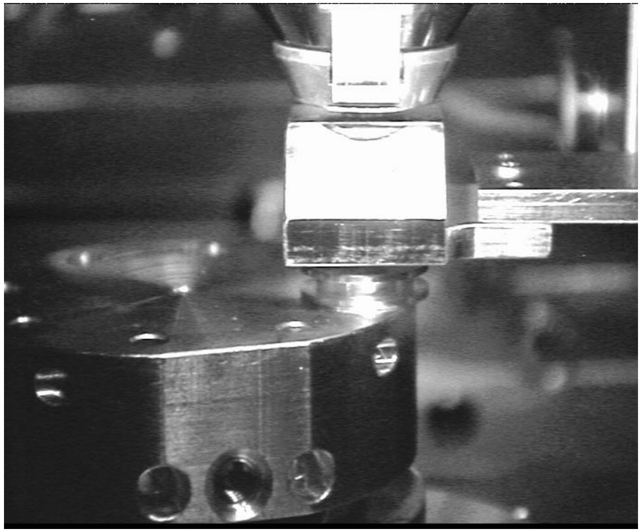
Generation of electron-hole pairs and subsequent radiative recombination by band-edge transitions and at defect states in band gaps as well as an excitation of discrete electronic states

Local polarization of the material emitting light directly or coupling to other electromagnetic modes (surface plasmons)

CL

Adjustable parabolic mirror, focusing lens, Czerny-Turner monochromator, CCD and 4 different PMT detectors (UV/VIS to IR), beam blanker

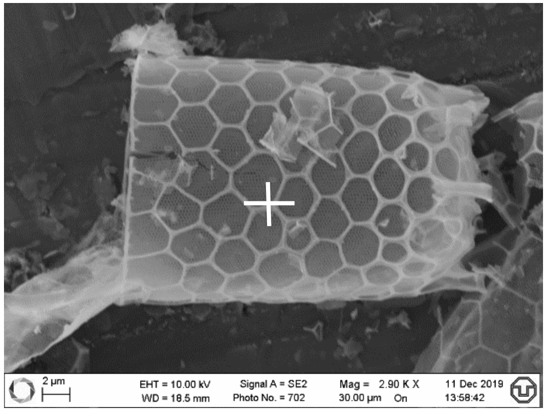


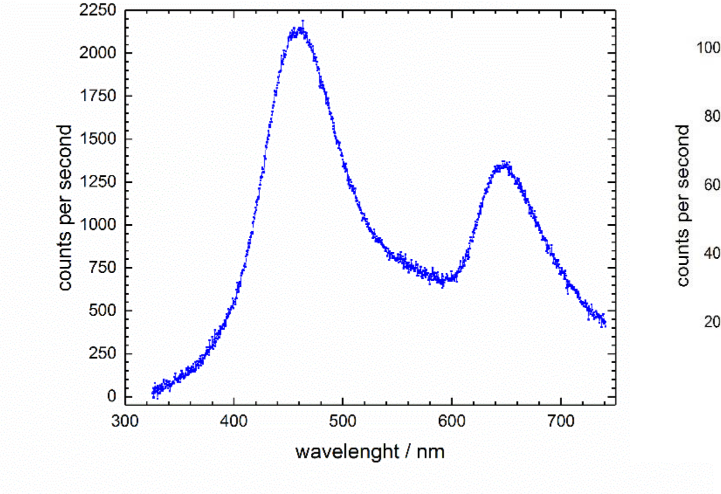


- cathodoluminescence (CL) spectroscopy
- time-resolved CL spectroscopy (TR-CL)
- mono- and panchromatic CL imaging

CL spectral investigations

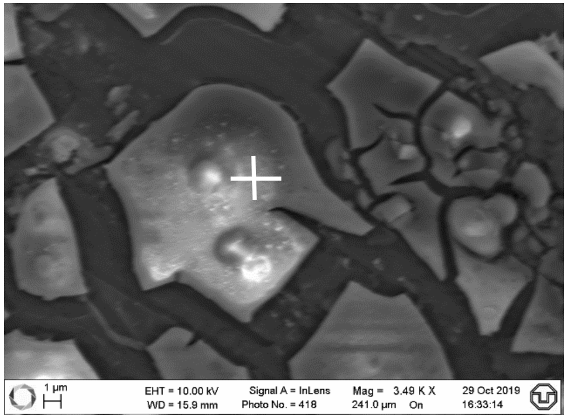
point like excitation

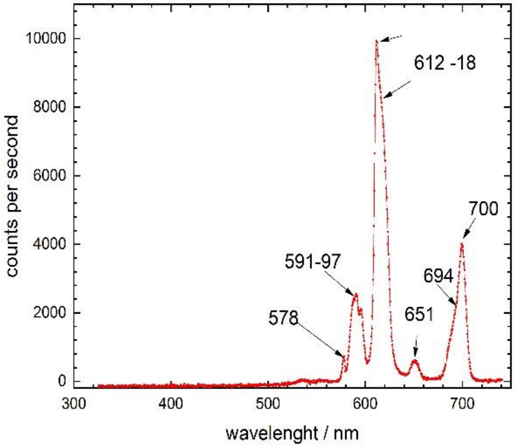




pure diatom

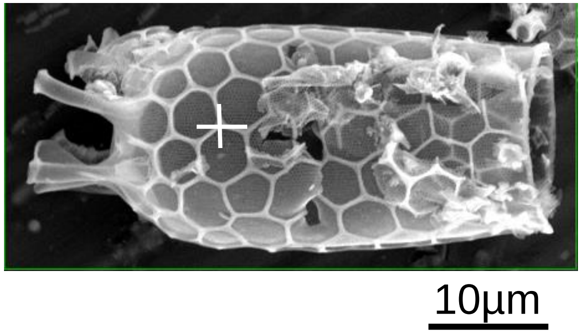
(defect correlated luminescence)

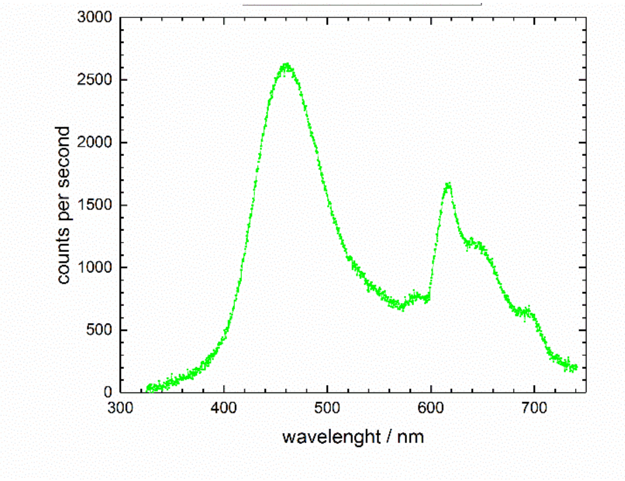




EuCl$_3$

(radiative transitions of *4f* electrons)

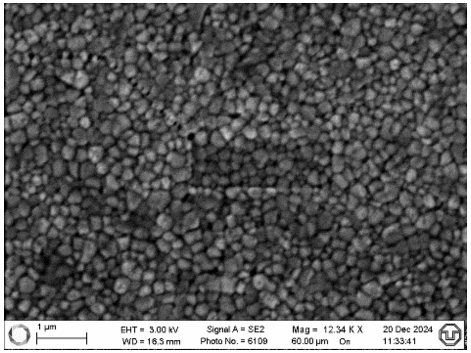


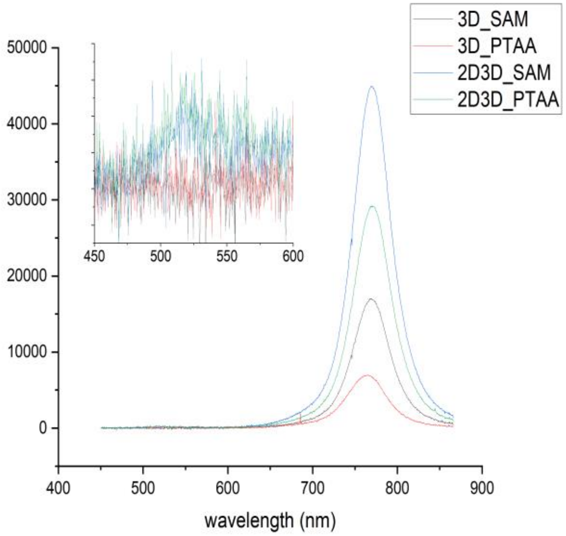


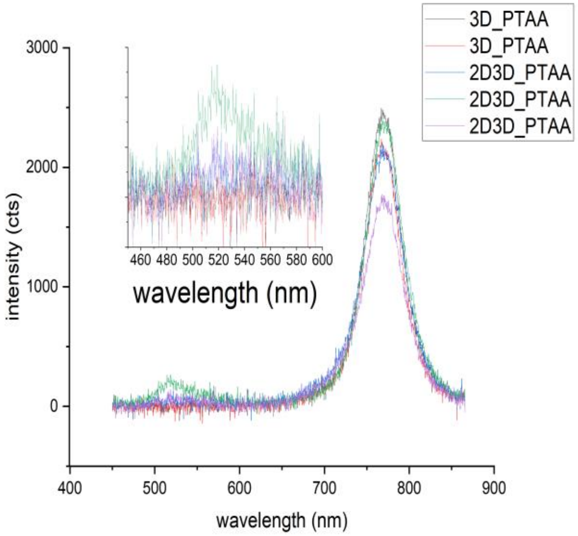
diatom after Eu sorption

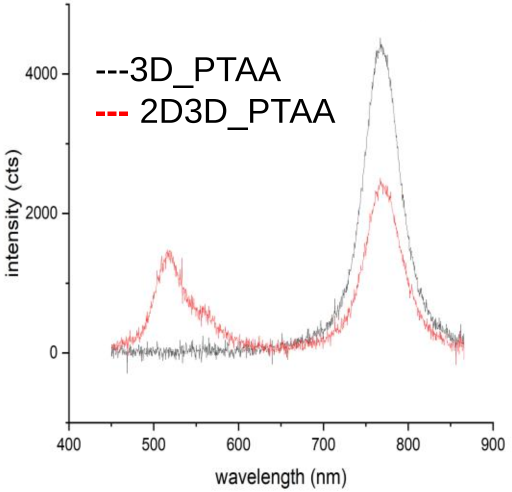
(superposition of diatom and Eu spectra)

CL spectra by scanning electron beam on 2D / 3D perovskite sample (IFW)





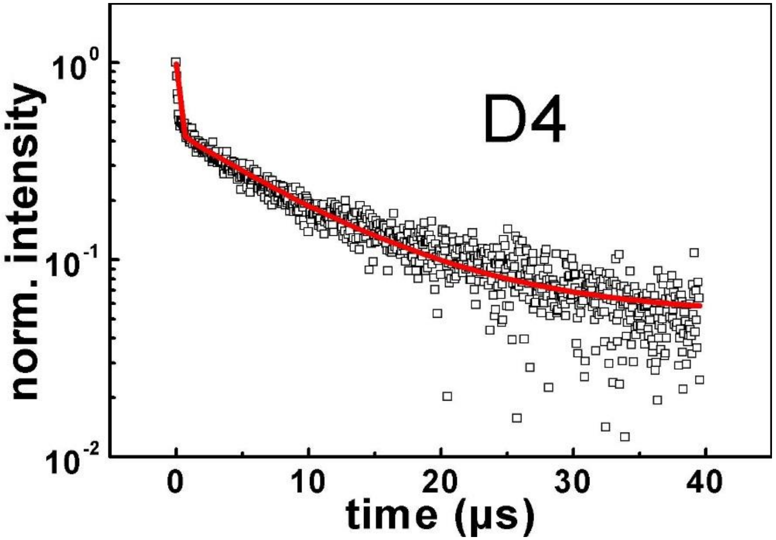




acceleration voltage EHT:	3 kV	2 kV	1 kV
depth of CL generation:	85 nm	45 nm	20 nm

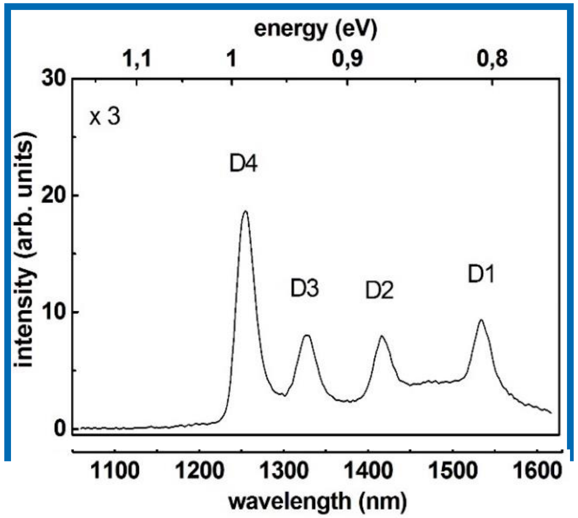
Time-resolved CL investigations

plastically deformed Si single crystals



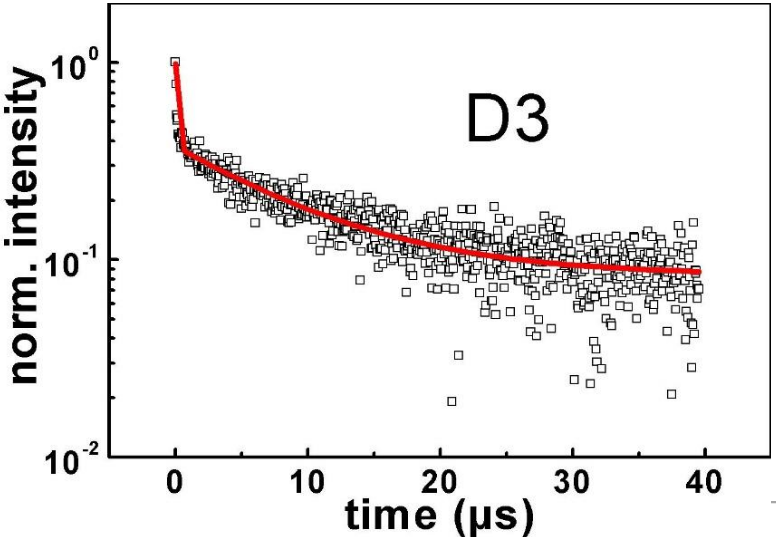
Fit for bi-exponential decay yields:

$$\tau_1 = 195\ \mathrm{ns}$$
$$\tau_2 = 2{,}9\ \mu\mathrm{s}$$

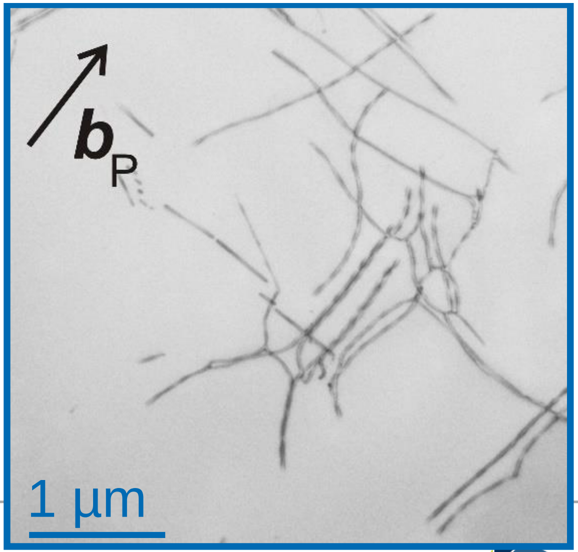


D4: recombination of an exciton bound to a shallow defect at a band gap reduced by the distortion field of dislocations

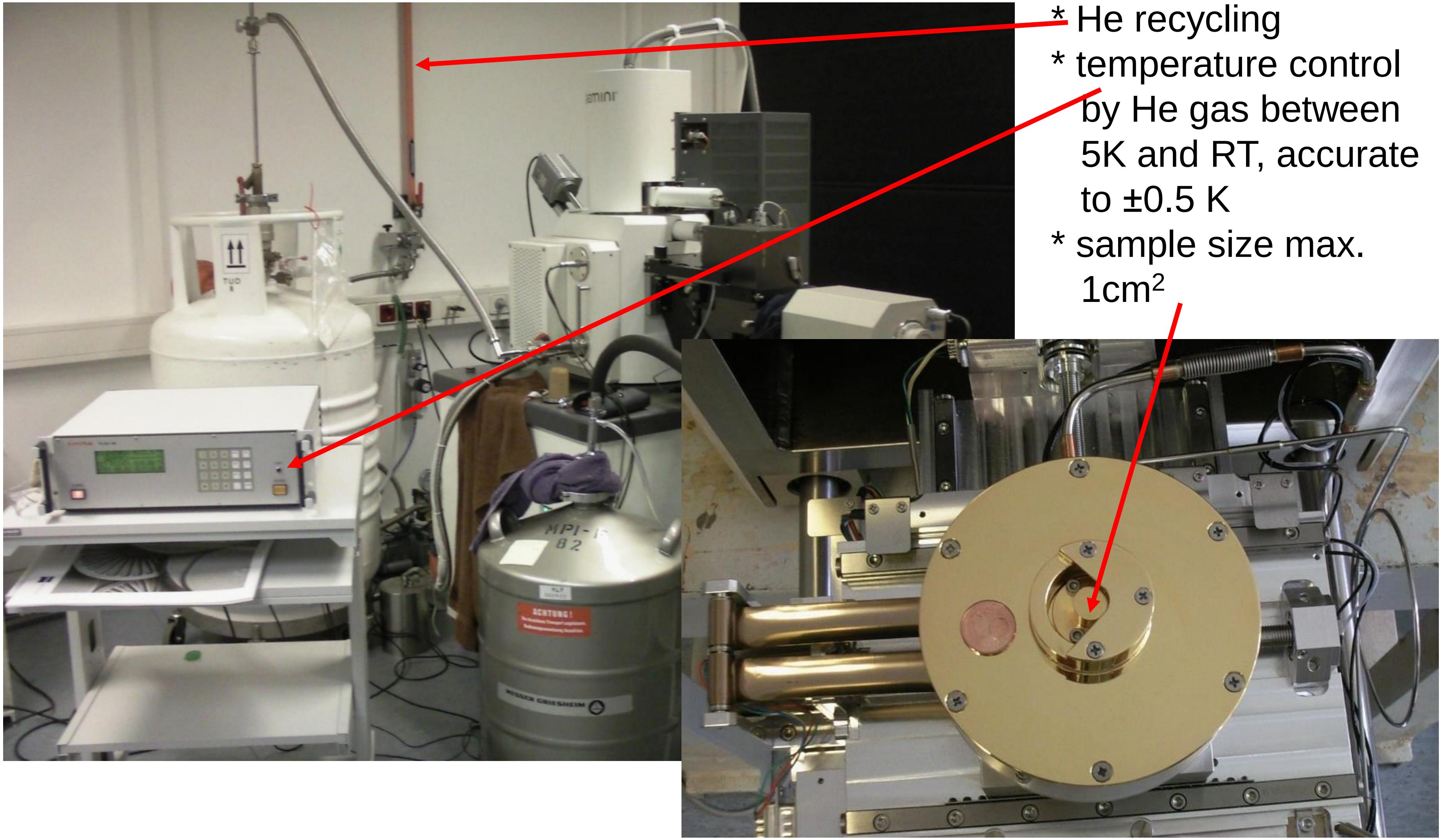
D3: TO phonon replica of D4



$$\tau_1 = 90\ \mathrm{ns}$$
$$\tau_2 = 2{,}6\ \mu\mathrm{s}$$



2.5 Implementation of experiments at low temperatures

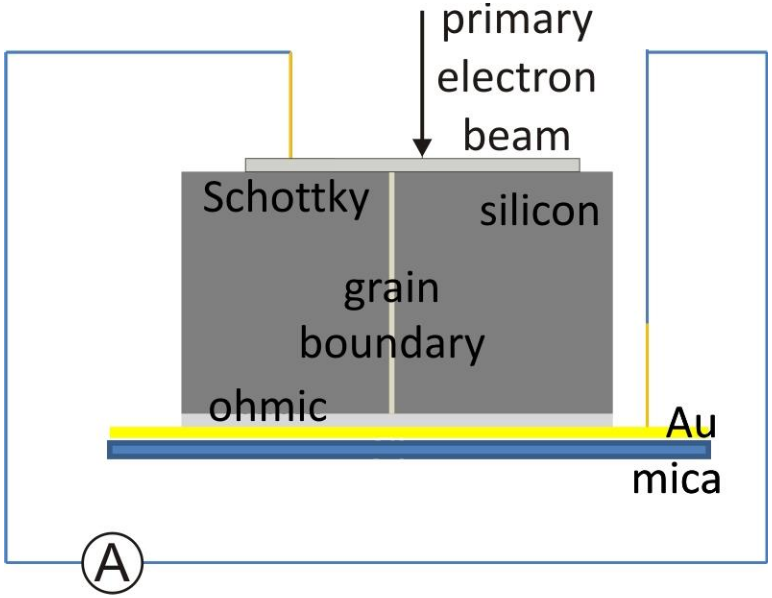


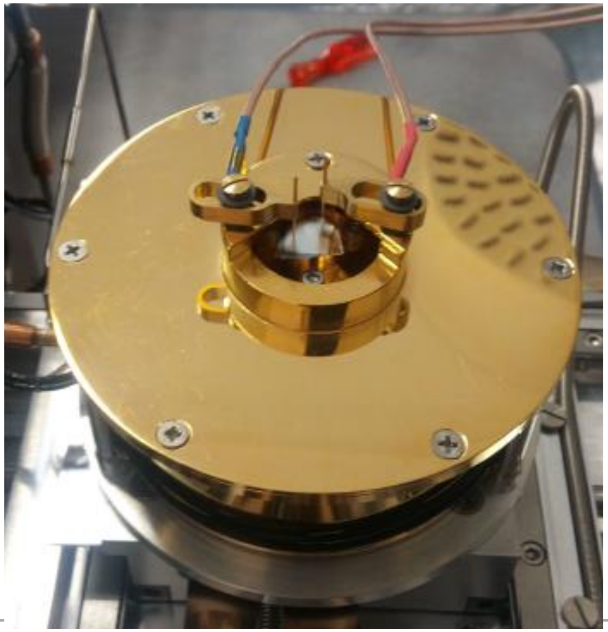
- He recycling
- temperature control by He gas between 5K and RT, accurate to ±0.5 K
- sample size max. 1cm^2

2.6 Examination of electrical properties

a current results from field-assisted spatial separation of electron-hole pairs, generated by inelastic interaction of primary electrons in semiconductor materials;
the current magnitude varies locally due to varying charge carrier recombination at defects

➔ Electron Beam Induced Current (EBIC)





EBIC imaging

Investigation of grain boundaries in Si

EBSD (z IPF)

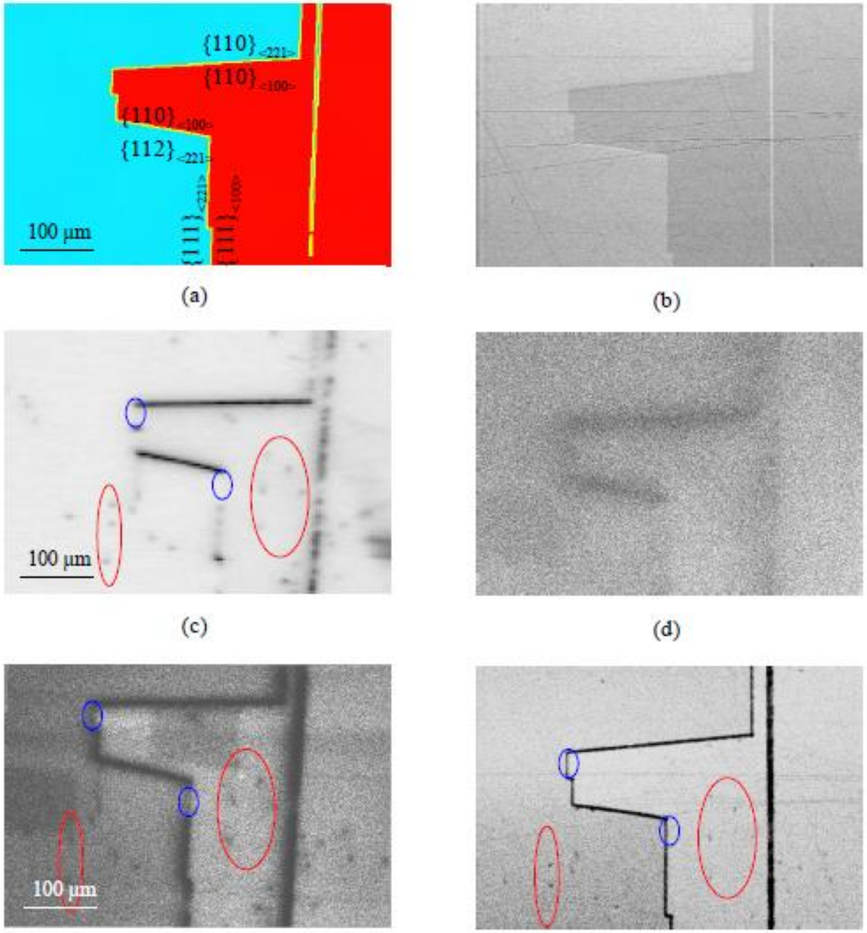
BSE

EBIC (RT)

Pan-CL (RT)

Pan-CL (20 K)

Pan-CL (4,5 K)

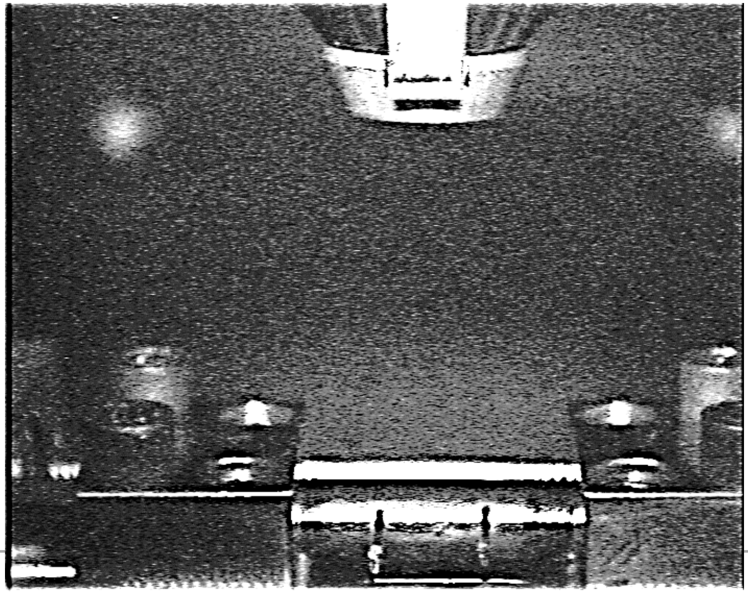


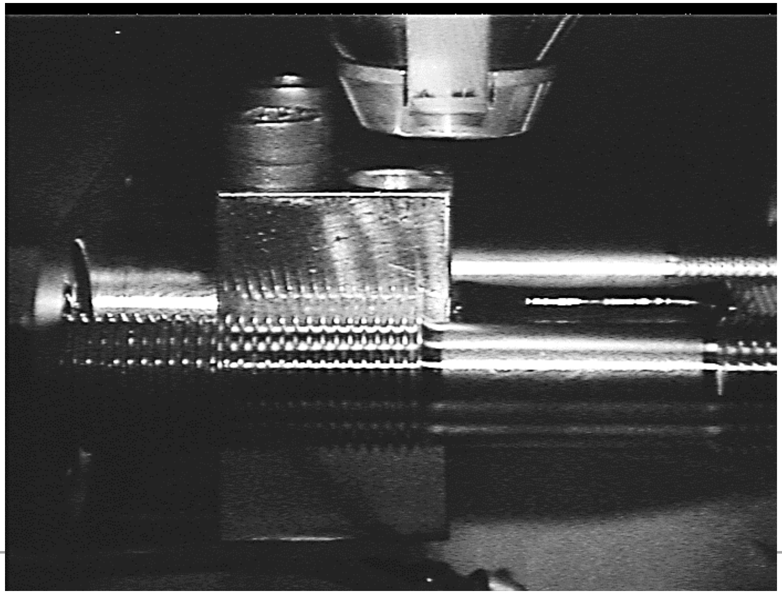
(a) (b) (c) (d)

➔ detection and quantitative characterization of (charged) crystal defects, diffusion length, lifetime & mobility of minority charge carriers at RT and LT

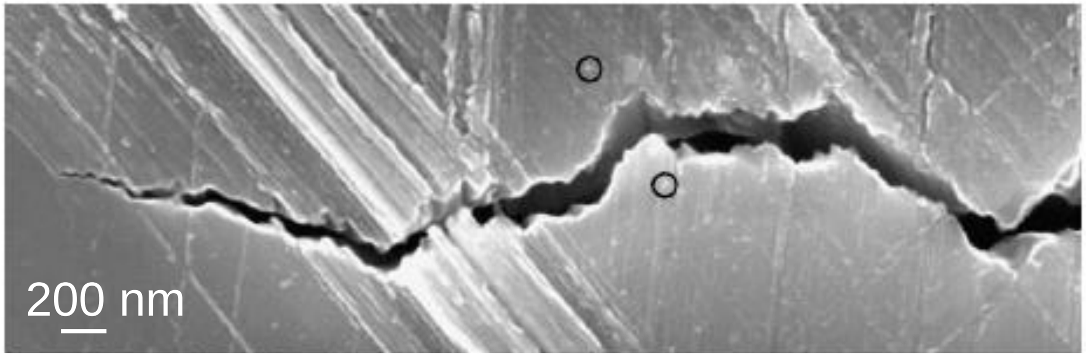
2.7 Elastic and plastic deformation of materials and *in-situ* observation

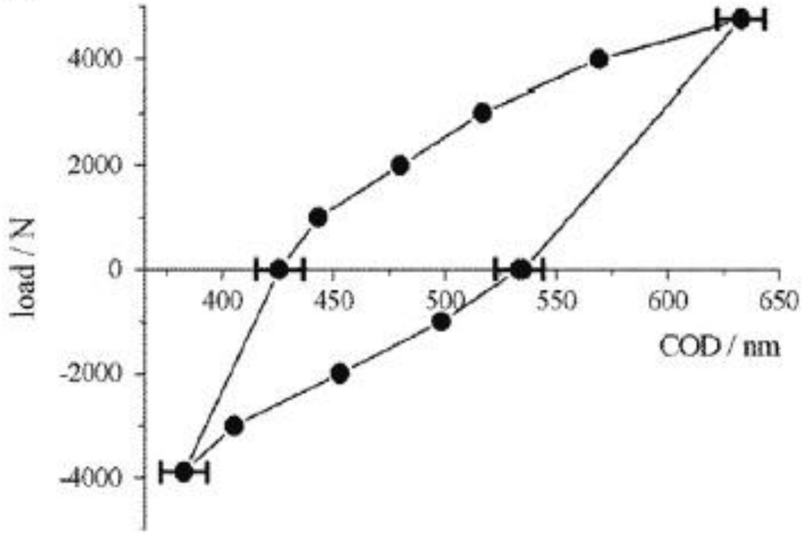
Push-pull testing device (10 kN)
by Kammrath & Weiss



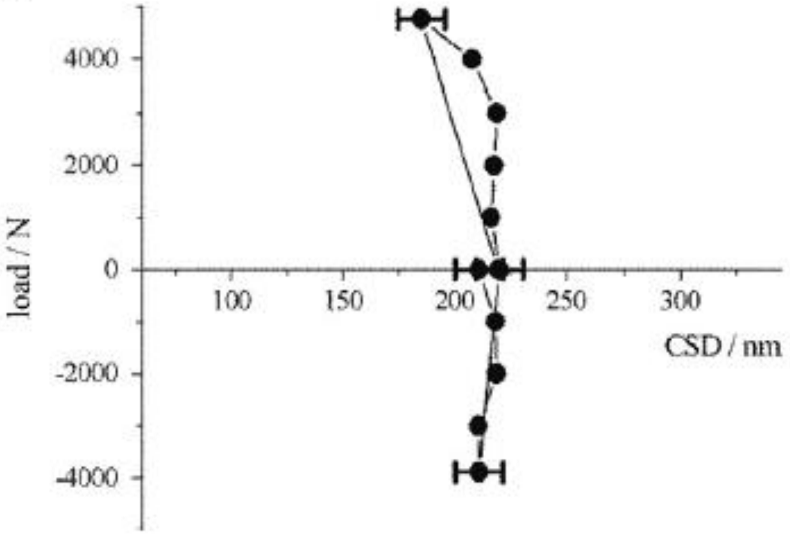


Investigation of crack behavior at an austenitic-ferritic duplex steel





crack opening displacement vs. load



crack sliding displacement vs. load

➔ *in-situ* investigations (cracks, slip activity) at RT and slightly elevated temperatures

3 Access to the SEM lab and future activities

- Currently 12 authorized users
- Institutions: IFMP & IAP @ TU, IFW Dresden, HTWD, HZDR
- Integration of the SEM into advanced practical course of BA students in physics (EBSD) and chemical engineering MA (imaging, EDX, EBSD)
- Access for users from outside the IFMP/IAP must be agreed with the responsible persons (cost sharing agreement with J. Geck)
- Written instructions for all techniques available, videos
  https://tud.link/hk2m , https://tud.link/94p2 , http://www.jove.com/video/53872
- Weekly scheduling on Thursdays for the following week
- Hygiene rules (if possible, individual work is preferred)

Lab Contact & Support:

- Ellen Hieckmann (ellen.hieckmann@tu-dresden.de) 4633 6051, Nadine Kruse (nadine.kruse@tu-dresden.de) 4633 7643
- Instruction of new users by Ellen Hieckmann (Nadine Kruse) into all techniques
- Independent basic operation of the SEM possible after three measuring shifts under guidance
- Scientific services can be offered to a certain extent, e.g. for the imaging of any (conductive) samples, EBSD, EDX and CL
- General aim: every user should work independently in the SEM lab
- The SEM operation should also continue to a large extent during the construction phase in the Recknagel building in 2025
- We are always looking for users with new research topics! Your are welcome!

Do not hesitate to contact us if you want to create a perfect image of your sample!