BORESKOV INSTITUTE OF CATALYSIS

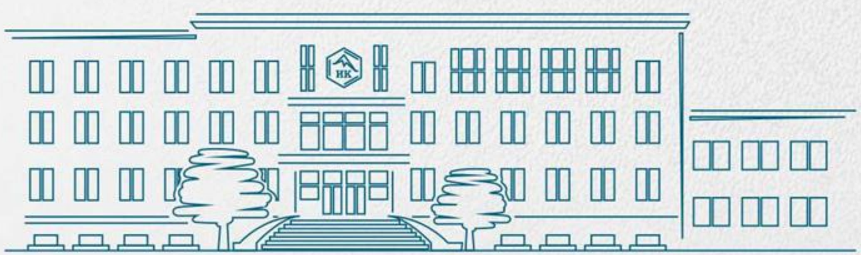
SFR-2020

СКИФ

“Electronic Structure” beamline 1-6 at SKIF synchrotron facility.

A.V. Bukhtiyarov, A.D. Nikolenko, I.P. Prosvirin,

R.I. Kvon, O.E. Tereshchenko



Boreskov Institute of Catalysis
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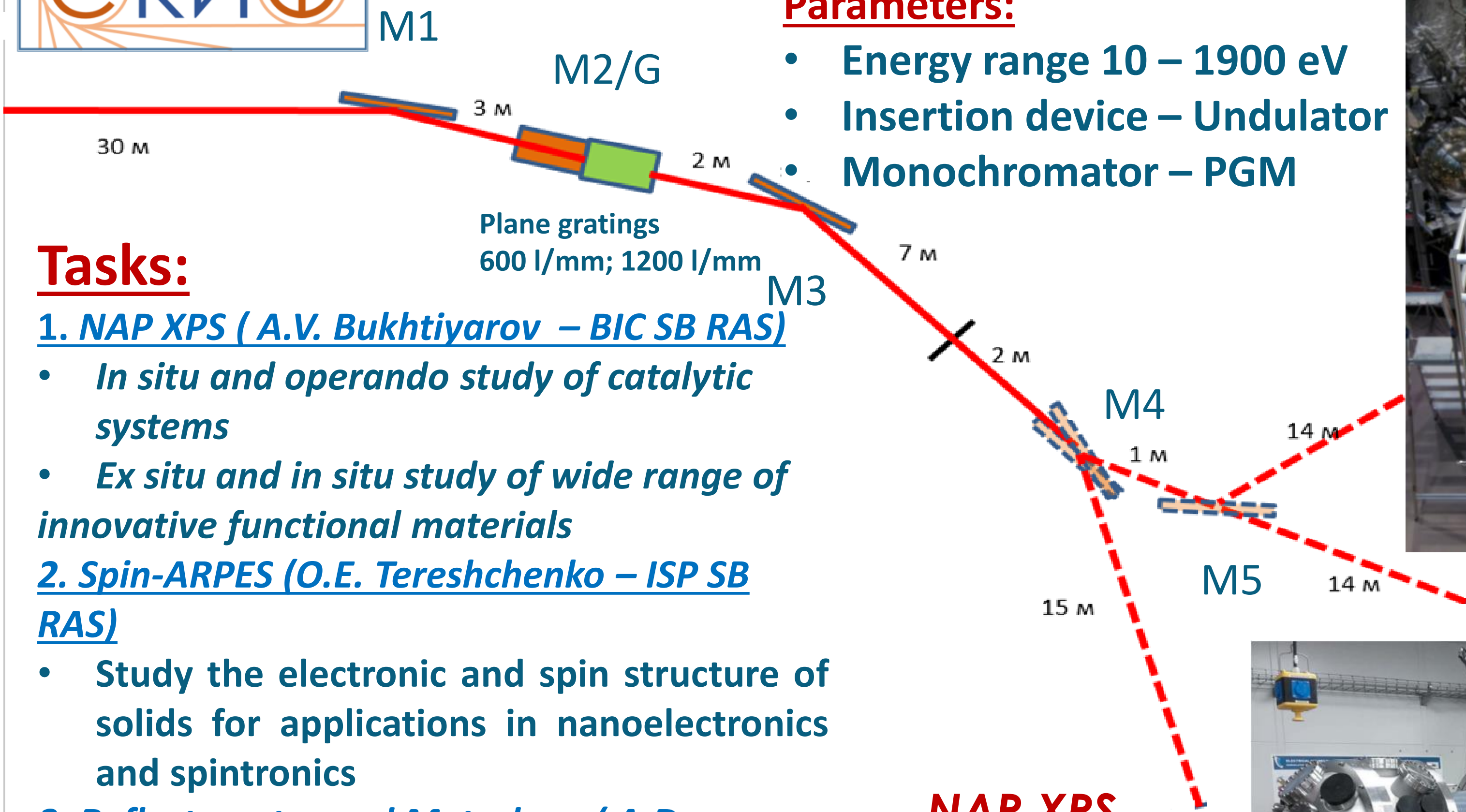
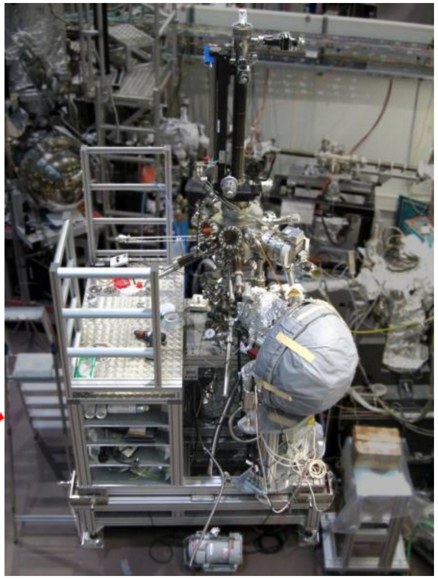
“Electronic Structure” beamline



Parameters:

- Energy range 10 – 1900 eV
- Insertion device – Undulator
- Monochromator – PGM

Spin-ARPES



Tasks:

1. NAP XPS (A.V. Bukhtiyarov – BIC SB RAS)

- *In situ and operando study of catalytic systems*
- *Ex situ and in situ study of wide range of innovative functional materials*

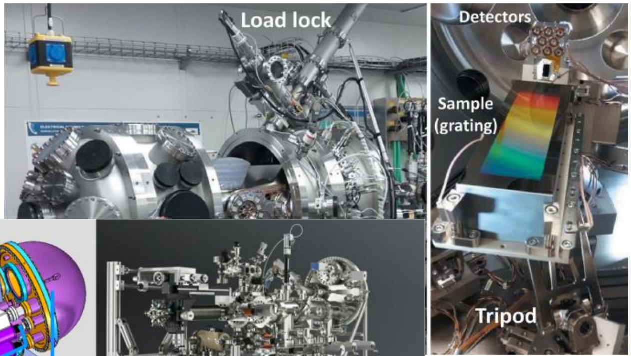
2. Spin-ARPES (O.E. Tereshchenko – ISP SB RAS)

- Study the electronic and spin structure of solids for applications in nanoelectronics and spintronics

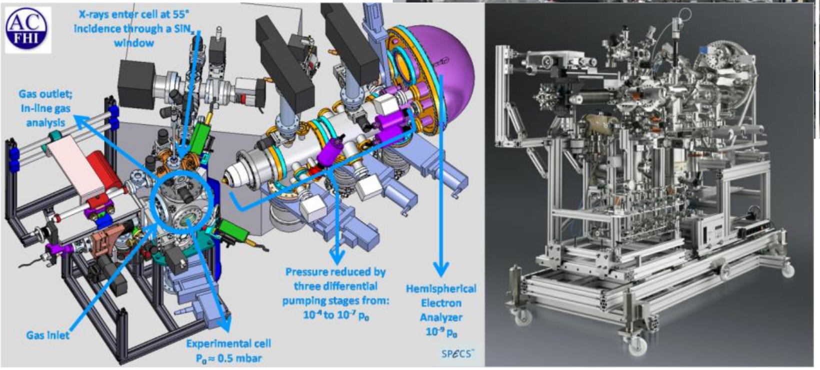
3. Reflectometry and Metrology (A.D. Nikolenko – Budker INP SB RAS)

- certification of spectral optical elements, focusing elements and X-ray detectors
- reference detector method for absolute calibration of the spectral sensitivity of various kinds of X-ray radiation receivers in the VUV and Soft X-Rays ranges

Reflectometer



NAP XPS



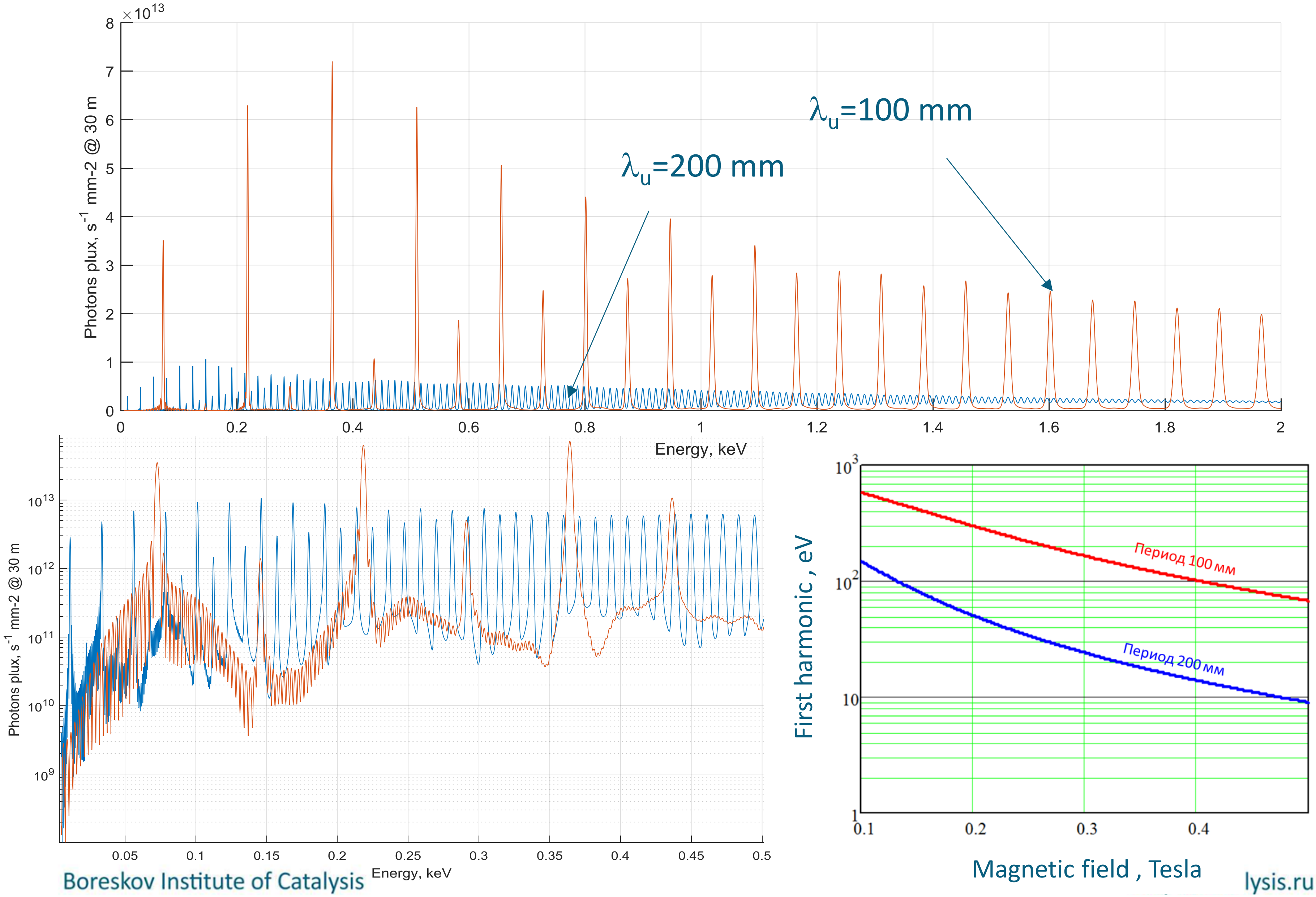
Undulator

The beamline is based on the use of an electromagnetic undulator with ability to change the period. It should allow to generate the synchrotron radiation in VUV and Soft X-Ray range (10-2000 eV).

The main parameters	Undulator	Wiggler
Period, mm	100	200
The number of periods	26	13
The total amount of poles	56	56
Total length, mm	2900	2900
Max magnetic field, Tesla	0.5	0.5
Min magnetic field, Tesla	0.05	0.05
Max current, A	270	250
Max power consumption, kW	20	17
Max phase error	2^o	2^o
First harmonic (Lowest photon energy), eV	67.6	9
Emitted power in aperture 6x10 mm (27 m)	227 Вт	54 Вт

More details @ poster section from Mr Anatoly Utkin
Poster Session: SR and FEL sources and centers #112

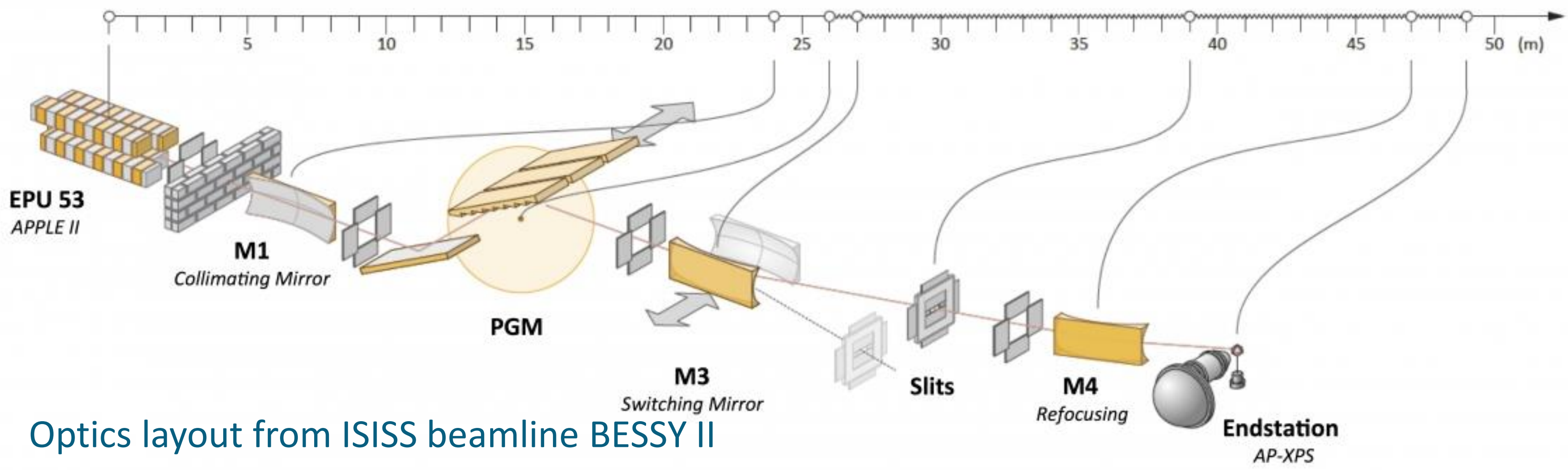
Undulator



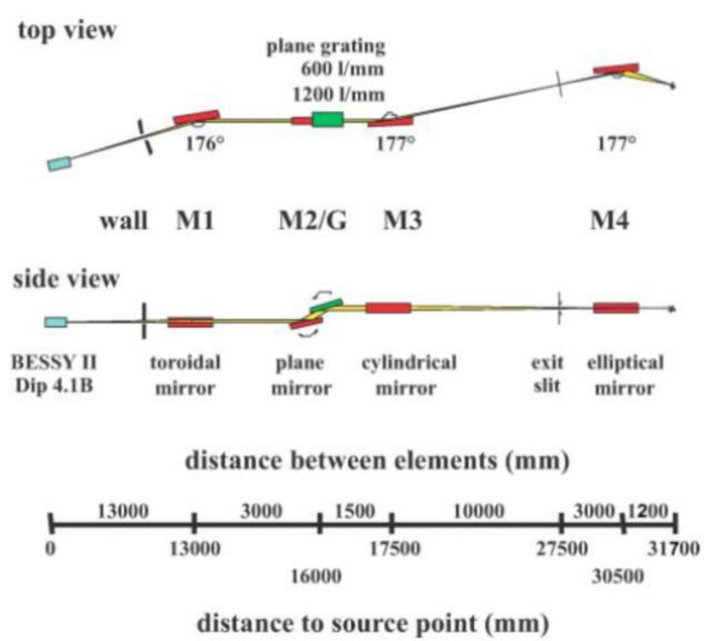
×10^13
Photons plux, s^-1 mm-2 @ 30 m
λu=100 mm
λu=200 mm
Energy, keV
Photons plux, s^-1 mm-2 @ 30 m
Energy, keV
First harmonic , eV
Период 100 мм
Период 200 мм
Magnetic field , Tesla

Optics layout of beamline: Background

Beamline outline of the HIPPIE beamline @ MAX IV



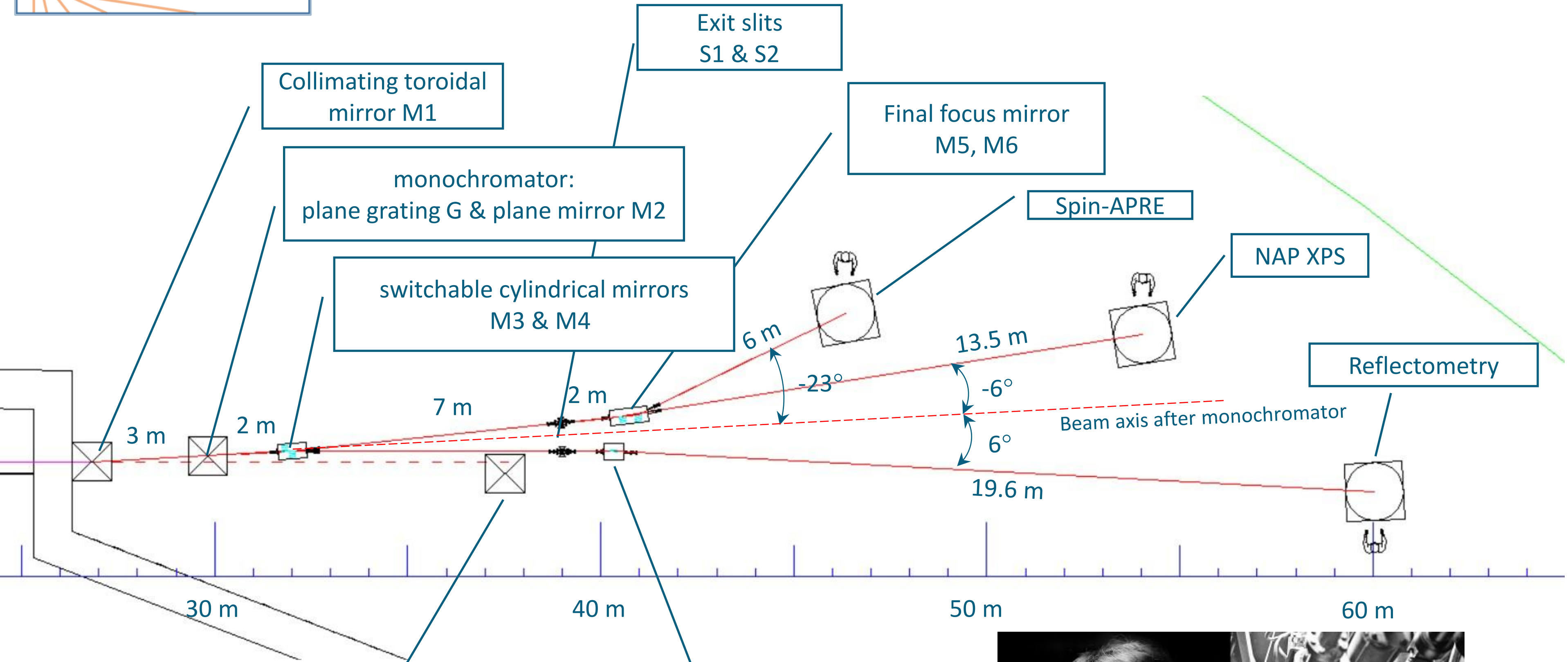
Optics layout from ISISS beamline BESSY II



Usually, to implement methods where it is necessary to use the Soft X-ray range (15–2000 eV) the optical scheme is based on a monochromator with a plane grating and focusing mirrors.

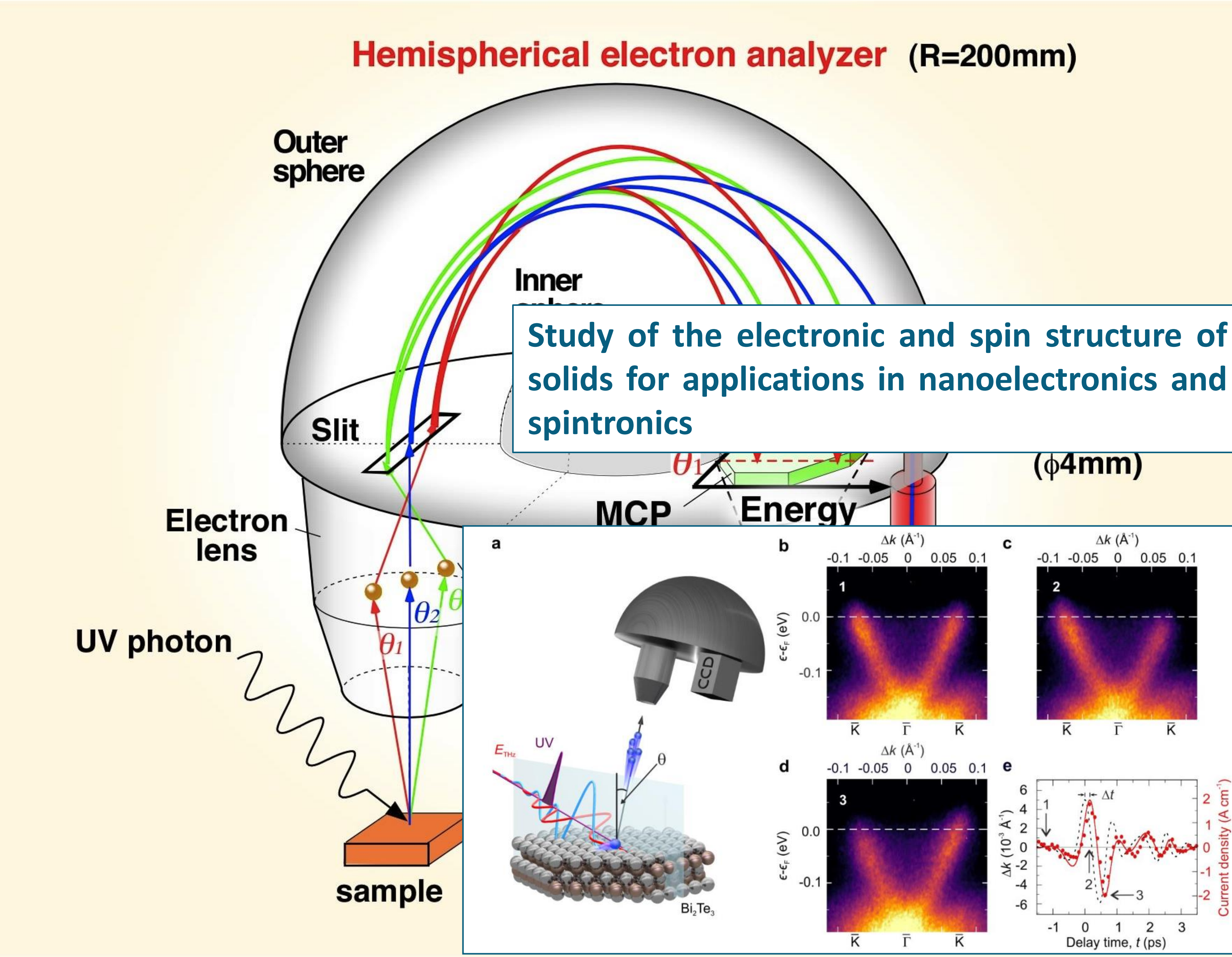
The use of this approach is well established and widely used on many Soft X-ray beamlines at different synchrotron facilities.

Optics layout of beamline



More details @ poster section from Dr. A. D. Nikolenko
Poster Session: SR technological application and X-ray apparatus #16

Spin-ARPES

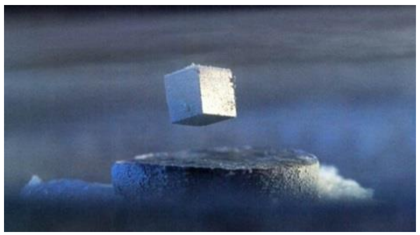


Study of the electronic and spin structure of solids for applications in nanoelectronics and spintronics

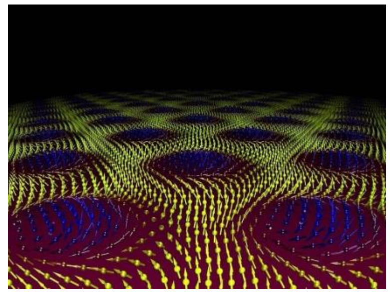
Nature **562**, p.396 (2018).

Spin- and Angle-resolved photoemission spectroscopy (SARPES) allows to study

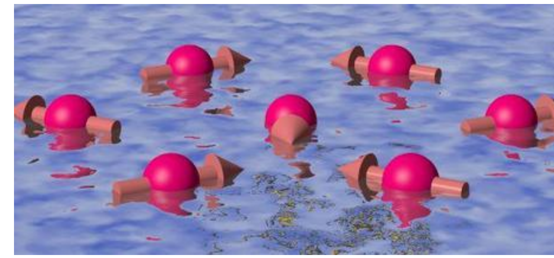
Many phenomena in quantum materials



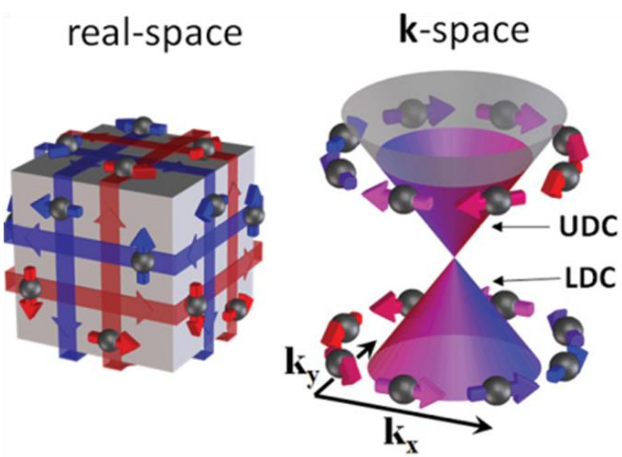
Superconductivity



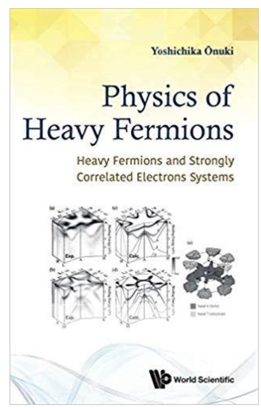
Various types of magnetic order



Quantum spin liquids



Topological Insulators

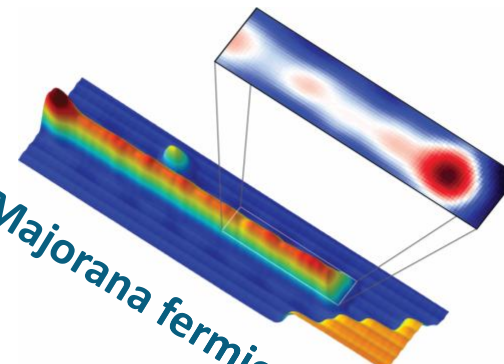

Yoshichika Ōnuki

Physics of Heavy Fermions

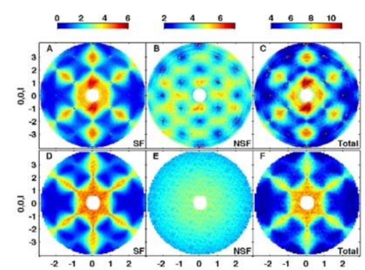
Heavy Fermions and Strongly Correlated Electrons Systems

World Scientific

Light-like electrons

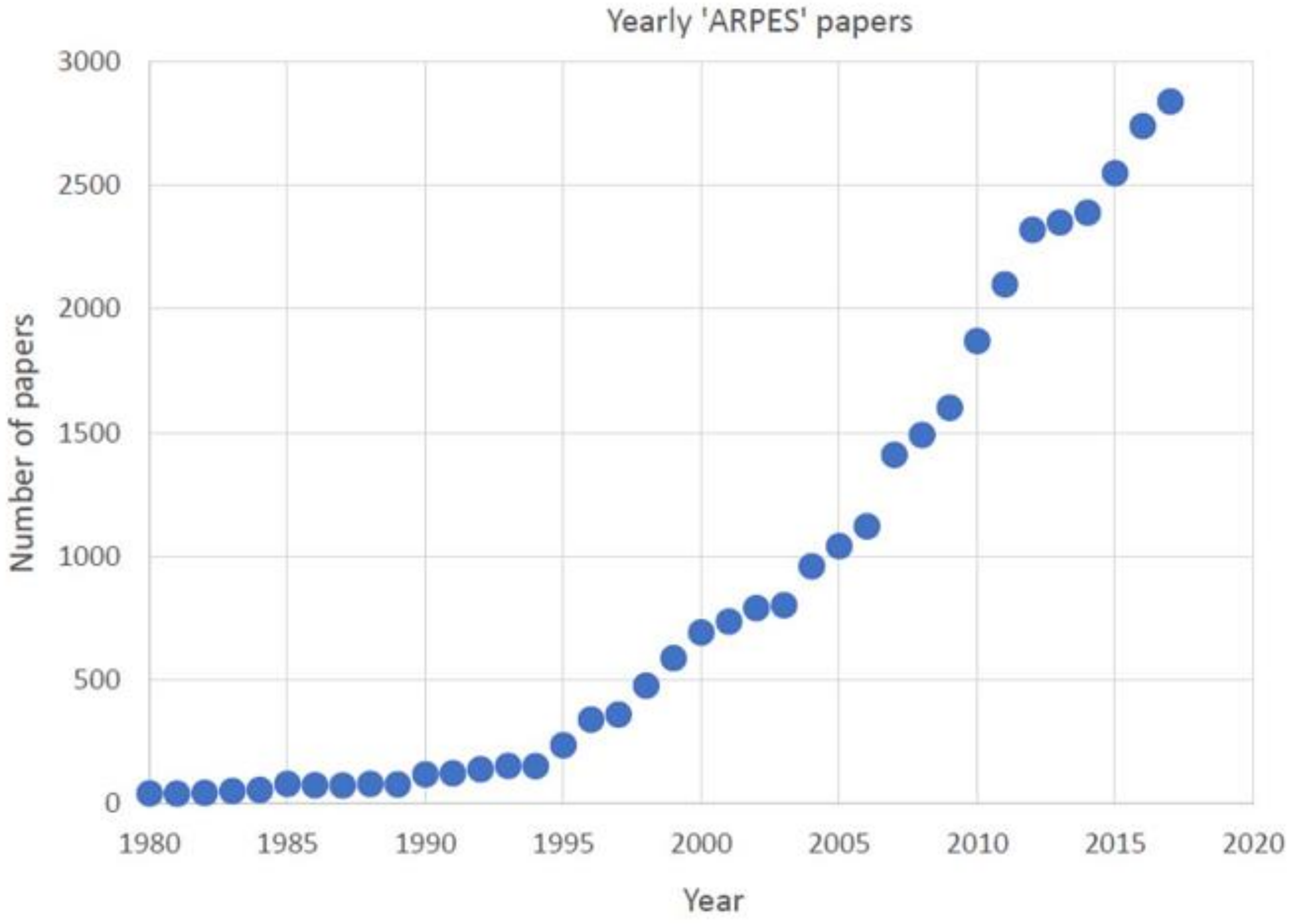


Majorana fermions



Magnetic monopoles

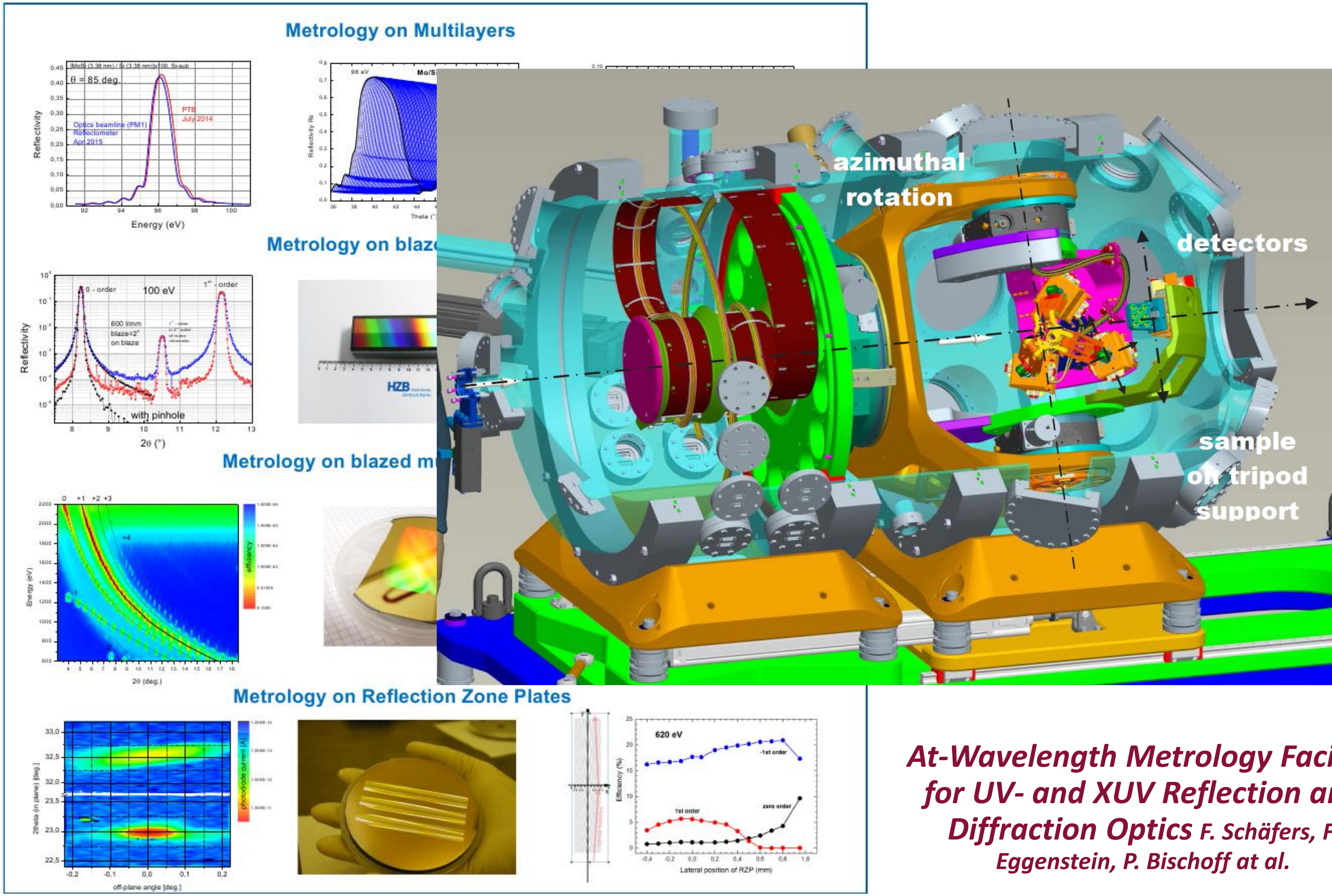
A growing experimental technique



UV- and XUV Reflectometer

Reflectometry allow certification of spectral optical elements, focusing elements and X-ray detectors

Reflectometry Station @ BESSY II (Berlin, Germany)

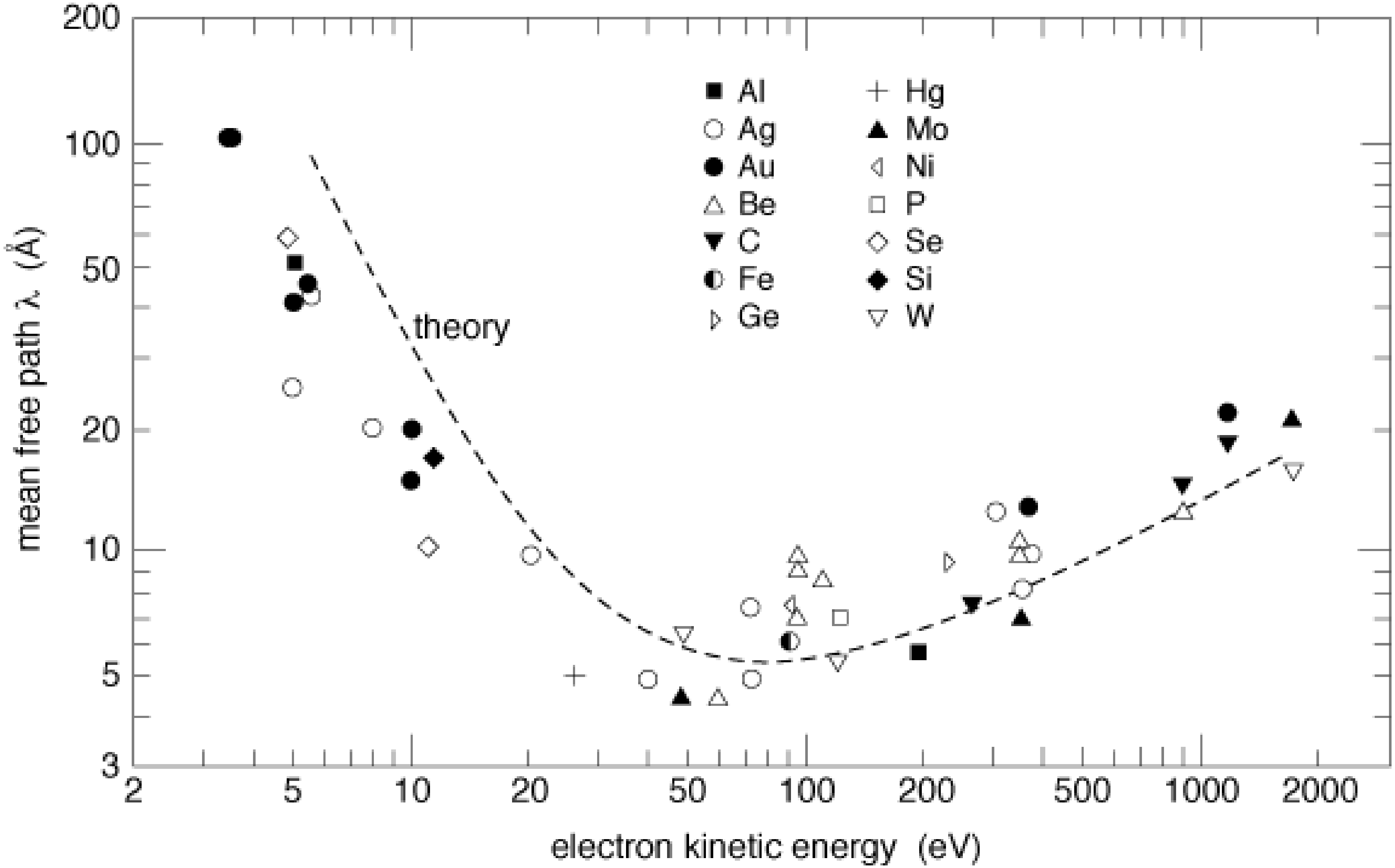


***At-Wavelength Metrology Facility for UV- and XUV Reflection and Diffraction Optics** F. Schäfers, F. Eggenstein, P. Bischoff at al.*

Advantages of synchrotron based XPS

- High brilliance and photon flux of X-rays
  - Short measurement time with excellent noise to signal ratio
  - Trace analysis of elements possible
  - *In-situ/operando* spectroscopy possible
- Collimated energy of incident X-rays and the narrow beam energy dispersion makes it possible to distinguish chemical shifts easier
- Polarization of X-rays
- Tunable photon energy
  - non destructive depth profiling

Advantages of synchrotron based XPS

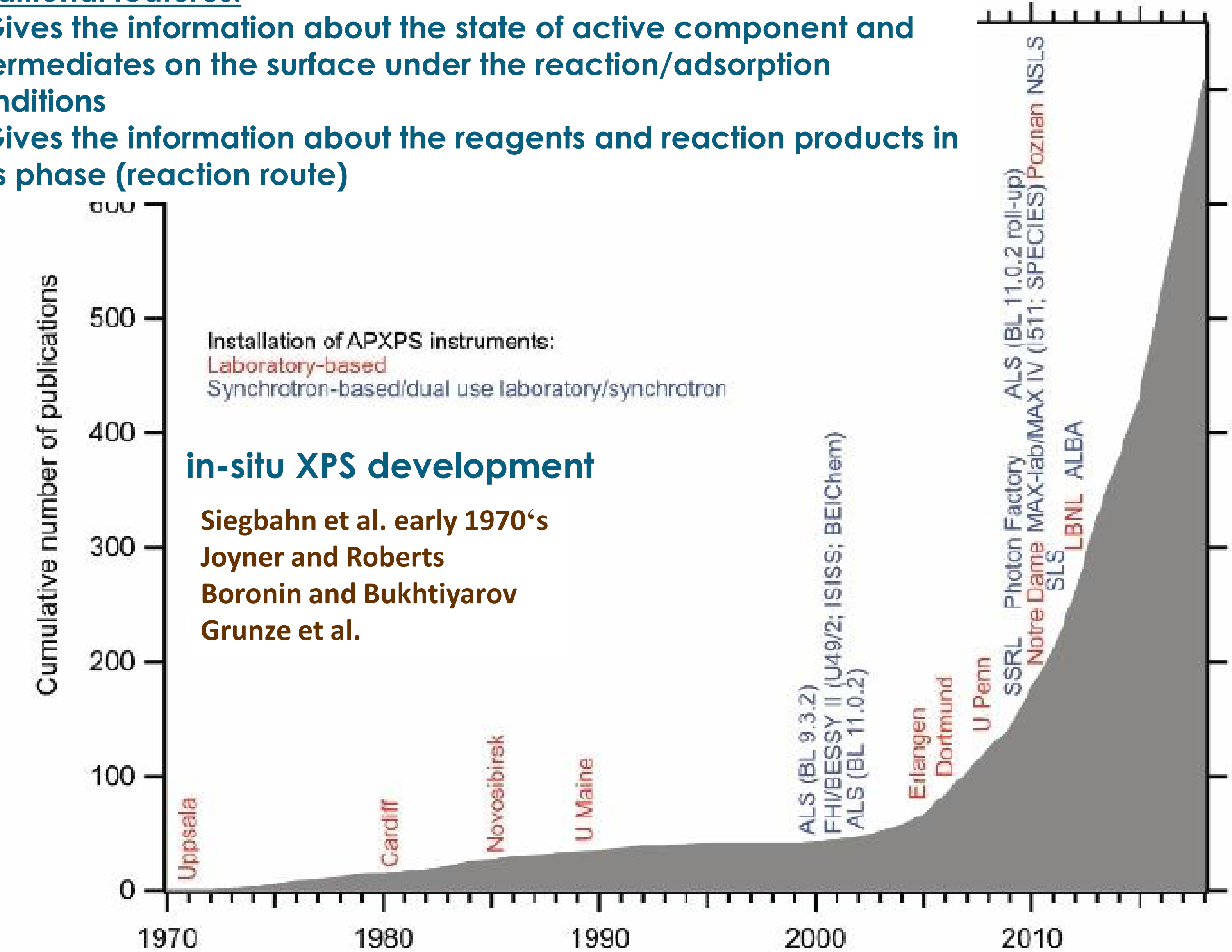


Possibility to provide the non-destructive depth profiling by variation of the excitation energy (electron kinetic energy)

Ambient pressure XPS systems

Additional features:

- Gives the information about the state of active component and intermediates on the surface under the reaction/adsorption conditions
- Gives the information about the reagents and reaction products in gas phase (reaction route)



in-situ XPS development

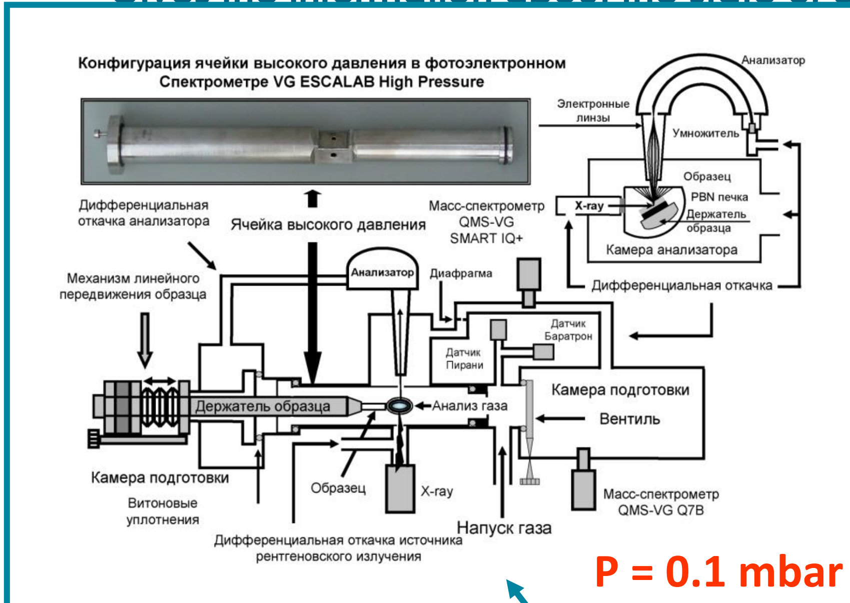
Siegbahn et al. early 1970's
Joyner and Roberts
Boronin and Bukhtiyarov
Grunze et al.

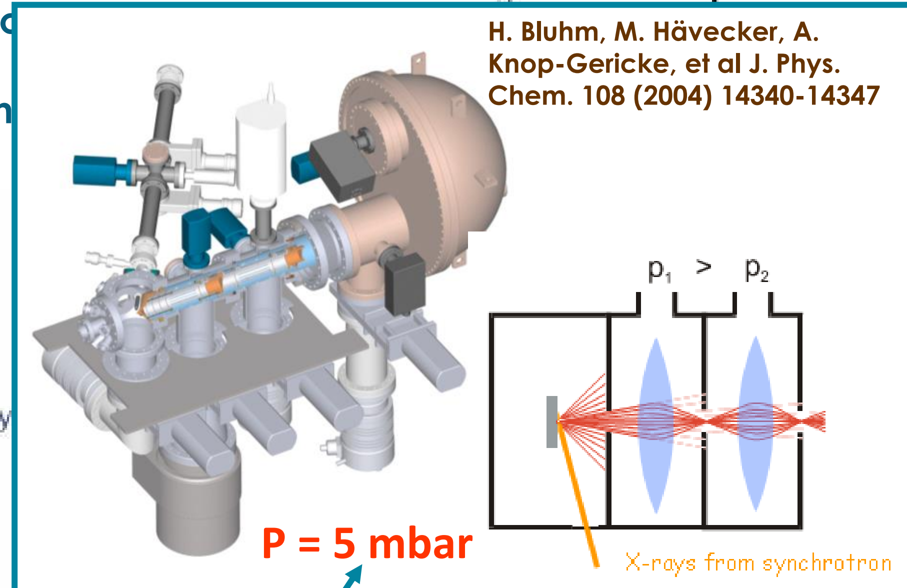
Joachim Schnadt et al 2020 J. Phys.: Condens. Matter https://doi.org/10.1088/1361-648X/ab9565

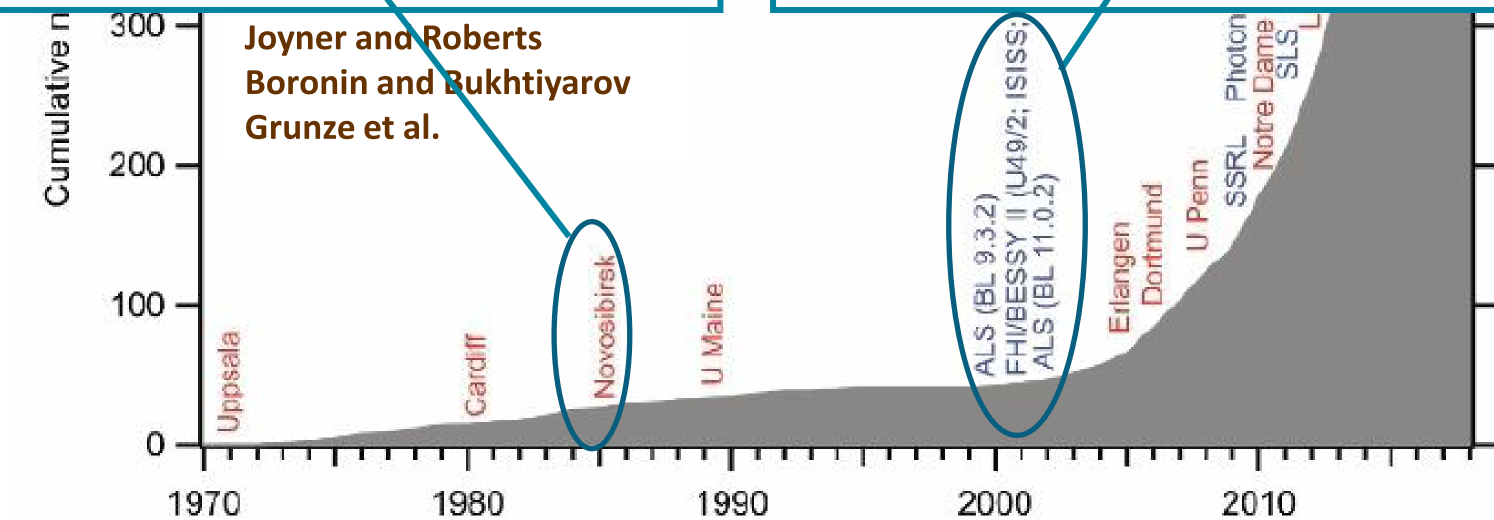
Ambient pressure XPS systems

Additional features:

- Gives the information about the state of active component and

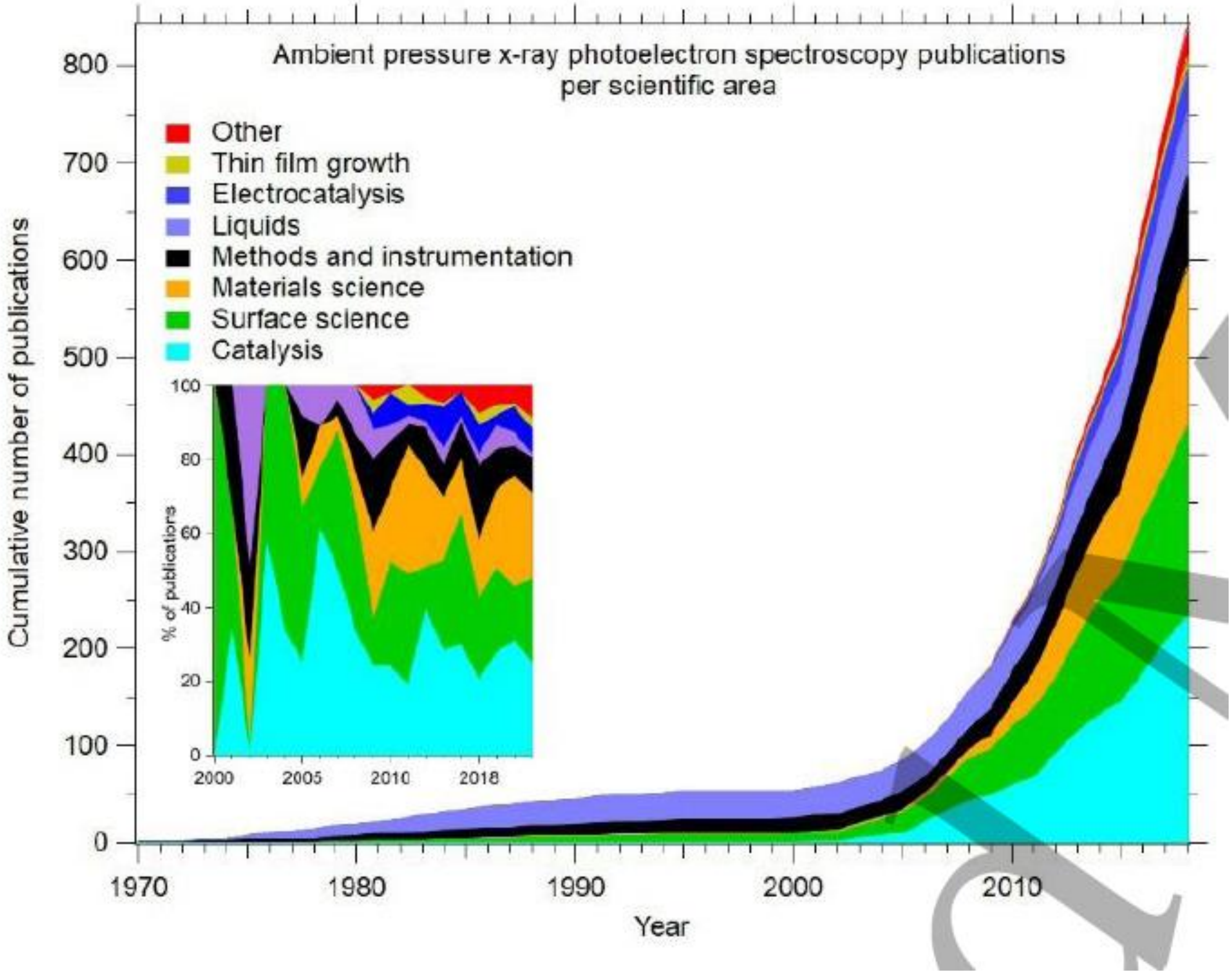






Joachim Schnadt et al 2020 J. Phys.: Condens. Matter https://doi.org/10.1088/1361-648X/ab9565

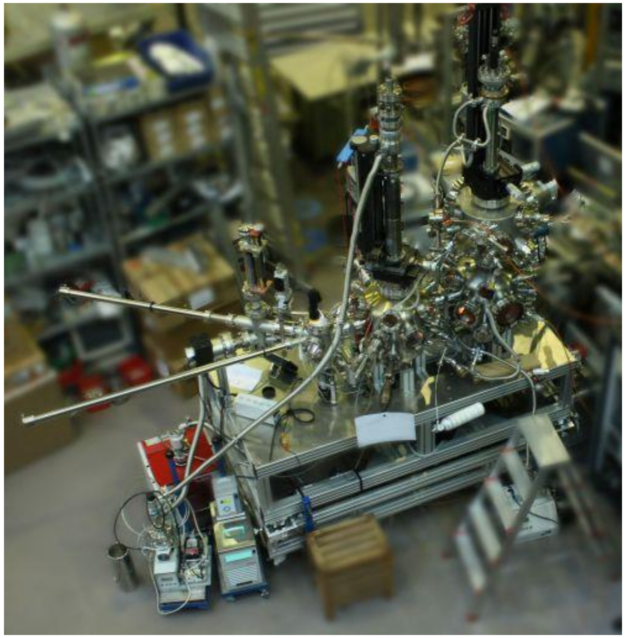
Ambient pressure XPS systems

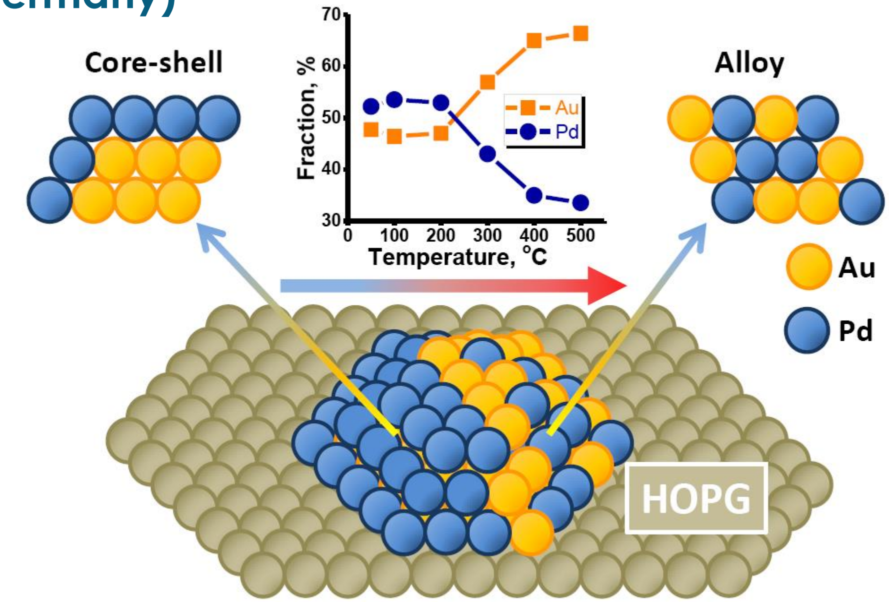


Joachim Schnadt et al 2020 J. Phys.: Condens. Matter https://doi.org/10.1088/1361-648X/ab9565

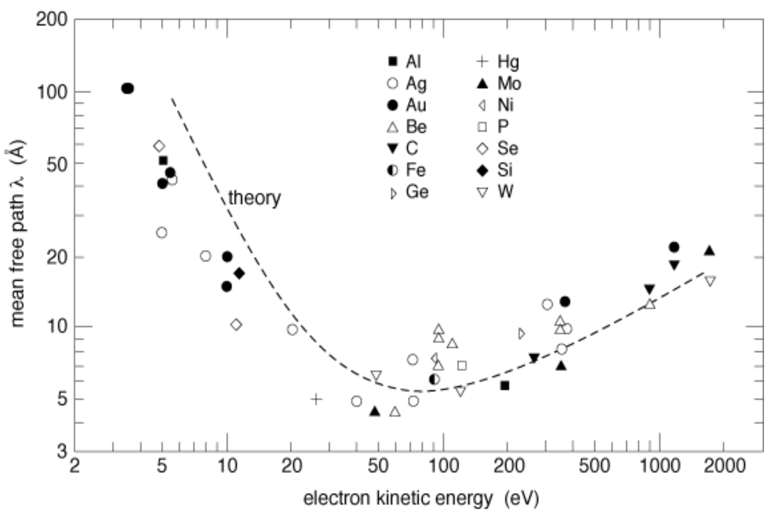
Model Pd-Au/HOPG catalysts: from “core-shell” structure to alloy

Russian-German Lab @ BESSY II (Berlin, Germany)





A.V. Bukhtiyarov, I.P. Prosvirin, V.I. Bukhtiyarov // Appl. Surf. Sci. 367 (2016) 214.

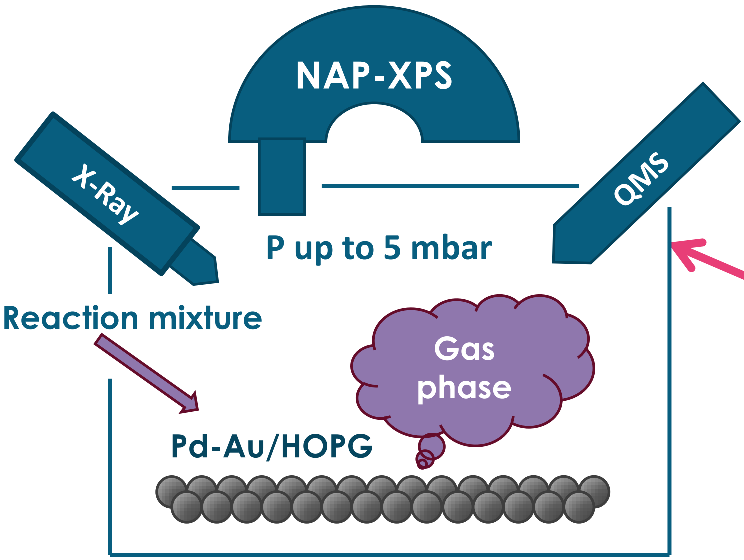


Preparation procedure

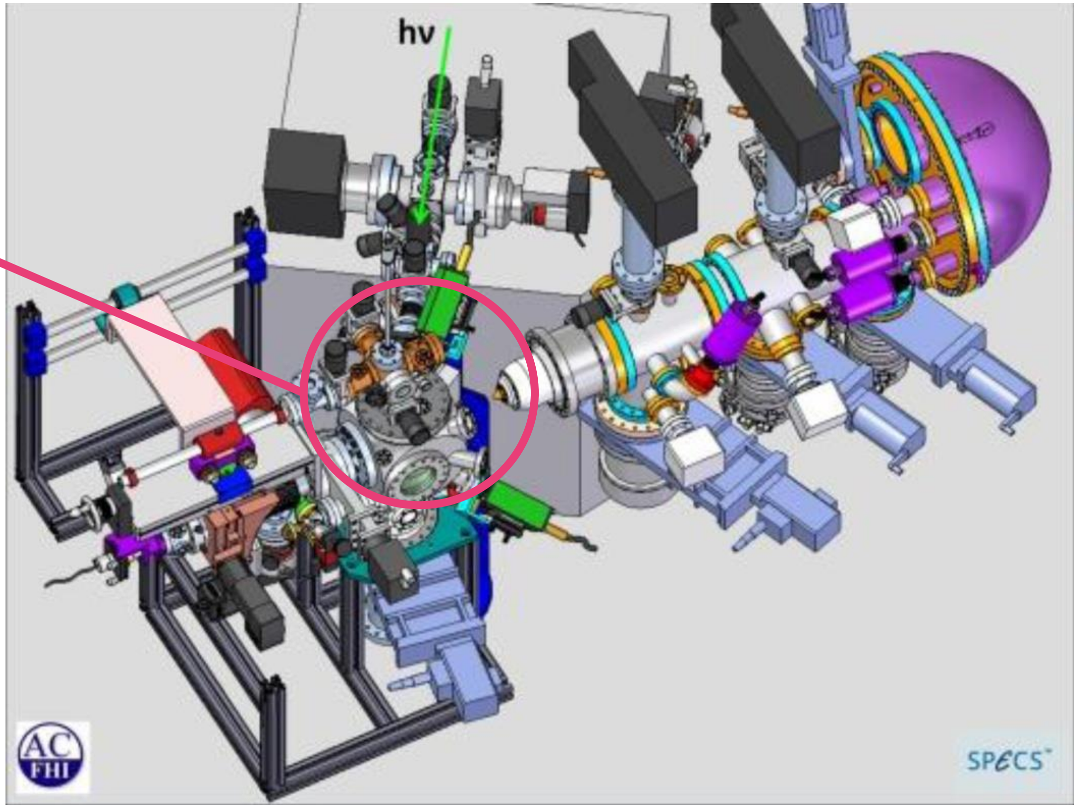
1. Soft Ar^+ sputtering
2. Metal - 1 (Pd or Ag) deposition
3. Annealing in UHV @ 300°C
4. Metal - 2 (Ag or Pd) deposition
5. Alloy formation (Annealing in UHV up to T - ?)

The optimal temperature for PdAu alloy formation ~ 400°C

CO oxidation over PdAu alloy particles. NAP XPS operando study



ISSIS @ BESSY II (Berlin, Germany) for NAP XPS measurements



CO oxidation

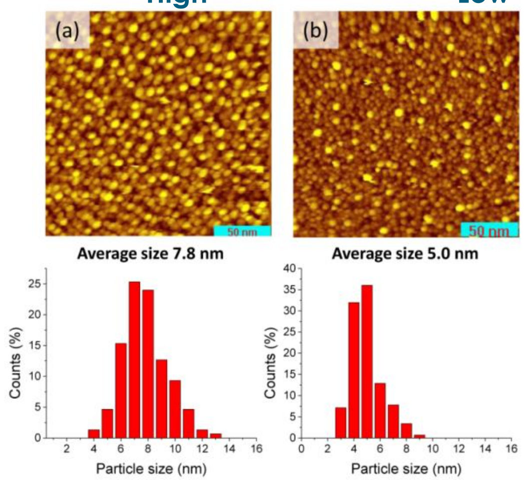
$CO/O_2 = 1/2$; $P_{total} = 0.25$ mbar

Does alloy exist under reaction conditions?

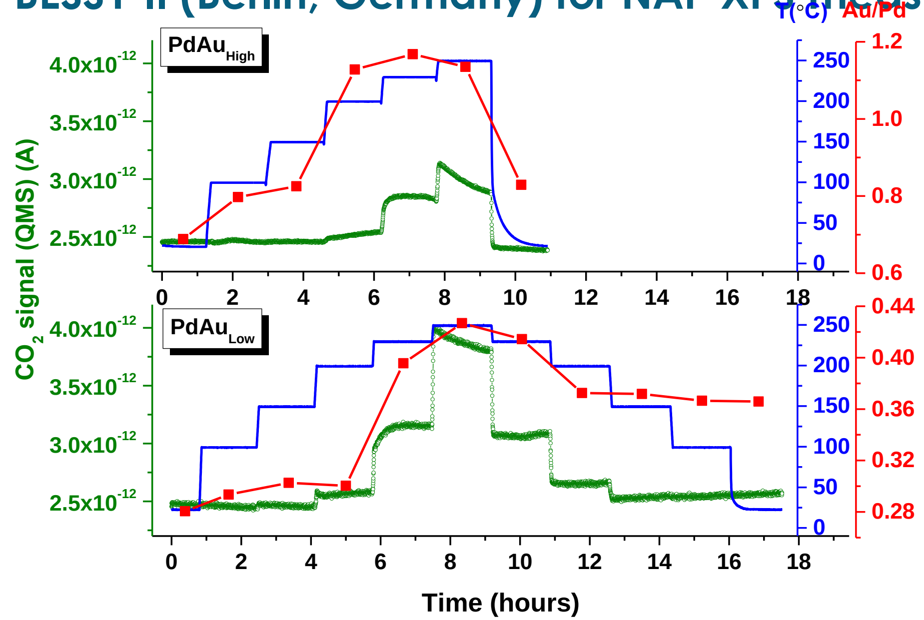
Restructuring of PdAu alloy particles in CO oxidation

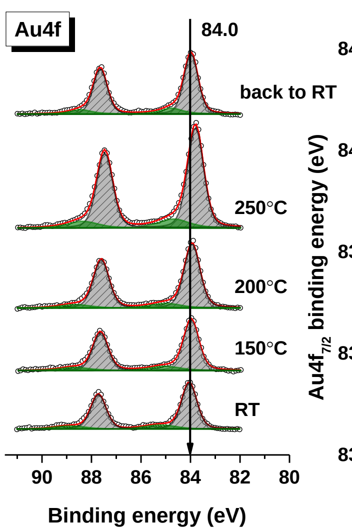
ISSIS @ BESSY II (Berlin, Germany) for NAP XPS measurements

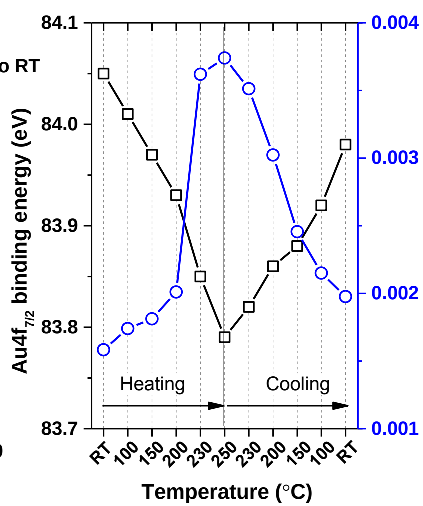
$PdAu_{High}$ $PdAu_{Low}$

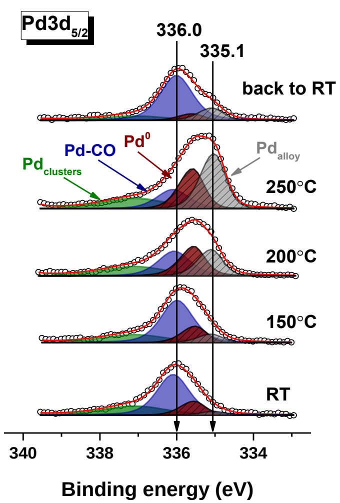


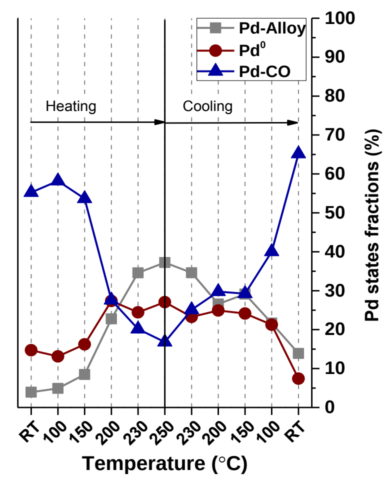
Au/Pd=1.61 **Au/Pd=0.37**







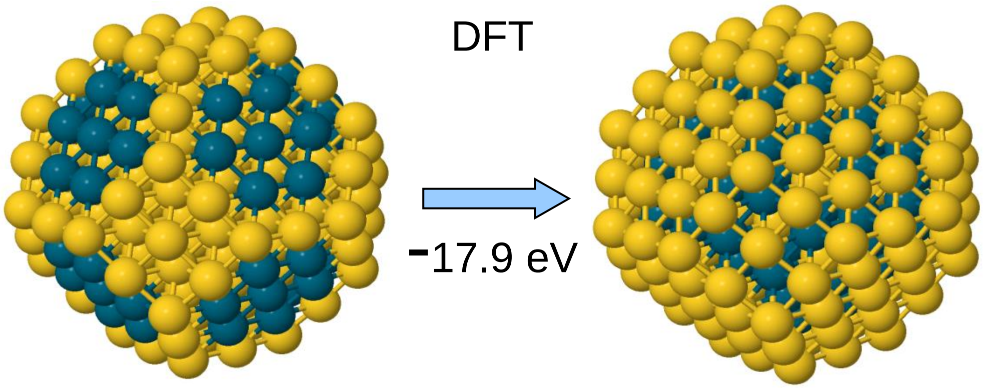




A.V. Bukhtiyarov, I.P. Prosvirin, A.A. Saraev, A.Yu. Klyushin, A. Knop-Gericke, V.I. Bukhtiyarov, Faraday Discuss., 2018, 208, 255-268.

CO Adsorption on Pd vs. Au Sites of Pd-Au Nanoparticles: DFT

$$E_{\mathrm{TOP}} = E_0 + \varepsilon_{\mathrm{bond}}^{\mathrm{Au-Pd}} N_{\mathrm{bond}}^{\mathrm{Au-Pd}} + \varepsilon_{\mathrm{corner}}^{\mathrm{Au}} N_{\mathrm{corner}}^{\mathrm{Au}} + \varepsilon_{\mathrm{edge}}^{\mathrm{Au}} N_{\mathrm{edge}}^{\mathrm{Au}}$$



$Pd_{56(out)}Au_{145}$ $Pd_{56(in)}Au_{145}$

Two homotops of the $Pd_{56}Au_{145}$ NPs: with all Pd atoms (blue spheres) inside the particles or with all Pd atoms in surface positions on (111) terraces

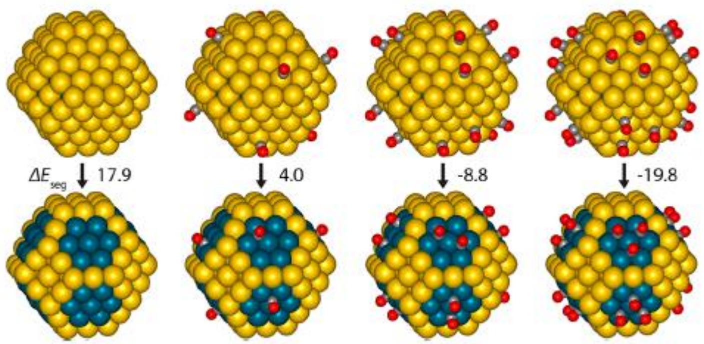
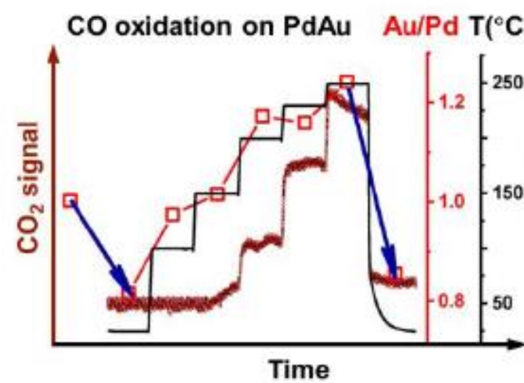
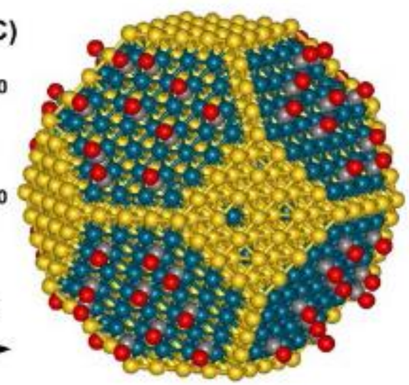


Figure 6. Structures of NP_{201} ($Pd_{56}Au_{145}$): $Pd_{in\text{-}core}$ (top row) and Pd_{terr} (bottom row) homotops with CO adsorbate on (111) terraces (0, 8, 16, and 24 CO molecules from left to right, respectively). The corresponding segregation energy, ΔE_{seg}, is given in eV.

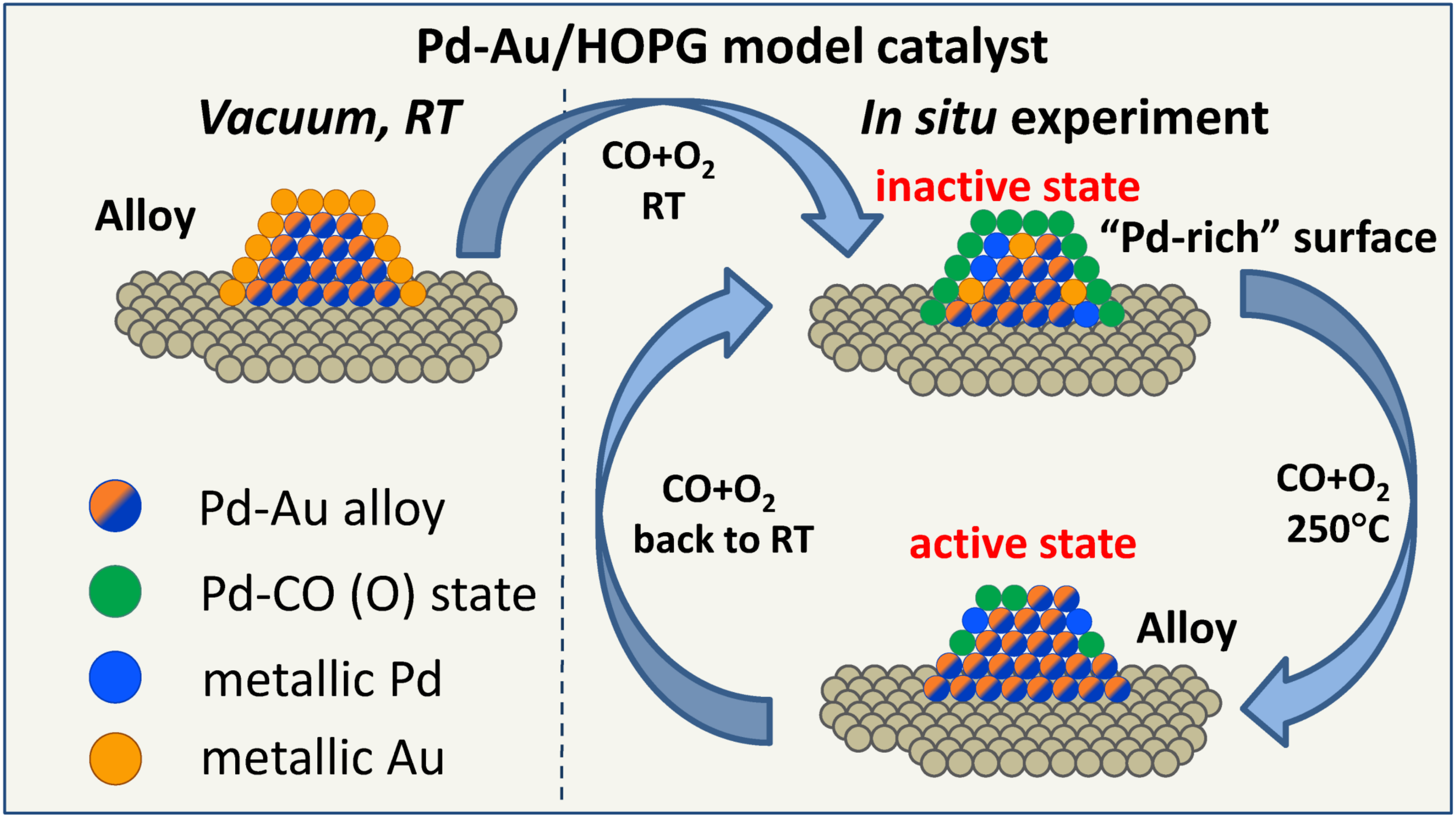




~10 adsorbed CO enough to surface segregate all Pd atoms of $Pd_{56}Au_{145}$

M. Mamatkulov, I.V. Yudanov, A.V. Bukhtiyarov, I.P. Prosvirin, V.I. Bukhtiyarov, and K.M. Neyman // J. Phys. Chem. C 2018, DOI: 10.1021/acs.jpcc.8b07402

Restructuring of PdAu alloy particles in CO oxidation



***in situ* techniques are necessary in order to investigate the active sites of bimetallic Pd-Au alloyed catalysts**

СКИФ

Thank you for your kind attention