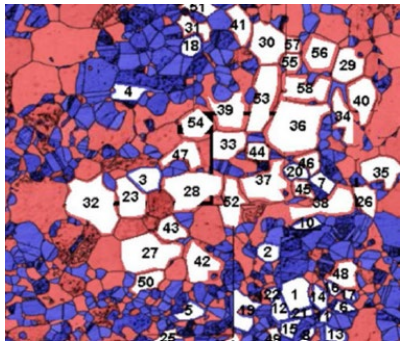
Workshop on Sachsen-DESY-Kooperationszentrum



DESY NanoLab

Andreas Stierle

Centre for X-Ray and Nanoscience (CXNS), DESY

nanolab.desy.de

NanoLab@DESY: Centre for X-Ray and Nanoscience

Science City Hamburg Bahrenfeld (SCHB)

HELMHOLTZ ASSOCIATION

CXNS DESY.

The DESY NanoLab

Providing on-site methods for nanoscience complementary to DESY photon science techniques at PETRA III(IV) and FLASH

Open for:

- **External users in the framework of accepted proposals or “around” X-ray beamtimes *https://door.desy.de***
- **Support of DESY in-house research (collaborative / contributive level)**
- **European users in the framework of the access program**

  Nanoscience Foundries and Fine Analysis
  https://www.nffa.eu/
- **Industrial users (DESY ITT + NFFA)**

HELMHOLTZ ASSOCIATION

How to Access the DESY NanoLab

During online PETRA III/FLASH proposal submission via DOOR

Open cycle flow cryostat (4-300K)	○ Yes ◉ No
Polarization	☑ standard ☐ variable linear ☐ circular
Polarization analyzer	○ Yes ◉ No

DESY NANOLAB (CONSULT DESY NANOLAB INSTRUMENTATION PAGE)	
Access to DESY NanoLab	Yes
Brief description of planned activities	
Microscopy: AFM (air)	No
Microscopy: Dual-beam focused ion beam (FIB)	No
Microscopy: High-resolution scanning electron microscope (SEM)	No
Microscopy: Variable temperature UHV STM/AFM	No
Surface spectroscopy: UHV-reflection absorption IR spectroscopy	Yes
Surface spectroscopy: X-ray photoelectron spectroscopy (XPS)	No
UHV Sample preparation	No
X-ray diffraction: Reflectometer	No
X-ray diffraction: Six-circle diffractometer	No

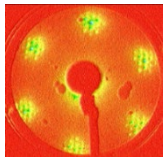
MAIN SAVE & CONTINUE

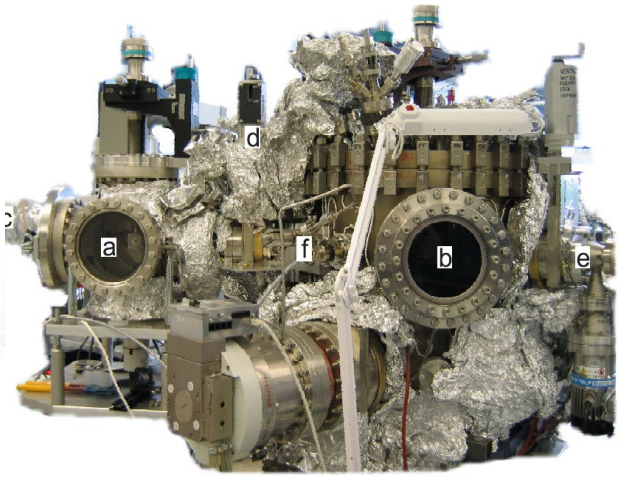
nanolab@desy.de

DESY NanoLab Techniques

Spectroscopy & Growth (H. Noei)

- UHV sample preparation chambers with LEED / AES
- XPS, FT-IR, STM, MBE growth





X-ray diffraction (V. Vonk)

- Reflectometer
- Six circle diffractometer
- Sample environments (het. catalysis, heating, UHV, electrochemistry)

Microscopy & Structuring (T. Keller)

- AFM, STM, optical
- SEM + FIB + EBSD + EDX (tomography)
- Lithography (CHyN)
- Scanning Auger Electron Microscope

Electrochemistry (M. Kohantorabi)

- Dedicated chemistry lab
- Potentiostats
- Induction oven / gases
- Solid / liquid FT-IR (FAU Erlangen)

Magnetic Characterization (L. Bocklage)

- Physical properties measurement system
- Kerr Microscope

nanolab.desy.de

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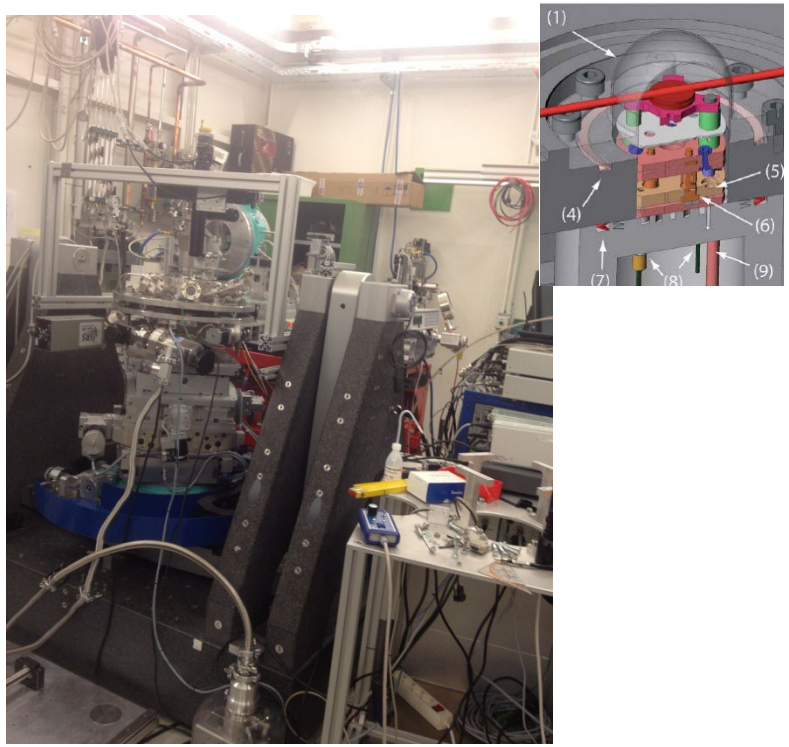
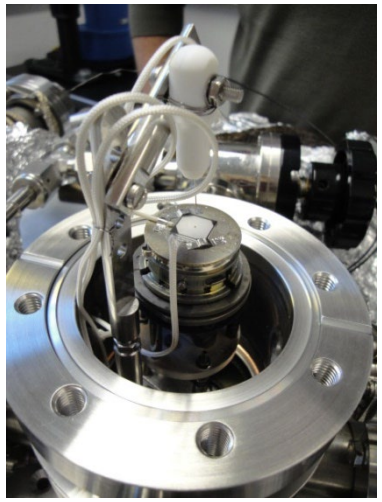
X-Rays: In-situ and Operando Experiments

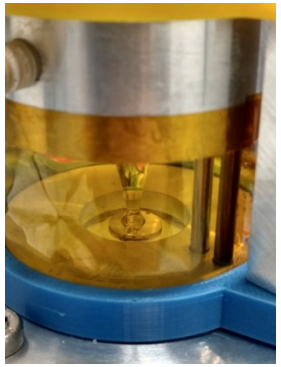
Mobile in-situ X-ray Diffraction Sample Environments

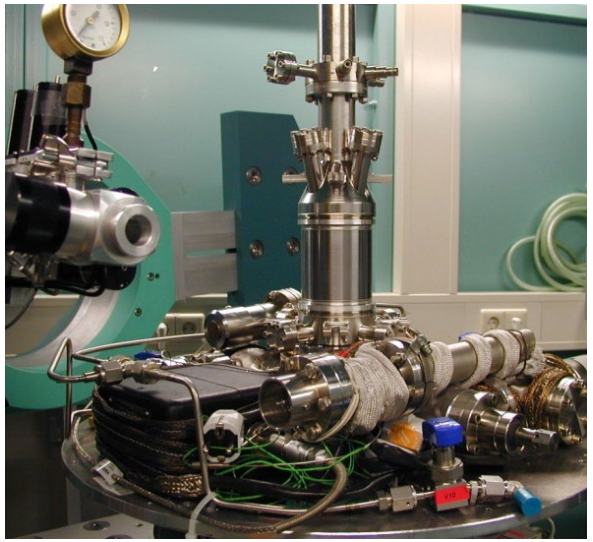


In-situ UHV / flow reaction chamber for catalysis



In-situ solid state electrochemistry Chamber, EC cells, RDE





In-situ UHV / HP chambers
RT-900 K
UHV - 1 bar
RT- 1500 K
UHV – 10^{-4} mbar

X-Rays: In-situ and Operando Experiments

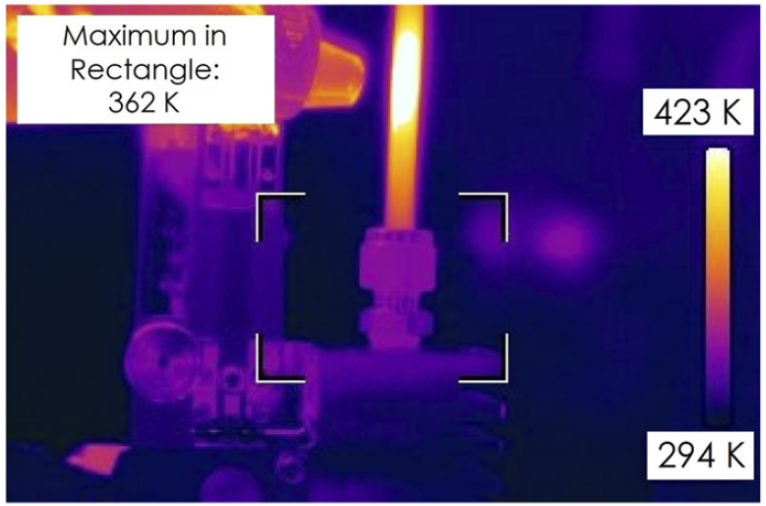
RESEARCH ARTICLE | JULY 06 2022

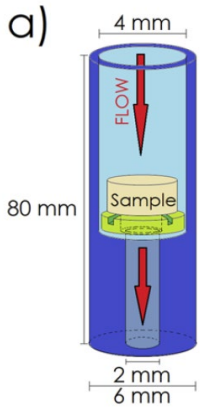
Operando reaction cell for high energy surface sensitive x-ray diffraction and reflectometry

R. Gleißner; E. E. Beck; Simon Chung; ... et. al

Check for updates

Rev Sci Instrum 93, 073902 (2022)
https://doi.org/10.1063/5.0098893



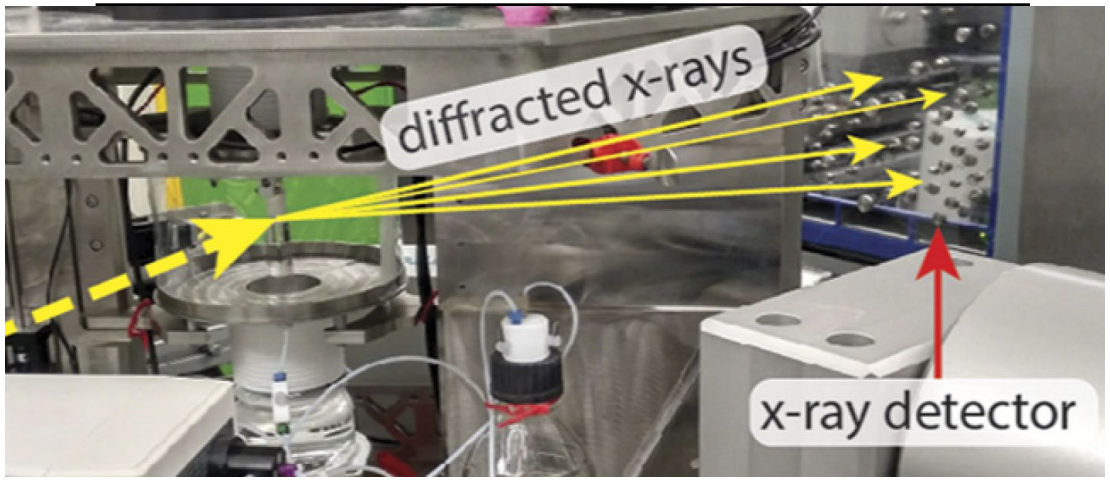


Catalysis SXRD flow cell
RT-700 K
50 bar

A combined rotating disk electrode–surface x-ray diffraction setup for surface structure characterization in electrocatalysis

Cite as: Rev. Sci. Instrum. **93**, 065111 (2022); https://doi.org/10.1063/5.0087864
Submitted: 10 February 2022 • Accepted: 22 May 2022 • Published Online: 22 June 2022

Leon Jacobse, Ralf Schuster, Johannes Pfrommer, et al.



Combined setup for SXRD and FT-IRRAS under EC conditions
Collaboration with Libuda group (FAU), in preparation

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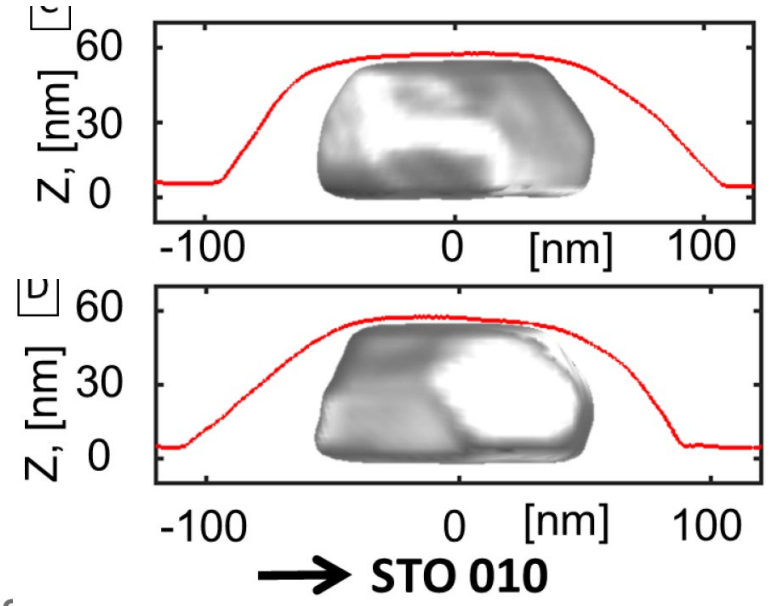
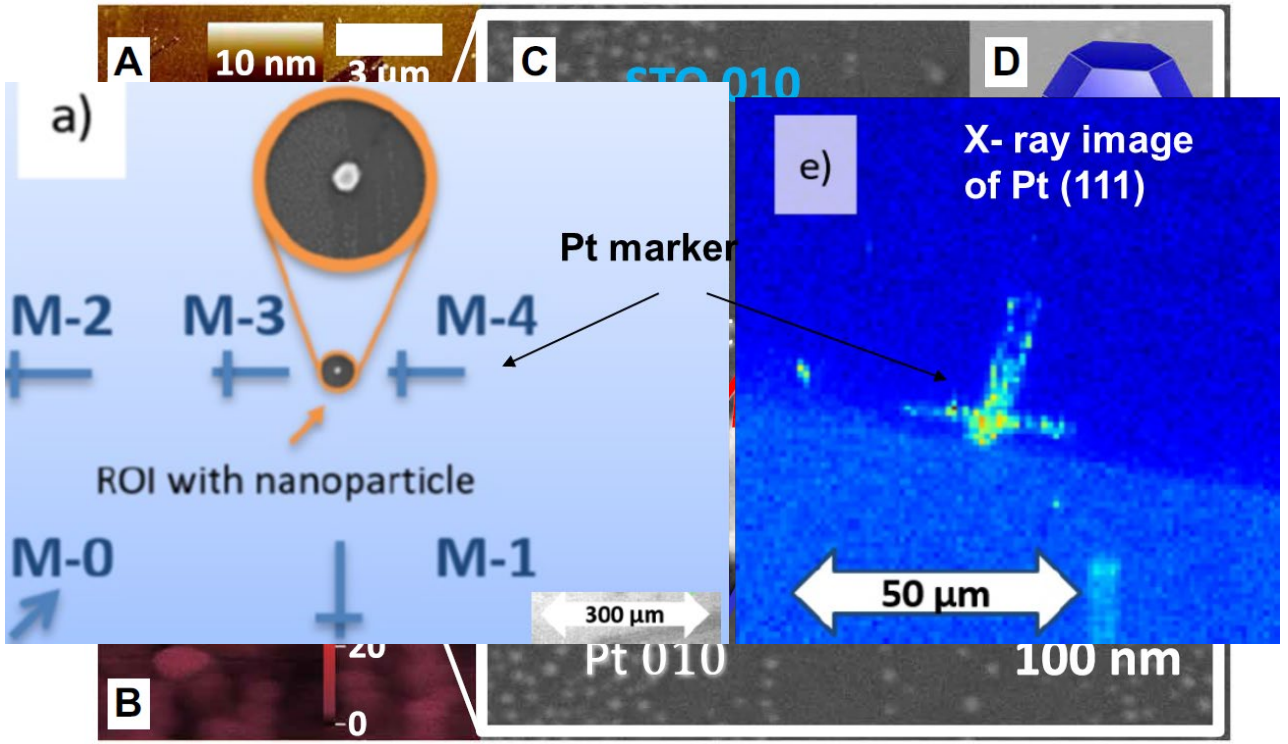
X-ray Imaging of Selected Single Nanoparticles for Het. Catalysis

Sample preparation

- $SrTiO_3$(001) substrate (TiO_2 terminated)
- Pt and Rh codeposition at 1100 K
- Post growth annealing at 1473 K
- Composition: 60% Pt 40% Rh
- Nano particle preselection by SEM and AFM
- Pt marker deposition



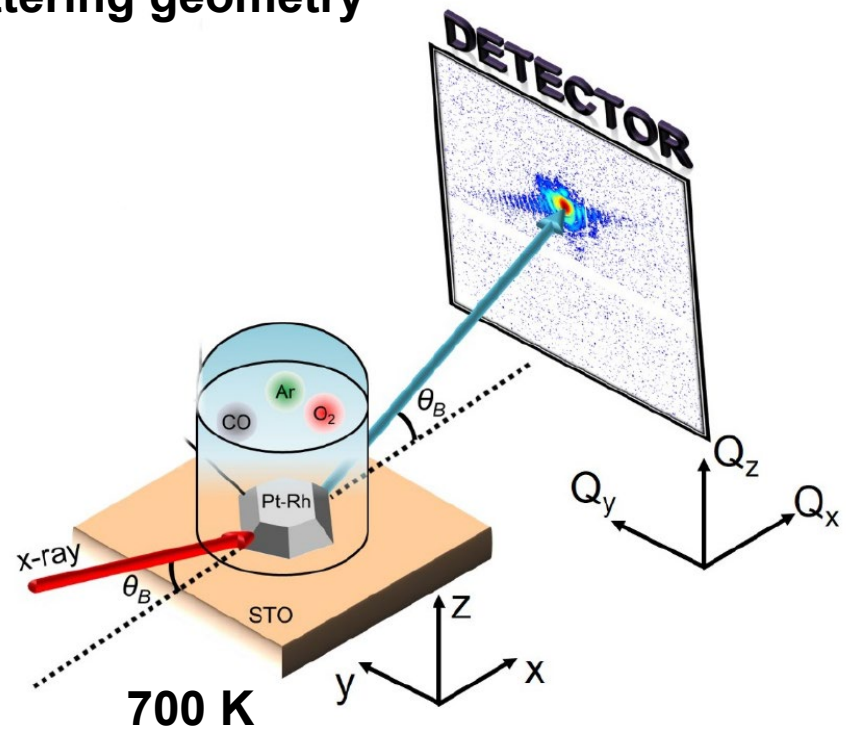


Nanoparticle height ~ 50 nm
diameter ~ 100 nm

Y. Y. Kim, T. F. Keller, T. J. Gonvalves, M. Abuin, H, Runge, L. Gelisio, J. Carnis, V. Vonk, P. N. Plessow, I. A. Vartaniants, A. Stierle, Science Advances, Vol 7, 40 (2021)

Bragg Coherent Diffraction Imaging

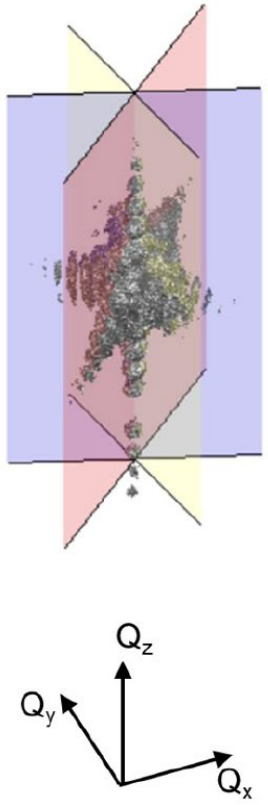
Scattering geometry

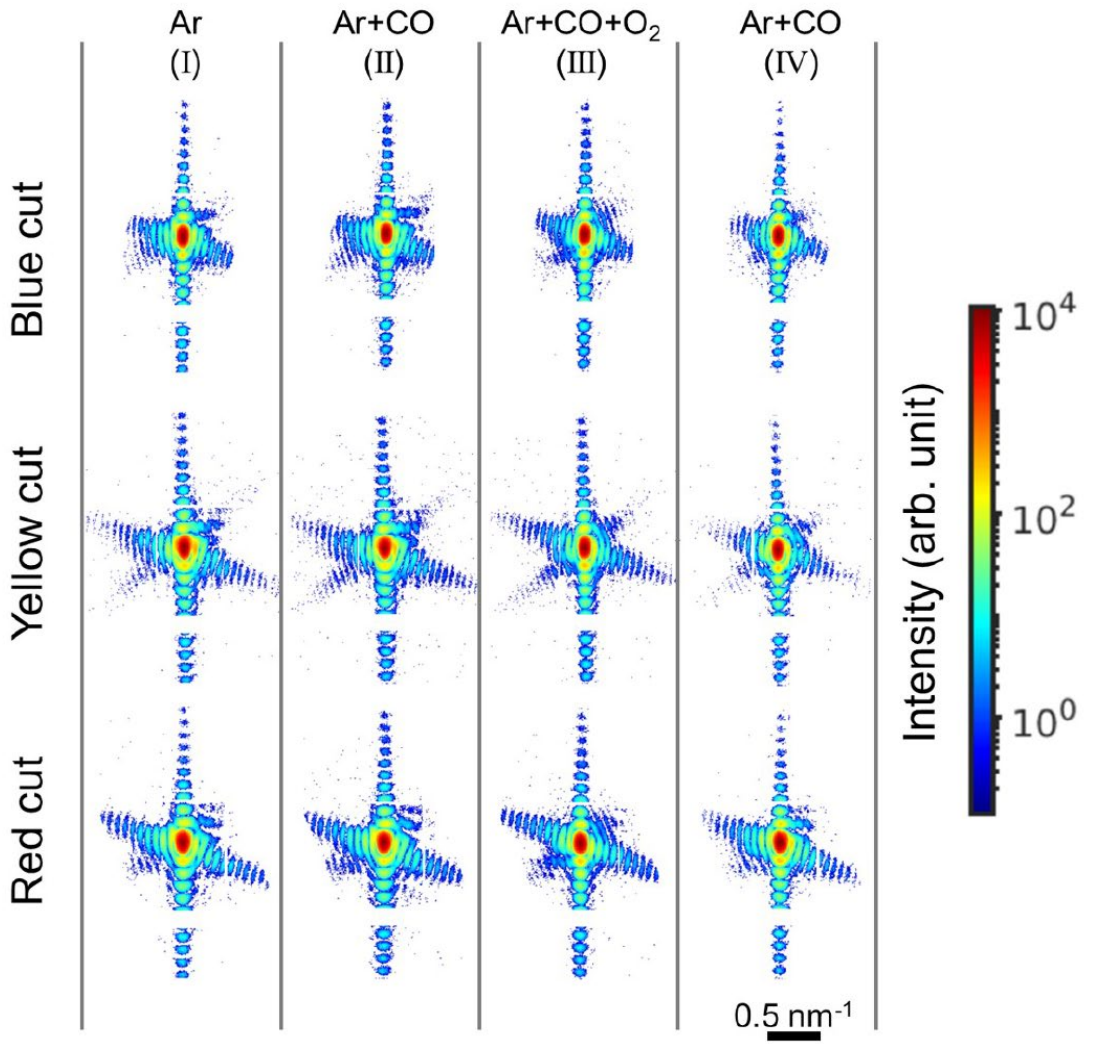


700 K

θ rocking scans: +- 1°
around the Pt (111) reflection

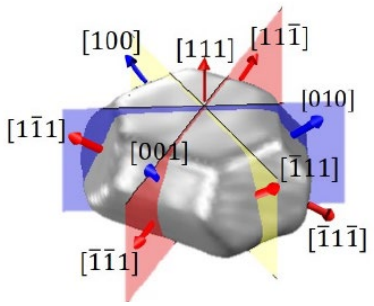
Evolution of the diffraction signal

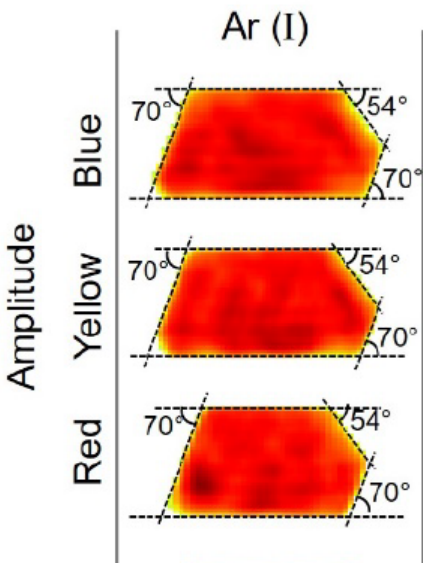


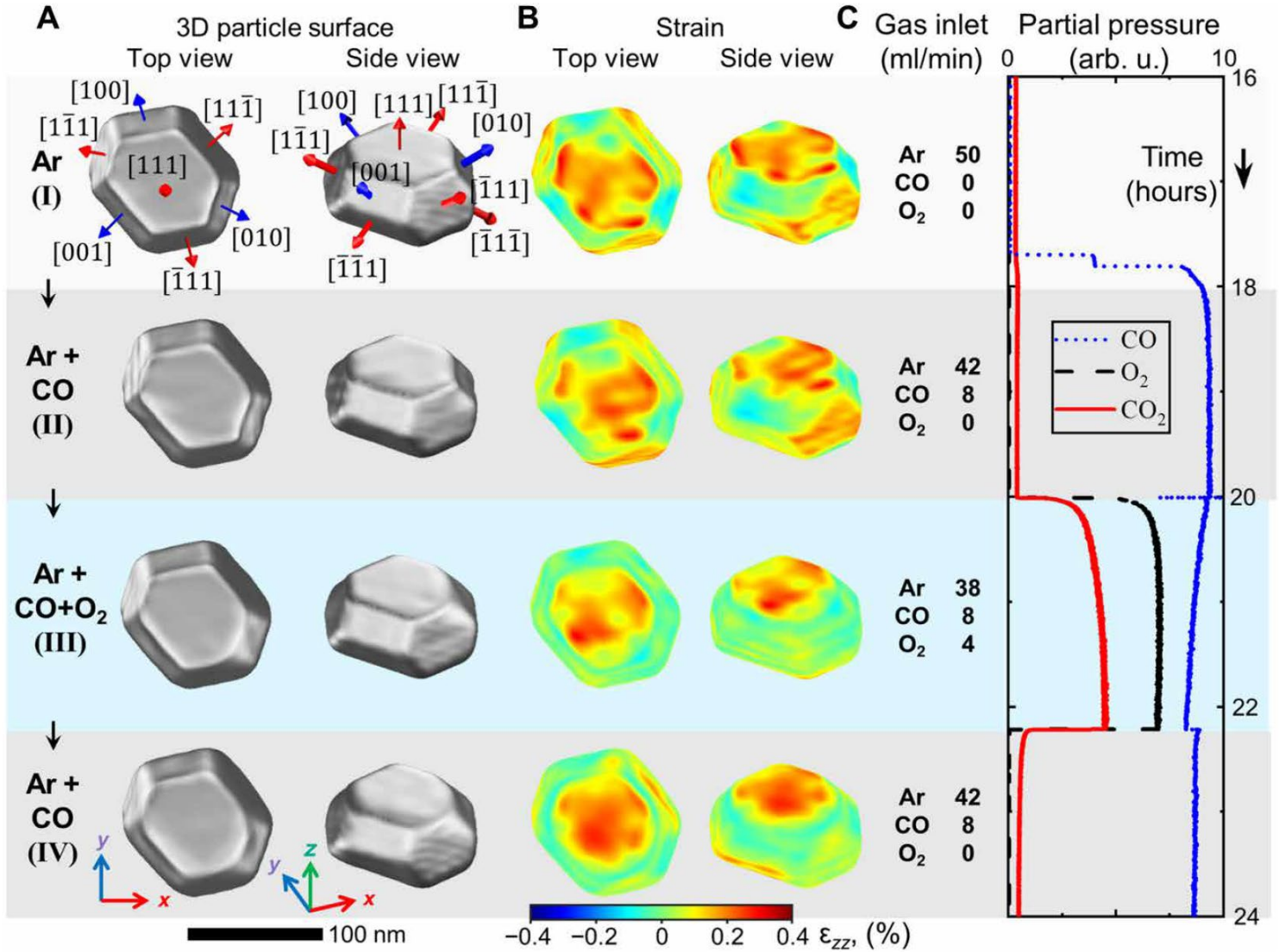


ESRF, ID01

Amplitude and Strain Reconstruction by Phase Retrieval Algorithms







55 nm high, 95 nm wide, and 120 nm long

Positive strain more pronounced on <111> Type facets

More equal strain distribution on side facets

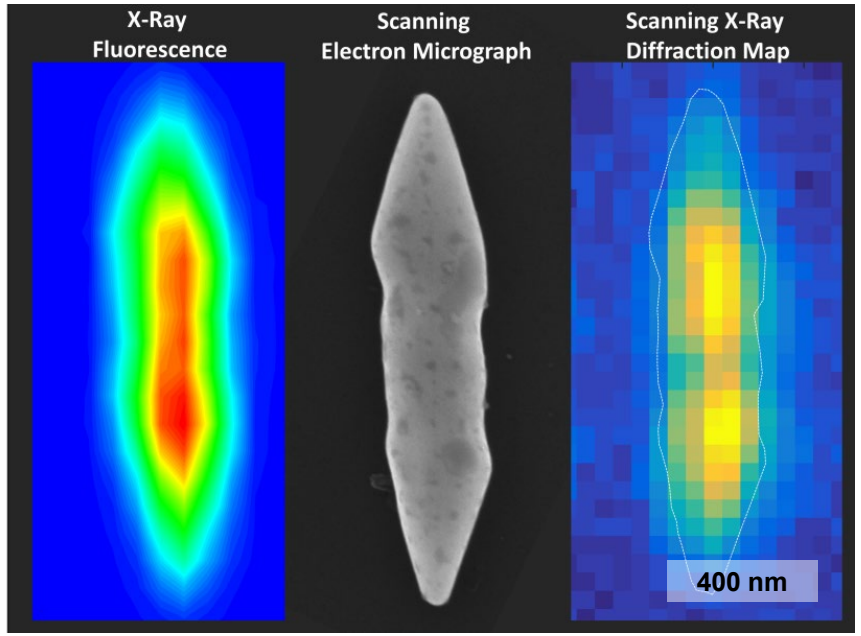
Change in strain state not reversible when back to pure CO

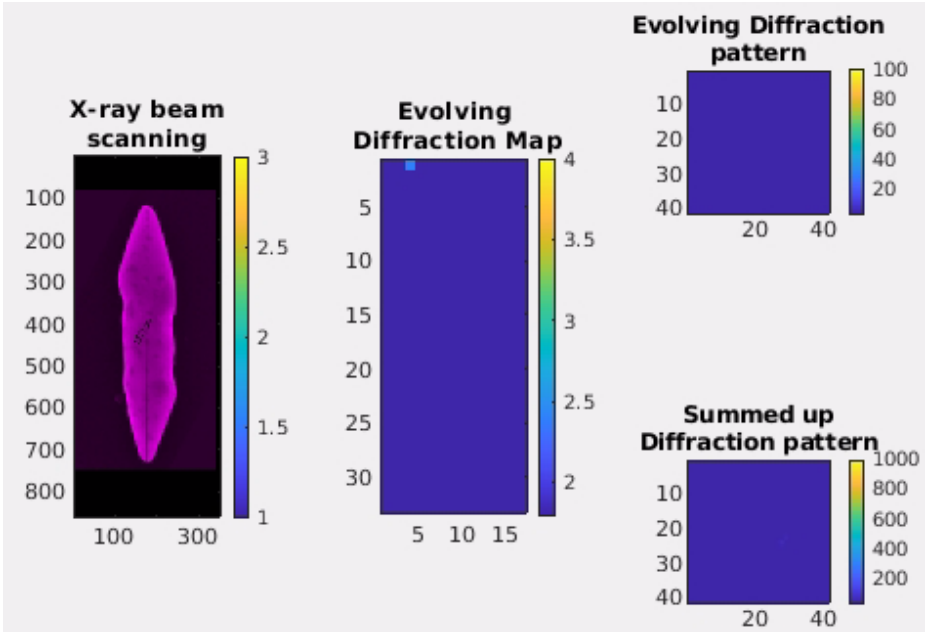
HELMHOLTZ ASSOCIATION

CNS DESY.

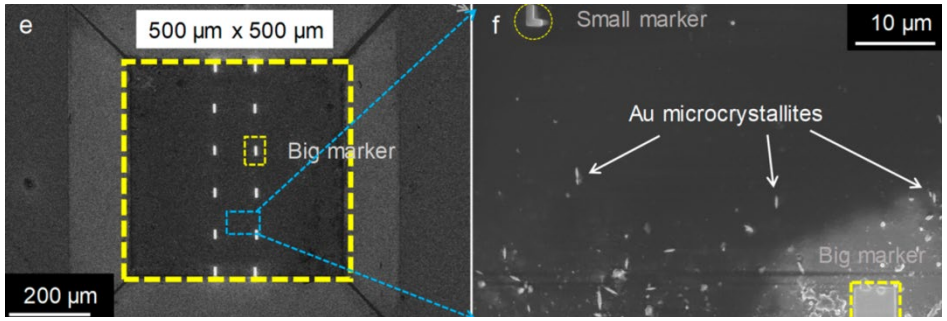
Multimodal Scanning X-ray Diffraction Microscopy

Spatial Distribution of Catalytic Non-Cubic Au in a Bipyramid Crystal at P06





Courtesy of A. Sarma



C. Sow, A. Sarma, et al., ACS Nano 2020, 14, 8, 9456.

MULTIMODAL MICROSCOPY

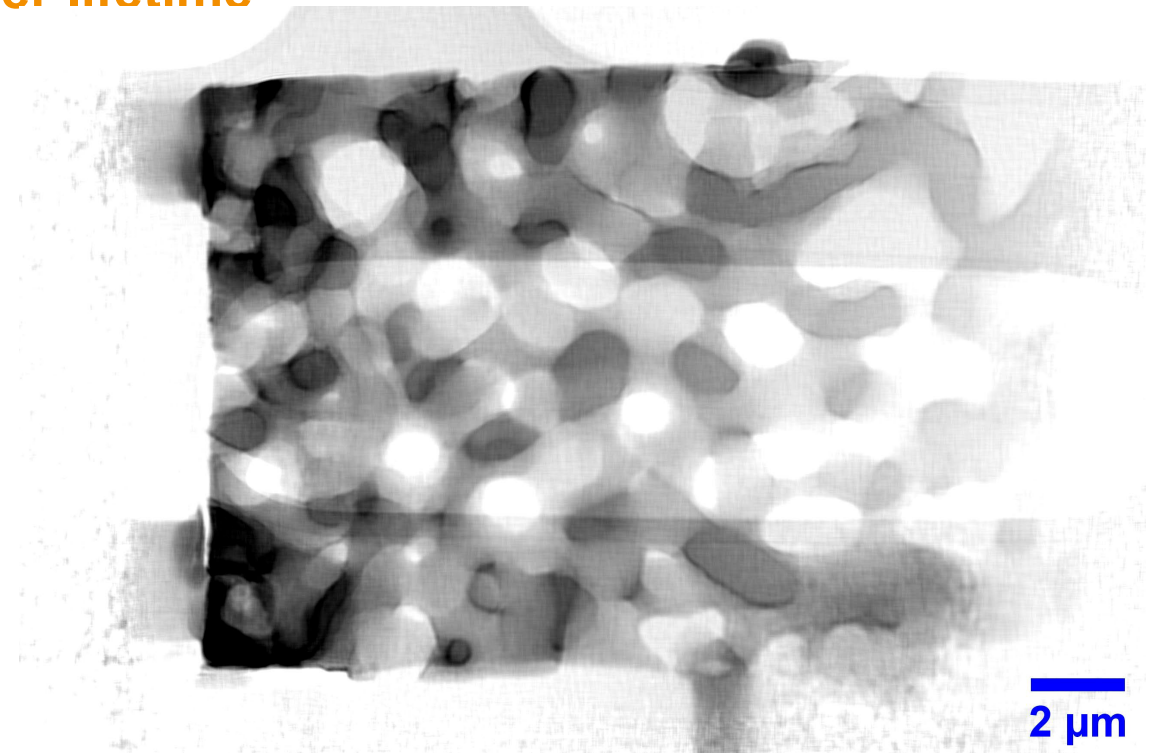
- **X-ray Diffraction and Fluorescence**
- **Hierarchical markers**
- **One-to-one (SEM @ DESY NanoLab)**

⇨ **Crystal tips are FCC rich**

Live View into Catalyst Materials

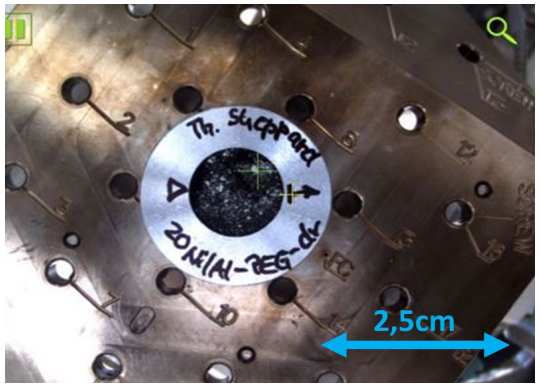
For more efficient, selective catalysts with longer lifetime

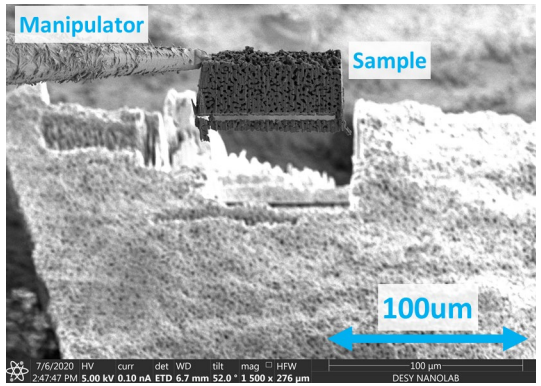
30 °C - 600 °C

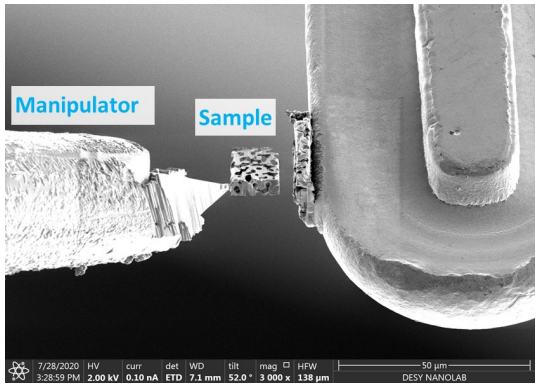


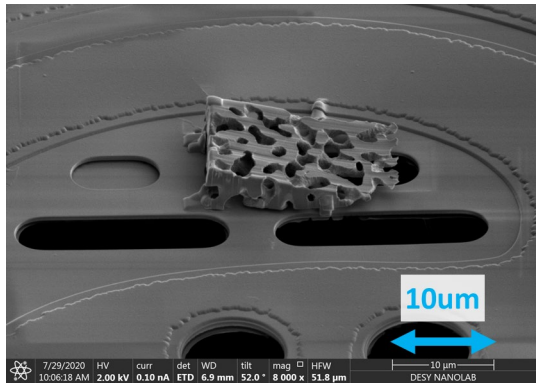
P06 / PETRA III

Hierarchical Nickel–Alumina catalysts for CO_2 methanation reaction









⇨ **X-ray imaging during calcination reveals an extensive volume shrinkage.**

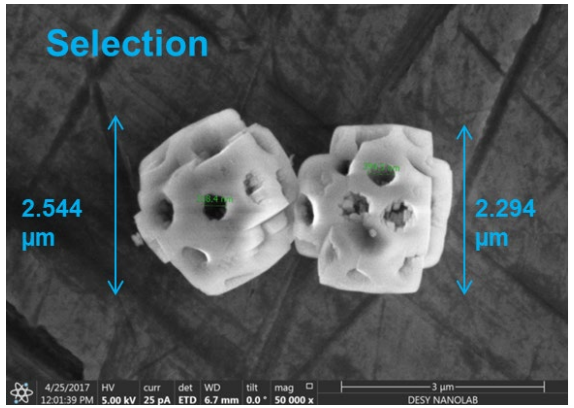
Collaboration within SFB 1441 "tracking the active site" coordinated by KIT

S. Weber et al., Advanced Science **2022**, 2105432.

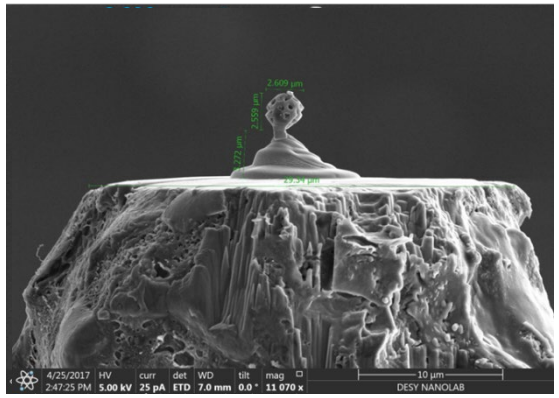
Combination of DESY NanoLab and PETRA III(IV)

Imaging a microporous zeolite carrier for catalytic nanoparticles

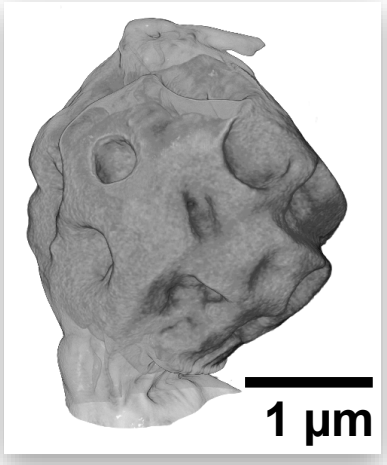
1. Selection in FIB / SEM



2. Mounting on tomography tip



3. 3D Ptycho- tomography + fluorescence (P06)



3D animation of reconstruction of zeolite, P06 ptycho-tomography

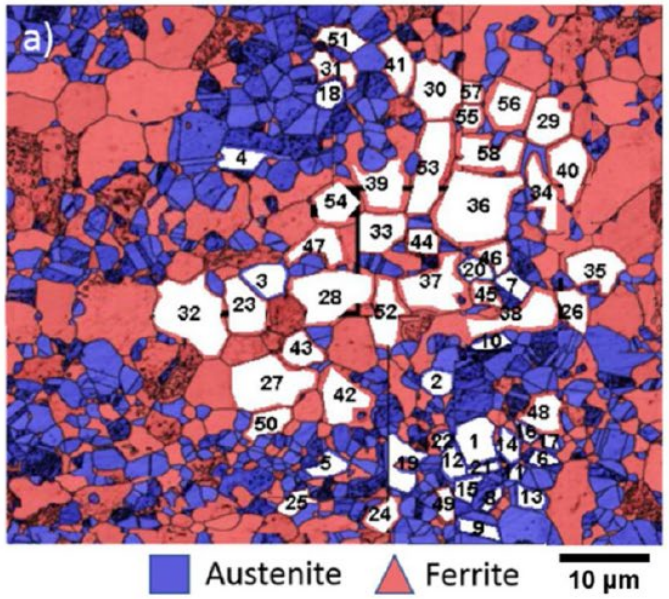
4. Post experimental FIB/SEM slice and view tomography

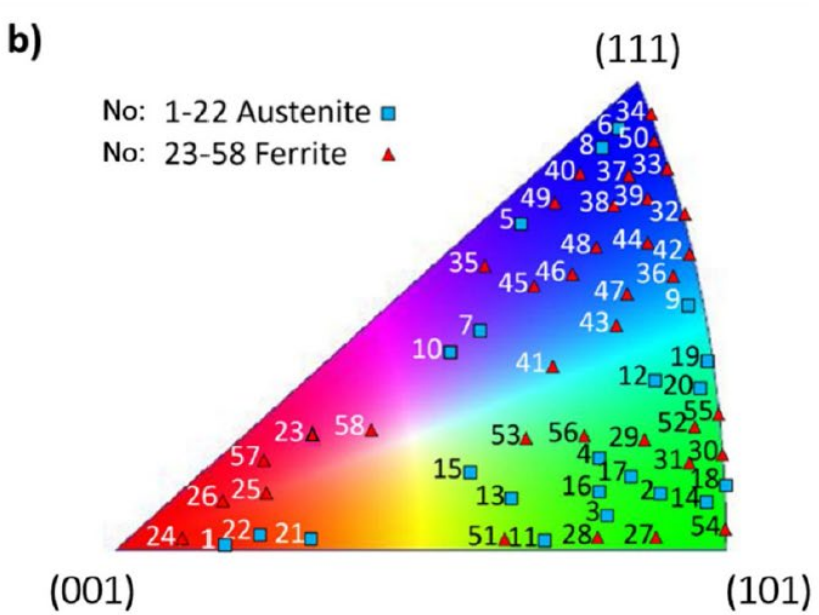
M. Seyrich, M. Kahnt, A. Schropp, C. Schroer, DESY
S. Kulkarni, T. F. Keller, A. Stierle, DESY
T. Sheppard, J.-D. Grunwaldt, KIT

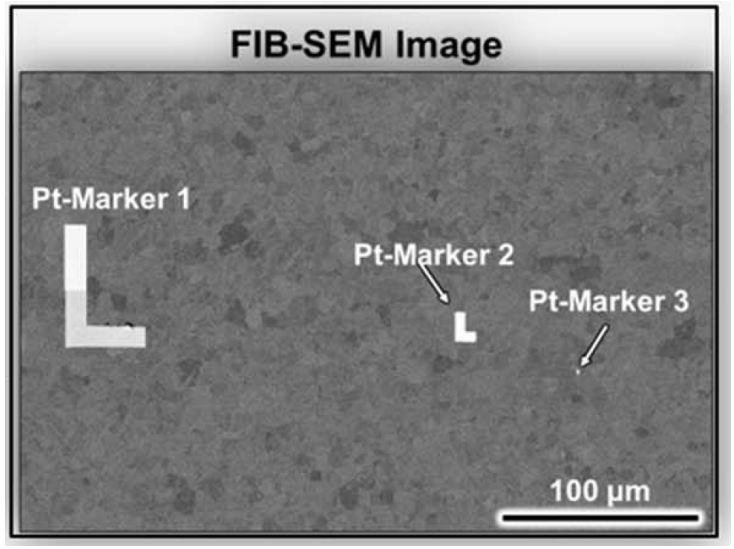
Combination of DESY NanoLab and PETRA III

Characterization of Native Oxide & Passive Film in Duplex Stainless Steel

Duplex stainless steel: high corrosion resistance combined with high mechanical strength
ferrite (more Fe, Cr and Mo) and austenite phase (enriched in N and Ni)





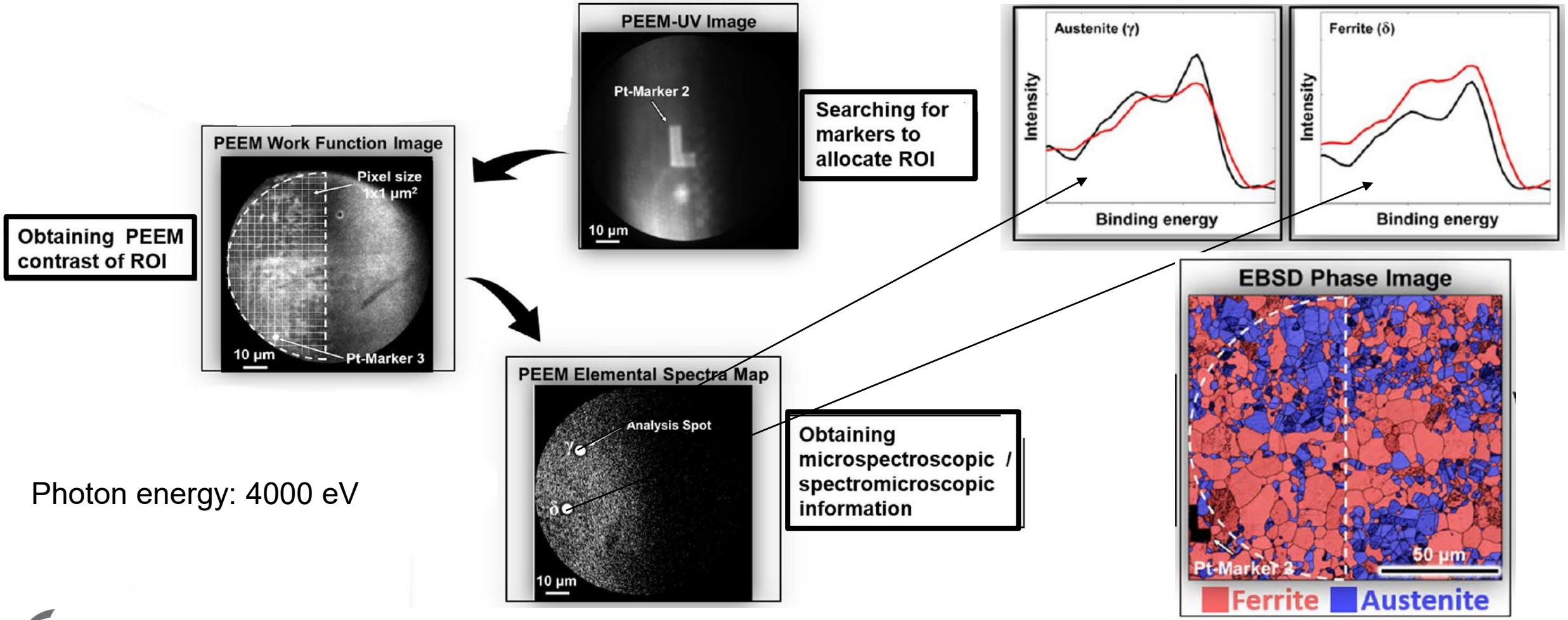


DESY NanoLab: Electron Backscatter Diffraction (EBSD): phase and grain orientation determination
Pt marker deposition from Pt containing precursor

M. Långberg, et al., J. Electrochemical Society 2019, 166, C3336
M. Långberg, et al., Corrosion Science, 2020, 174, 108841

Combination of DESY NanoLab and PETRA III

Hard X-ray photoelectron microscopy (HAXPEEM) at P22 (Hard X-ray photoemission beamline at PETRA III)



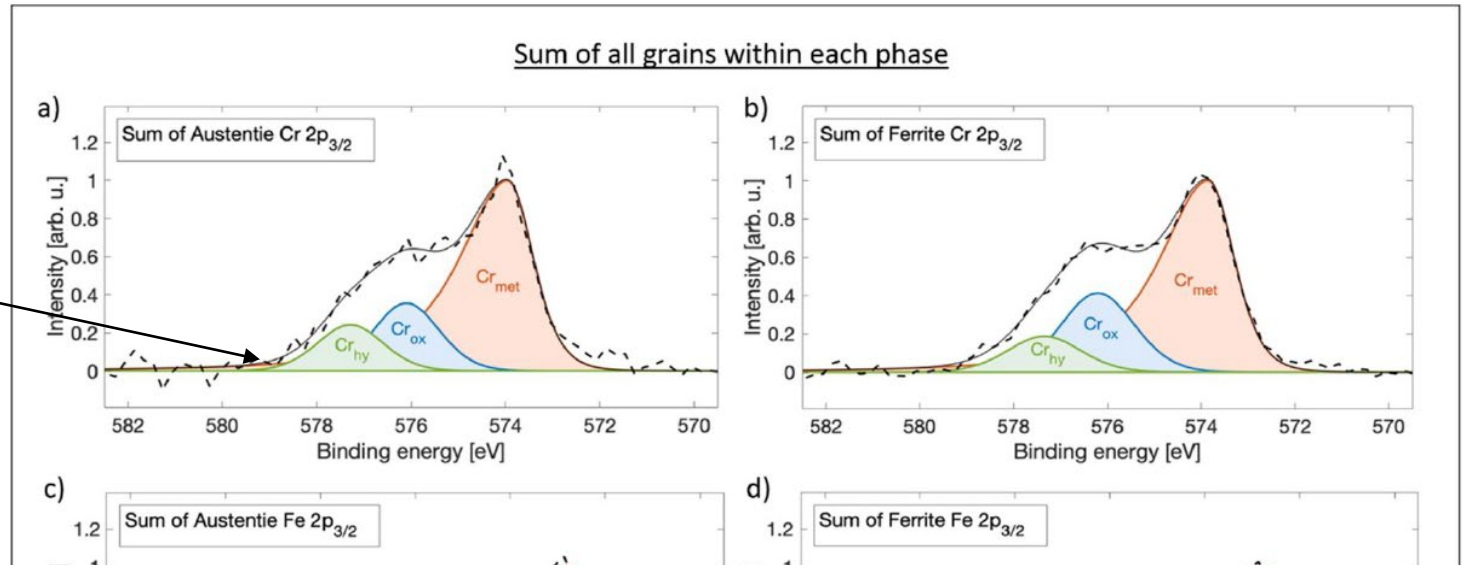
Photon energy: 4000 eV

M. Långberg, et al., J. Electrochemical Society 2019, 166, C3336
collaboration with KTH Stockholm and FZ Jülich

Combination of DESY NanoLab and PETRA III

Phase and orientation dependent natural oxide characterization on duplex stainless steel

more Cr hydroxide

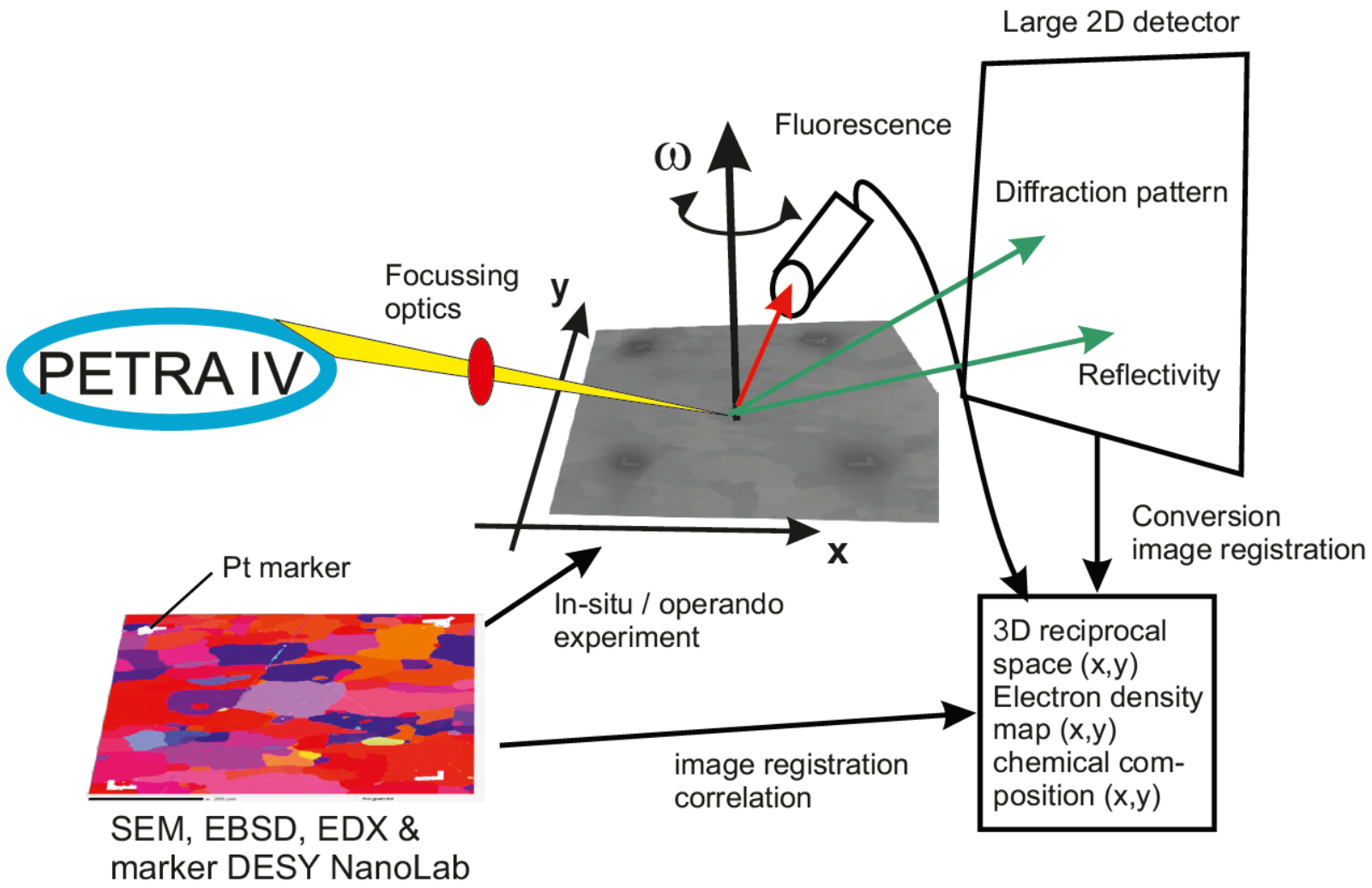


	Thickness (nm)			Cr content (at%)		
	Inner layer	Outer layer	Total	Inner layer	Outer layer	Average
Austenite	1.0	0.8	1.8	66	84	75
Ferrite	1.0	0.6	1.6	75	83	79

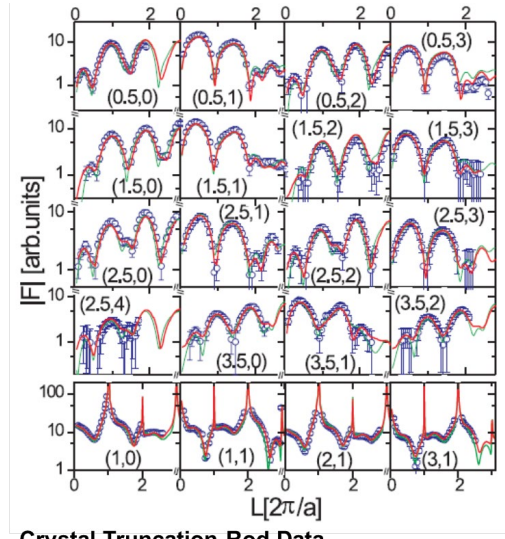
M. Långberg, et al., Corrosion Science, 2020, 174, 108841

The Future: Surface Sensitive X-ray Diffraction with nm Resolution

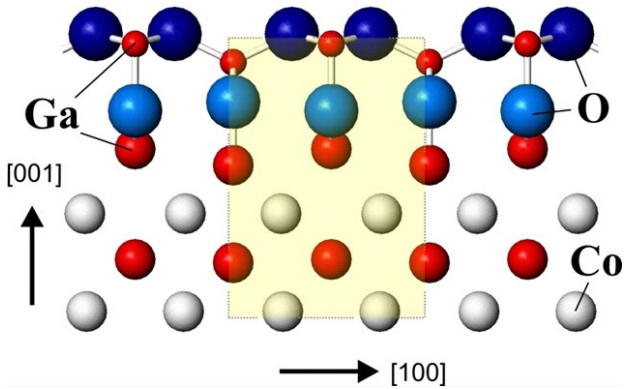
Fully automatized in-situ and operando experiments



SXRD



Crystal Truncation Rod Data
Fit: red line (DFT model: green line)

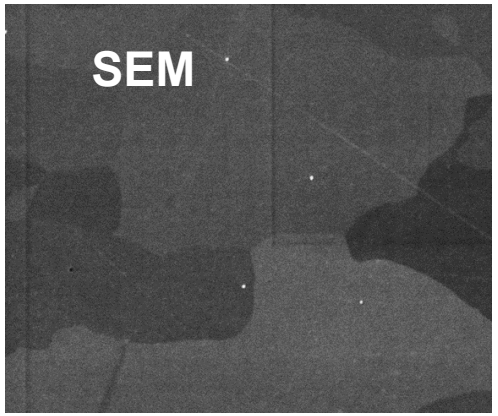


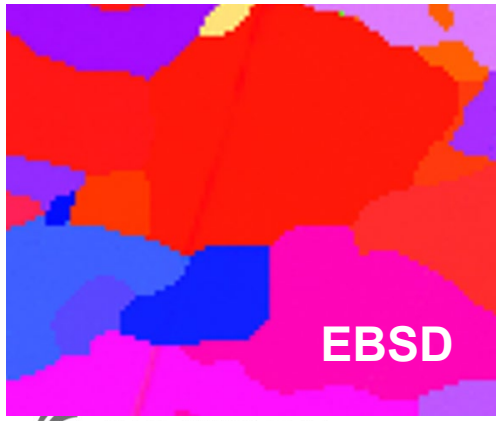
Structural Model:
Ga_4O_4 on CoGa(100)

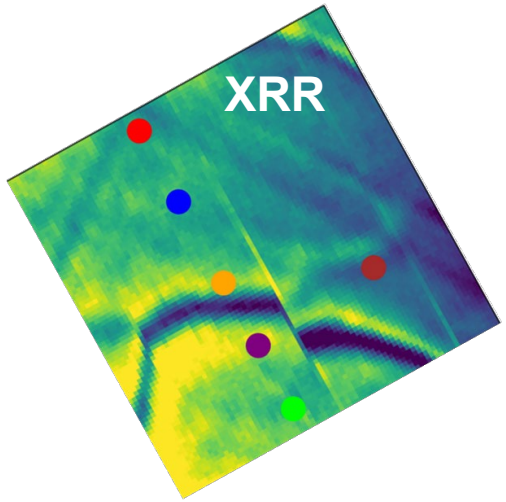
The Future: Surface Sensitive X-ray Diffraction with nm Resolution

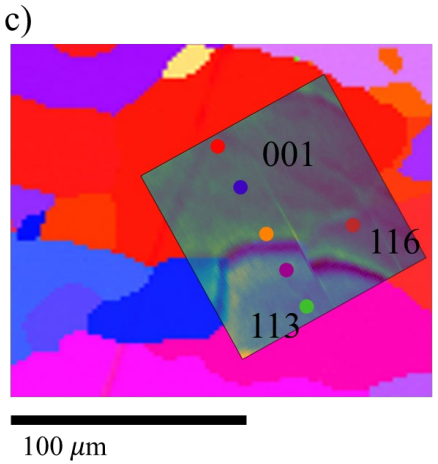
First XRR experiments: ESRF ID1 on fine grain Nb foils for SC RF cavity applications

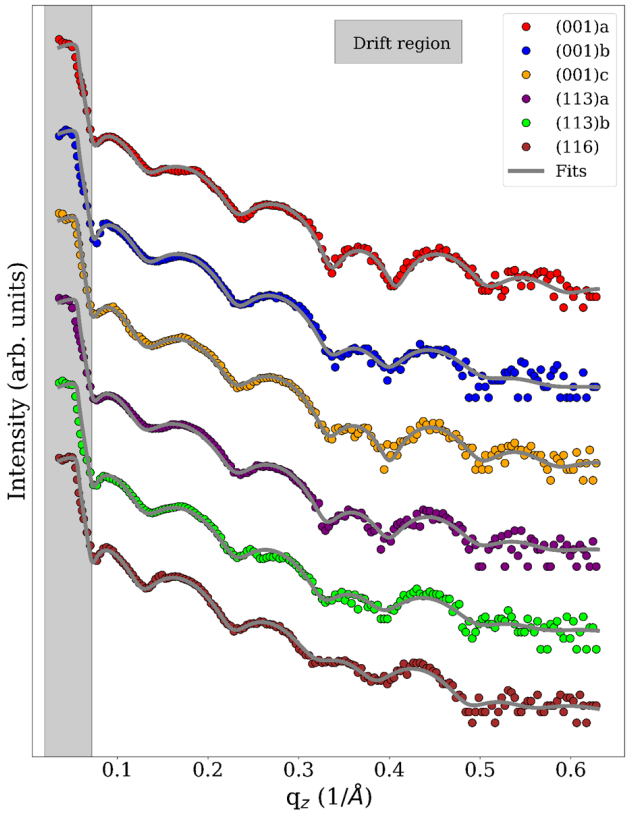
Photon energy 10 keV
70 nm X-ray beam size

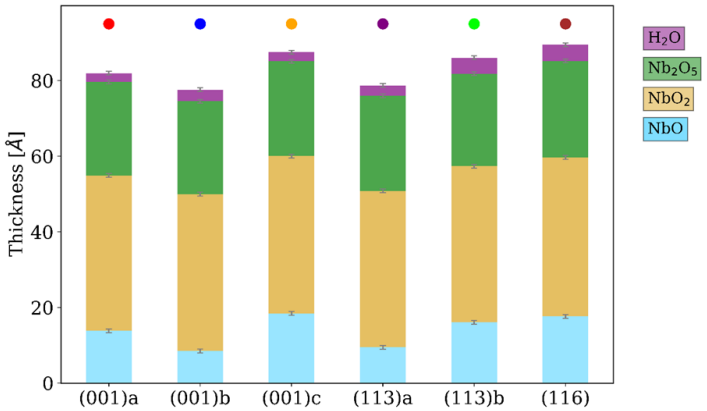












G. Dalla Lana, et al., in preparation

HELMHOLTZ ASSOCIATION

Thanks to:

DESY: The DESY NanoLab group, beamlines P07, P08, P09, P21, P22, P23, P24, P06, P01, P03, P10

KTH: Pan group

FZ Jülich: Schneider group

ESRF: ID1

FAU: Libuda group

Synchrotron sources for beamtime: PETRA III, ESRF

Financial support:

HELMHOLTZ ASSOCIATION

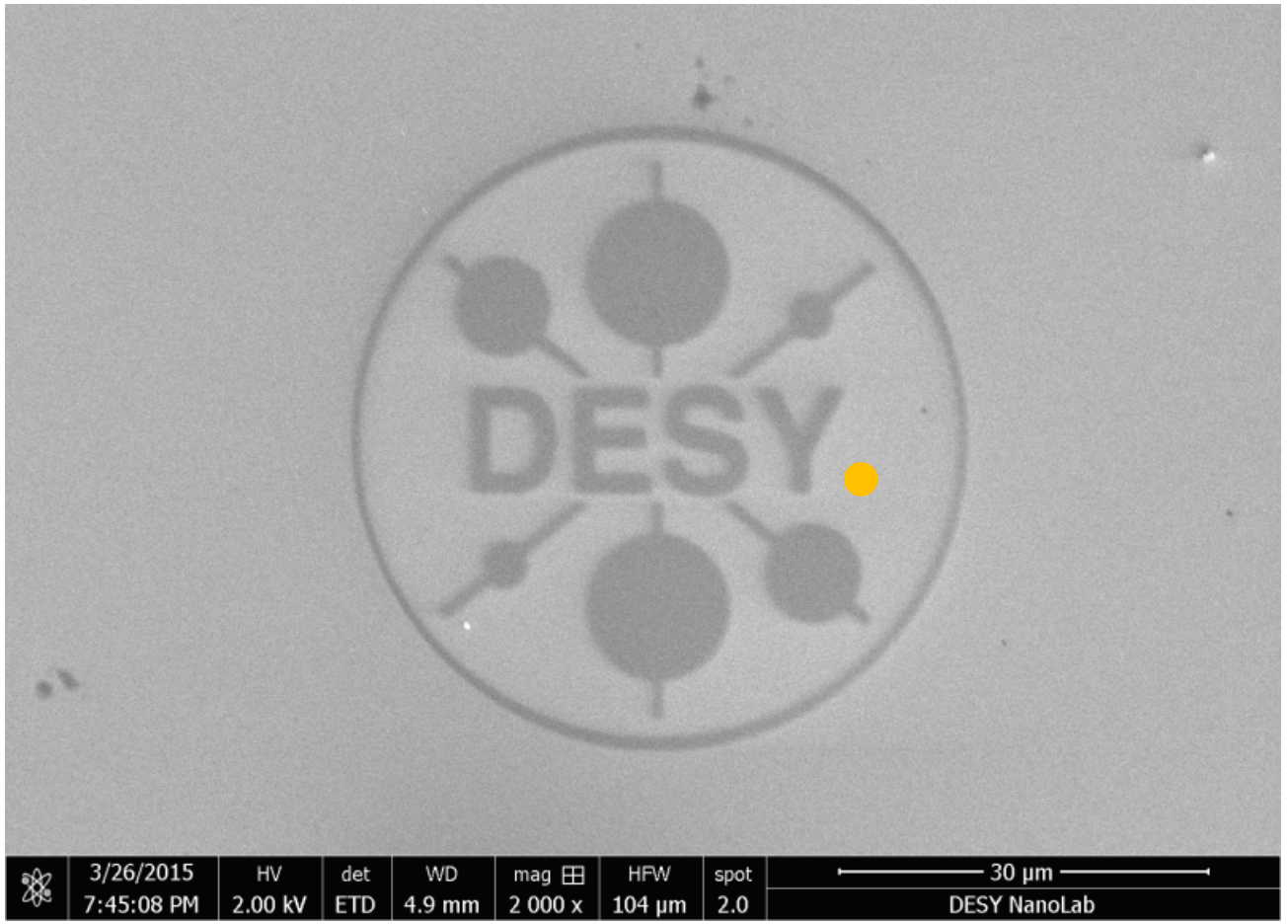
SEVENTH FRAMEWORK PROGRAMME

nffa.eu nanoscience foundries & fine analysis

Bundesministerium für Bildung und Forschung

PETRA III

Thank You for Your Attention



HELMHOLTZ ASSOCIATION

CNS DESY.