Surface Diffraction with high energy X-rays

X-ray Diffraction with E ≥ 50 keV

P08/7 team at PETRA III

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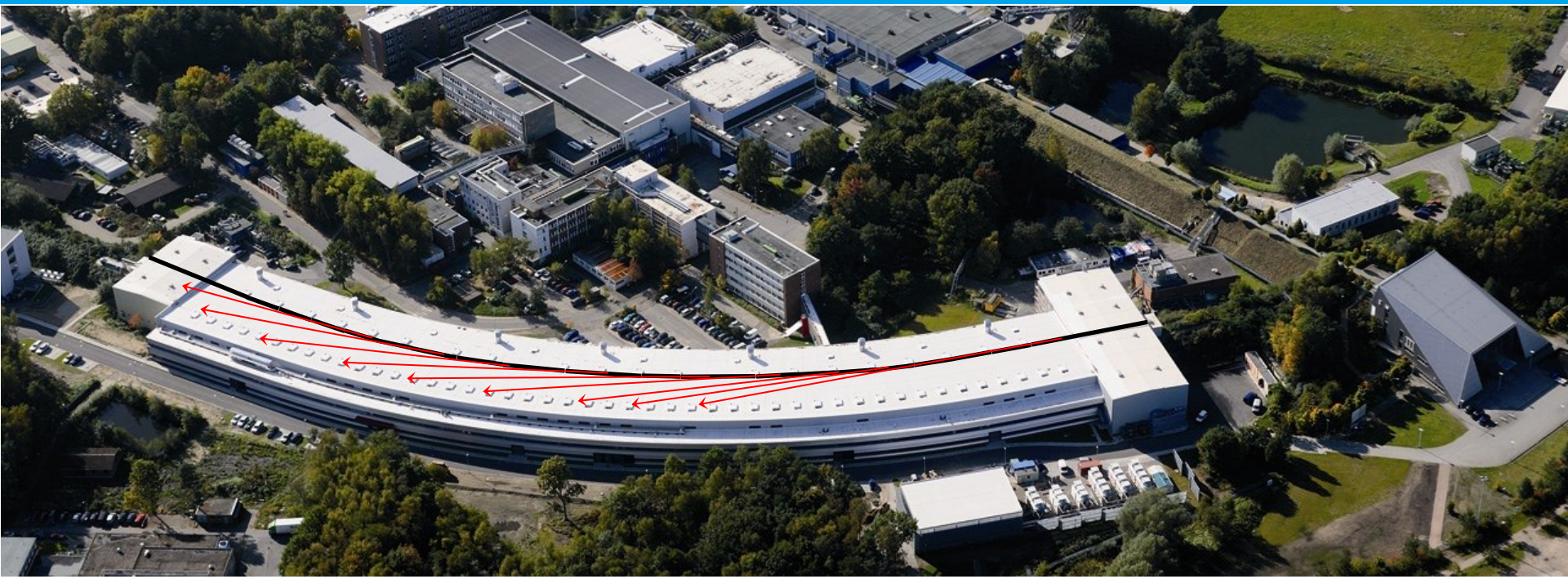
- Motivation for high energy surface diffraction
- Time resolved *in situ* experiments
- Nanostructured surfaces studied with hard X-rays
- Outlook marriage of high energy X-rays and very high emittance

HELMHOLTZ | GEMEINSCHAFT

DESY

PETRA III



- particle energy: 6 GeV
- stored current: 100 mA (top-up)
- emittance: 1 nm rad
- coupling: 1%
- circumference: 2304 m
- source size, high ß: 142 x 5 μm^2
- source size, low ß: 35 x 6 μm^2
- X-ray wavelength: 10 – 0.06 Å
- beamline length: 70-100 m
- annual operation: 5000 h (5216 h in 2013)

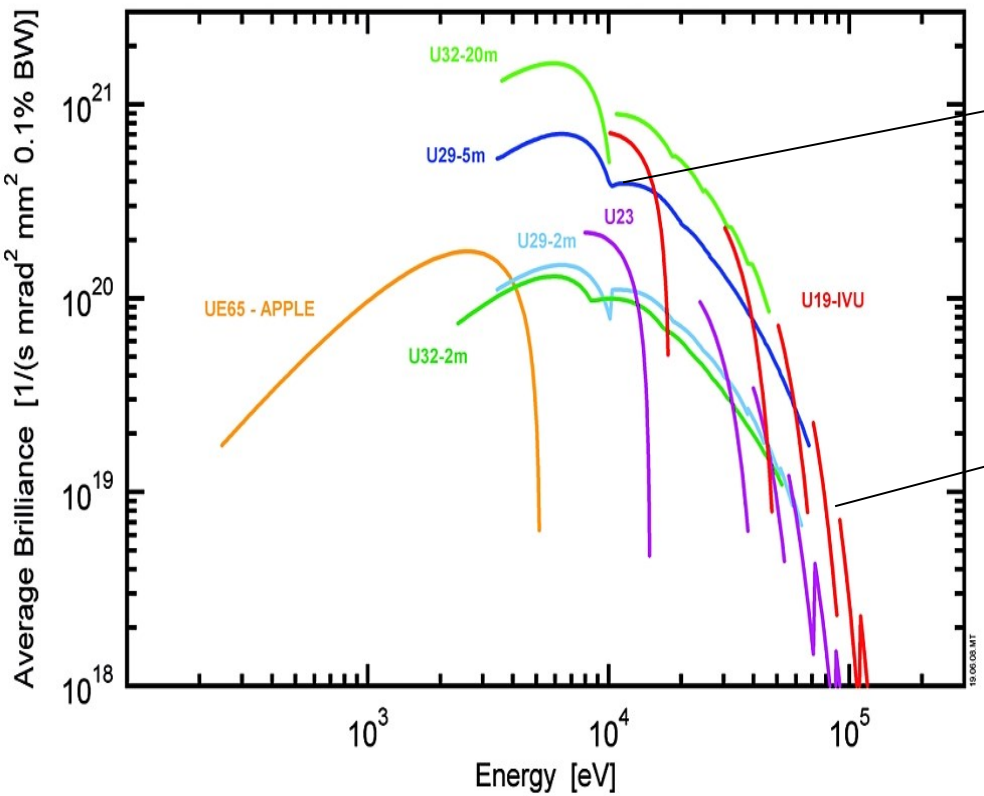
- built in 1978
- rebuilt as a synchrotron radiation source in 2007
- commissioning since 2009
- user operation since 2010
- largest storage ring of its kind

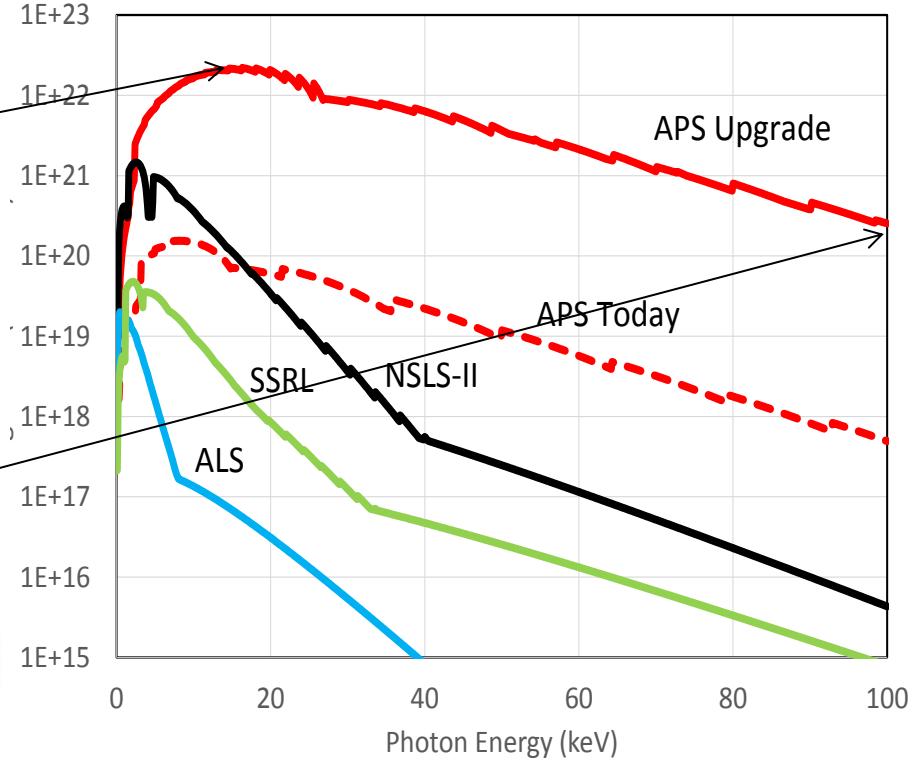
Comparison PETRA III ↔ APS-U

PETRA III

x 100

APS

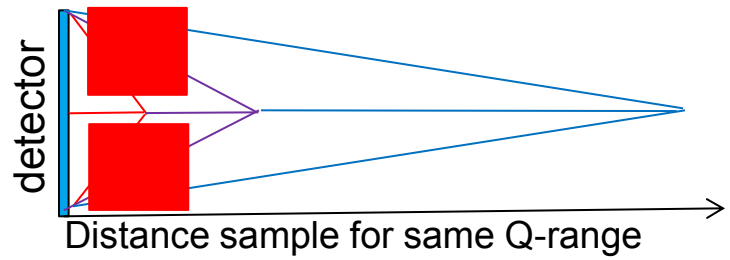




Why high energy X-rays?

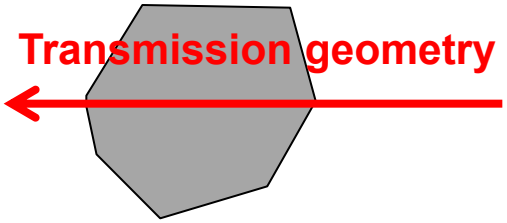
Low absorption → High penetration power ($1/\mu_{Fe,\ 100\ keV}$=3.1 mm)
Small correction factors

Small Bragg angle → High momentum transfer (30 $Å^{-1}$ easily achievable)
Small windows
Small correction factors



Small Darwin width → High angular resolution
Low extinction

Large crystals → Volume enhancement of weak intensities

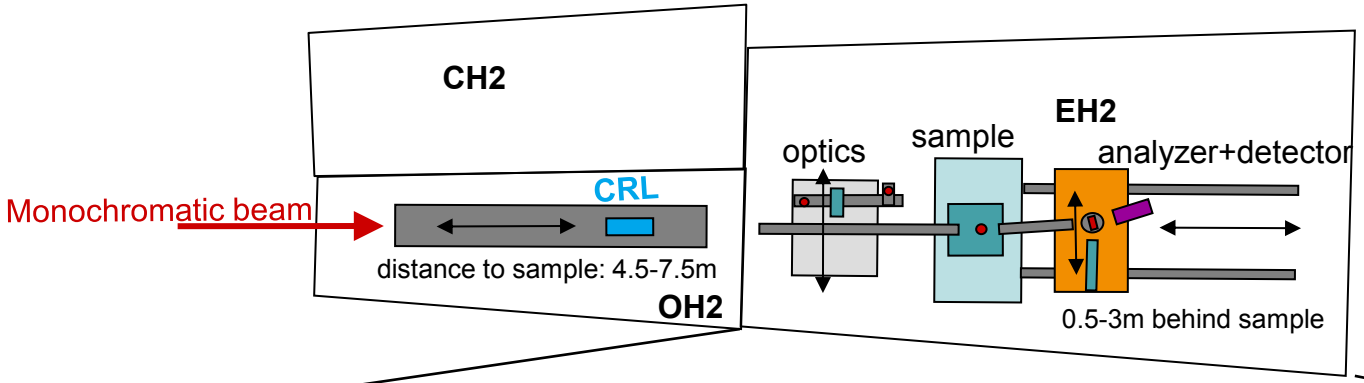


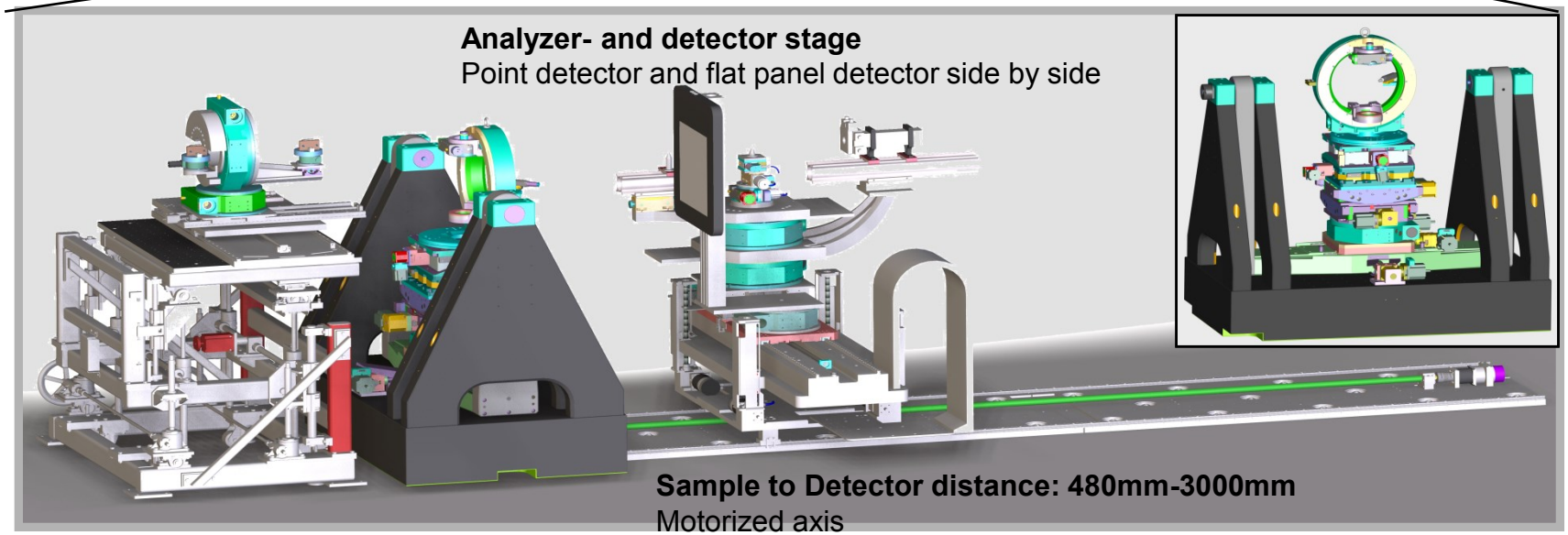
BUT:

Less intensity in incident beam

Scattered intensity proportional to λ^2

Outline of the Physics Hutch at P07





Optical table
Slits, Absorber, Diode
CCD Camera
Monochromator for liquids

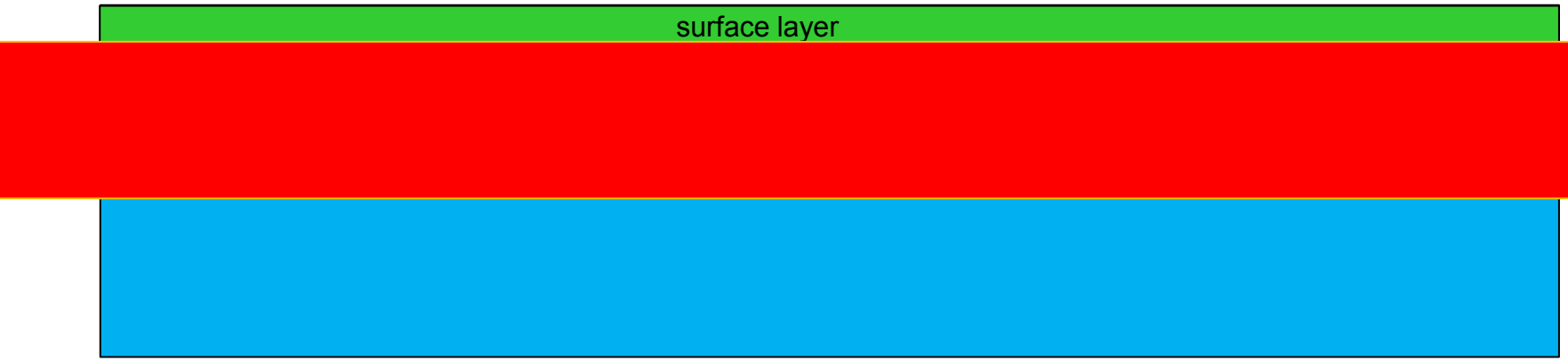
Sample stage
Eulerian cradle (removable)
High precision X-, y- and z-translation
4 rotations
Up to 300 kg load

Surface, interface and surface near regions

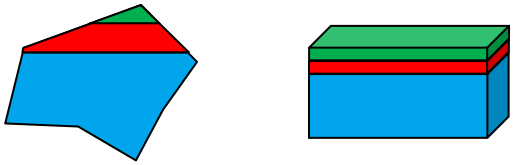
Laue geometry!

- Study of μm-size layers
- Depth dependencies

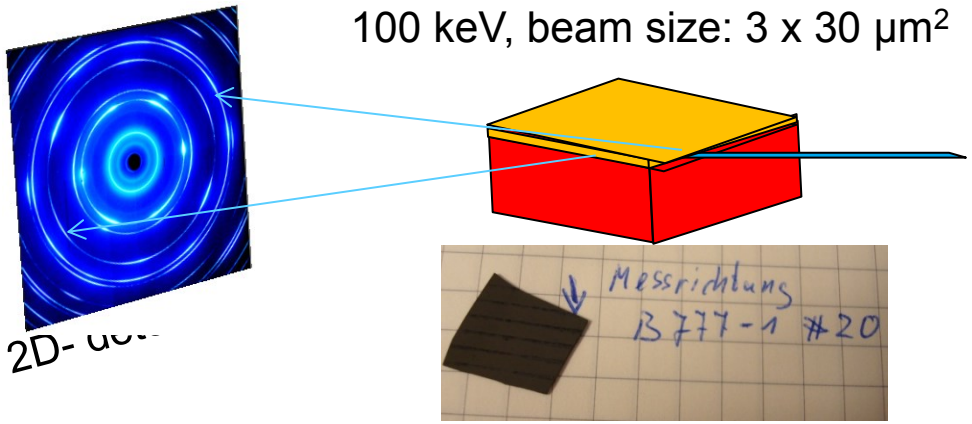
Beam height ≈ 2 μm,
Beam divergence < 100 μrad
Focus length > 20 mm

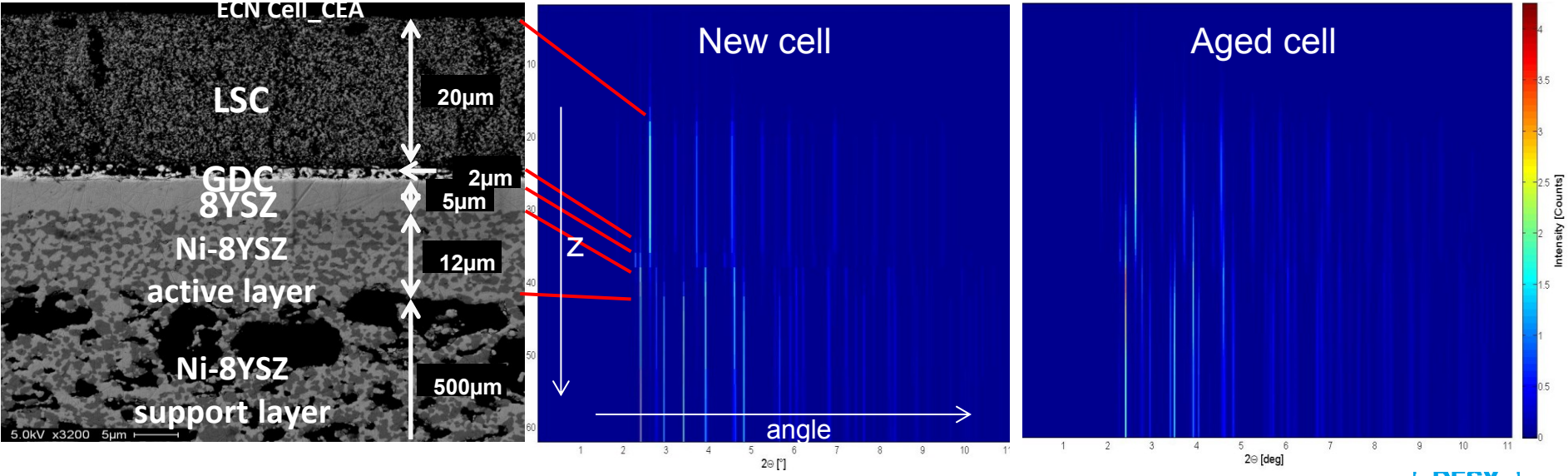


parallel layers



Example: Intermediate Temperature Steam Electrolyser (ITSE)





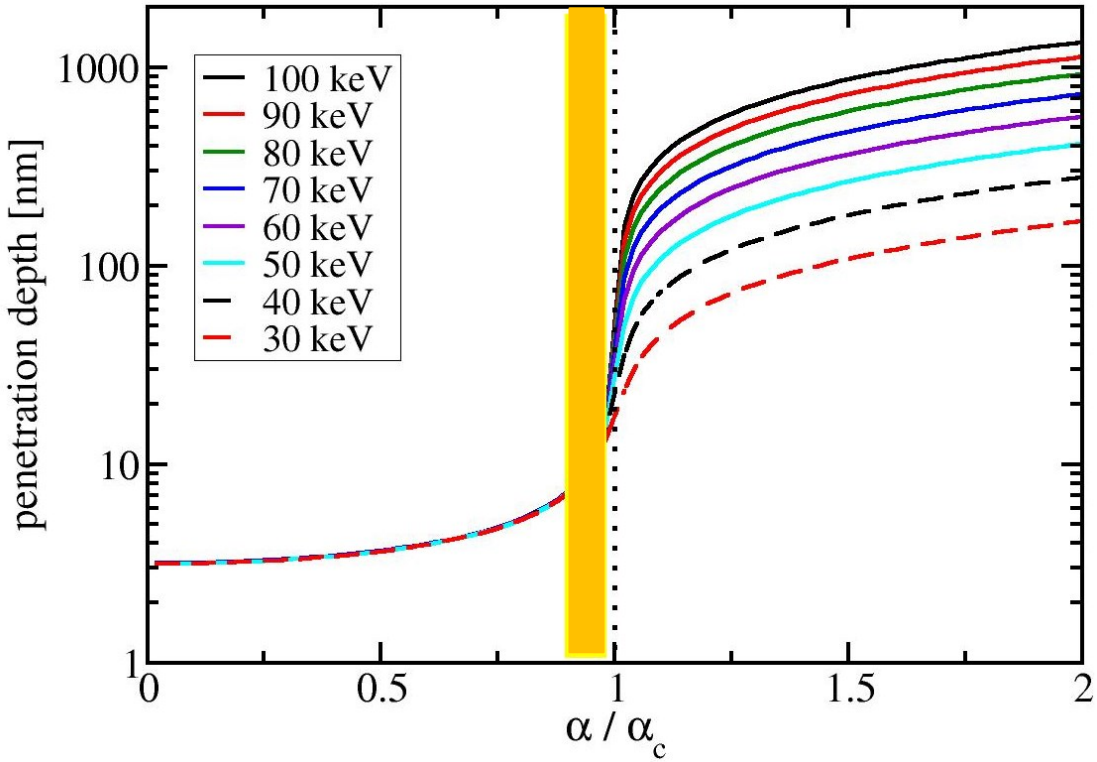
“Real” surfaces?

Incident angle $\alpha < \alpha_{critical}$ → very limited penetration, energy independent (!)

nm-sensitivity

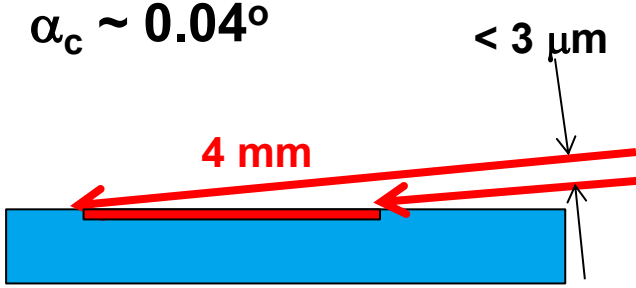
Problem: $\alpha_{critical} \propto \lambda$

→ round corners, edges, dents, scratches, bumps



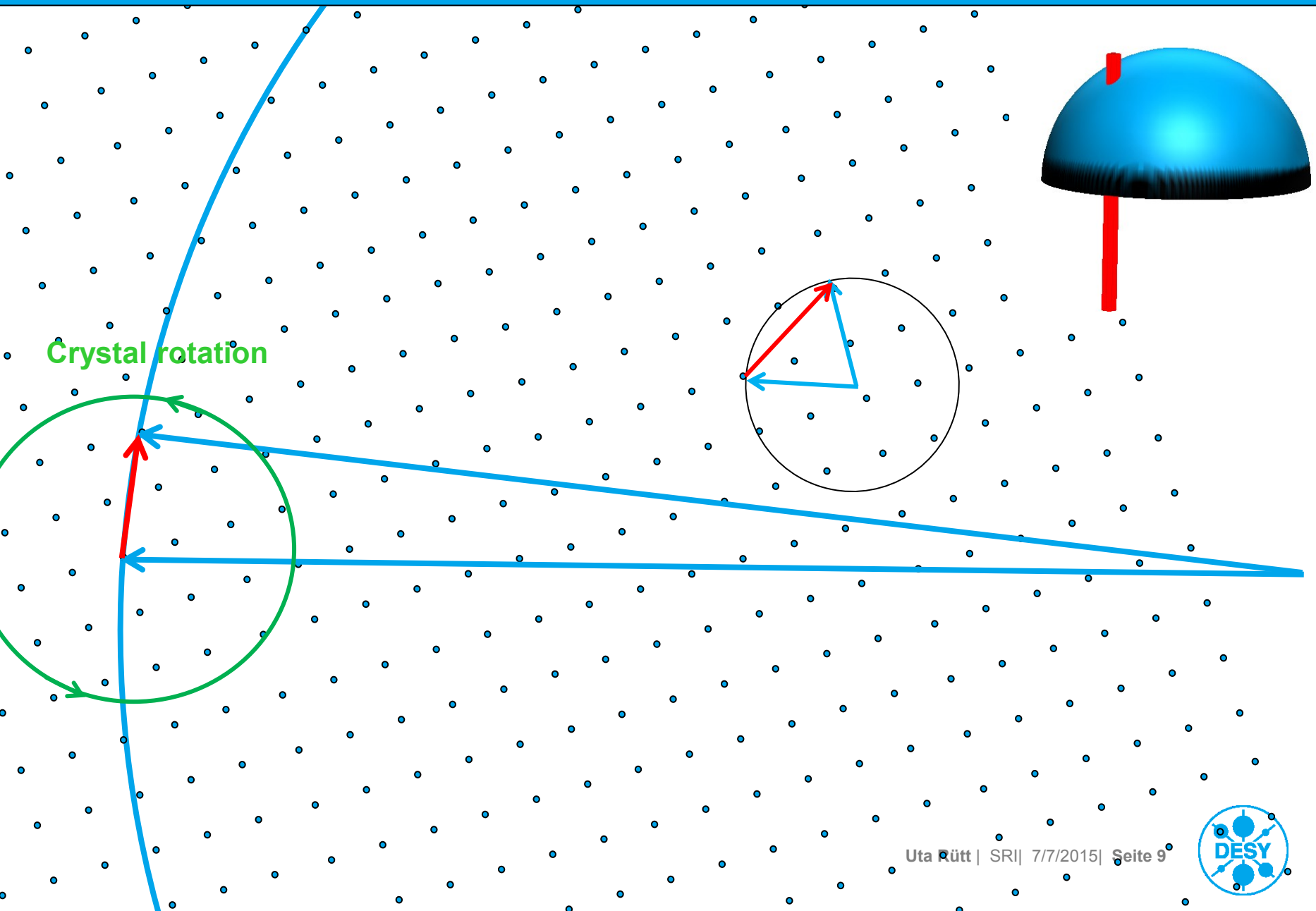
Pd, 85 keV

α_c ~ 0.04°

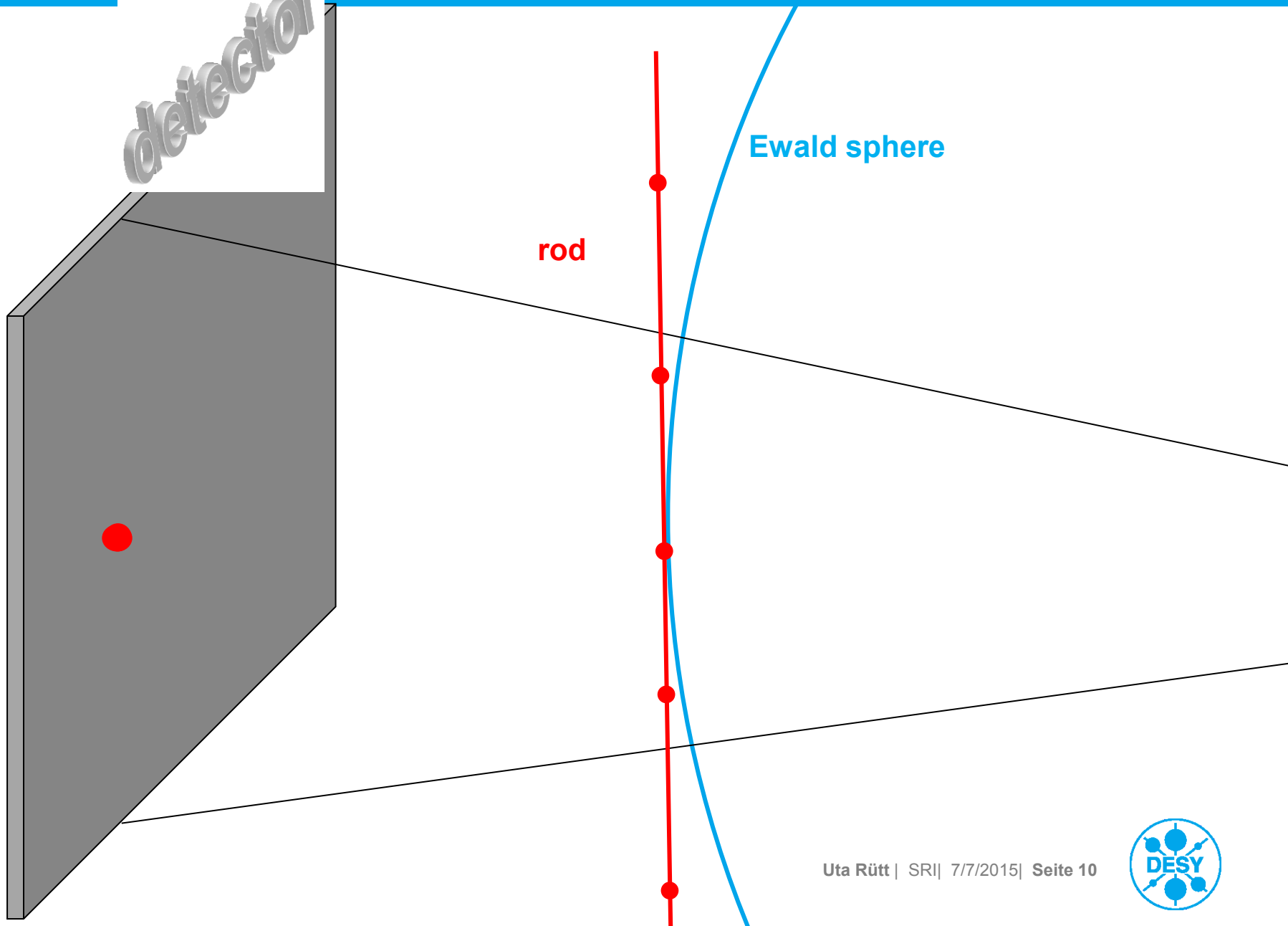


F = 3μm / sin(0.04°) = 4 mm

Surface diffraction and crystal truncation rods

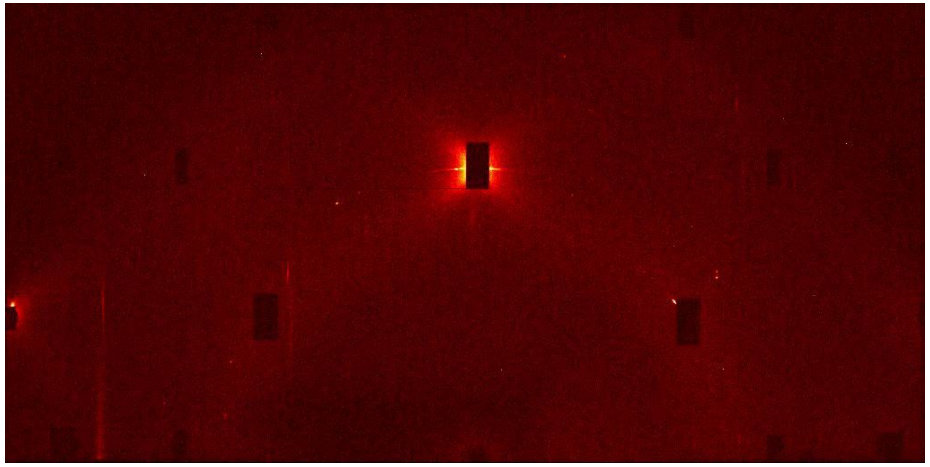


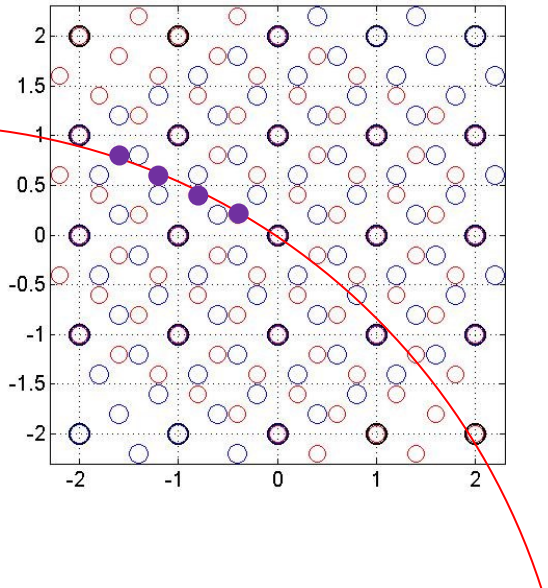
Crystal truncation rod from the side
rotation of crystal perpendicular to the plane

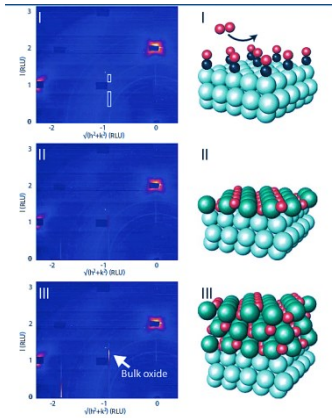


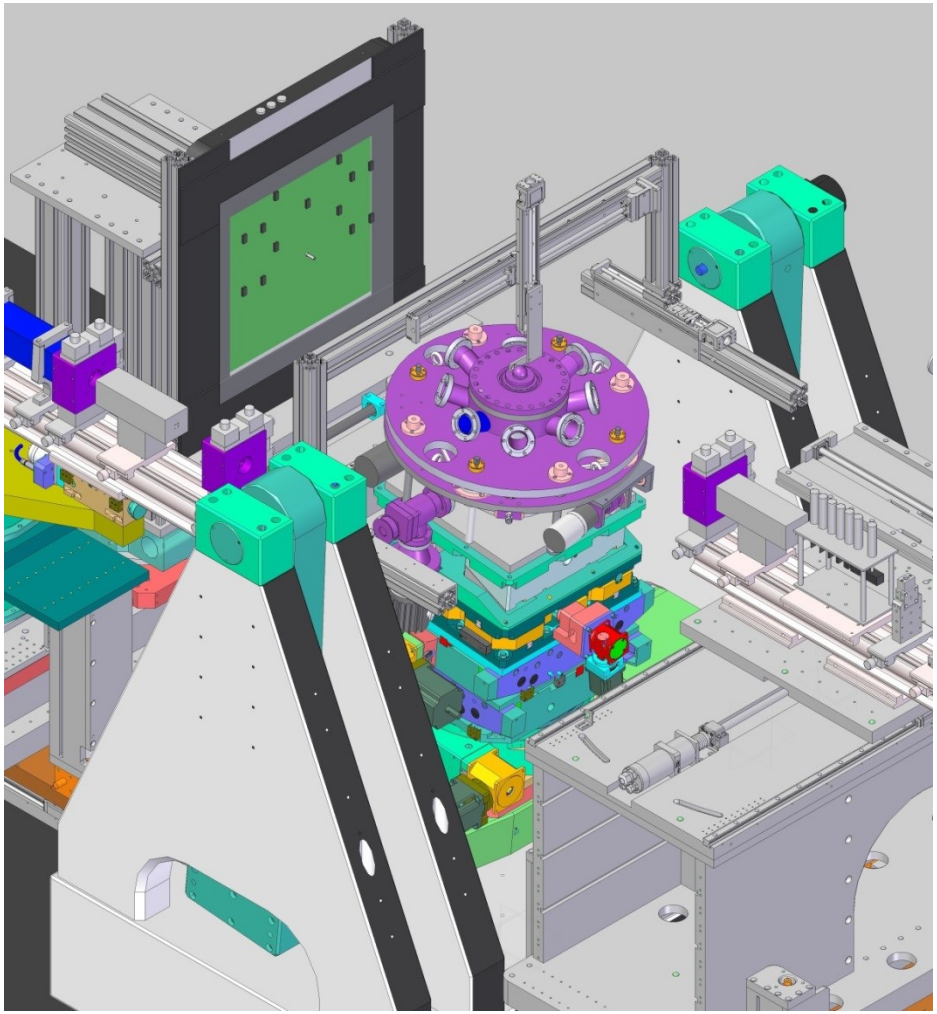
Catalysis: Pd-single crystal for CO_2 production

J. Gustafson, M. Shiplin, C. Zhang et al. (2014) **Science** 343, p.758

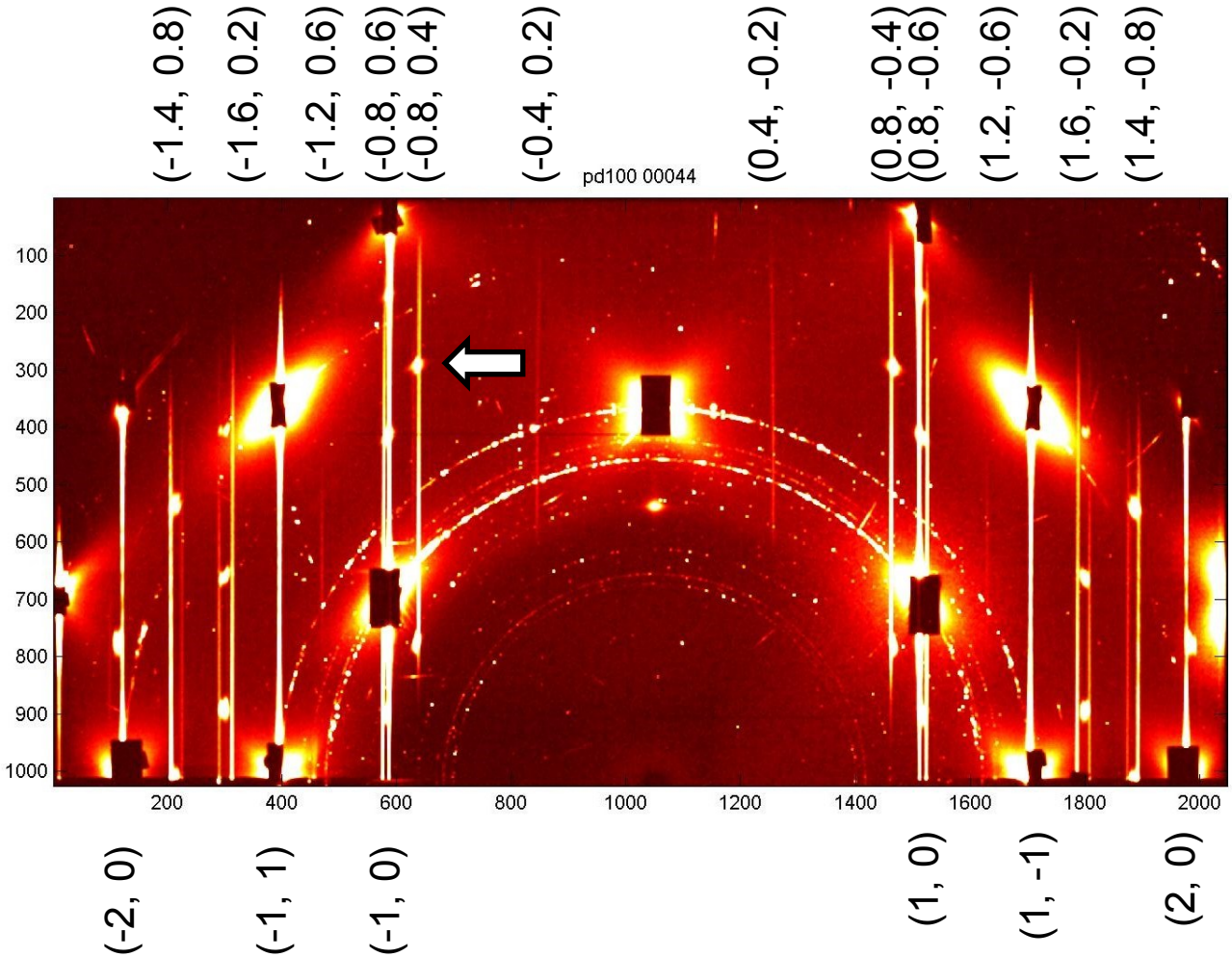


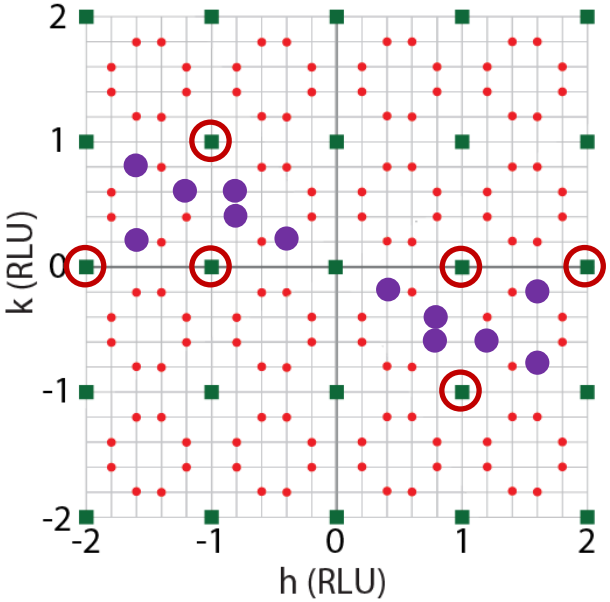






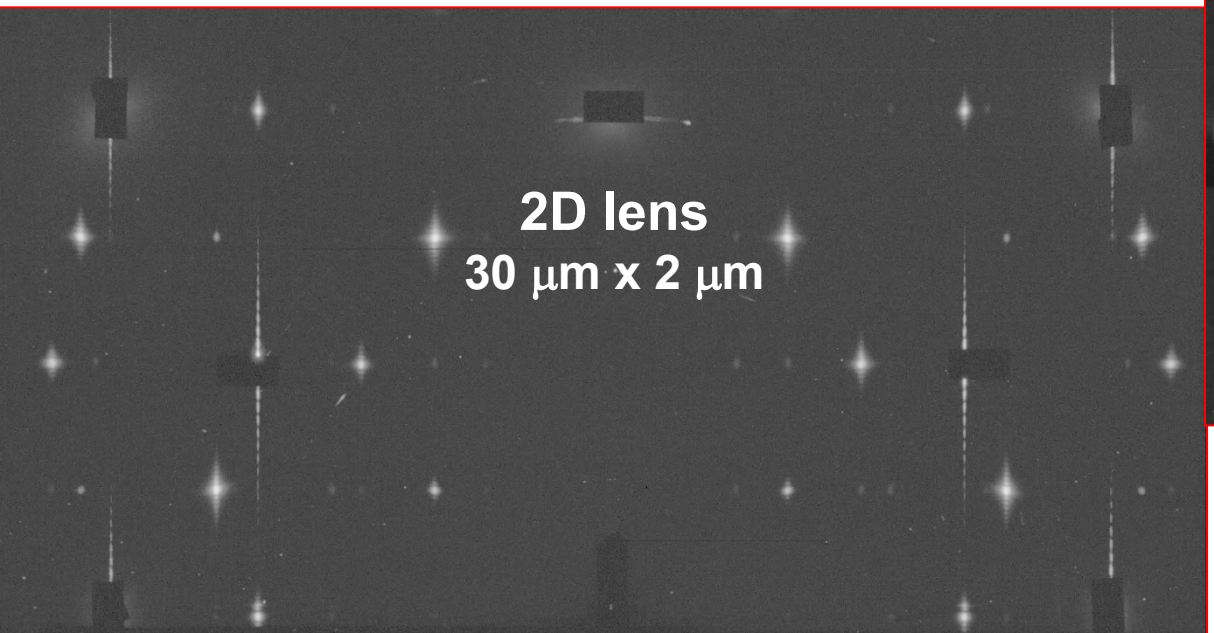
Oxidation of Pd(100) surface

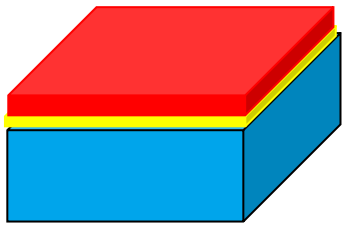


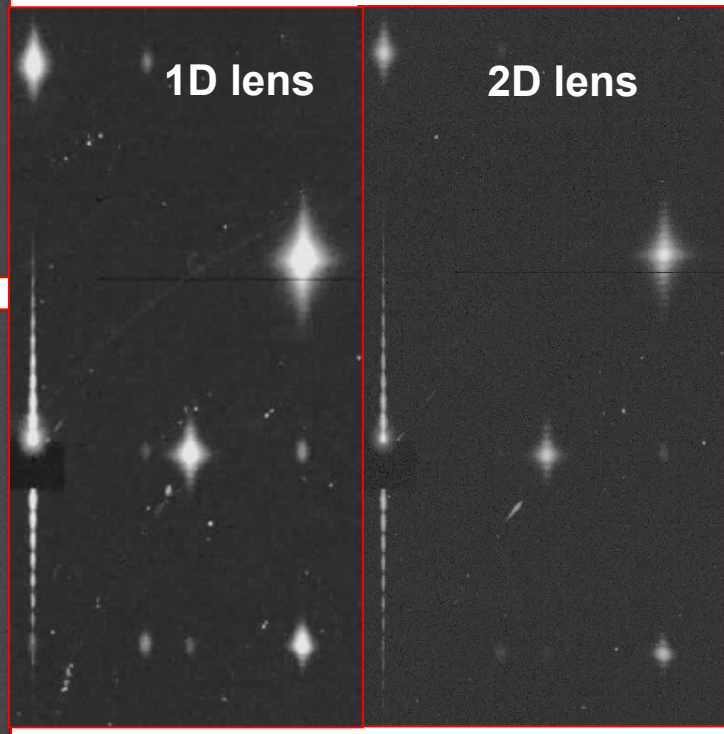


Pd(100)-(5 Pd/100)-surface oxide

Comparsion 1D-2D lenses ultrathin film at 78 keV

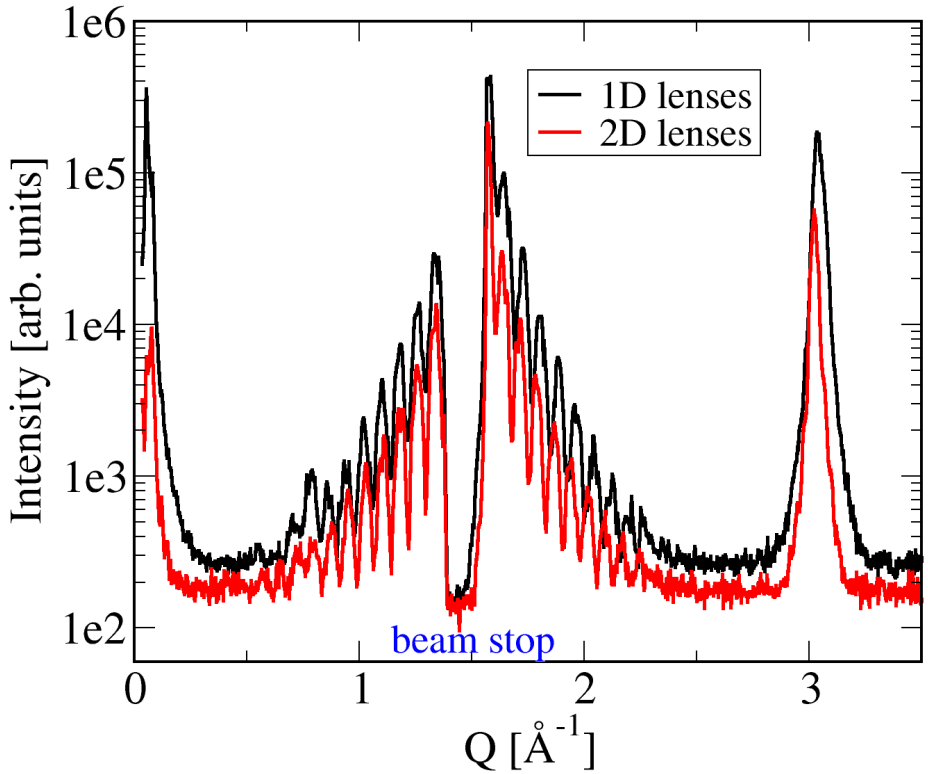


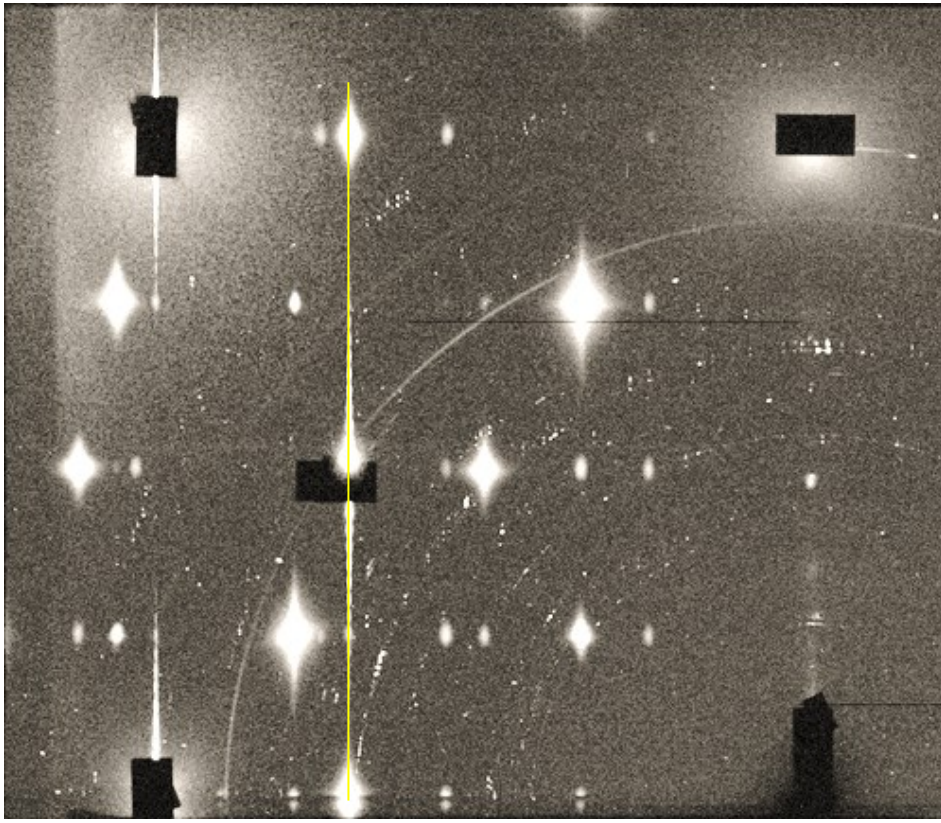




Whole pattern taken in 2 minutes

Comparsion 1D-2D lenses ultrathin film





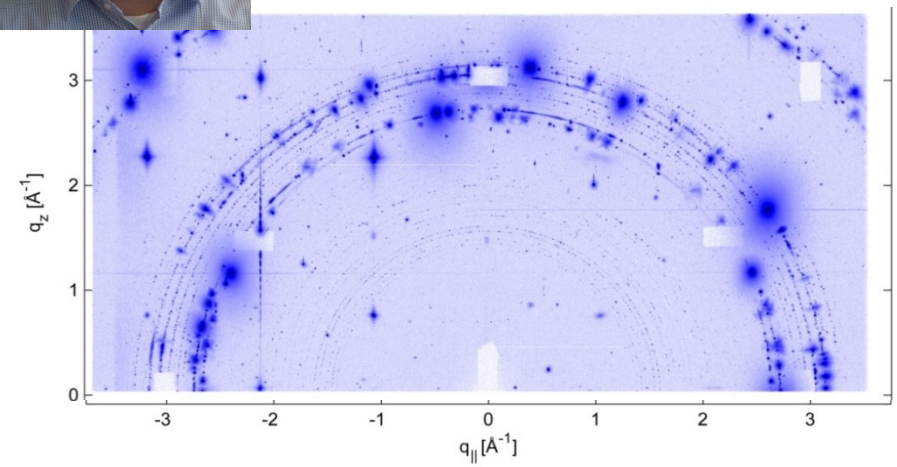
→ More flux of about factor 4
→ Less resolution

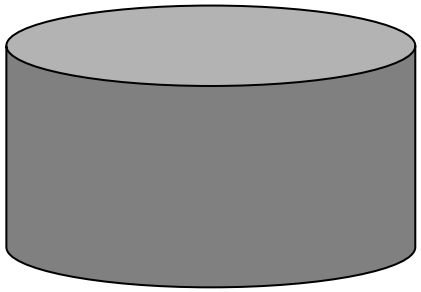
Approach for better time resolution for *in situ* studies

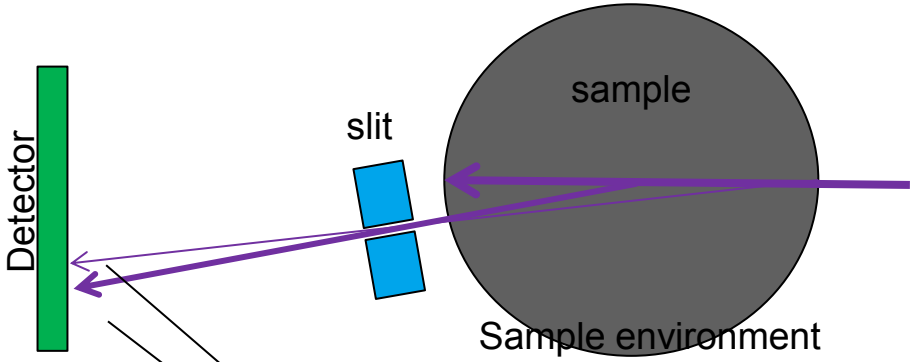
Florian Bertram: In situ MBE of FeO

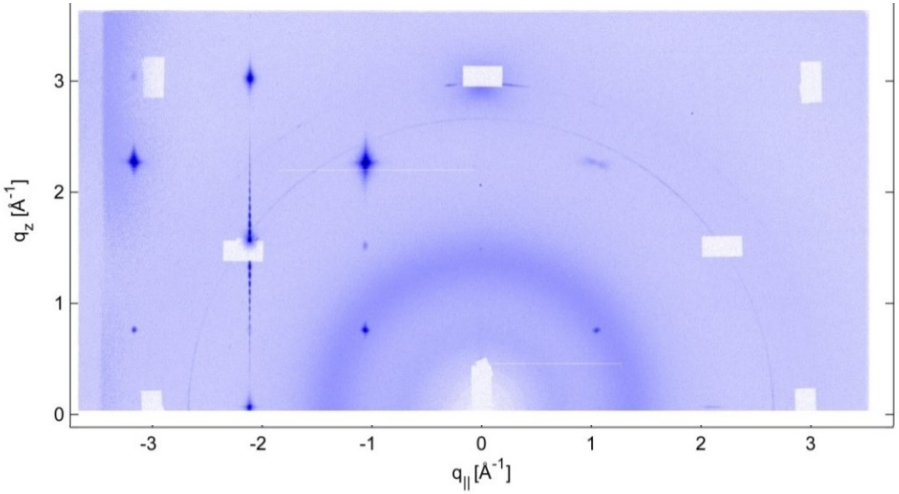
Concentration on 1 CTR → 2 seconds instead of 2 minutes

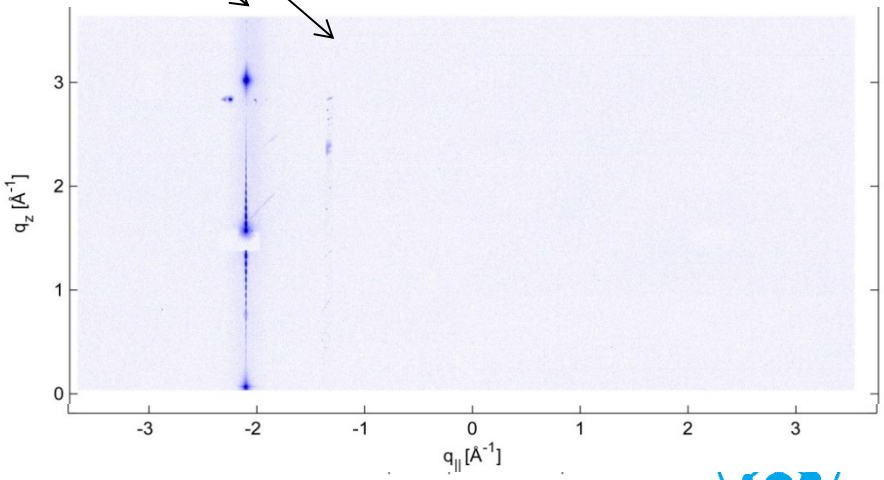
Better suppression of sample environment with slit behind sample











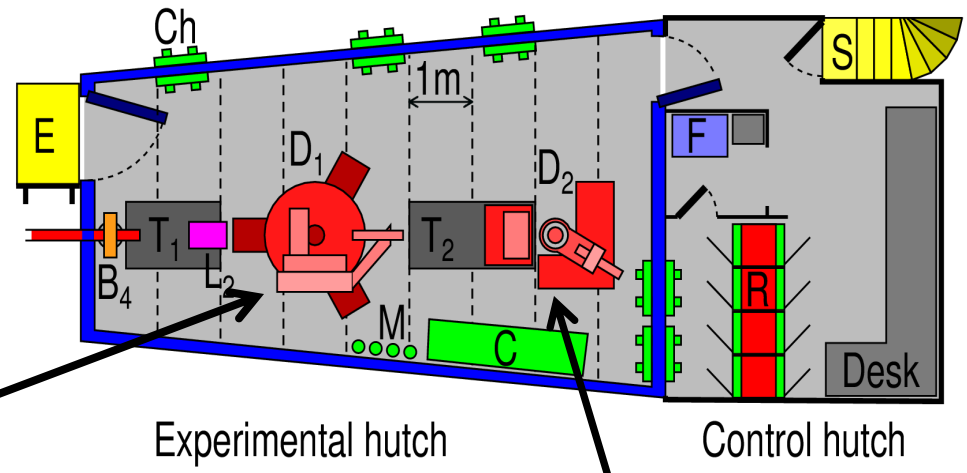
Nanostructures on wafer

- Nanoparticles and quantum dots for
  - Catalysis
  - fuel cells
  - LEDs
  - photovoltaic cells
  - batteries
  - Supercapacitors
  - …
- *In situ* and *in operando* detailed observations
  - in special atmosphere
  - with electrical contacts
  - in magnetic fields
  - In furnaces and cryostats
  - …..

P08: High Resolution Diffraction at 6-25 keV

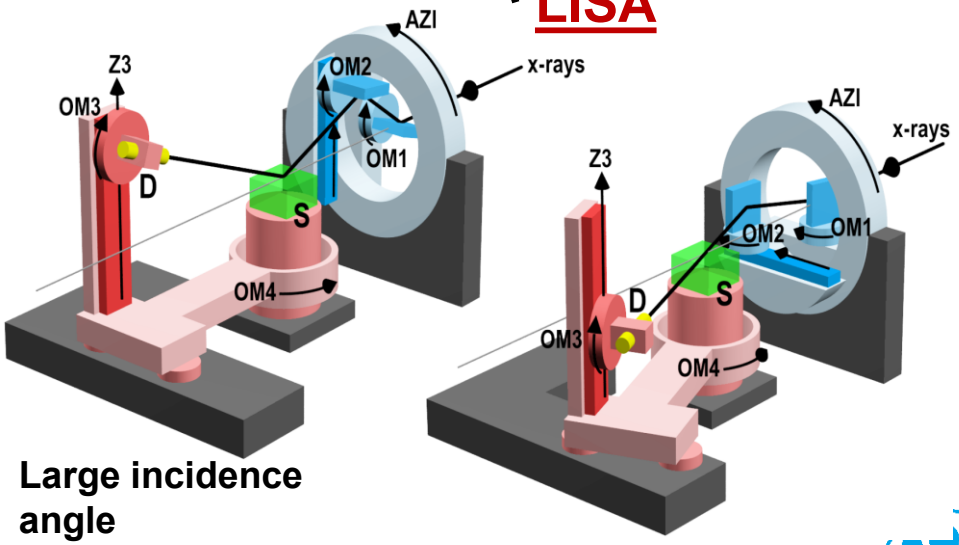
Beamline design: OH Seeck

P08



High resolution diffractometer

LISA

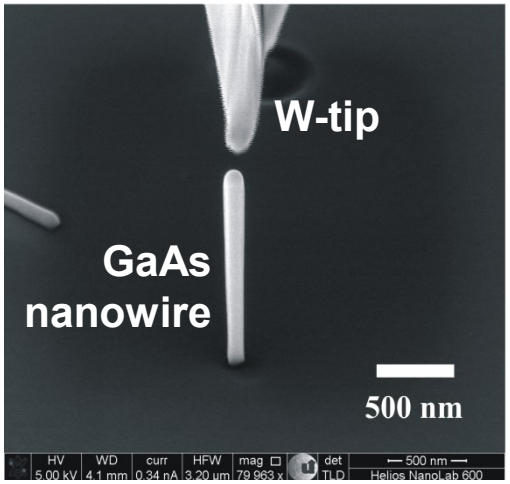


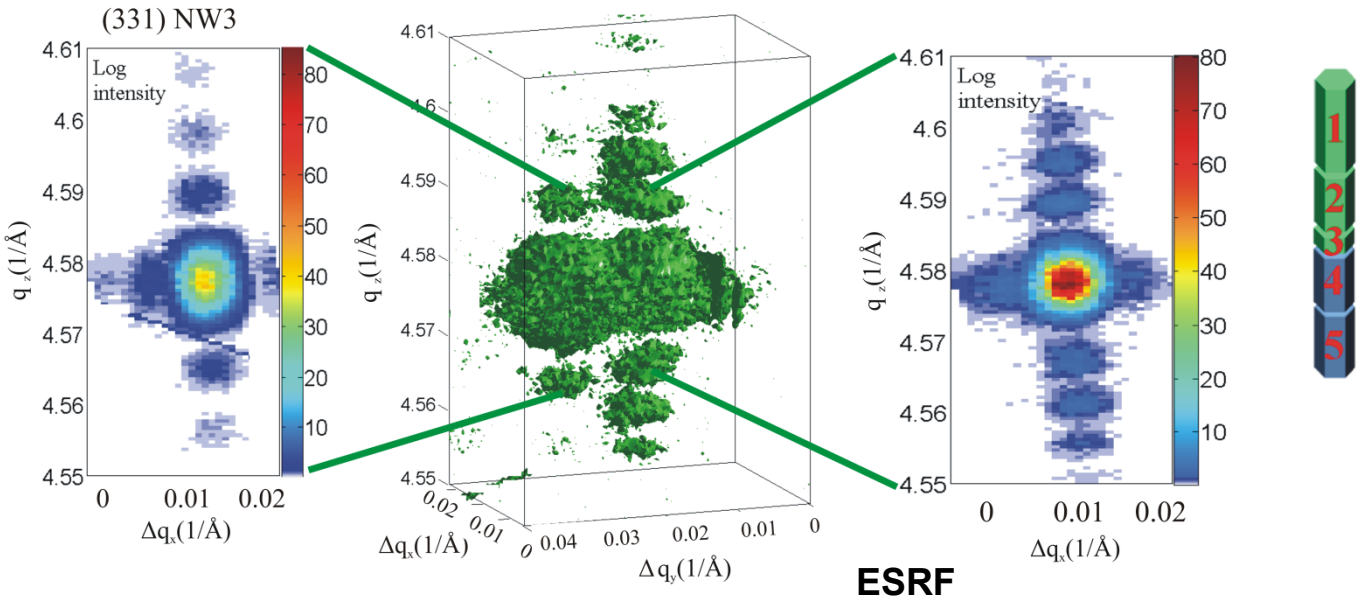
Large incidence angle

GaAs Nanowires on Si(111)

Genziana Bussone et al., NanoLett (2015), **15**, 981

Correlation of electrical and structural properties: stacking faults and interface along the wire





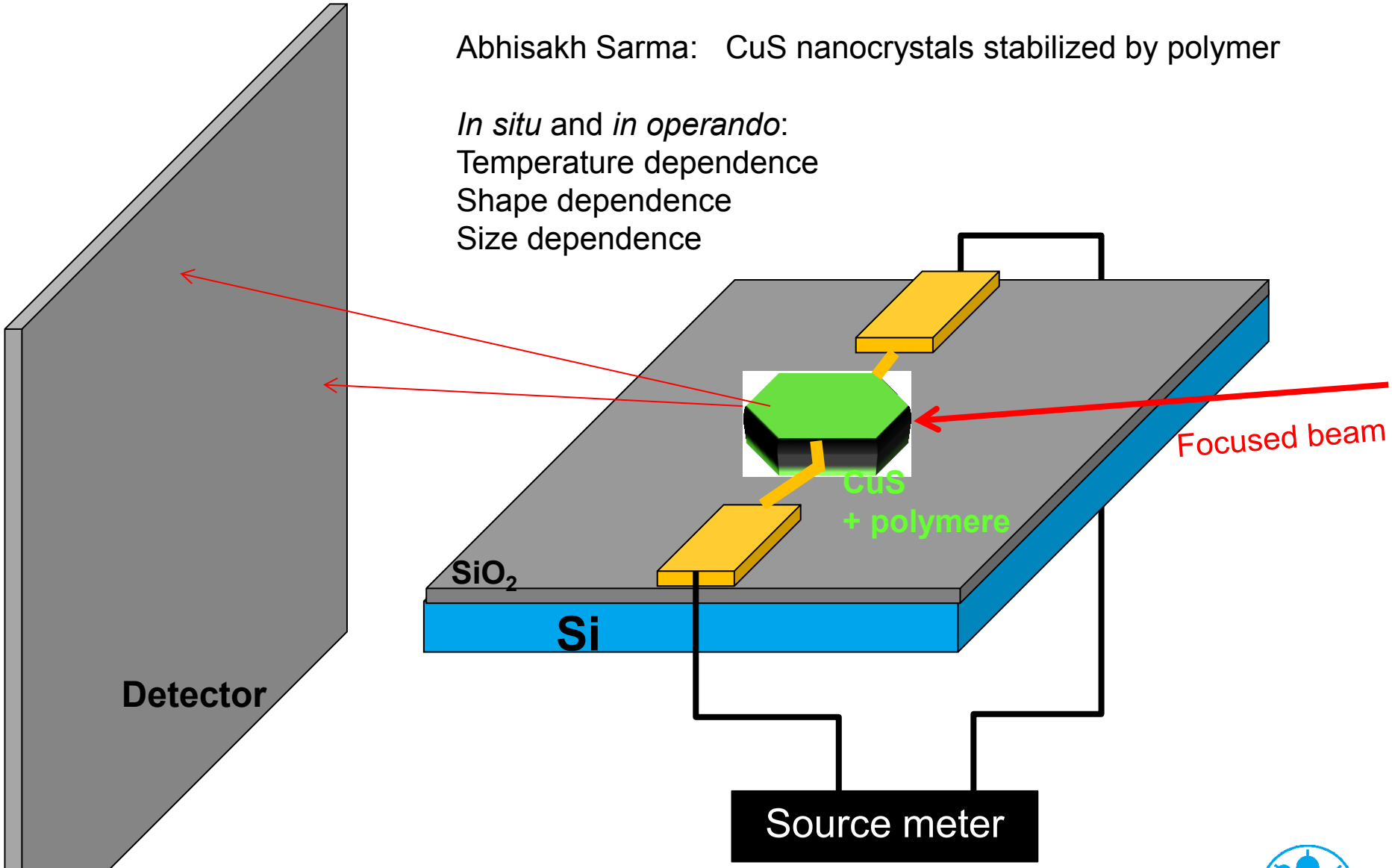
ESRF

Illumination over the full length
→ Averaging along the wire

Superionic nanostructures

Abhisakh Sarma: CuS nanocrystals stabilized by polymer

In situ and *in operando*:
Temperature dependence
Shape dependence
Size dependence

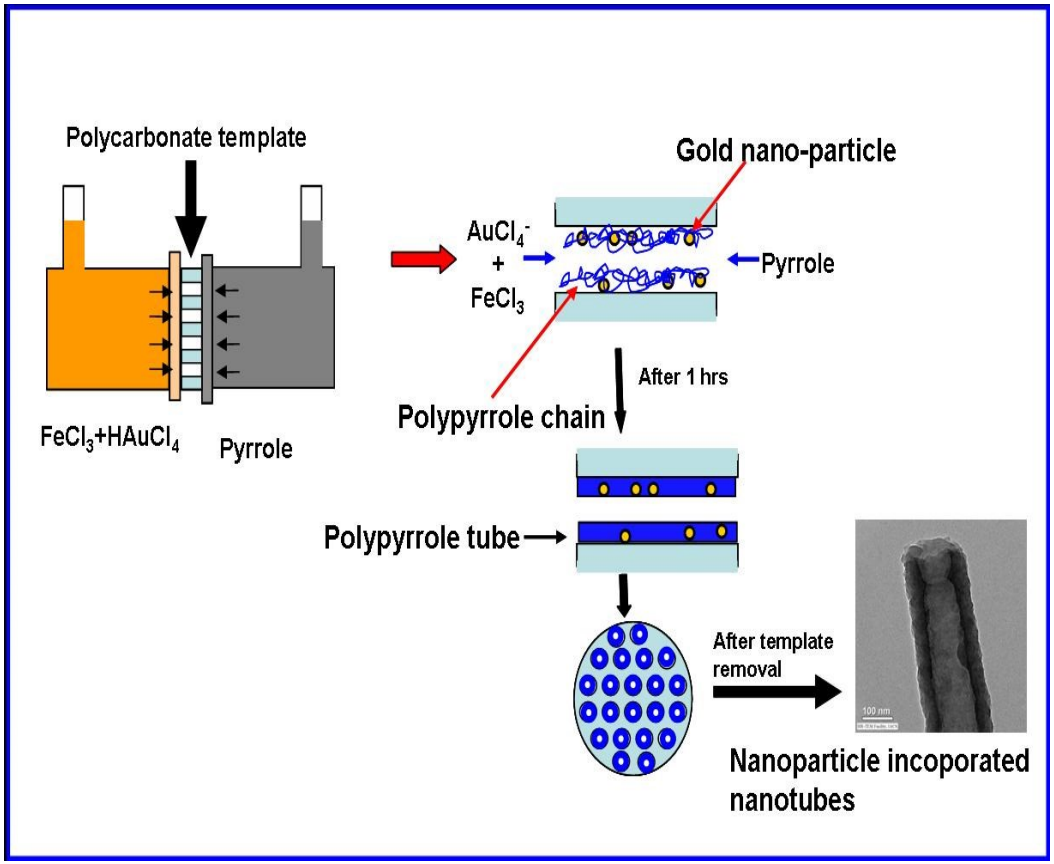


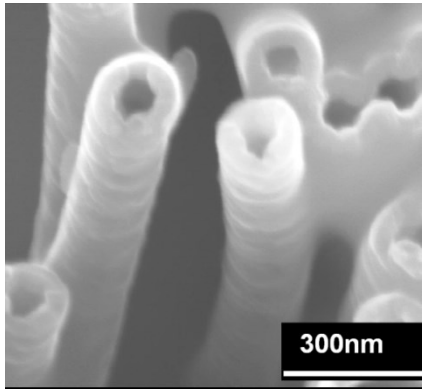
Charge density wave state

Abhisakh Sarma: Gold in Polypyrrole nanotubes

Colossal dielectric constant → Energy storage device

Resistance Switching → Memory devices





Dielectric constant vs.

- Gold particle size
- Gold microstructure
- Gold amount
- Nanotube size

High energies and high brilliance

High Energy Surface Diffraction: fast overview in reduced angular space

- Large q-space → precise study of local structural ordering
  → overview of reciprocal space, long range ordering
- Large reciprocal space measured in 2 minutes
- CTR measured in seconds

Smaller Emittance

- More flux in small focus → faster measurements
- Smaller focus → higher real space resolution, individual nanostructures
- Coherent beam → ptychography

***In situ* and *in operando* studies of nanostructures under "nanoscope"**

Thanks!

Christian Schroer (PETRA)	1D lenses
Jens Patommel (TU Dresden)	1D lenses
Norbert Schell (P07-HZG)	management of sector 5
Andreas Stierle (Nanolab)	catalysis chamber

Thank you for your attention!