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

**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

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**“Electronic Structure” beamline**

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

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http://catalysis.ru
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).

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

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

Magnetic field, Tesla

\( \lambda_u = 100 \text{ mm} \)

\( \lambda_u = 200 \text{ mm} \)

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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

Collimating toroidal mirror M1

monochromator: plane grating G & plane mirror M2

switchable cylindrical mirrors M3 & M4

Final focus mirror M7

Exit slits S1 & S2

Final focus mirror M5, M6

Spin-APRE

NAP XPS

Reflectometry

Beam axis after monochromator

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

Institut катализа им. Г.К. Борескова СО РАН
Spin-ARPES

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

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
- Light-like electrons
- Magnetic monopoles
- Majorana fermions

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http://catalysis.ru
A growing experimental technique
Reflectometry allow certification of spectral optical elements, focusing elements and X-ray detectors. Reflectometry Station @ BESSY II (Berlin, Germany)
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)
**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.
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).

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Joyner and Roberts
Boronin and Bukhtiyarov
Grunze et al.

P = 0.1 mbar

P = 5 mbar

Siegbahn et al. early 1970's

X-rays from synchrotron
Ambient pressure XPS systems

Ambient pressure x-ray photoelectron spectroscopy publications per scientific area

- Other
- Thin film growth
- Electro catalysis
- Liquids
- Methods and instrumentation
- Materials science
- Surface science
- Catalysis

Cumulative number of publications

Year


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

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

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

\[ \text{CO} / \text{O}_2 = 1/2; \ P_{\text{total}} = 0.25 \text{ mbar} \]

Does alloy exist under reaction conditions?
Restructuring of PdAu alloy particles in CO oxidation

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


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http://catalysis.ru
CO Adsorption on Pd vs. Au Sites of Pd-Au Nanoparticles: DFT

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

-17.9 eV

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

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


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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
Thank you for your kind attention