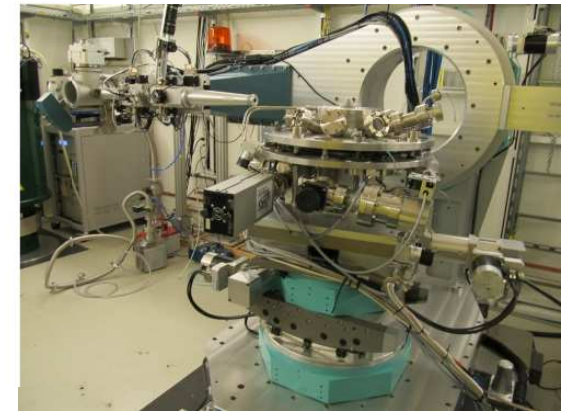
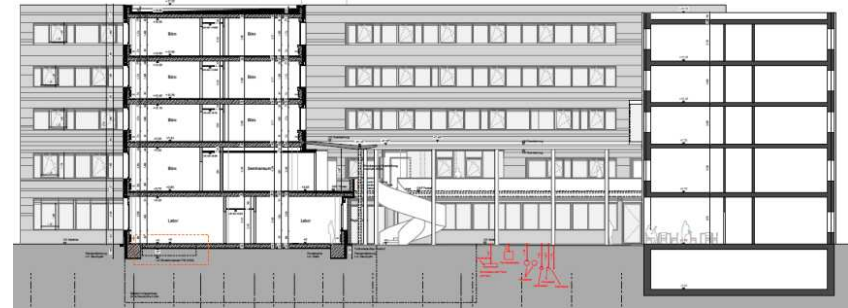
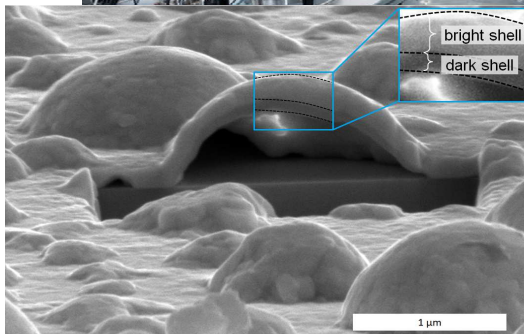


Management of Nanotechnology Laboratory as a RI



Andreas Stierle

DESY FS-NL and University of Hamburg (INF)



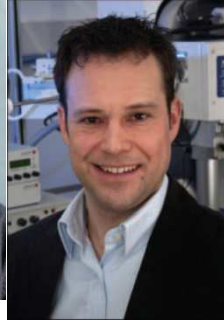
<http://nanolab.desy.de/>



The DESY NanoLab Team

Scientists

Thomas Keller
Vedran Vonk
Heshmat Noei
Andreas Stierle



Postdocs

Mike Wagstaffe
Simon Chung
Elin Granaes
Arti Dangwal-Pandey

Guests

Mikhail
Shipilin
(Stockholm
University)



PhD & Master students

Konstantin Krausert
Primin Lakner, Steffen Tober
Henning Runge
Guilherme dalla Lana
Björn Arndt Marcus Creutzburg
Robert Gleissner

Administrative & Technical support

Katharina Roeper
Simon Geile
Satish Kulkarni
Arno Jeromin
Dennis Renner



Mission of the DESY NanoLab

Providing on-site methods for nanoscience complementary to DESY photon techniques at PETRA III, FLASH and X-FEL especially for experiments with nano-focussed beams

Open for:

- external users in the framework of accepted proposals or “around” beamtimes
- Support of DESY and campus wide in-house research (collaborative / contributive level)
- European users in the framework of access program nano + x-rays NFFA EUROPE (Nano Foundaries and Fine Analysis)
- X-FEL users on collaborative level, full access depending on negotiations with X-FEL GmbH



DESY NanoLab Location



<http://desy.de/~videoweb/cxns/aktuell.html>

DESY Site – Campus Bahrenfeld



How to Access the DESY NanoLab

1. DESY NanoLab box in „DOOR“ for beamtime proposals

The screenshot shows a web browser window with the URL `application.php?FLAVOR=user&FACTYPE=SR&PRC`. The browser's address bar and tabs are visible. The main content area is a form titled "DESY NANOLAB (CONSULT DESY NANOLAB INSTRUMENTATION PAGE)". The form contains a table with two columns: a list of activities and a "Yes/No" response column. The activities listed are: "Access to DESY NanoLab", "Brief description of planned activities", "Microscopy: AFM (air)", "Microscopy: Dual-beam focused ion beam (FIB)", "Microscopy: High-resolution scanning electron microscope (SEM)", "Microscopy: Variable temperature UHV STM/AFM", "Surface spectroscopy: UHV-reflection absorption IR spectroscopy", "Surface spectroscopy: X-ray photoelectron spectroscopy (XPS)", "UHV Sample preparation", "X-ray diffraction: Reflectometer", and "X-ray diffraction: Six-circle diffractometer". The responses are: "Yes", "", "No", "No", "No", "No", "Yes", "No", "No", "No", and "No". Below the table is a section titled "OPTIONAL ADDITIONAL MESSAGE" with a text input field containing "10-15 user groups / PETRA III call, success rate 1:3". A note below the input field states "(*) these fields are mandatory." At the bottom of the form are two buttons: "MAIN" and "SAVE & CONTINUE".

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

OPTIONAL ADDITIONAL MESSAGE

10-15 user groups / PETRA III call, success rate 1:3

(*) these fields are mandatory.

MAIN SAVE & CONTINUE

How to Access the DESY NanoLab

2. European access project NFFA

The screenshot displays the nffa.eu website. At the top, there is a navigation bar with links for 'intranet', 'login/register', 'your wishlist', and 'submit your application'. Below this is the nffa.eu logo and a menu with categories: 'ABOUT the project', 'OFFER tools catalogue', 'APPLY guidelines', 'OUTCOMES the latest outcomes', and 'NEWS events & highlights'. The main content area features a large blue box on the left with the text: 'The widest range of tools for research at the nanoscale. Free of charge access for academia and industry thanks to Horizon 2020. BROWSE THE OFFER & APPLY FOR FREE ACCESS. Next deadline 16 July 2018 at 17:00 (CEST)'. To the right, under the heading 'the latest outcomes', there are several featured articles. One article, dated Jan 08, 2018, is titled 'Plasma etching and pyrolysis contribute to size reduction of laser polymerized 3D structures' and is categorized as 'FROM OUR JOINT RESEARCH'. Another article is titled 'Deep neural networks lead to nanoscience images classification software' and is also categorized as 'FROM OUR JOINT RESEARCH'. A third article is titled 'One step preparation of ZnFe₂O₄/Zn₅(OH)₆(CO₃)₂ nanocomposite with improved As(V) removal capacity' and is categorized as 'FROM OUR USERS'. At the bottom right, there is a section for 'INDUSTRY CORNER'.

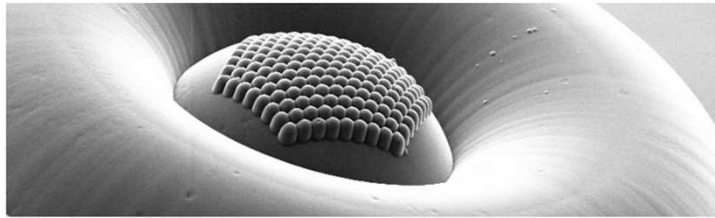
DESY NanoLab methods + beamtime at PETRA III
www.nffa.eu, questions: thomas.keller@desy.de



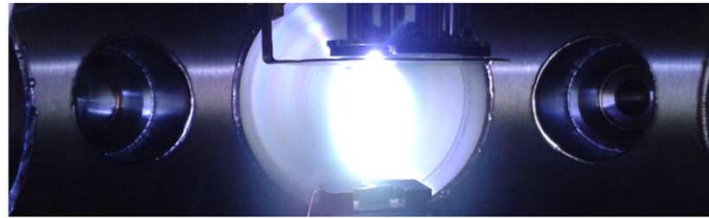
2. European access project NFFA

Regulated access to state of the art nanoscience facilities

Integrated with the large scale infrastructures for fine analysis



**LITHOGRAPHY &
PATTERNING**



**GROWTH &
SYNTHESIS**



**THEORY &
SIMULATION**

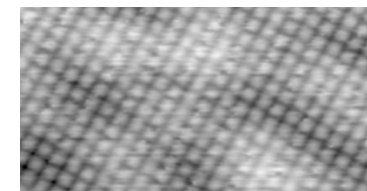
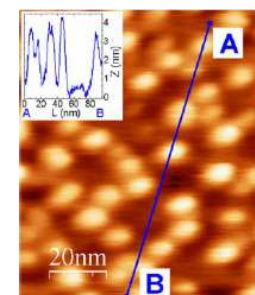
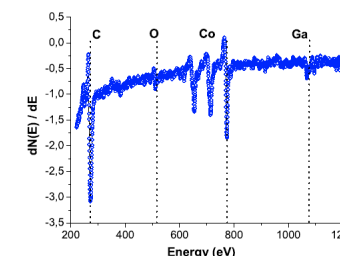
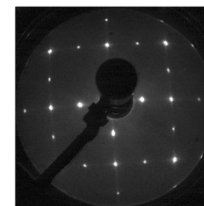


CHARACTERISATION

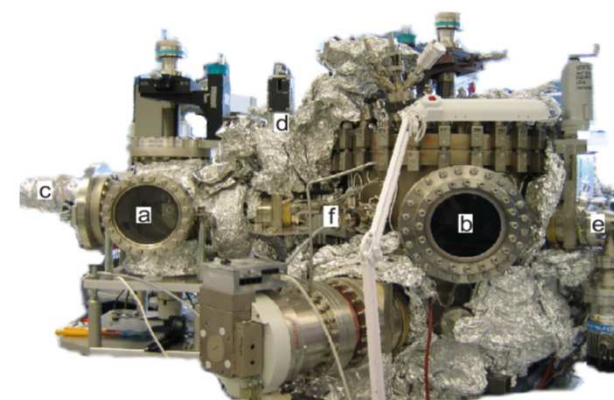
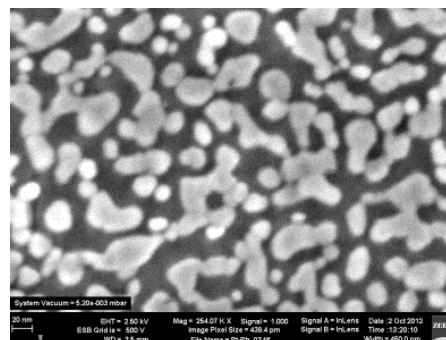
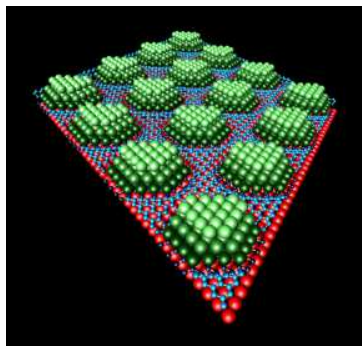
DESY NanoLab Strategy

Implementation of

- nano characterization techniques (atomic scale structure, chemistry and magnetism)
- nano structuring techniques
- nano synthesis techniques



Development of well defined sample transfer protocols between NanoLab and beamlines (nano-PS)



Overview DESY NanoLab Techniques

Spectroscopy & Growth (H. Noei)

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

X-ray diffraction (V. Vonk)

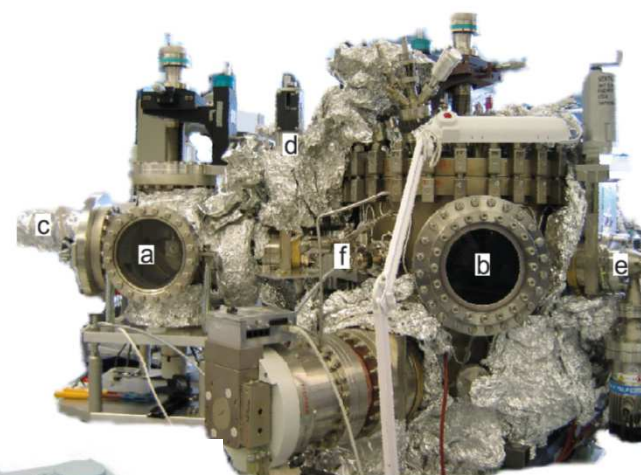
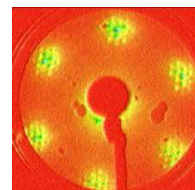
- Reflectometer
- Six circle diffractometer

Microscopy & Structuring (T. Keller)

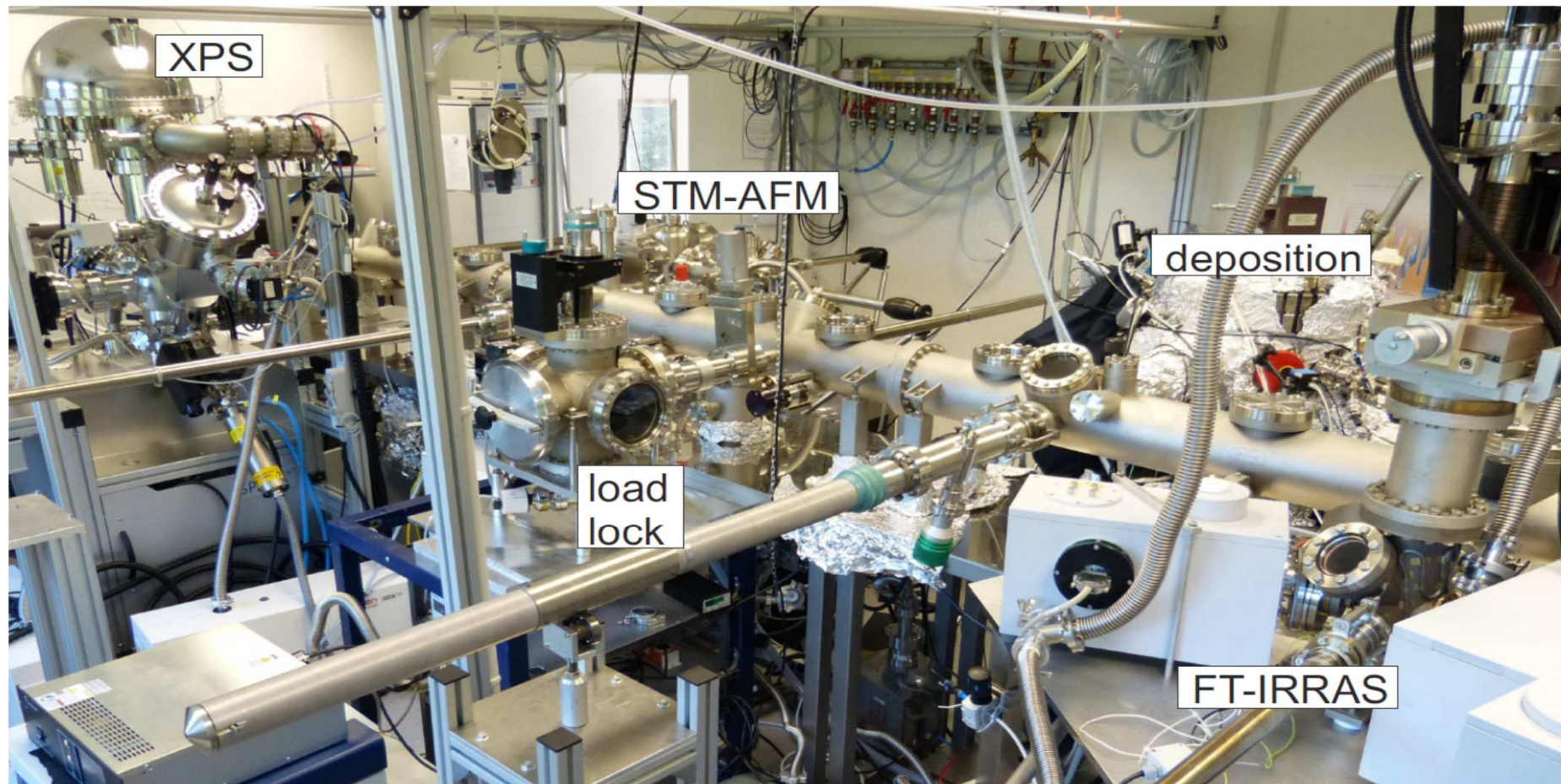
- AFM, STM, optical
- SEM + FIB + Lithography (future)

Magnetic Characterization (R. Röhlberger)

- Physical properties measurement system (CHyN)
- Kerr Microscope (future)



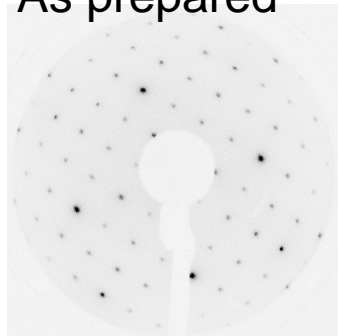
UHV Lab: Structure, Composition and Growth



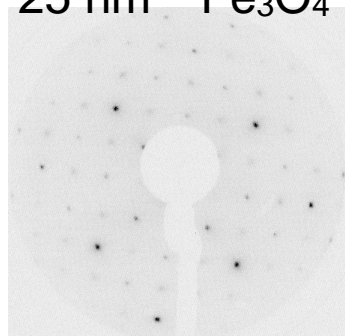
Multi method UHV laboratory

UHV Lab: Structure, Composition and Growth

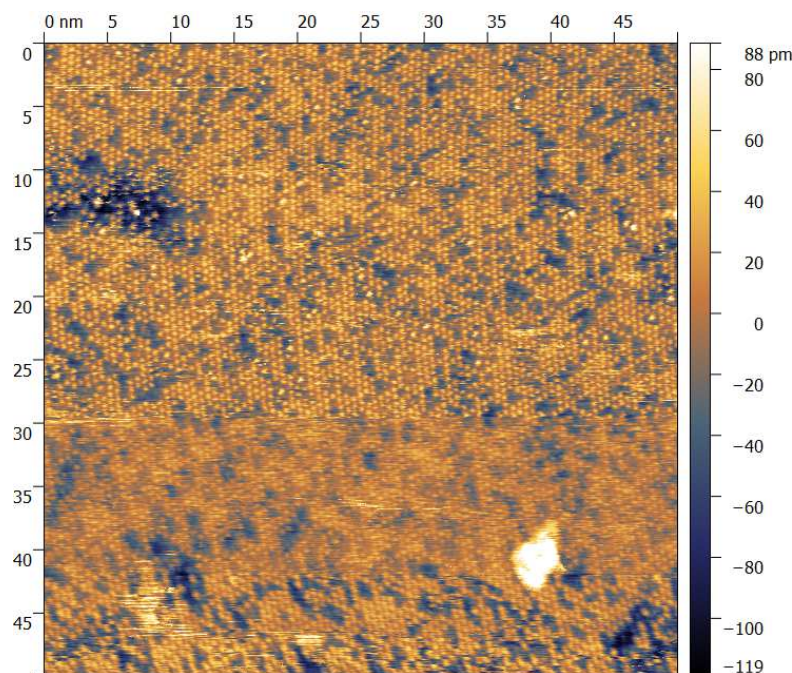
As prepared



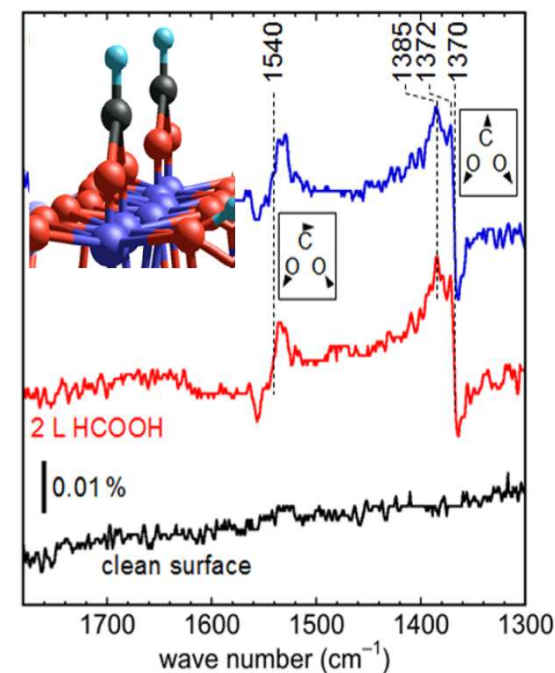
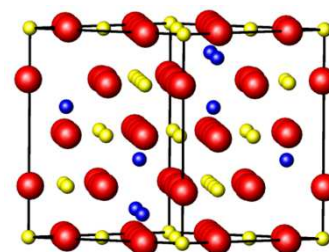
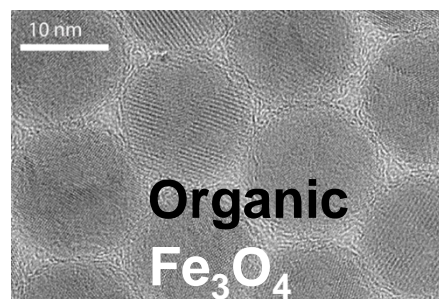
25 nm $^{57}\text{Fe}_3\text{O}_4$



LEED-patterns before and after growth of 25 nm $^{57}\text{Fe}_3\text{O}_4$ (S. Tober)



FT-IR spectroscopy of formic acid on magnetite (001), XPS: oxidation state



O. Gamba, H. Noei, A. Stierle, *et al.*,
J. Phys. Chem. C 119, 20459 (2015)
A. Dreyer, ..., H. Noei, A. Stierle, *et al.*,
Nature Materials 15 522-528 (2016)

STM on magnetite (111)
M. Creutzburg, E. Granäs,
SFB 986, TU HH



X-ray diffraction & Sample Environments

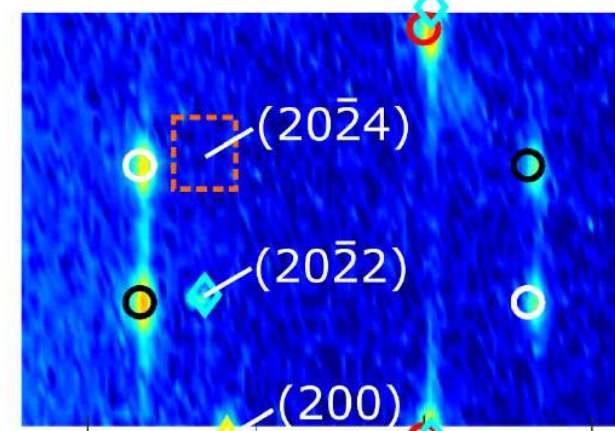
Reflectometer: *Roughness, film thickness*

- **Source:** Mo-K α
- Spot size: 12x0.3 mm² (h \times v)
- Divergence: 0.4 mrad² (v)
- **Type:** θ -2 θ scans
- Control software: SPEC
- Detectors: Point detector, Strip detector (Mythen)



Diffractometer: *Crystallinity, crystal phases and orientations*

- **Source:** Cu-K α
- Spot size: 0.25x0.25 mm² (h \times v)
- Divergence: 5x5 mrad² (h \times v)
- **Type:** full reciprocal space access
- Control software: SPEC
- Detectors: Point detector or Strip detector (Mythen)

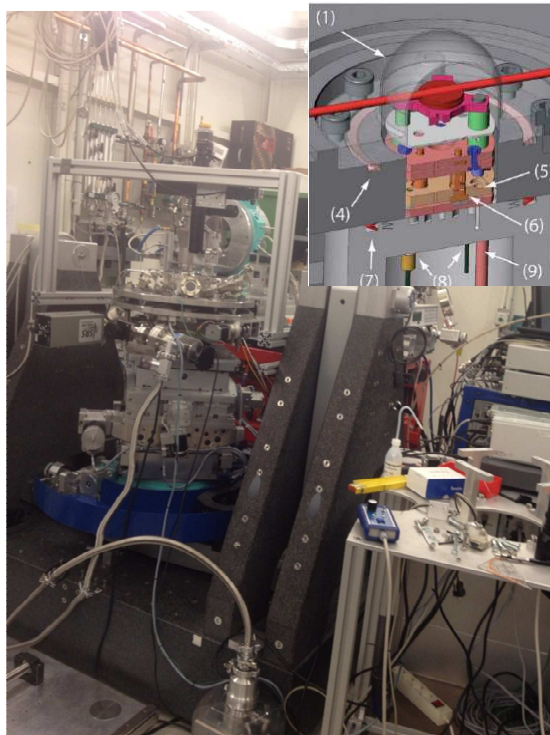


Andreas Stiene DESY Hamburg 2010 Page 12
2.6 nm high Pt NPs / Al₂O₃

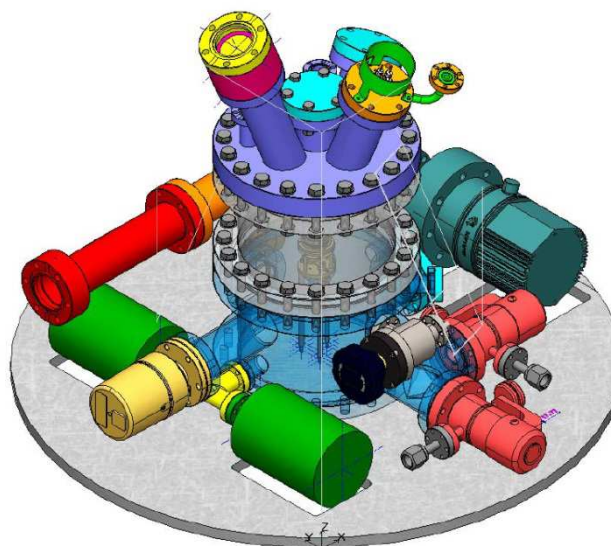
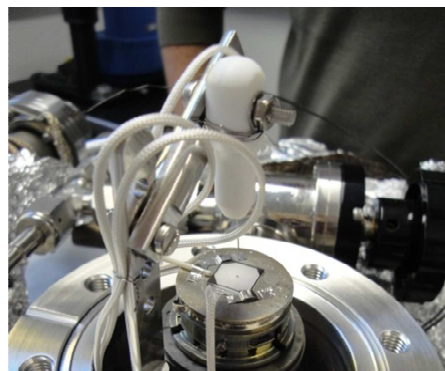
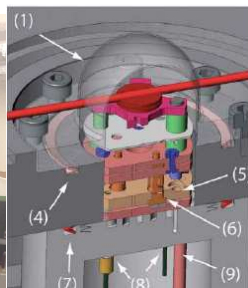


X-ray diffraction & Sample Environments

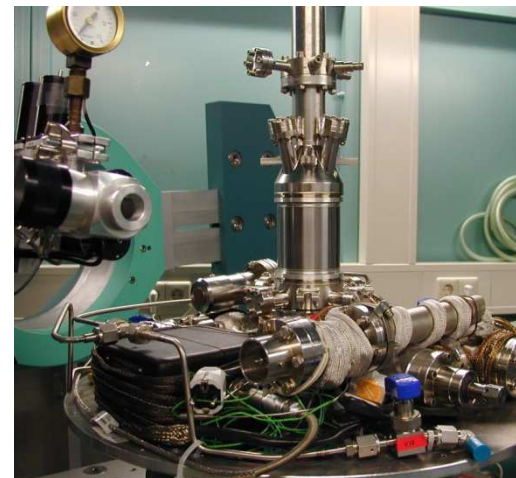
Mobile in-situ X-ray Diffraction Sample Environments



In-situ UHV / flow reaction chamber (BMBF NanoXcat) for catalysis



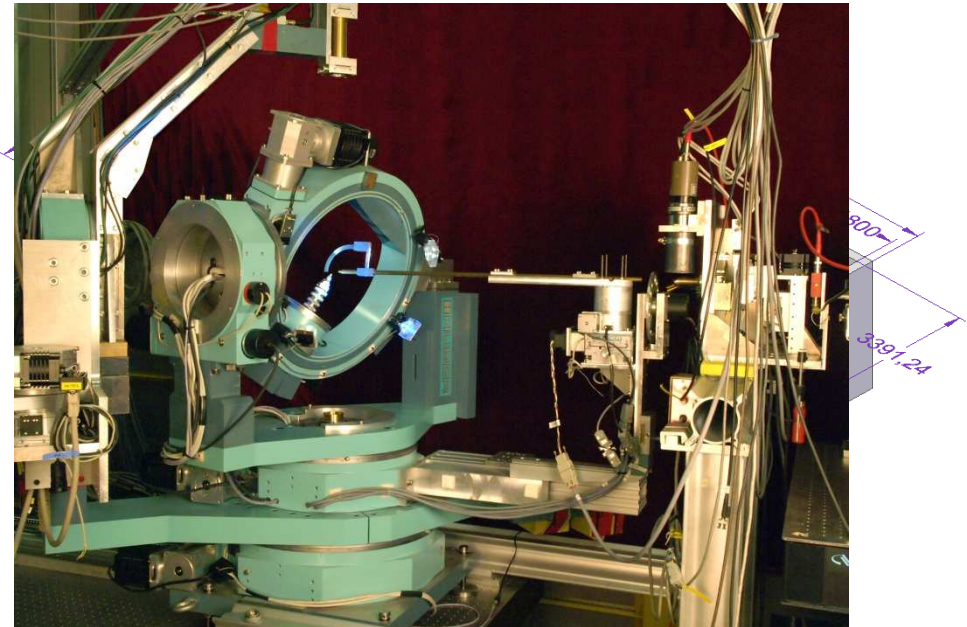
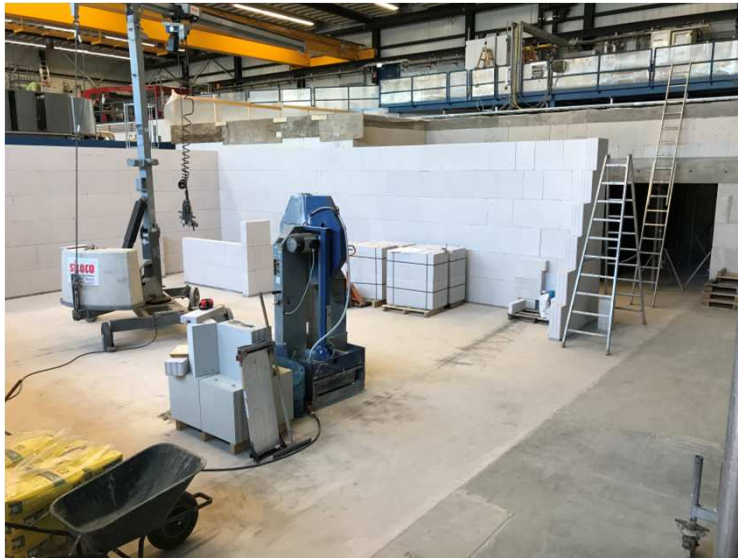
In-situ solid state electrochemistry Chamber, EC cells



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

X-ray Diffraction & Sample Environments

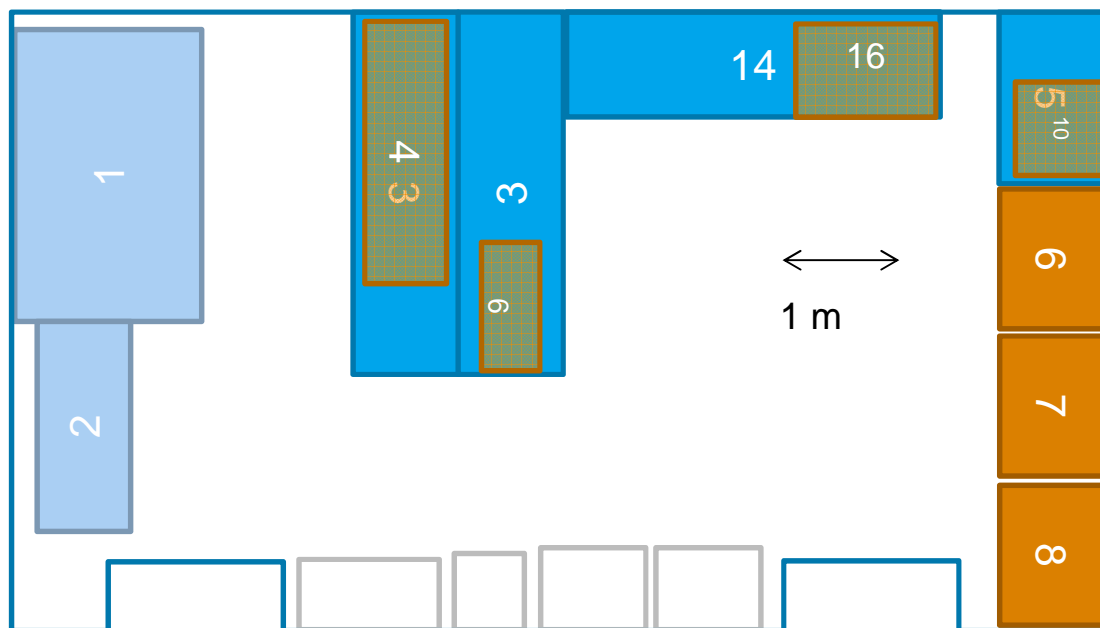
Future location: DORIS hall building 25



1. Reflectometer
2. Six circle diffractometer (z axis style) (GIXRD, SXRD)
3. Six axis diffractometer (with Eulerian cradle), old Weckert instrument (GISAXS, GIXRD)

X-ray Diffraction & Sample Environments

Future Electrochemistry laboratory in building 25b



In cooperation with CAU
Kiel, Ruprecht Haensel
Labor

- Vakuumsysteme
- Labormöbel
- Abzüge
- Geräte auf Laborbänken
- Lager

1. UHV System (Präparationskammer, Transfersystem, Rack) 2.6 m x 1.6 m (4.0 m²) / Nanolab
2. Glovebox Elektrochemie 1.8 m x 0.8 m (1.5 m²) / Nanolab
3. Langmuir Trog (Filmwaage) in Einhausung + Elektronik 3.0 m x 0.9 m / CAU Kiel
4. HF Abzug mit Spüle und Neutralisationsanlage im Unterschrank 1.2 m x 0.9 m
5. Abzug anorganische Chemie (Lager im Unterschrank) 1.2 m x 0.9 m
6. Abzug organische Chemie (Abfälle im Unterschrank) 1.2 m x 0.9 m
7. Laminar flow bench 1.1 m x 0.5 m / CAU Kiel
8. Reinstwasser Anlage 0.8 m x 0.8 m
9. Kühl-Gefrier Kombination Labor 0.6 m x 0.6 m
10. Laborbank 3.2 m x 0.9 m
11. Induktionsofen 1.2 m x 0.8 m / CAU Kiel

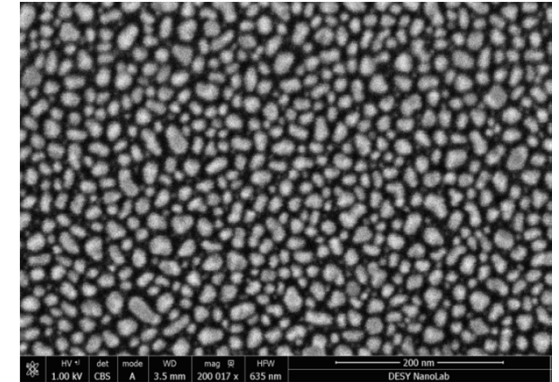
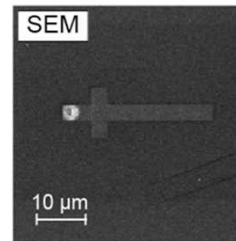


Microscopy & Structuring



Nova Nano SEM 450, FEI

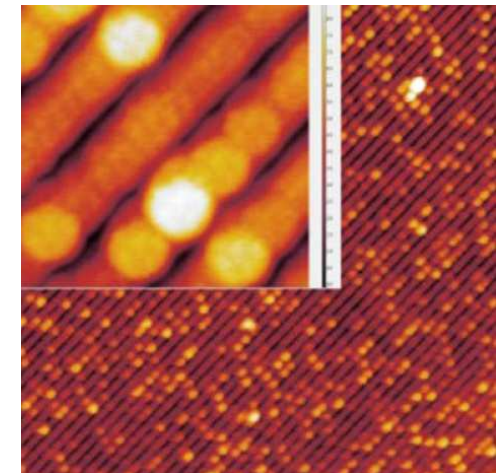
- Highest lateral resolution
1.0 nm @ 15 kV, 1.4 nm @ 1 kV
- Operation at 30 kV - 200 V
for anti-charging: deceleration mode
- Multi detection mode
- EDX
- Pt marker deposition



Dimension Icon AFM

Major operating modes:

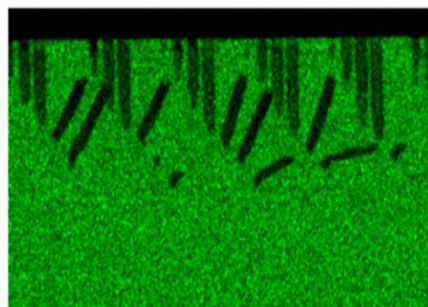
- tapping / contact mode
- STM mode
- Force spectroscopy
- Lateral force microscopy (LFM)
- Magnetic force microscopy (MFM)
- KPFM module (Kelvin probe force microscopy)
- Liquid cell
- Electrochemical cell with potentiostat



SEM and EDX application: Filling of porous materials



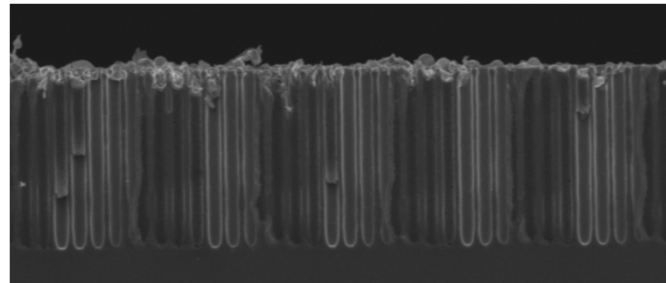
Porous silicon filled with
Semiconducting polymer



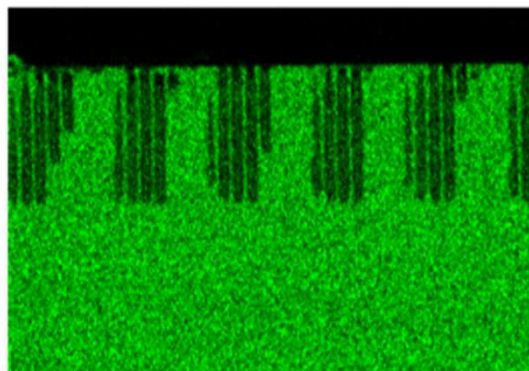
Si K α 1

Energy-dispersive spectroscopy

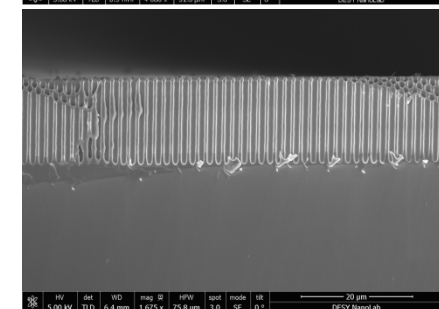
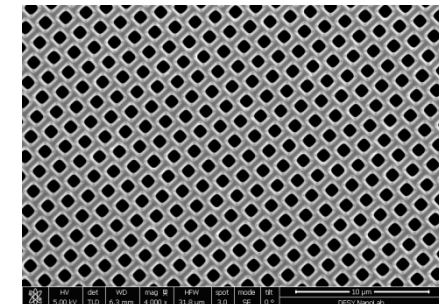
*Surface topography
and chemical structure*



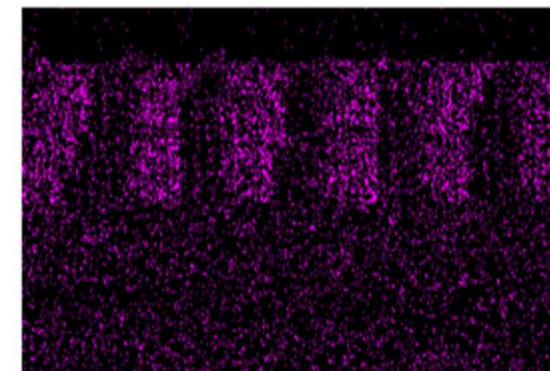
Si K α 1



10 μm



N K α 1_2



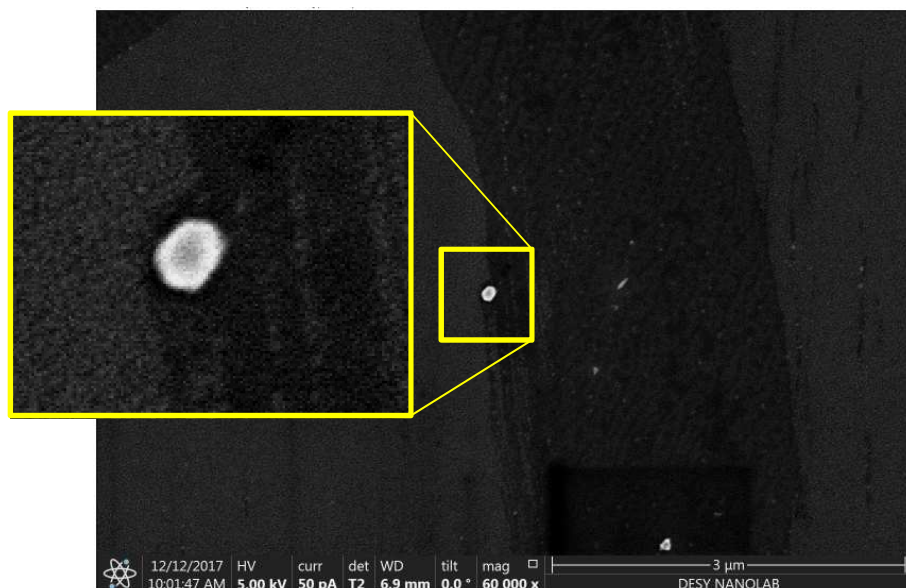
10 μm



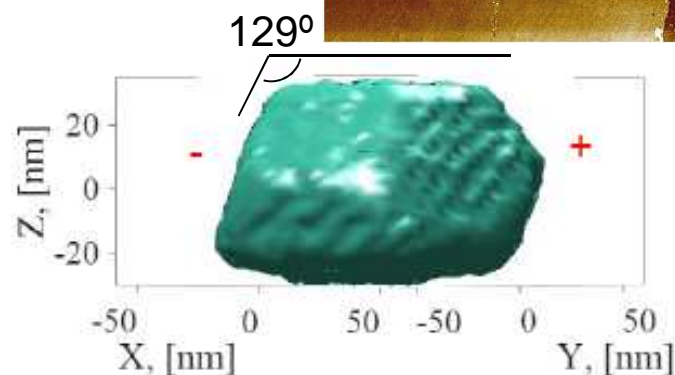
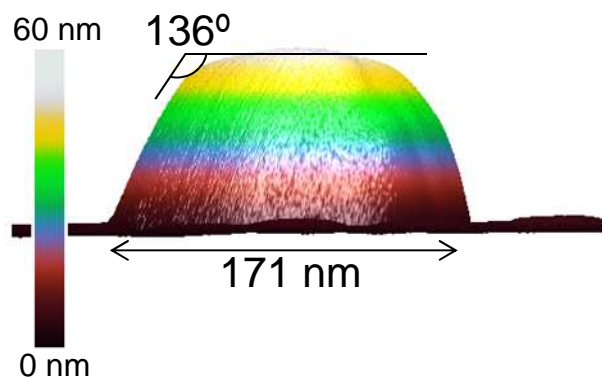
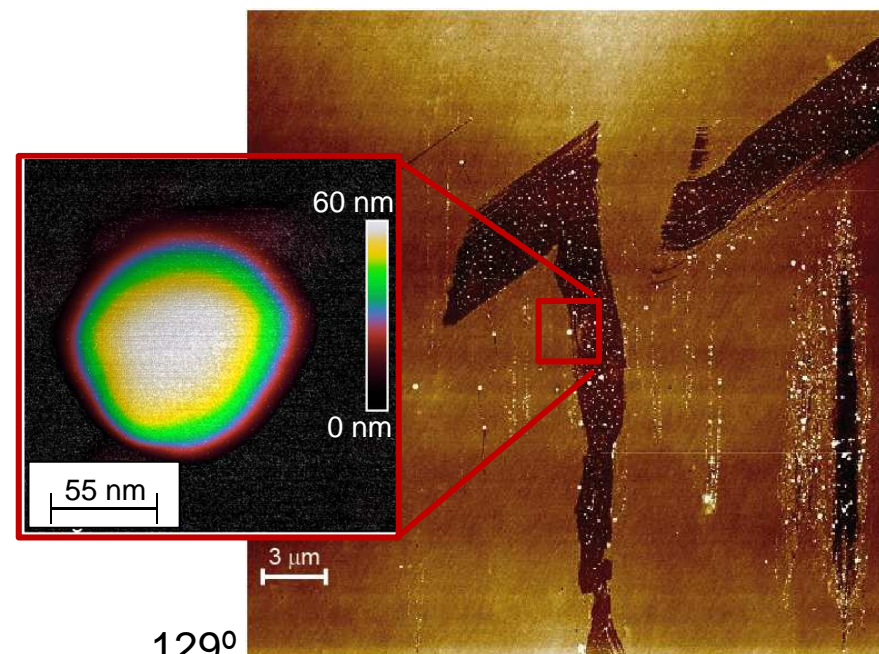
Microscopy & Structuring

Nano transfer & positioning of a PtRh catalyst nanoparticle

SEM image:



AFM image:



Bragg CDI
reconstruction
under NAP
CO oxidation
conditions

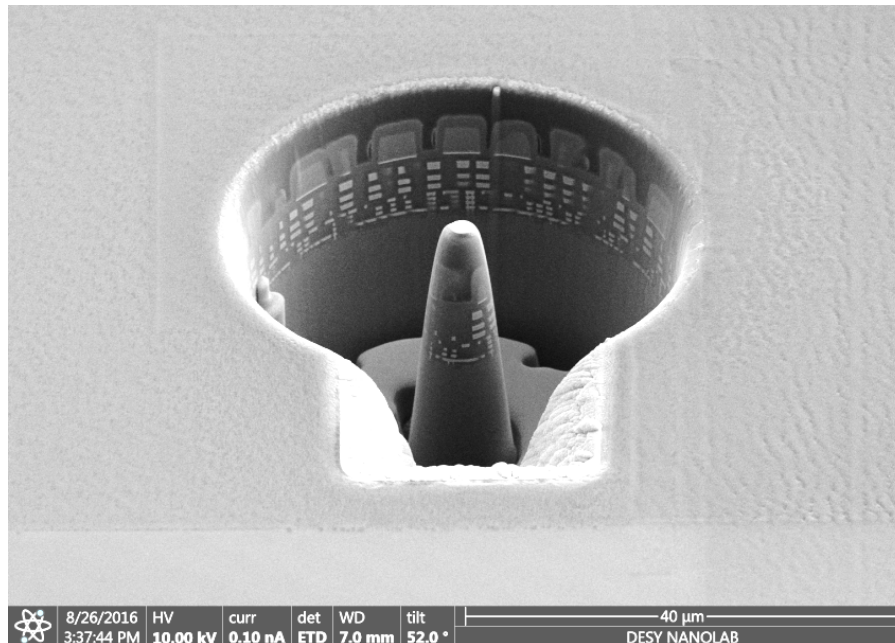


M. Abuín, S. Kulkarni, T. Keller, cooperation with I. Vartanians group

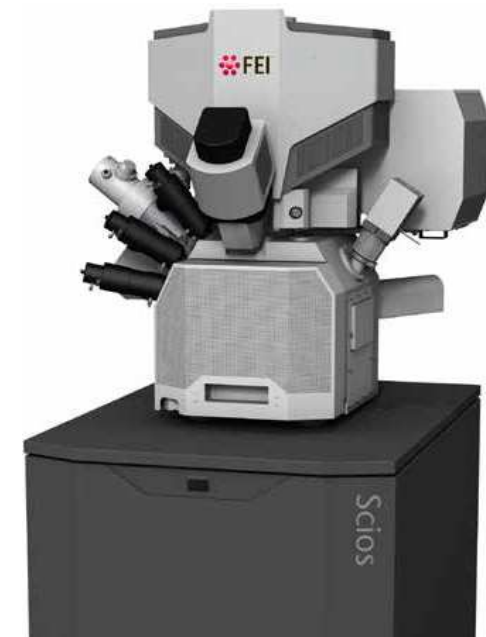
Microscopy & Structuring

FIB - Dual electron and Ga focused ion beam

- cross section sample preparation
- 3D tomographic imaging (IC structure, Ch. Schroer group)
- gas dosing system for marker fabrication and manipulation
- EDX analysis



**(future) E-beam lithography in collaboration
with University of Hamburg (CHYN)**



courtesy of FEI Deutschland GmbH

Funded by BMBF /
University of Bayreuth
Collaboration with
L. Dubrovinsky and P02
diamond anvil HP cell
preparation

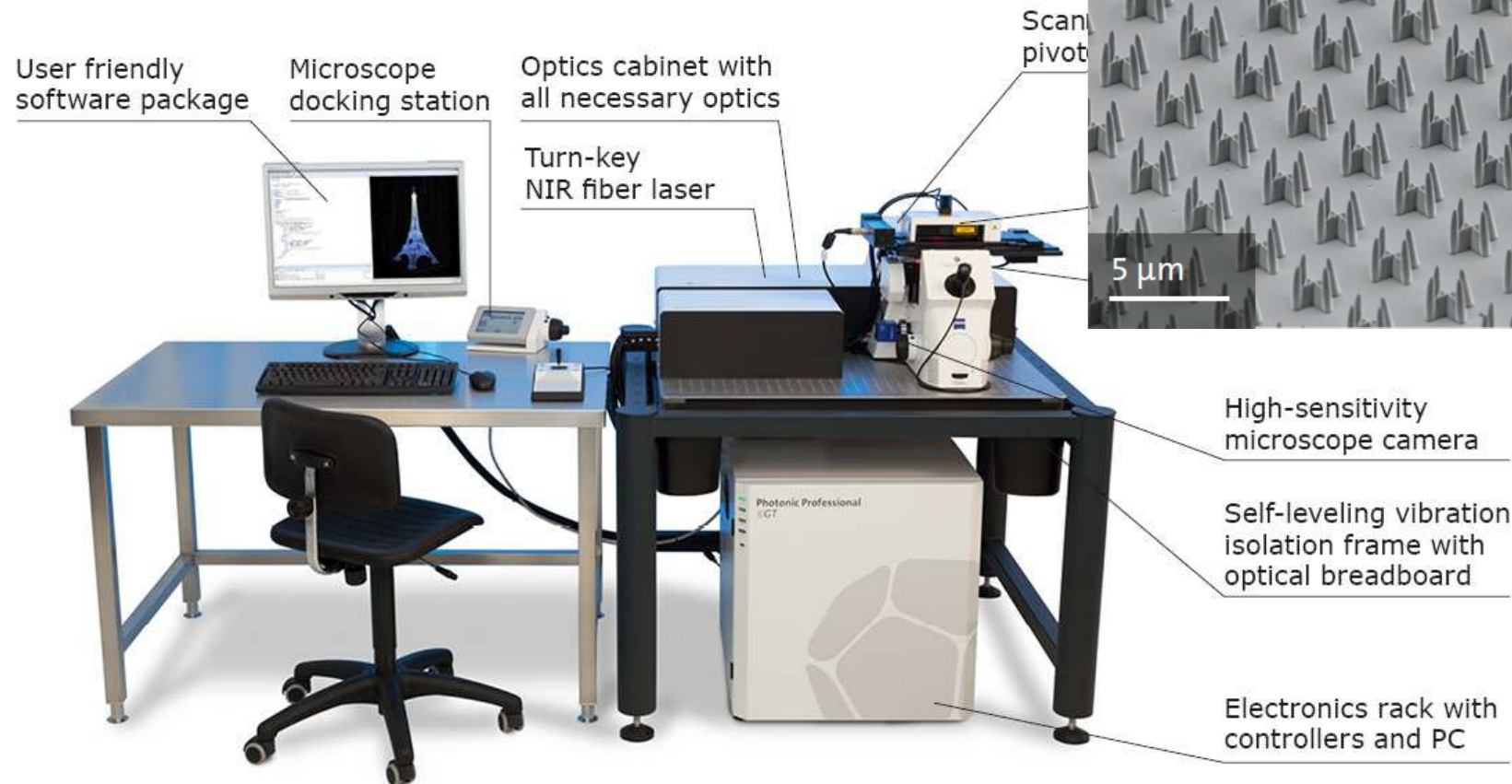


Microscopy & Structuring

Future instruments

Scanning Auger microscope

High-resolution 3D printer (150 nm smallest features)

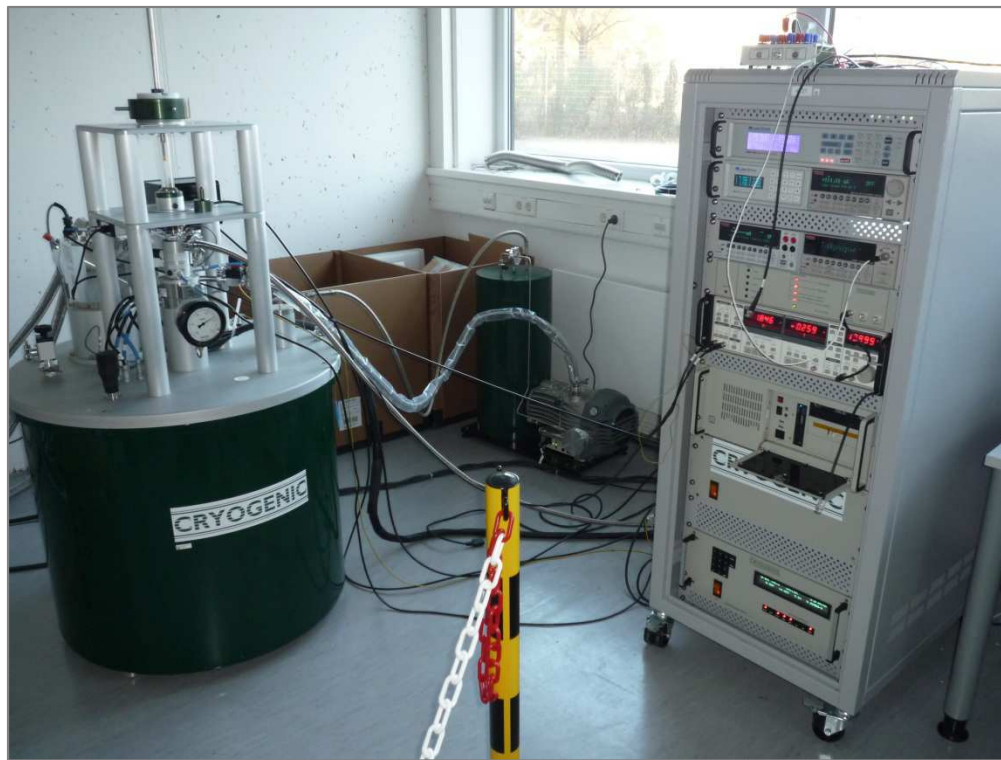


Courtesy of Prof. Mu Wang, Nanjing University, China



Magnetic Characterization and Transport

CFMS - cryogen free measurement system by Cryogenic Ltd,



Cryogen free magnet:

- Field range ± 14 T, 20 bit resolution
- 4-quadrant low field option ± 38 mT

Integrated sample cryostat:

- Temperature range: 1.6 – 400 K

Measurement options:

- vibrating sample magnetometry
- AC susceptibility
- AC and DC resistivity and Hall effect
- AC calorimetry
- thermal transport

Responsible: cornelius.strohm@desy.de

Future instrumentation: Kerr microscope

Thank You for Your Attention

